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Environmental Technology Verification Report

Field-Portable Gas Chromatograph/ Mass Spectrometer

Inficon, Inc., HAPSITE

by

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Notice

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**FIELD-PORTABLE GAS CHROMATOGRAPH/ MASS SPECTROMETER**
APPLICATION:	**MEASUREMENT OF CHLORINATED VOLATILE ORGANIC COMPOUNDS IN WATER**
TECHNOLOGY NAME:	**HAPSITE with Headspace Sampling Accessory**
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PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative environmental technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies.

Under this program, in partnership with recognized testing organizations, and with the full participation of the technology developer, the EPA evaluates the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing the demonstration results, and preparing reports. The testing is conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible. The EPA National Exposure Research Laboratory, in cooperation with Sandia National Laboratories, the testing organization, evaluated field-portable systems for monitoring chlorinated volatile organic compounds (VOCs) in water. This verification statement provides a summary of the demonstration and results for the Inficon HAPSITE field-portable gas chromatograph/mass spectrometer (GC/MS) system.

DEMONSTRATION DESCRIPTION

The field demonstration of the HAPSITE portable GC/MS was held in September 1997. The demonstration was designed to assess the instrument's ability to detect and measure chlorinated volatile organic compounds in groundwater at two contaminated sites: the Department of Energy's Savannah River Site, near Aiken, South Carolina, and the McClellan Air Force Base, near Sacramento, California. Groundwater samples from each site were supplemented with performance evaluation (PE) samples of known composition. Both sample types were used to assess instrument accuracy, precision, sample throughput, and comparability to reference laboratory results. The primary target compounds at the Savannah River Site were trichloroethene and tetrachloroethene. At McClellan Air

Force Base, the target compounds were trichloroethene, tetrachloroethene, 1,2-dichloroethane, 1,1,2-trichloroethane, 1,2-dichloropropane, and *trans*-1,3-dichloropropene. These sites were chosen because they contain varied concentrations of chlorinated VOCs and exhibit different climatic and geologic conditions. The conditions at these sites are typical, but not inclusive, of those under which this technology would be expected to operate. A complete description of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report, Field-Portable Gas Chromatograph/Mass Spectrometer, Inficon, Inc., HAPSITE.* (EPA/600/R-98/142).

TECHNOLOGY DESCRIPTION

GC/MS is a proven laboratory analytical technology that has been used for environmental characterization and monitoring for many years. The combination of gas chromatography and mass spectrometry allows the rapid separation and identification of compounds in complex mixtures. The gas chromatograph separates the sample into individual components. These components are introduced into the electron impact source module of the spectrometer, where the molecules are fragmented into ions by an electron beam. The ion fragments are further separated by mass and detected by an electron multiplier. The resulting mass spectrum is characteristic of a particular compound and can be used to identify each component in the sample extract through comparison with a reference spectral library. Quantitation is achieved by comparing the abundance of ions which are characteristic of a specific compound with the detector response from the analysis of a standard mixture. Field-portable GC/MS is a versatile technique that can be used to provide rapid screening data or laboratory-quality analyses. As with many field analytical studies, it may be necessary to send a portion of the samples to an independent laboratory for confirmatory analyses.

The Inficon HAPSITE with a headspace sampling accessory is a commercially available GC/MS system that provides laboratory-grade performance in a field-transportable package. The instrument, including the on-board computer, is designed for field use and is encapsulated in a weather-resistant case. The GC/MS unit weighs about 35 pounds and the headspace sampling accessory weighs about 15 pounds. Both units can be easily transported and operated in the rear compartment of a minivan or station wagon. The instrument utilizes an equilibrium headspace technique for the analysis of VOCs in water. Instrument detection limits for most chlorinated VOCs in water are in the range of 5 to 10 µg/L. At the time of the demonstration, the cost of the HAPSITE with headspace accessory was in the range of $75,000 to $95,000, depending upon instrument options. Operational costs, which include consumable supplies but not labor costs, are on the order of $150 per 8-hour day.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the HAPSITE were observed:

Sample Throughput: Throughput was approximately two to three water samples per hour. This rate includes the periodic analysis of blanks and calibration check samples.

Completeness: The HAPSITE reported results for all but one of the 166 PE and groundwater samples provided for analysis at the two demonstration sites. One sample was dropped during preparation.

Analytical Versatility: The HAPSITE detected all of the compounds in the PE samples for which it was calibrated. Its calibration included 84% (27 of 32) of all chlorinated and nonchlorinated volatile hydrocarbon compounds included in the PE samples at the demonstration. Additional compounds could have been detected with a longer GC/MS run time and a reduced sample throughput. The HAPSITE detected all (59 of 59) of the groundwater contaminants in excess of 5 µg/L reported by the reference laboratory at both sites. A total of 68 contaminants, at concentration levels of 1 µg/L or higher, were detected by the reference laboratory in all groundwater samples.

Precision: Precision was determined by analyzing sets of four replicate samples from a variety of PE mixtures containing known concentrations of chlorinated VOCs. The results are reported as relative standard deviations (RSD). The RSDs compiled for all reported PE compounds from both sites had a median value of 12% and a 95th percentile value of 29%. By comparison, the compiled RSDs from the reference laboratory had a median value of

7% and a 95th percentile value of 25%. The ranges of HAPSITE RSD values for specific target compounds were as follows: trichloroethene 7 to 18%, tetrachloroethene, 6 to 22%; 1,2-dichloroethane, 2 to 12%; 1,1,2-trichloroethane, 8 to 28%; 1,2-dichloropropane, 7 to 21%; and *trans*-1,3-dichloropropene, 7 to 17%.

Accuracy: Instrument accuracy was evaluated by comparing HAPSITE results with the known concentrations of chlorinated organic compounds in PE mixtures. Absolute percent difference (APD) values from both sites were calculated for all analytes in the PE mixtures. The APDs for all reported compounds from both sites had a median value of 8% and a 95th percentile value of 27%. By comparison, the compiled APDs from the reference laboratory had a median value of 7% and a 95th percentile value of 24%. The ranges of HAPSITE APD values for target compounds were as follows: trichloroethene, 1 to 20%; tetrachloroethene, 6 to 33%; 1,2-dichloroethane, 2 to 20%; 1,1,2-trichloroethane 1 to 21%; 1,2-dichloropropane, 3 to 21%; and *trans*-1,3-dichloropropene, 1 to 15%.

***Comparability*:** A comparison of HAPSITE and reference laboratory data was based on 33 groundwater samples analyzed at each site. The correlation coefficients (r) for all compounds detected by both the HAPSITE and laboratory at or below 100 µg/L concentration levels were 0.983 at Savannah River and 0.978 at McClellan. The r values for compounds detected at concentration levels in excess of 100 µg/L were 0.996 for Savannah River and 1.000 for McClellan. These correlation coefficients reveal a highly linear relationship between HAPSITE and laboratory data. The median absolute percent difference between groundwater compounds mutually detected by the HAPSITE and reference laboratory was 13%, with a 95th percentile value of 60%.

Deployment: The system was ready to analyze samples within 30 minutes of arrival at the site. At both sites, the instrument was transported in a minivan and was operated in its rear luggage compartment. The instrument was powered by self-contained batteries or from line ac power. The recommended training interval for routine sample processing is about 3 days for a chemist with limited GC/MS experience. Method development and analysis of very complex samples requires a higher level of operator training and experience in GC/MS data interpretation.

The results of this demonstration show that the HAPSITE can provide useful, cost-effective data for environmental site screening and routine monitoring. This instrument could be employed in a variety of applications, ranging from producing rapid analytical results in screening investigations, to producing accurate and precise data that are directly comparable with that obtained from an off-site laboratory. These data could be used to develop risk assessment information, support a remediation process, or fulfill monitoring requirements. In the selection of a technology for deployment at a site, the user must determine what is appropriate through consideration of instrument performance and the project's data quality objectives.

Gary J. Foley, Ph. D.	Samuel G. Varnado
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NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable federal, state and local requirements.

Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's natural resources. The National Exposure Research Laboratory (NERL) is the EPA center for the investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. The NERL research goals are to (1) develop and evaluate technologies for the characterization and monitoring of air, soil, and water; (2) support regulatory and policy decisions; and (3) provide the science support needed to ensure effective implementation of environmental regulations and strategies.

The EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through verification of performance and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. It is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies.

Candidate technologies for this program originate from the private sector and must be market ready. Through the ETV Program, developers are given the opportunity to conduct rigorous demonstrations of their technologies under realistic field conditions. By completing the evaluation and distributing the results, the EPA establishes a baseline for acceptance and use of these technologies.

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Executive Summary

The U.S. Environmental Protection Agency, through the Environmental Technology Verification Program, is working to accelerate the acceptance and use of innovative technologies that improve the way the United States manages its environmental problems. As part of this program, the Consortium for Site Characterization Technology was established as a pilot program to test and verify field monitoring and site characterization technologies. The Consortium is a partnership involving the U.S. Environmental Protection Agency, the Department of Defense, and the Department of Energy. In 1997 the Consortium conducted a demonstration of five systems designed for the analysis of chlorinated volatile organic compounds in groundwater (GW). The developers participating in this demonstration were Electronic Sensor Technology, Perkin-Elmer Photovac, and Sentex Systems, Inc. (field-portable gas chromatographs); Inficon, Inc. (field-portable gas chromatograph/mass spectrometer, GC/MS); and Innova AirTech Instruments (photoacoustic infrared analyzer). This report documents demonstration activities, presents demonstration data, and verifies the performance of the Inficon HAPSITE field-portable GC/MS. Reports documenting the performance of the other technologies have been published separately.

The demonstration was conducted at two geologically and climatologically different sites: the U.S. Department of Energy's Savannah River Site, near Aiken, South Carolina, and McClellan Air Force Base, near Sacramento, California. Both sites have groundwater resources that are significantly contaminated with a variety of chlorinated volatile organic compounds. The demonstrations to evaluate the capabilities of each field-portable system were conducted in September 1997 and were coordinated by Sandia National Laboratories.

The demonstration provided adequate analytical and operational data with which to evaluate the performance of the Inficon HAPSITE GC/MS system. Instrument precision and accuracy were determined from analyses of replicate samples from 16 multicomponent standard mixtures of known composition. The relative standard deviations obtained from analyses of 4 replicate samples from each of the 16 standard mixtures were used as measures of precision. The distribution of relative standard deviations from all compounds had a median value of 12% and a 95th percentile value of 29%. Accuracy was expressed as the absolute percent difference between the HAPSITE measured value and the true value of the component in the standard mixtures. The distribution of absolute percent difference values for all compounds in all standard mixtures had a median value of 8% and a 95th percentile value of 27%. A comparison of HAPSITE and reference laboratory results from groundwater samples at each site resulted in a median absolute percent difference of 13%, with a 95th percentile value of 60%. A correlation analysis between HAPSITE and laboratory results indicates a high degree of linear correlation (r >0.98) at both low (≤100 µg/L) and high (>100 µg/L) contaminant concentrations. The sample throughput rate of the HAPSITE was determined to be two to three samples per hour.

The HAPSITE detected all of the groundwater contaminants reported by the reference laboratory at both sites which were present at concentration levels in excess of 5 µg/L. The results of the demonstration show that the Inficon HAPSITE field-portable GC/MS with its headspace sampling accessory can provide useful, cost-effective data for environmental site characterization and routine monitoring. As with any technology selection, the user must determine whether the technology is appropriate for the application by taking into account instrument performance parameters and the project's data quality objectives.

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Acronyms and Abbreviations

ac	alternating current
amu	atomic mass unit
APD	absolute percent difference
BNZN	benzene
°C	degrees centigrade
CCC	calibration check compounds
CCL4	carbon tetrachloride
CLFRM	chloroform
dc	direct current
11DCA	1,1-dichloroethane
12DCA	1,2-dichloroethane
DCE	dichloroethene
11DCE	1,1-dichloroethene
c12DCE	*cis*-1,2-dichloroethene
t12DCE	*trans*-1,2-dichloroethene
DCL	DataChem Laboratories
DOE	Department of Energy
EPA	Environmental Protection Agency
ETV	Environmental Technology Verification Program
eV	electron-volt
GC	gas chromatograph
GW	groundwater
GC/MS	gas chromatograph/mass spectrometer
hp	horsepower
Hz	hertz, cycles per second
i.d.	inside diameter
L	liter
m	meter
mg	milligram
mg/L	milligram per liter
mL	milliliter
mm	millimeter
MAFB	McClellan Air Force Base
MCL	maximum concentration level
MDL	method detection limit
MS	mass spectroscopy
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology

NR	not reported
PC	personal computer
PCE	tetrachloroethene (perchloroethene)
PE	performance evaluation
ppb	parts per billion
ppm	parts per million
PQL	practical quantitation limit
PVC	poly (vinyl chloride)
QA	quality assurance
QC	quality control
r	correlation coefficient
RPD	relative percent difference
RSD	relative standard deviation
SPCC	system performance check compounds
SRS	Savannah River Site
TCA	trichloroethane
111TCA	1,1,1-trichloroethane
TCE	trichloroethene
V	volts
V ac	volts alternating current
VOA	volatile organics analysis
VOC	volatile organic compound
XYL	xylene
µg	microgram
µg/L	microgram per liter
µL	microliter

Acknowledgments

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The participation of personnel from Inficon, Inc. in this technology demonstration is also acknowledged. Chuck Sadowski and co-workers operated the instrument during the demonstrations.

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Chapter 1
Introduction

Site Characterization Technology Challenge

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through verification of performance and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. It is intended to assist and inform those involved in the design, distribution, permitting, purchase, and use of environmental technologies. The ETV Program capitalizes on and applies the lessons that were learned in the implementation of the Superfund Innovative Technology Evaluation Program to twelve pilot programs: Drinking Water Systems, Pollution Prevention for Waste Treatment, Pollution Prevention for Innovative Coatings and Coatings Equipment, Indoor Air Products, Advanced Monitoring Systems, EvTEC (an independent, private-sector approach), Wet Weather Flows Technologies, Pollution Prevention for Metal Finishing, Source Water Protection Technologies, Site Characterization and Monitoring Technology, Climate Change Technologies, and Air Pollution Control.

For each pilot, the EPA utilizes the expertise of partner “verification organizations” to design efficient procedures for performance tests of the technologies. The EPA selects its partners from both public and private sectors, including federal laboratories, states, and private sector entities. Verification organizations oversee and report activities based on testing and quality assurance protocols developed with input from all major stakeholder and customer groups associated with the technology area. The U.S. Department of Energy’s (DOE’s) Sandia National Laboratories in Albuquerque, New Mexico, served as the verification organization for the demonstration described in this report.

The performance verification reported here is based on data collected during a demonstration of technologies for the characterization and monitoring of chlorinated volatile organic compounds (VOCs) in groundwater. Rapid, reliable, and cost-effective field screening and analysis technologies are needed to assist in the complex task of characterizing and monitoring hazardous and chemical waste sites. Environmental regulators and site managers are often reluctant to use new technologies that have not been validated in an objective EPA-sanctioned testing program or other similar process. Until the field performance of a technology can be verified through objective evaluations, users will remain skeptical of innovative technologies, despite the promise of better, less expensive, and faster environmental analyses. This demonstration was administered by the Site Characterization and Monitoring Technology Pilot Program, which is also known as the Consortium for Site Characterization Technology. The mission of the Consortium is to identify, demonstrate, and verify the performance of innovative site characterization and monitoring technologies. The Consortium also disseminates information about technology performance to developers, environmental remediation site managers, consulting engineers, and regulators.

Technology Verification Process

The technology verification process consists of the four key steps shown here and discussed in more detail in the following paragraphs:

1. identification of needs and selection of technology;
2. planning and implementation of demonstration;
3. preparation of report; and
4. distribution of information.

Identification of Needs and Selection of Technology

The first aspect of the verification process is to determine the technology needs of the EPA and the regulated community. The EPA, the U.S. Department of Energy, the U.S. Department of Defense, industry, and state agencies are asked to identify technology needs for site characterization and monitoring. Once a need is recognized, a search is conducted to identify suitable technologies that will address this need. This search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searching industry and trade publications, attending related conferences, and following up on suggestions from technology developers and experts in the field. Candidate characterization and monitoring technologies are evaluated against the following criteria:

- may be used in the field or in a mobile laboratory;
- has a regulatory application;
- is applicable to a variety of environmentally affected sites;
- has a high potential for resolving problems for which current methods are unsatisfactory;
- has costs that are competitive with current methods;
- has performance as good or better than current methods in areas such as data quality, sample preparation, and/or analytical turnaround time;
- uses techniques that are easier and safer than current methods; and
- is a commercially available, field-ready technology.

Planning and Implementation of Demonstration

After a technology has been selected, the EPA, the verification organization, and the developer(s) agree on a strategy for conducting the demonstration and evaluating the technology. A conceptual plan for designing a demonstration for a site characterization technology has been published by the Site Characterization and Monitoring Technology Pilot Program (EPA, 1996a). During the planning process, the following steps are carried out:

- identification of at least two demonstration sites that will provide the appropriate physical or chemical attributes in the desired environmental media;
- identification and definition of the roles of demonstration participants, observers, and reviewers;
- determination of logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network);
- arranging for field sampling and reference analytical laboratory support; and

- preparation and implementation of a demonstration plan that addresses the experimental design, sampling design, quality assurance and quality control (QA/QC), health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements.

Preparation of Report

Each of the innovative technologies is evaluated independently and, when possible, against a reference technology. The technologies are operated in the field by the developers in the presence of independent observers who are provided by the EPA or the verification organization. Demonstration data are used to evaluate the capabilities, limitations, and field applications of each technology. Following the demonstration, all raw and reduced data used to evaluate each technology are compiled in a technology evaluation report, which is a record of the demonstration. A data summary and detailed evaluation of each technology are published in an environmental technology verification report. The report includes a verification statement, which is a concise summary of the instrument's performance during the demonstration.

Distribution of Information

The goal of the information distribution strategy is to ensure that environmental technology verification reports and accompanying verification statements are readily available to interested parties through traditional data distribution pathways, such as printed documents. Related documents and updates are also available on the World Wide Web through the ETV Web site (*http://www.epa.gov/etv*) and through a Web site supported by the EPA Office of Solid Waste and Emergency Response Technology Innovation Office *(http://clu-in.com)*. Additional information at the ETV Web site includes a summary of the demonstration plan, test protocols (where applicable), demonstration schedule and participants, and in some cases a brief narrative and pictorial summary of the demonstrations.

The Wellhead VOC Monitoring Demonstration

In August 1996, the selection of a technology for monitoring chlorinated VOCs in water was initiated by publication in the *Commerce Business Daily* of a solicitation and notice of intent to conduct such a technology demonstration. Potential participants were also solicited through manufacturer and technical literature references. The original demonstration scope was limited to market-ready *in situ* technologies; however, only a limited response was obtained, so the demonstration scope was expanded to include technologies that could be used to measure groundwater (GW) at or near the wellhead. The final selection of technologies was based on the readiness of the technologies for field demonstration and their applicability to the measurement of chlorinated VOCs in groundwater at environmentally affected sites.

For this demonstration, five instrument systems were selected. Three of them were field-portable gas chromatographs with various detection systems: one with a surface acoustic wave detector from Electronic Sensor Technology, one with dual electron capture and photoionization detectors from Perkin-Elmer Photovac, and one with an argon ion/electron capture detector from Sentex Systems. The fourth instrument was a field-portable gas chromatograph/mass spectrometer (GC/MS) from Inficon, and the fifth was a photoacoustic infrared spectrometer from Innova AirTech Instruments. This report documents demonstration activities, presents demonstration data, and verifies the performance of the Inficon, Inc., HAPSITE field-portable gas chromatograph/mass spectrometer. Reports documenting the performance of the other four technologies have been published separately.

The demonstration was conducted in September 1997 at the DOE Savannah River Site (SRS) near Aiken, Georgia, and at McClellan Air Force Base (MAFB), near Sacramento, California. Both sites have subsurface plumes of chlorinated VOCs and extensive networks of groundwater monitoring wells. The demonstrations were coordinated by Sandia National Laboratories with the assistance of personnel from the Savannah River Site.

The primary objective of this demonstration was to evaluate and verify the performance of field-portable characterization and monitoring technologies for analysis of chlorinated VOCs in groundwater. Specific demonstration objectives were to:

- verify instrument performance characteristics that can be directly quantified (such factors include response to blank samples, measurement accuracy and precision, sample throughput, and data completeness);
- verify instrument characteristics and performance in various qualitative categories such as ease of operation, required logistical support, operator training requirements, transportability, versatility, and other related characteristics; and
- compare instrument performance with results from standard laboratory analytical techniques currently used to analyze groundwater for chlorinated VOCs.

The goal of this and other ETV demonstrations is to verify the performance of each instrument as a separate entity. Technologies are not compared with each other in this program. The demonstration results are summarized for each technology independent of other participating technologies. In this demonstration, the capabilities of the five instruments varied and in many cases were not directly comparable. Some of the instruments are best suited for routine monitoring where compounds of concern are known and there is a maximum contaminant concentration requirement for routine monitoring to determine regulatory compliance. Other instruments are best suited for characterization or field-screening activities where groundwater samples of unknown composition can be analyzed in the field to develop an improved understanding of the type of contamination at a particular site. This field demonstration was designed so that both monitoring and characterization technologies could be verified.

Chapter 2
Technology Description

This chapter was provided by the developer and was edited for format and relevance. The data presented include performance claims that may not have been verified as part of the demonstration. Chapters 5 and 6 report instrument features and performance observed in this demonstration. Publication of this material does not represent EPA approval or endorsement.

Technology Overview

The Inficon HAPSITE is a field-portable gas chromatograph/mass spectrometer that can be operated from battery or ac line power. The basic instrument was originally designed to analyze gas samples in the parts per billion (ppb) to parts per million (ppm) range. Sample components are separated by gas chromatography, and are detected with a conventional quadrupole mass spectrometer. The mass spectrometer is capable of scanning from 1 to 300 atomic mass units (amu) and employs a continuous dynode electron multiplier detector system. The primary application of the HAPSITE is for direct air measurements. The HAPSITE provides an MS-only mode of operation in which an air sample can be directly introduced into the mass spectrometer via a membrane interface without GC separation. An equilibrium headspace sample accessory can be used to concentrate volatile sample components from water, soil, or sludge matrices in the gas phase above the sample. The accessory automatically introduces a portion of the headspace gas into the GC/MS.

The HAPSITE GC/MS can be operated in a field-portable or transportable mode. In the transportable mode, a suitable working environment, such as a trailer or van equipped with 110 V ac line power, is required for operation. The HAPSITE GC/MS is mounted on a service module that contains a turbomolecular and roughing pump. The combined weight of the system in the transportable mode is 75 pounds.

In the field-portable mode, the HAPSITE utilizes a proprietary chemical getter pumping system to maintain the vacuum in the mass spectrometer. The pumping system, which contains no moving parts, provides a vacuum for 30 days, at 8 hours use per day, after which it must be replaced. In the field-portable mode, the weight of the GC/MS is 35 pounds. The unit is rugged and water resistant and is designed to be operated in the typical environment found in a manufacturing or chemical plant or at a hazardous waste site. It can withstand the normal shocks and bumps encountered during field use. Battery life in the field-portable mode is 2 to 3 hours. The system uses a self-contained carrier gas, and internal standard gases are used to tune and calibrate the mass spectrometer. The internal standards can also be coinjected with air samples. The carrier gas supply is suffficient for 8 hours of operation, and the internal standard gas supply will last 3 days at 8 hours per day of use. The headspace sampling accessory can be operated via battery or ac line power, weighs 15 pounds, and will equilibrate up to four samples simultaneously. A separate carrier gas supply is required for the headspace accessory.

The HAPSITE consists of an inlet system (heated transfer line, sample pump, gas sampling valve); gas chromatograph (isothermal oven and 27-m × 32-mm i.d. capillary column with Supelco SPB-1, 1-µm film coating, and 3-m precolumn for backflush), and mass spectrometer (membrane interface, quadrupole mass spectrometer, internal computer, and hard disk). An external notebook personal computer (PC) is included with the system and can be used for system control as well as to display and analyze data in real time. The external PC is not required for operation once methods have been developed and stored on the HAPSITE internal PC.

Principle of Operation

The headspace sampling accessory uses a temperature-controlled environment to equilibrate a water, soil, or sludge sample in a sealed vial. The volatile components in the sample matrix reach an equilibrium distribution between the water sample and the vapor headspace above the sample. A portion of the headspace gas is transferred to the gas sampling loop of the HAPSITE sample introduction system via a pump and carrier gas. The fixed volume of the loop is then injected onto the GC precolumn. The principle of headspace equilibration and subsequent headspace sampling is similar to SW-846 Method 3810 (EPA, 1986). The GC is operated isothermally at 60 °C and the analytes are separated during an 11-minute run. Compounds that would elute after 11 minutes are backflushed from the precolumn. Components elute from the GC column and enter the mass spectrometer ionizer assembly through a poly(dimethylsilicone) membrane interface, which excludes most of the nitrogen carrier gas. The membrane is maintained at 60 °C.

The separated compounds produce a characteristic 70-eV electron impact spectrum. When tuned to the manufacturer's specifications, the spectrometer will produce consistent, National Institute of Standards and Technology (NIST), library-searchable spectra for compounds in the low parts-per-billion to parts-per-million range. The mass spectrometer can be operated in a full scan or selected ion mode. In this demonstration, the unit was operated in full scan mode. Target compounds are identified by their GC retention time and comparison of their mass spectra with a target compound library of spectra collected during calibration. Spectra of unknown compounds can be compared with spectra in the NIST mass spectral library for tentative identification.

Quantification is accomplished by applying a relative response factor from a daily calibration standard. For the headspace method, internal standards and surrogates are used to identify and compensate for matrix effects. Internal and surrogate standards used in this demonstration were toluene-d_8, chlorobenzene-d_5, fluorobenzene, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, dibromofluoromethane, and dichlorobenzene-d_4.

History of the Technology

Inficon, Inc. is one of the world's largest manufacturers of quadrupole mass spectrometers for application in a variety of manufacturing processes. The mass spectrometer in the HAPSITE is an adaptation of this industrial spectrometer product line. The HAPSITE GC/MS was originally designed to meet the requirements for source emission testing as specified in the 1990 Clean Air Act amendments. The EPA has provisionally approved a Method-301 validation for use of the HAPSITE in source-emission testing. The method has been approved for source measurements of mineral calciners and is pending extension as a general source-testing method for gaseous organic compounds.

Applications

The HAPSITE GC/MS and headspace sampling accessory are designed to measure the presence and concentration of volatile organic compounds in water, soil, and sludge. The technology is applicable to site investigation and characterization and to periodic monitoring to determine the migration of volatiles at remediation sites. Site engineers charged with definition of site contamination and monitoring the effectiveness of remediation techniques comprise the largest group of potential users.

Advantages

The primary advantage of the HAPSITE is its ability to provide on-site results of a quality comparable to conventional laboratory GC/MS. Decisions can be made in a cost-effective manner in regard to the need for further sampling or the use of high-cost field equipment, such as drilling rigs. The initial cost is comparable to a laboratory GC/MS equipped with a purge-and-trap accessory. Field-portable gas chromatographs with nonspecific detectors are less costly but lack the ability of the GC/MS to identify and quantitate the organic components in complicated sample matrices.

Limitations

The major limitation of the HAPSITE is the isothermal GC oven. Standard chromatographic run times of 10 minutes must be extended to 20 minutes in order to detect the dichlorobenzenes. The last five analytes from the EPA Method 8260A list of compounds—1,2-dibromo-3-chloropropane, 1,2,4-trichlorobenzene, naphthalene, hexachlorobutadiene, and 1,2,3-trichlorobenzene—are not compatible with this instrument. The technique is limited to the determination of those compounds with sufficient volatility to be removed from the sample in detectable concentrations using the equilibrium headspace technique.

Performance Characteristics

The HAPSITE/headspace GC/MS method is applicable to a wide range of organic compounds that have sufficiently high volatility to be effectively removed from water, soil, or sludge samples via equilibrium headspace. The chemical compounds shown in Table 2-1 have been evaluated by Inficon personnel during method development and are suitable for analysis with the HAPSITE.

Practical Quantitation Limits and Method Detection Limits

The practical quantitation limits (PQLs) for chemical analytes in water are also listed in Table 2-1. The practical quantitation limit is the lower bound of the calibration range and represents a peak-to-peak signal-to-noise ratio of 10:1. This signal level provides acceptable and reproducible (±20%) signal integration with the HAPSITE software. The method detection limit (MDL) is estimated at one half the PQL.

Accuracy

The HAPSITE GC/MS headspace system is expected to perform at an accuracy level of ±25% or better over the calibration range 95% of the time.

Precision

The precision, as represented by the relative standard deviation (RSD) on replicate measurements, is expected to be ≤20% over the working range of the instrument.[1]

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[1] The relative standard deviation is the sample standard deviation divided by the mean value and multiplied by 100.

Table 2-1. Inficon HAPSITE GC/MS Analyte List

Compound	CAS[a] Number	PQL[b] (μg/L)	Quant. Mass[c]
Benzene	71-43-2	5	78
Bromobenzene	108-86-1	10	77
Bromochloromethane	74-97-5	15	49
Bromodichloromethane	75-27-4	5	83
Bromoform	75-25-2	15	173
Bromomethane	74-83-9	5	94
Carbon tetrachloride	56-23-5	5	117
Chlorobenzene	108-90-7	5	112
Chloroethane	75-00-3	10	64
Chloroform	67-66-3	5	83
Chloromethane	74-87-3	5	50
Dibromochloromethane	124-48-1	5	129
1,2-Dibromoethane	106-93-4	5	107
Dibromomethane	95-50-1	5	174
Dichlorodifluoromethane	75-71-8	10	85
1,1-Dichloroethane	75-35-3	5	63
1,2-Dichloroethane	107-06-2	5	62
1,1-Dichloroethene	75-35-4	5	61
cis-1,2-Dichloroethene	156-59-2	5	61
trans-1,2-Dichloroethene	156-60-5	5	61
1,2-Dichloropropane	78-87-5	10	63
2,2-Dichloropropane	594-20-7	10	77
1,1-Dichloropropene	563-58-6	10	75
cis-1,3-Dichloropropene	10061-01-5	10	75
trans-1,3-Dichloropropene	10061-02-6	10	75
Ethyl benzene	100-41-4	5	91
Isopropyl benzene	98-82-8	10	105
Methylene chloride	75-09-2	5	49
Styrene	100-42-5	5	104
1,1,1,2-Tetrachloroethane	630-20-6	20	131
1,1,2,2-Tetrachloroethane	79-34-5	20	83
Tetrachloroethene	127-18-4	5	166
Toluene	108-88-3	5	91
1,1,1-Trichloroethane	71-55-6	5	97
1,1,2-Trichloroethane	79-00-5	5	97
Trichloroethene	79-01-6	5	130
Trichlorofluoromethane	75-69-4	5	101
1,2,3-Trichloropropane	96-18-4	15	75
Vinyl chloride	75-01-4	5	62
ortho-Xylene	95-47-6	5	91
meta-Xylene	108-38-3	5	91
para-Xylene	106-42-3	5	91

Notes: This table was provided by the instrument developer. The PQL is defined as a peak-to-peak signal-to-noise ratio of 10:1.

[a] CAS = *Chemical Abstracts* Service.

[b] PQL = practical quantitation limit.

[c] Quant. mass = quantification mass.

Instrument Working Range

The HAPSITE can measure the volatile organics listed in Table 2-1 over a dynamic range of 10^4. For tetrachloroethene, this would represent a working range of 5 µg/L to 50 mg/L. The working range of the instrument can be adjusted from the lower limit upward by controlling the injection volume. For a sample containing tetrachloroethene in the 10- to 100-mg/L range, the injection volume could be reduced by 50% to allow measurement within the linear dynamic range of the instrument.

Comparison with Reference Laboratory Analyses

The HAPSITE GC/MS analytical results for VOCs in water are expected to differ from reference laboratory measurements, using Method 8260A (EPA, 1986b), by no more than ±35%, 95% of the time.

Data Completeness

Analysis and valid results will be reported for 90% or more of the samples presented for analysis during the demonstration.

Other Analytical Performance Characteristics and Requirements

An MS tune check is performed every 12 hours to manufacturer's specification using the tuning compounds 1,3,5 tris-(trifluoromethyl) benzene and bromopentafluorobenzene. This tune check verifies the stability of the instrument. The system must pass the tune check prior to sample analysis. In addition, the initial calibration curve for all target analytes must generate a relative standard deviation of 30% or less for each compound in the calibration. A GC/MS calibration check is performed at least once during every 12 hours of operation. Specific analytes from the initial calibration curve are designated as calibration check compounds (CCCs). All CCC sample results must be within 25% of initial calibration results. System blanks must also be run prior to field sample analysis. Instrument carryover from a high concentration sample to a low concentration sample will be less than 0.25% of the high sample. For example, a 5-mg/L sample of tetrachloroethene should generate a result of less than 12.5 µg/L in a blank sample immediately following the high-level sample.

Other Field Performance Characteristics

The following performance parameters are provided by the instrument developer.

Instrument Setup and Disassembly Time

The HAPSITE GC/MS requires 30 minutes for setup and disassembly. The HAPSITE and headspace accessory can be shipped or carried as checked baggage. The carrier and internal standard gas canisters must be shipped as hazardous materials.

Instrument Calibration Frequency During Field Use

An MS tune check is required at startup and after every 12 hours of operation. A daily calibration check is also required at startup and after every 12 hours of operation.

Ancillary Equipment Requirements

A power source of 110-V 60-Hz ac is desirable for initial startup of the instrument. Approximately 40% of the battery life is expended in startup. Normal operation is to start the instrument on ac power prior to taking it to the field. During field use, the instrument is powered by battery. Carrier gas and tuning gases are required. A 20-mL Luer lock syringe, 40-mL volatile organic analysis (VOA) vials, and 10-μL syringes for internal standards and surrogates are also required.

Field Maintenance Requirements

Battery life is 3 hours; carrier gas must be replaced every 8 hours. Operation in the field requires a nonevaporative getter or chemical pump. The usable life on the pump is 30 days at 8 hours of operation per day.

Sample Throughput Rate

Initial headspace analysis equilibration time for the first sample is 30 minutes. Analysis time is 15 minutes per sample. Up to four samples can be equilibrated simultaneously while an analysis is being carried out on a fifth sample.

Operator Training Requirements and Ease of Operation

The HAPSITE GC/MS requires 3 days of training for technical personnel familiar with GC/MS operation. This training includes setup and maintenance of the instrument and methods. Training for field operation of the instrument (sample preparation and injection only) requires 1 day.

Chapter 3
Demonstration Design and Description

Introduction

This chapter summarizes the demonstration objectives and describes related field activities. The material is condensed from the Demonstration Plan for Wellhead Monitoring Technology Demonstration (Sandia, 1997), which was reviewed and approved by all participants prior to the field demonstration.

Overview of Demonstration Design

The primary objective was to test and verify the performance of field-portable characterization and monitoring technologies for the analysis of chlorinated VOCs in groundwater. Specific demonstration objectives are listed below:

- verify instrument performance characteristics that can be directly quantified; such factors include response to blank samples, measurement accuracy and precision, data completeness, sample throughput, etc.;
- verify instrument characteristics and performance in various qualitative categories such as ease of operation, required logistical support, operator training requirements, transportability, versatility, and other considerations; and
- compare instrument results with data from standard laboratory analytical methods currently used to analyze groundwater for chlorinated VOCs.

The experimental design included a consideration of both quantitative and qualitative performance factors for each participating technology.

Quantitative Factors

The primary quantitative performance factors that were verified included such instrument parameters as precision and accuracy, blank sample response, instrument performance at sample concentrations near its limit of detection, sample throughput, and comparability with reference methods. An overview of the procedures used to determine quantitative evaluation factors is given below.

Precision

Measurement uncertainty was assessed over the instrument's working range by the use of blind replicate samples from a number of performance evaluation (PE) mixtures. Eight PE mixtures containing chlorinated VOCs at concentrations ranging from 50 µg/L to over 1000 µg/L were prepared and distributed at each site. The mixtures were prepared from certified standard mixes with accompanying documentation giving mixture content and purity. The relative standard deviation was computed for each compound contained in each set of replicate PE samples and was used as a measure of instrument precision.

Accuracy

Instrument accuracy was also evaluated by using results from the PE samples. A mean recovery was computed for each reported compound in each PE mixture. The average instrument result for each compound, based on four blind replicate sample analyses, was compared against the known concentration in the PE mixture and reported as the average percent recovery and the absolute percent difference.

Blank Sample Response

At least two blank groundwater samples were analyzed with each instrument system per demonstration day. These were distributed as blind samples in the daily set of samples provided to each instrument operator. The results from these samples were used to assess the degree to which instrument contamination and sample-to-sample carryover resulted in a false positive.

Low-Level Sample Response

The scope of this demonstration did not include an exhaustive determination of instrument detection limits. However, 10 replicate spiked samples at concentrations near typical regulatory action limits were provided for analysis at each site to validate the instrument performance at these low concentration levels. The results from these analyses were compiled as detects and nondetects and were used to calculate the percentage of correct determinations and false negatives.

Sample Throughput

Sample throughput takes into account all aspects of sample processing, including sample preparation, instrument calibration, sample analysis, and data reduction. The multiday demonstration design permitted the determination of sample throughput rates over an extended period. Thus the throughput rates are representative of those likely to be observed in routine field use of the instrument.

Laboratory–Field Comparability

The degree to which the field measurements agree with reference laboratory measurements is a useful parameter in instrument evaluation. In this demonstration, comparisons were made on groundwater samples by computing the absolute percent difference between laboratory and field technology results for all groundwater contaminants detected. Linear regression of the two data sets was also carried out to determine the strength of the linear correlation between the two data sets.

Qualitative Factors

Key qualitative instrument performance factors observed during the demonstration were instrument portability, logistical support requirements, operator training requirements, and ease of operation. Logistical requirements include the technology’s power requirements, setup time, routine maintenance, and the need for other equipment or supplies, such as a computers, reagent solutions, or gas mixtures. Qualitative factors were assessed during the demonstration by review of vendor information and on-site audits. Vendors provided information concerning these factors during preparation of the demonstration plan. Vendor claims regarding these specifications and requirements are included in Chapter 2. During the field demonstration phase, auditors from the verification organization observed instrument operation and documented the degree of compliance with the instrument specifications and methodology. Audit results are included in Chapter 6.

Site Selection and Description

Two sites—the DOE Savannah River Site near Aiken, South Carolina, and McClellan Air Force Base near Sacramento, California—were chosen for this demonstration. This section provides a brief history of each site, a discussion of important geological features, and an outline of the nature and extent of contamination at each site. The sites chosen met the following selection criteria:

- presence of chlorinated VOCs in groundwater;
- multiple wells at the site with a variety of contaminants and depths;
- documented well-sampling history with characterization and monitoring data;
- convenient access; and
- support facilities and services at the site.

Savannah River Site

The Savannah River Site is operated under contract by the Westinghouse Savannah River Company. The complex covers 310 square miles in western South Carolina, adjacent to the Savannah River, as shown in Figure 3-1. The SRS was constructed during the early 1950s to produce the basic materials used in the fabrication of nuclear weapons, primarily tritium and plutonium-239. Production of weapons material at the SRS also produced unusable byproducts such as intensely radioactive waste. In addition to these high-level wastes, other wastes at the site include low-level solid and liquid radioactive wastes, transuranic waste, hazardous chemical waste, and mixed waste.

Figure 3-1. The general location of the Savannah River Site in the southeast United States.

Geological Characteristics

The SRS is located on the upper Atlantic Coastal Plain. The site is underlain by a thick wedge (approximately 1000 feet) of unconsolidated Tertiary and Cretaceous sediments that overlie Precambrian and Paleozoic metamorphic rocks and consolidated Triassic sediments (siltstone and sandstone). The younger sedimentary section consists predominantly of sand and sandy clay. The depth to the water table from the surface ranges from 50 to 170 feet for the wells used in this demonstration.

Groundwater and Monitoring Wells
The wells selected for sampling in this demonstration were in the A/M area, located in the northwest section of the site. This area encompasses an abandoned process transfer line that, beginning in 1958, carried wastewater for 27 years from M-area processing facilities to a settling basin. Site characterization data indicate that several leaks occurred in the transfer line, which is buried about 20 feet below the surface, producing localized contamination. Past industrial operations resulted in the release of chlorinated solvents, primarily trichloroethene (TCE), tetrachloroethene (PCE), and 1,1,1-trichloroethane, to the subsurface.

The A/M area monitoring-well network, shown in Figure 3-2, consists of approximately 400 wells. The dark squares in the figure indicate soil borings and the light squares indicate monitoring wells. The largest group of wells, comprising approximately 70% of the total, are associated with the plume originating from the process transfer lines and the settling basin. The majority of these wells are constructed of 4-inch poly(vinyl chloride) (PVC) casing with wire-wrapped screens varying in length from 5 to 30 feet. The wells are screened either in the water-table aquifer (M-area aquifer, well depths ranging from 30 to 170 feet), the underlying tertiary aquifer (Lost Lake aquifer, well depths ranging from 170 feet to 205 feet), or a narrow permeable zone within the confining unit above the cretaceous aquifer (Crouch Branch Middle Sand, well depths ranging from 215 to 260 feet). The wells are all completed with approximately 2.5 feet of standpipe above ground and a protective housing. Most wells are equipped with a dedicated single-speed centrifugal pump (1/2 hp Grundfos Model 10S05-9) that can be operated with a control box and generator. Wellhead pump connections also contain a flow meter and totalizer for monitoring pumped volumes.

All the wells are measured quarterly for water levels. On a semiannual basis, all point-of-compliance wells (41), plume definition wells (236), and background wells (6) are sampled to assess compliance with groundwater protection standards. Other water quality parameters such as conductivity, turbidity, temperature, and pH are

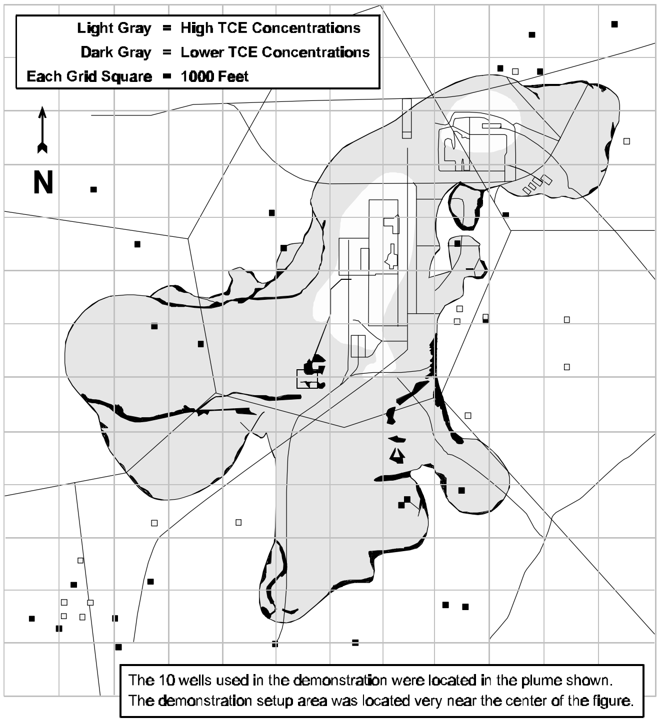

Figure 3-2. A map of the A/M area at the Savannah River Site showing the subsurface TCE plume.

also measured. As a part of the monitoring program, VOCs are measured using EPA Method 8260A at an off-site contract laboratory. The most recent (winter of 1996) quarterly water analysis results for the 10 wells used in this demonstration are shown in Table 3-1. Well cluster numbers shown in the table include a letter designation (A through D) that indicates the relative screening depth and aquifer zone. The A wells are the deepest of a cluster, while the D wells mark the shallowest.

Table 3-1. Quarterly Monitoring Results for SRS Wells Sampled in the Demonstration

Sample Description	Well Number	Compound	Qtrly. Results[a] (µg/L)
Very low 1	MSB 33B	Trichloroethene Tetrachloroethene	10 5
Very low 2	MSB 33C	Trichloroethene Tetrachloroethene	5 12
Low 1	MSB 18B	Trichloroethene Tetrachloroethene 1,1-Dichloroethene	12 12 3
Low 2	MSB 37B	Trichloroethene Tetrachloroethene Carbon tetrachloride	28 2 2
Mid 1	MSB 4D	Trichloroethene Tetrachloroethene	219 178
Mid 2	MSB 64C	Trichloroethene Tetrachloroethene 1,1-Dichloroethene	51 337 13
Very high 1	MSB 4B	Trichloroethene Tetrachloroethene	830 43
Very high 2	MSB 70C	Trichloroethene Tetrachloroethene 1,1-Dichloroethane 1,1,1-Trichloroethane	1290 413 61 17
Very high 1	MSB 14A	Trichloroethene Tetrachloroethene	3240 2440
Very high 2	MSB 8C	Trichloroethene Tetrachloroethene	3620 2890

[a] Winter 1996.

McClellan Air Force Base

McClellan Air Force Base is located 7 miles northeast of downtown Sacramento, California, as shown in Figure 3-3. The installation consists of about 3000 acres bounded by the city of Sacramento on the west and southwest, the city of Antelope on the north, the unincorporated areas of Rio Linda on the northwest, and North Highlands on the east.

McClellan has been an active industrial facility since its dedication in 1936, when it was called the Sacramento Air Depot. Operations have changed from maintenance of bombers during World War II and the Korean War, to maintenance of jet aircraft in the 1960s, and now include the maintenance and repair of communications equipment and electronics. McClellan currently operates as an installation of the Air Force Materiel Command and employs approximately 13,400 military and civilian personnel.

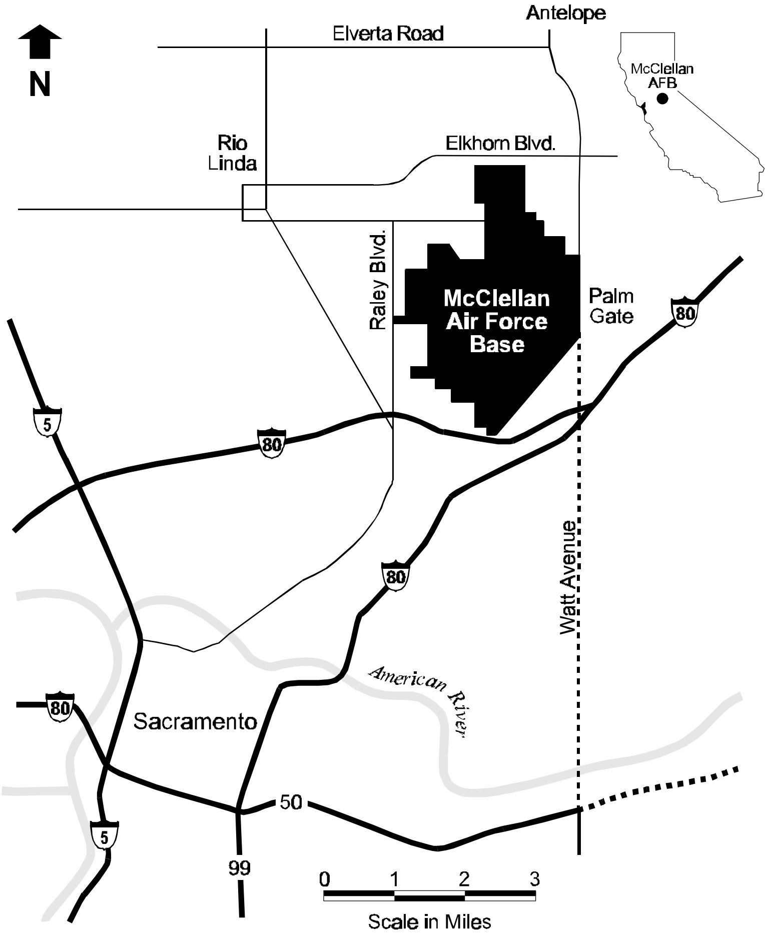

Figure 3-3. A map of Sacramento and vicinity showing the location of McClellan Air Force Base.

Currently, most of the industrial facilities are located in the southeastern portion of the base. The southwestern portion has both industrial and storage areas. In the far western part are vernal pools and wetland areas. Between these wetlands and the engine test cells along the taxiways is an open area that was used for disposal pits.

McClellan Air Force Base is listed on the EPA Superfund National Priorities List of hazardous waste sites. The most important environmental problem at MAFB is groundwater contamination caused by the disposal of hazardous wastes, such as solvents and oils, into unlined pits. Approximately 990 acres beneath McClellan are contaminated with volatile organic compounds. Remediation activities at MAFB include an extensive groundwater pump-and-treat network, as well as soil-vapor extraction systems.

McClellan has been designated a Chlorinated Hydrocarbons Remedial Demonstration Site as part of the National Environmental Technology Test Sites program. The Strategic Environmental Research and Development Program is the parent organization that provides support staff for the environmental technologies undergoing development and testing at MAFB.

Geological Characteristics

Surface features at MAFB include open grassland, creeks and drainages, and vernal pools, as well as industrial, residential, and runway areas. The land surface is a relatively flat plain that slopes gently to the west. Surface elevations range from about 75 feet above mean sea level on the eastern side of the base to about 50 feet above mean sea level on the western side.

Surface soils at MAFB are variable, but are generally sediments that have formed from stream erosion of granite rocks in the Sierra Nevada. Soil in the vadose zone—the unsaturated region between the surface and the groundwater table—is composed of interbedded layers of sands, silts, and clays. The vadose zone ranges from 90 to 105 feet. Clays and hardpan layers in this zone slow, but do not halt, infiltration of liquids into the underlying aquifer.

The groundwater beneath MAFB behaves as one hydrogeologic unit. This single aquifer has been divided into five groundwater monitoring zones, designated A, B, C, D, and E, from shallowest to deepest.

Groundwater and Monitoring Wells

An estimated 14 billion gallons of contaminated water underlie MAFB. Trichloroethene is the most frequently detected contaminant in the subsurface groundwater. Over 90% of the contaminant mass is located in the A zone, the shallowest portion of the aquifer. An estimated surface area of approximately 664 acres is underlain by a plume in the A zone that exceeds the 5-µg/L maximum contaminant level for TCE, as shown in Figure 3-4. Groundwater contaminants consistently detected above federal maximum concentration limits (MCLs) are shown in Table 3-2.

Other detected compounds that are either below regulatory levels or are not currently regulated are also shown in the table.

Monitoring wells at McClellan range from 2 to 8 inches in diameter. Well casings are Schedule 5 stainless steel (304) and the well screen is Johnson stainless steel (304) with a 0.01- or 0.02-inch screen slot size. The screen is surrounded by either 16 × 40 or 8 × 20 mesh gravel pack to a level about 3 feet above the screen. An approximately 3-foot sand bridge and 3-foot bentonite seal are placed above the gravel pack. A concrete sanitary seal containing about 3% bentonite powder is used to seal the well casing between the bentonite seal and the ground surface.

For this demonstration, monitoring wells that penetrate both A and B aquifer zones in operational units A and B were selected for sample collection. Quarterly monitoring data exist for 354 wells at the A and B zone aquifer levels in these operational units. Monitoring results for TCE were used to select ten wells. Groundwater TCE concentrations in the selected wells ranged from very low (~10 µg/L) to very high (>5000 µg/L) levels.

Wells that had multiple contaminants or nonchlorinated contaminants were given selection preference over those with only a few chlorinated hydrocarbons. The most recent (winter of 1996) monitoring results for the wells chosen for this demonstration are shown in Table 3-3.

Sample Set Descriptions

The experimental design of the demonstration specified the preparation and collection of an approximately equal number of PE samples and groundwater samples for distribution to the participants and reference laboratory. Descriptions of the PE and groundwater samples and their preparation are given below.

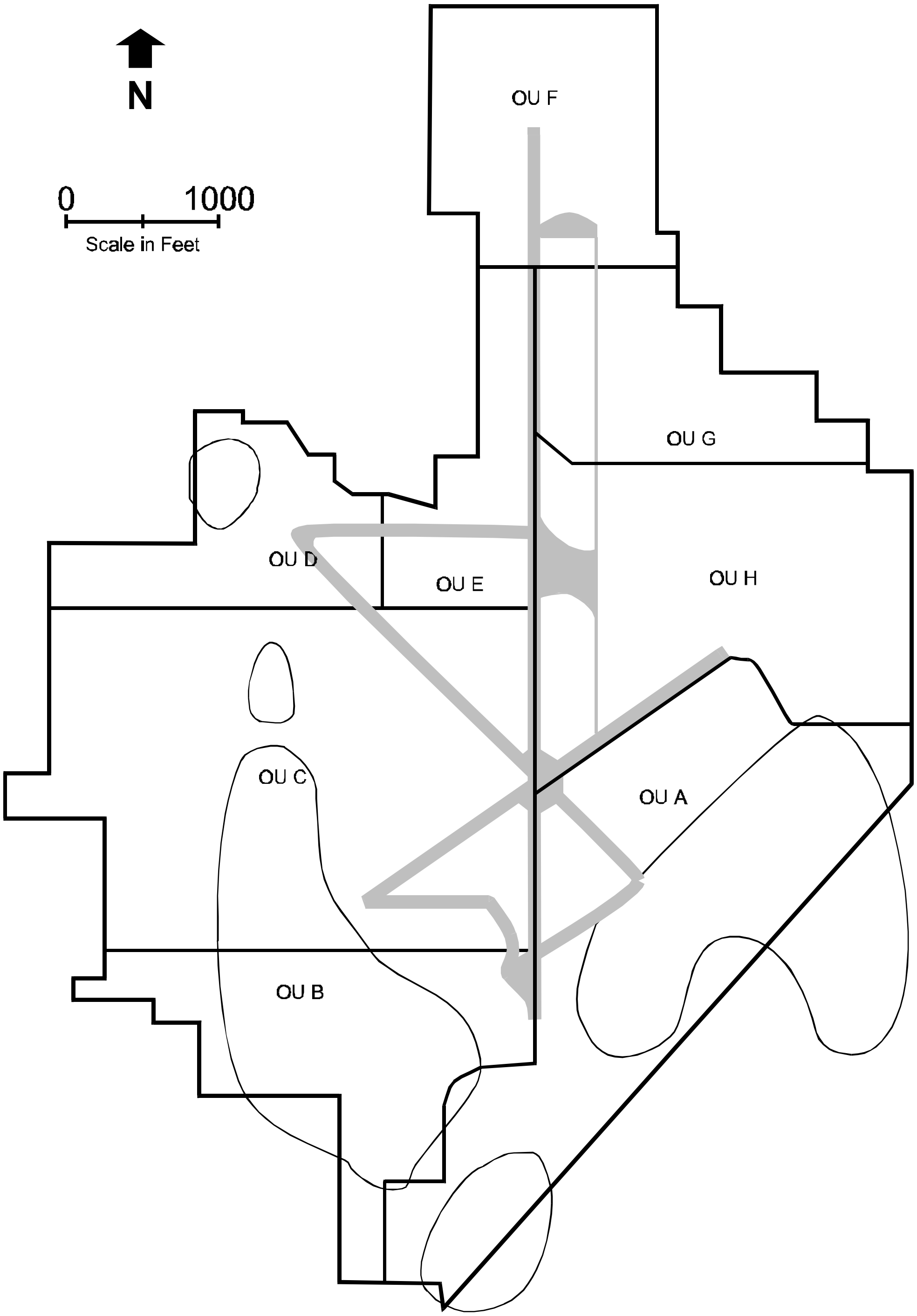

Figure 3-4. Subsurface TCE plumes at McClellan Air Force Base in the shallowest (A) aquifer layer. The circular lines enclose plume concentrations in excess of 5 µg/L TCE. OU refers to operational units. Monitoring wells used in the demonstration were primarily in OUs A and B. The demonstration setup area was very near OU D (upper left in the figure).

Table 3-2. Groundwater Contaminants at MAFB

Detected above MCL[a]	Detected below MCL	Detected – Not Regulated
Benzene	Bromodichloromethane	Acetone
Carbon tetrachloride	Trichlorofluoromethane	2-Butanone
Chloroform		1,1-Dichloroethane
1,2-Dichlorobenzene		4-Methyl-2-pentanone
1,2-Dichloroethane		Toluene
1,1-Dichloroethene		
1,2-Dichloroethene (cis and trans)		
Tetrachloroethene		
1,1,1-Trichloroethane		
Trichloroethene		
Vinyl chloride		

[a] MCL = maximum concentration limit.

Table 3-3. Quarterly Monitoring Results for MAFB Wells Sampled in the Demonstration

Sample Description	Well Number	Compound	Qtrly. Results[a] (μg/L)
Very low 1	EW-86	Trichloroethene 1,1-Dichloroethene	8 13
Very low 2	MW-349	Trichloroethene Tetrachloroethene Chloroform Acetone	9 5 8 9
Low 1	MW-331	1,1-Dichloroethane Carbon tetrachloride Chloroform Trichloroethene *cis*-1,2-Dichloroethene	16 5 7 19 41
Low 2	MW-352	1,1-Dichloroethane Tetrachloroethene Freon11	6 5 115
Mid 1	EW-87	1,1,1-Trichloroethane 1,1-Dichloroethene Trichloroethene *cis*-1,2-Dichloroethene	17 334 220 5
Mid 2	MW-341	Trichloroethene *cis*-1,2-Dichloroethene	350 18
High 1	MW-209	Chloroform Trichloroethene *cis*-1,2-Dichloroethene *trans*-1,2-Dichloroethene	53 586 80 13
High 2	MW-330	Chloroform Trichloroethene *cis*-1,2-Dichloroethene *trans*-1,2-Dichloroethene	44 437 64 9

Table 3-3. Quarterly Monitoring Results for MAFB Wells Sampled in the Demonstration (Continued)

Sample Description	Well Number	Compound	Qtrly. Results[a] (μg/L)
Very high 1	MW-334	1,1-Dichloroethene	1000
		Benzene	705
		Carbon tetrachloride	728
		Chloroform	654
		Dichloromethane	139
		Trichloroethene	20,500
		cis-1,2-Dichloroethene	328
		Xylene	59
Very high 2	MW-369	1,2-Dichloroethane	13
		Carbon tetrachloride	91
		Chloroform	84
		Tetrachloroethene	6
		Trichloroethene	10,200
		cis-1,2-Dichloroethene	246

[a] Winter 1996.

PE Samples and Preparation Methods

Three different commercially available (Supelco, Bellefonte, Pennsylvania) standard solutions of chlorinated VOCs in methanol were used to prepare the PE mixtures. The standard solutions were supplied with quality control documentation giving the purity and weight of the compounds in the mixture. The contents of the three mixtures, termed mix 1, mix 2, and mix 3, are given in Table 3-4. VOC concentration levels in these standard solutions were either 200 μg/L or 2000 μg/L. The PE mixtures were prepared by dilution of these standard solutions.

The number of replicate samples and the compound concentrations from each of the nine PE mixtures prepared at each site are given in Table 3-5 for the SRS and Table 3-6 for MAFB. Ten replicates of the mixture with the lowest concentration level were prepared so technology performance statistics near typical regulatory action levels could be determined. Four replicates were prepared for each technology and the reference laboratory from the other eight PE mixtures. The highest-level PE mixture, denoted “spike/low” in the tables, consisted of high-level (>1000 μg/L) concentrations of TCE and PCE (and other compounds at MAFB as noted in the table) in the presence of a low-level (50 or 100 μg/L) PE mixture background. Eight blank samples were also provided to each technology at each site. The blank samples were prepared from the same batch of deionized, carbon-filtered water used to prepare the PE mixtures.

Performance evaluation mixtures were prepared in either 8-L or 10-L glass carboys equipped with bottom spigots. Stock PE solutions were dispensed with microsyringes into a known volume of deionized, carbon-filtered water in the carboy. The mixture was gently stirred for 5 minutes with a Teflon-coated stir bar prior to dispensing samples from the bottom of the carboy. A twofold excess volume of PE mixture was prepared in order to ensure a sample volume well in excess of the required volume. The mixture was not stirred during sample dispensing to minimize headspace losses in the lower half of the carboy. Headspace losses that did occur during dispensing were limited to the top portion of the mixture, which was discarded after the samples were dispensed. Samples were dispensed into bottles specified by participants (40 mL, 250 mL, and 1 L) with zero

Table 3-4. Composition of PE Source Materials

PE Mix 1 - Purgeable A Supelco Cat. No. 4-8059 Lot LA68271	PE Mix 2 - VOC 3 Supelco Cat. No. 4-8779 Lot LA64701	PE Mix 3 - Purgeable B Supelco Cat. No. 4-8058 Lot LA 63978
Trichlorofluoromethane	1,1-Dichloropropene	1,2-Dichloroethane
1,1-Dichloroethane	1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
Dichloromethane	Trichloroethene	*cis*-1,3-Dichloropropene
1,1-Dichloroethene	1,2-Dichloropropane	*trans*-1,3-Dichloropropene
Chloroform	1,1,2-Trichloroethane	*trans*-1,2-Dichloroethene
Carbon tetrachloride	1,3-Dichloropropane	1,1,1-Trichloroethane
Trichloroethene	1,2-Dibromoethane	Benzene
1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	Bromodichloromethane
1,1,2-Trichloroethane	1,1,2,2-Tetrachloroethane	Toluene
Tetrachloroethene	1,2,3-Trichloropropane	Ethyl benzene
Dibromochloromethane	1,2-Dibromo-3-chloropropane	Bromoform
Chlorobenzene	*cis*-1,3-Dichloropropene	
1,2-Dichlorobenzene	*trans*-1,3-Dichloropropene	
2-Chloroethyl vinyl ether	Hexachlorobutadiene	

Table 3-5. PE Sample Composition and Count for SRS Demonstration

Sample Concentration Level	PE Mixture - Mixture Concentration[a]	No. of Replicates
Very low level	VOC Mix 1 - 10 µg/L	10
Low level	VOC Mix 1 - 50 µg/L	4
	VOC Mix 2 - 100 µg/L	4
Mid level	VOC Mix 1 - 200 µg/L	4
	VOC Mix 2 - 200 µg/L	4
High level	VOC Mix 1 - 600 µg/L	4
	VOC Mix 2 - 800 µg/L	4
Spike / low	1.02 mg/L TCE spike + 50 µg/L mix 1	4
	1.28 mg/L TCE and 1.23 mg/L PCE spike + 100 µg/L mix 2	4
Total number of samples		42

[a] TCE = trichloroethene; PCE = tetrachloroethene.

headspace. The samples for field analysis were not preserved with chemical additives since sterile, nutrient-free water was used in their preparation.

Reference laboratory samples were preserved by acidification as specified in Method 8260A. Following preparation, all samples were kept under refrigeration until they were distributed to participants. All PE mixtures were prepared and dispensed on the weekend before the demonstration week.

Table 3-6. PE Sample Composition and Count for MAFB Demonstration

Sample Concentration Level	PE Mixture - Mixture Concentration[a]	No. of Replicates
Very low level	VOC Mix 3 - 10 µg/L	10
Low level	VOC Mix 3 - 50 µg/L	4
	VOC Mix 2 - 100 µg/L	4
Mid level	VOC Mix 3 - 200 µg/L	4
	VOC Mix 2 - 300 µg/L	4
High level	VOC Mix 1 - 600 µg/L	4
	VOC Mix 2 - 800 µg/L	4
Spike / low	1.22 mg/L TCE, 1.00 mg/L PCE, 0.50 mg/L 11DCA, and 0.50 mg/L BNZN spike + 100 µg/L mix 3	4
	1.04 mg/L 11DCA, 0.86 mg/L BNZN, 0.57 mg/L TCE, and 0.51 mg/L PCE spike + 50 µg/L mix 2	4
Total number of samples		42

[a] TCE = trichloroethene; PCE = tetrachloroethene; 11DCA = 1,1-dichloroethane; BNZN = benzene.

Groundwater Samples and Collection Methods

A total of 33 groundwater samples were provided to each participant and reference laboratory at each demonstration site. These samples were collected from 10 wells selected to cover TCE concentrations ranging from 10 µg/L to >1000 µg/L. The presence of other groundwater contaminants was also considered in well selection, as noted previously. Samples from each well were prepared in either triplicate or quadruplicate to allow statistical evaluation of instrument precision and accuracy relative to the reference laboratory results.

Groundwater at both sites was sampled by the same contract personnel who conduct sampling for quarterly well monitoring. Site-specific standard operational procedures, published in the demonstration plan, were followed at both sites. The sampling procedure is briefly summarized in the next paragraph.

The wells were purged with three well volumes using a submersible pump. During the purge, pH, temperature, and conductivity were monitored. Following well purge, pump flow was reduced and the purge line was used to fill a 10-L glass carboy. This initial carboy volume of groundwater was discarded. The carboy was filled to between 9 and 10 L a second time at a fill rate of 2 to 3 L/minute with the water stream directed down the side of the carboy for minimal agitation. The filled carboy was gently mixed with a Teflon stir bar for 5 minutes. Zero-headspace samples were immediately dispensed from the carboy while it was at the wellhead in the same manner as PE samples. Either three or four replicate samples were prepared for each technology and the reference laboratory. Following dispensing, the sample bottles were placed in a cooler and held under refrigeration until they were distributed to the participants. Groundwater sampling was completed during the first 2 days of each demonstration. Lists of the sampled wells and quarterly monitoring results are given in Tables 3-1 and 3-3 for the SRS and MAFB, respectively.

Sample Handling and Distribution

The distribution and status of all samples were tracked with chain-of-custody forms. Samples were dispensed to participants in small coolers containing a supply of blue ice. Normally, two sets of either 10 or 11 samples were distributed to participants each day during the 4 days of the demonstration, for a total of 83 samples, including blanks, at each site.

Some of the participants required information concerning the content of the samples prior to carrying out an analysis. This information was noted on the chain-of-custody form for each PE and groundwater sample, and was made available to the participants. Recorded information included:

- number of contaminants in the sample;
- list of contaminants in the sample;
- boiling point range of sample constituents; and
- approximate concentration range of contaminants in sample (low, mid, high).

The type of information provided during this demonstration would be required by the technology as a part of its normal operational procedure and did not compromise the results of the test. The information provided to each of the participants is documented in Chapter 5.

Field Demonstration Schedule and Operations

The following schedule was followed at both sites. The field team arrived on the Thursday prior to the demonstration week. Performance evaluation samples were prepared on Friday, Saturday, and Sunday. Technology participants arrived at the site on Monday morning and immediately began instrument setup. The first set of PE samples was normally distributed to all participants by midday Monday. The groundwater sampling crew, consisting of at least two on-site contractors and at least one ETV field-team member, carried out sampling of the 10 wells on Monday and Tuesday. The first groundwater samples were distributed on Wednesday. Thursday was reserved as a visitor day during which local and regional regulatory personnel and other potential instrument users were invited to hear presentations about instrument capabilities as well as to view the instruments in operation. Sample analysis was also performed on Thursday. On Friday, the final day of the demonstration, participants finished sample analysis, packed up, and departed by midafternoon.

Site Operations and Environmental Conditions

Instruments were deployed in parking lots or open fields adjacent to the well networks sampled during each demonstration. All participants came to the site self-equipped with power and shelter. Some came with field-portable generators and staged under tent canopies; others operated their instruments inside vehicles and used dc-to-ac power inverters connected to the vehicle’s battery. Tables were provided for those participants who required a work space. Each team provided its own instrument operators. Specifics regarding instrument setup and the qualifications, training, and experience of the instrument operators are given in Chapter 6.

The SRS demonstration took place on September 8 through 12, 1997, and the MAFB demonstration on September 22 through 26, 1997. The verification organization team staged its operations out of a tent at the SRS and out of a mobile laboratory at MAFB. The PE mixtures at the SRS were prepared at a nearby SRS laboratory facility and in the mobile laboratory at MAFB. Refrigerators at on-site facilities of the groundwater sampling contractors were used to store the samples at both sites prior to their distribution.

Environmental conditions at both sites are summarized in Table 3-7. Conditions at SRS were generally hot and humid. Sporadic rain showers were encountered on one of the test days, but did not impede demonstration activities. Conditions at MAFB were initially hot and progressed to unseasonably hot. Moderately high winds were also encountered during the last 2 days at MAFB.

Table 3-7. Weather Summary for SRS and MAFB During Demonstration Periods

Site/Parameters	Mon	Tue	Wed	Thu	Fri
SRS					
Temperature range (°C)	20 – 34	21 – 33	21 – 28	18 – 30	19 – 33
Relative humidity range (%)	25 – 68	28 – 67	51 – 71	40 – 70	26 – 70
MAFB					
Temperature range (°C)	17 – 33	18 – 36	18 – 37	24 – 35	24 – 35
Relative humidity range (%)	17 – 72	25 – 47	15 – 59	17 – 67	31 – 83
Wind speed range (knots)	0 – 7	3 – 6	1 – 6	4 – 13	2 – 11

Note: Ranges are given for the 7 a.m. to 7 p.m. time interval.

Field Audits

Field auditors were used to observe and record specific features of technology operations. The demonstration goal was to have at least two auditors observe each technology over the course of the two field demonstrations. Audit results are documented in Chapter 6. The following checklist was used by the audit team as a guideline for gathering information during the audit:

- description of equipment used;
- logistical considerations, including size and weight, shipping and power requirements, other required accessories;
- historical uses and applications of the technology;
- estimated cost of the equipment and its field operation;
- number of operators required;
- required operator qualifications;
- description of data produced;
- compounds that the equipment can detect;
- approximate detection limits for each compound, if available;
- initial calibration criteria;
- calibration check criteria;
- corrective actions for unacceptable calibrations;
- specific QC procedures followed;
- QC samples used;
- corrective action for QC samples;
- sample throughput rate;
- time requirements for data analysis and interpretation;
- data output format and description;
- specific problems or breakdowns occurring during the demonstration;
- possible sample matrix interference; and
- other auditor comments and observations.

Data Collection and Analysis

The analytical results were collected in hardcopy format at the end of each day. These results were used to document sample completion and throughput. The participants also provided a compilation of their results on computer disks at the conclusion of each demonstration week. No feedback on analytical results or performance was given to the participants during the course of either demonstration week. Following the SRS demonstration, and only after all results were submitted, was qualitative verbal feedback given to each participant concerning their accuracy and precision on SRS PE sample results. This was reasonable since a well-defined monitoring plan would use preliminary samples to determine control limits and to make system modifications or refinements prior to advancing to the next phase of sampling and analysis. Three weeks following the MAFB demonstration, copies of all submitted data were entered into spreadsheets by the verification organization and transmitted to participants for final review. This gave each participant the opportunity to detect and change calculation or transcription errors. If other more substantive changes were proposed, they were submitted to the verification organization, along with documentation outlining the rationale for the change. Following this final data review opportunity, no other data changes were permitted. The extent and nature of any changes are discussed in Chapter 6.

Demonstration Plan Deviations

The following deviations from the written demonstration plan were recorded during the field demonstration. The impact of each deviation on the overall verification effort, if any, is also included.

- Five blank samples were submitted to the reference laboratory from the SRS demonstration instead of the 8 samples specified in the demonstration plan. The impact on the verification effort was minimal since a total of 13 blanks (8% of the total field sample count) were analyzed by the reference laboratory.
- During groundwater sampling of SRS well MSB 14A, two 250-mL sample bottles were not filled. Omission of this sample resulted in a double replicate sample set instead of a triple replicate for Electronic Sensor Technology and Sentex. The impact on the study was insignificant since this omission accounted for only 1 sample out of a total groundwater sample count of 33.
- The demonstration plan specified that only two VOC mixtures would be used at each demonstration site. In fact, three mixtures were used at the MAFB demonstration (Table 3-6) to add complexity to the sampling. This change caused some minor confusion with one of the developers, who was not expecting this particular set of compounds at MAFB. The most significant impact of this change was a loss of time for the affected developer as a result of extended data review of the unanticipated mixture. The misunderstanding was verbally clarified and no further problems were encountered. The results from the high-level VOC mix 1 were not used in the statistical analyses.

Chapter 4
Laboratory Data Results and Evaluation

Introduction

A reference laboratory was used to verify PE sample concentrations and to generate analytical results for all groundwater samples using EPA Method 8260A. This chapter includes a brief description of the reference laboratory and its data quality control program; the methodology and accompanying quality control procedures employed during sample analysis; and laboratory results and associated measures of data quality for both demonstration sites.

Reference Laboratory

DataChem Laboratories (DCL) in Salt Lake City, Utah, was chosen as the reference laboratory for both phases of this demonstration. This is a full-service analytical laboratory with locations in Salt Lake City and Cincinnati, Ohio. It provides analytical services in support of environmental, radiological, mixed-waste, and industrial hygiene programs. DataChem's qualifications include U.S. EPA Contract Laboratory Program participation in both inorganic and organic analysis and American Industrial Hygiene Association accreditation, as well as U.S. Army Environmental Center and U.S. Army Corps of Engineers (Missouri River Division) certification. State-specific certifications for environmental analytical services include Utah, California, Washington, New Jersey, New York, Florida, and others.

Laboratory Selection Criteria

Selection criteria for the reference laboratory included the following: relevant laboratory analytical experience, adequacy of QC documentation, turnaround time for results, preselection audit results, and cost. Early discussions with DCL revealed that the laboratory conducts a high number of water analyses using Method 8260A. Prior to laboratory selection, a copy of the DataChem Quality Assurance Program Plan (DataChem, 1997) was carefully reviewed. This document outlines the overall quality assurance program for the laboratory and provides specific quality control measures for all the standard analytical methods used by the laboratory. Laboratory analysis and reporting time for sample analysis was 21 days, with a per-sample cost of $95.

In June 1997, Sandia sent several PE water samples to DCL for evaluation. Laboratory performance on these samples was reviewed during an audit in June 1997. The laboratory detected all compounds contained in the PE mixtures. Reported concentration levels for all compounds in the mixtures were within acceptable error margins. The audit also indicated that the laboratory conducted its operations in accordance with its QA plan. The results of this preliminary investigation justified the selection of DCL as the reference laboratory and provided ample evidence of the laboratory's ability to correctly use Method 8260A for the analysis of demonstration samples.

Summary of Analytical Work by DataChem Laboratories

In addition to the preselection audit samples noted above, DCL also analyzed predemonstration groundwater samples collected at SRS in August 1997. During the demonstration phase, DCL was sent split samples of all PE and groundwater samples given to the demonstration participants from both the Savannah River and McClellan sites. A total of 90 and 91 samples from the SRS and MAFB demonstrations, respectively, were received and analyzed by the laboratory. Over the course of 1 month, demonstration samples were run in 9 batches of approximately 20 samples per batch. The results were provided in both hardcopy and electronic format. The hard copy included all paperwork associated with the analysis, including the mass spectral information for each compound detected and complete quality control documentation. The electronic copy was provided in spreadsheet format and included only the computed result for each target compound in each sample.

Preselection evaluation of DCL established their competence in the use of Method 8260A. In light of these findings and in an effort to expedite laboratory analysis of demonstration samples, an estimate of the concentration levels of target compounds in both PE and groundwater samples was provided to the laboratory with each batch of samples. With a knowledge of the approximate concentration range of the target compounds, the analyst was able to dilute the sample appropriately, thereby eliminating the need to do multiple dilutions in order to obtain a suitable result within the calibrated range of the instrument.

Summary of Method 8260A

Method 8260A, which is included in the EPA SW-846 compendium of methods, is used to measure volatile organic compounds in a variety of solid waste matrices, including groundwater (EPA, 1996b). The method can be used to quantify most volatile organic compounds with boiling points below 200 °C that are either insoluble or only slightly soluble in water. The method employs a chromatography/mass spectrometric procedure with purge-and-trap sample introduction. An inert gas is bubbled through a vessel containing the water sample. The volatile organic compounds partition into the gas phase and are carried to a sorbent trap, where they are adsorbed. Following the purge cycle, the sorbent trap is heated and the volatile compounds are swept into the GC column, where they are separated according to their boiling points. The gas chromatograph is interfaced directly to a mass spectrometer that bombards the compounds with electrons as they sequentially exit the GC column. The resulting fragments, which possess charge and mass characteristics that are unique for each compound, are detected by the spectrometer's mass detector. The signal from the mass detector is used to build a compound mass spectrum that is used to identify the compound. The detector signal intensities for selected ions unique to each target compound are used to quantify the amount of the compound in the sample.

Method 8260A Quality Control Requirements

Method 8260A specifies a number of quality control activities to be carried out in conjunction with routine sample analysis. These activities are incorporated into DCL QA documentation and are summarized in Table 4-1 (DataChem, 1997). Corrective actions are specified in the event of failure to meet QC criteria; however, for the sake of brevity they are not given in the table. In most cases the first corrective action is a calculation check. Other corrective actions include system recalibration, sample rerun, batch rerun, or flag data.

Summary of Laboratory QC Performance

The following sections summarize the QC activities and results that accompanied the analysis of each sample batch.

Table 4-1. Method 8260A Quality Control Summary

Activity	Frequency	Data Acceptance Criteria
Spectrometer tune check	Bromofluorobenzene standard every 12 hours	Relative abundance; range of characteristic mass fragments meets specifications.
System performance check	SPCC[a] sample every 12 hours	Compound relative response factors must exceed required minimums.
System calibration check	CCC[b] sample every 12 hours	Response factor of CCC varies by no more than ±25% from initial calibration. Internal standard retention time within 30 seconds of last check. Internal standard area response within –50 to 100% of last check.
Lab method blank	One or more per batch (approx. 20 samples)	≤ 3× Detection limit.
Field blank	One or more per batch	≤ 3× Detection limit.
Laboratory control standard	One or more per batch	Compound recovery within established limits.[c]
Matrix spike	One or more per batch	Spike recovery within established limits. [c]
Matrix spike duplicate	One or more per batch	Relative percent difference of check compounds ≤50%.
Surrogate standards	Included in every sample	Recovery within established limits. [c]
Internal standards	Included in every sample	Recovery within established limits. [c]

[a] SPCC = system performance check compounds.

[b] CCC = calibration check compounds.

[c] The laboratory generates control limits that are based on 100 or more analyses of designated compounds. The upper and lower acceptable recovery limits are based on a 3-standard-deviation-interval about the mean recovery from the multiple analyses. The result from a single analysis must fall within these control limits in order to be considered valid.

Target Compound List and Method Detection Limits

The method detection limits and practical quantitation limits for the 34 target compounds used in this demonstration are given in Table 4-2. The PQL marks the lower end of the calibrated working range of the instrument and indicates the point at which detection and reported results carry a 99% certainty. Detects reported between the MDL and PQL carry less certainty and are flagged accordingly in the tabulated results.

Sample Holding Conditions and Times

Method 8260A specifies a maximum 14-day holding time for refrigerated water samples. All samples prepared in the field were kept under refrigeration before and during shipment to the laboratory. Upon receipt at the laboratory, they were held under refrigeration until analysis. All samples were analyzed within the 14-day time period following their preparation or collection.

System Calibration

Method 8260A stipulates that a five-point calibration be carried out using standard solutions for all target compounds across the working range of the instrument. Each mix of compounds is run five times at each of the five points in the instrument range. For an acceptable calibration, precision from these multiple analyses, as

Table 4-2. Reference Laboratory Method Detection Limits for Target Compounds

Target Compound	Method Detection Limit (µg/L)	Practical Quantitation Limit (µg/L)
Trichlorofluoromethane	0.15	1
1,1-Dichloroethane	0.08	1
Methylene chloride	0.10	1
1,1-Dichloroethene	0.08	1
Chloroform	0.07	1
Carbon tetrachloride	0.10	1
1,1-Dichloropropene	0.10	1
1,2-Dichloroethane	0.04	1
Trichloroethene	0.14	1
1,2-Dichloropropane	0.04	1
1,1,2-Trichloroethane	0.09	1
Tetrachloroethene	0.10	1
1,3-Dichloropropane	0.06	1
Dibromochloromethane	0.08	1
1,2-Dibromoethane	0.09	1
Chlorobenzene	0.06	1
1,1,1,2-Tetrachloroethane	0.05	1
1,1,2,2-Tetrachloroethane	0.07	1
1,2,3-Trichloropropane	0.50	1
1,2-Dibromo-3-chloropropane	0.62	1
Hexachlorobutadiene	0.10	1
cis-1,3-Dichloropropene	0.17	1
trans-1,3-Dichloropropene	0.08	1
1,2-Dichlorobenzene	0.17	1
trans-1,2-Dichloroethene	0.17	1
1,1,1-Trichloroethane	0.26	1
Benzene	0.12	1
Bromodichloromethane	0.11	1
Toluene	0.15	1
Ethyl benzene	0.14	1
Bromoform	0.10	1
cis-1,2-Dichloroethene	0.14	1
ortho-Xylene	0.11	1
Acetone	2.9	5

Notes: Detection limits are given for an undiluted 5-mL sample volume. Detection limits are determined annually using the method outlined in 40 CFR Part 136 Appendix B (seven replicates of deionized water spiked at 1 µg/L concentration level). Dilutions of the original sample raise the MDL and PQL values accordingly. Surrogate standards used in the analyses were 1,2-dichloroethane-d_4, toluene-d_8, and 4-bromofluorobenzene. Internal standards were fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4.

given by the relative standard deviation, must be 30% or less. A minimum instrument response factor[1] is also prescribed by the method for a designated subset of compounds termed system performance check compounds (SPCC). The five-point calibration curve from the most recent instrument calibration met the specified precision criteria. The system performance check compound response factors also met method criteria.

Daily Instrument Performance Checks

Daily mass spectrometer tune checks as well as other system performance and calibration checks noted in Table 4-1 were carried out for each of the nine sample batches and met Method 8260A on quality control criteria.

Batch-Specific Instrument QC Checks

Method Blanks

All method blank analyses met established criteria (Table 4-1), with one exception. Hexachlorobutadiene, one of the demonstration target compounds, was detected in two of the method blanks at levels in excess of 3 times the MDL. This compound was a component in one of the standard mixes used in preparing the PE samples because reference laboratory data for this compound were not used in the study. Only one of the participating technologies was calibrated to detect this particular compound. Occasional detection of this compound as a minor instrument contaminant does not adversely affect the analytical results for other target compounds.

Laboratory Control Standard

At least one laboratory control standard was run with each of the nine batches of samples. Recovery values for each component in the mixture are given in Figure 4-1 for SRS analyses and Figure 4-2 for MAFB analyses. Recovery values were all within the laboratory-specific control criteria.

Matrix Spike and Matrix Spike Duplicate

The compounds in the matrix spike were the same as those in the laboratory control standard. Computed matrix spike and matrix spike duplicate recoveries were all within the recovery ranges noted in Table 4-1. The relative percent differences (RPDs)[2] calculated for the matrix spike and matrix spike duplicate samples also met the laboratory criteria of ≤50%. All RPD values from matrix spike analyses were less than 10% for the SRS samples and less than 13% for MAFB samples.

Sample-Specific QC Checks

Internal Standard

All samples met internal standard acceptance criteria except one. All three internal standards in sample SP31 failed to meet area response criteria and results from that sample were not included in the reference data set.

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[1] The response factor is the ratio of instrument response for a particular target compound to the instrument response for an internal standard.

[2] The relative percent difference between two samples is the absolute value of their difference divided by their mean and multiplied by 100.

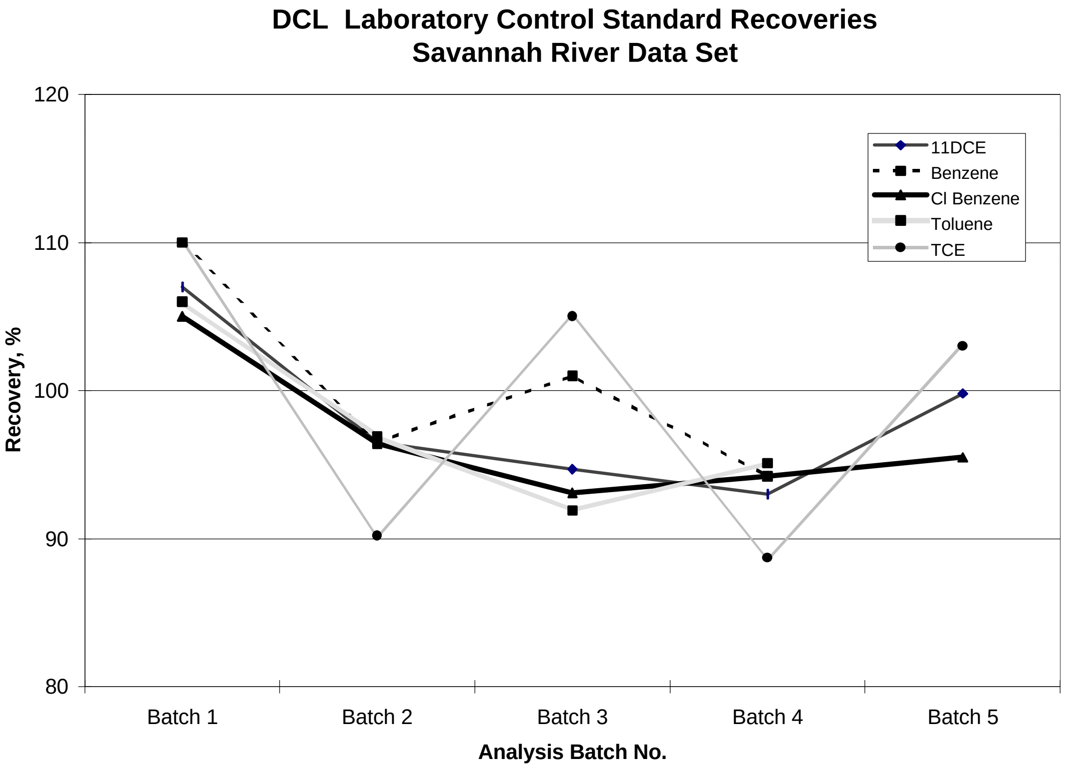

Figure 4-1. Laboratory control standard recovery values for SRS analyses.

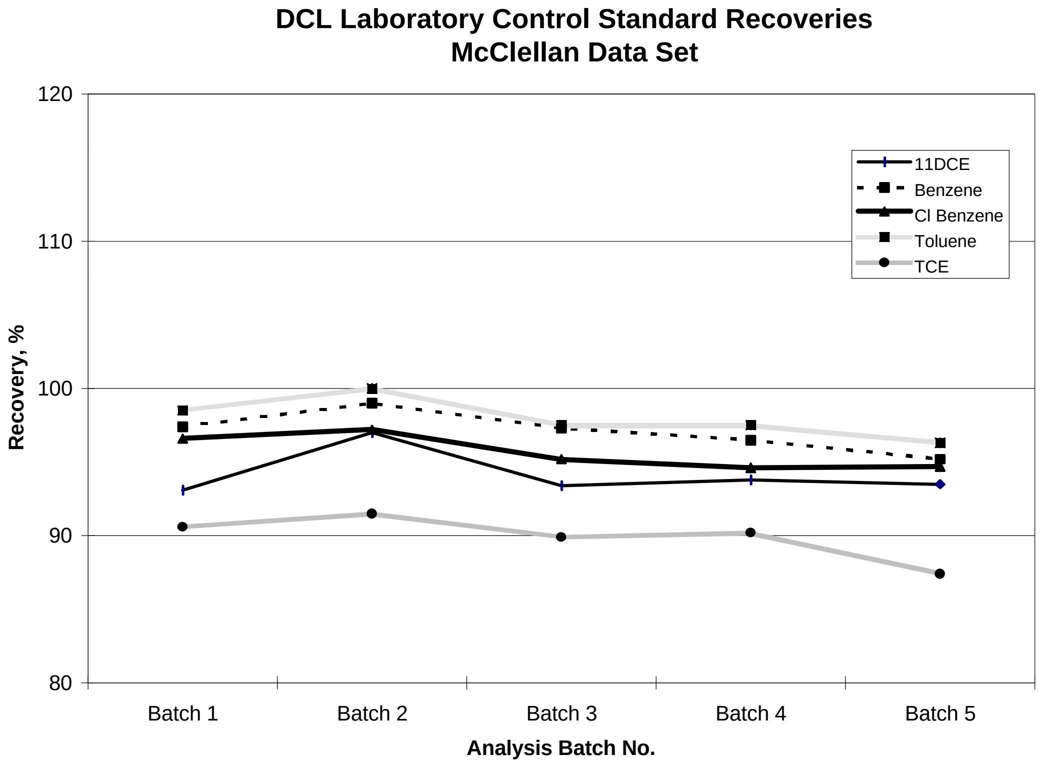

Figure 4-2. Laboratory control standard recovery values for MAFB analyses.

Surrogate Standard

With the following exceptions, surrogate standard recoveries met the criteria established by the laboratory, as noted in Table 4-1. Six samples (SP12, SP16, SP26, SP29, SP33, and SP65) failed surrogate recovery criteria for 1,2-dichloroethane-d_4 and passed recovery criteria for 4-bromofluorobenzene and toluene-d_8. The actions taken are noted in Table 4-3.

Summary of Analytical and QC Deviations

A summary of QC deviations as well as other analytical errors or omissions is given in Table 4-3. The actions taken with regard to the affected data and the reference data set are also tabulated, along with a brief rationale.

Table 4-3. Summary of Reference Laboratory Quality Control and Analytical Deviations

Deviation or QC Criteria Failure	Action
Required dilution not made on two samples (SP20 and SP21). Some compounds were present above instrument linear range.	Data Included: Data values for affected samples fall in the range of the other three replicate samples.
Three field blanks were not sent to DCL from SRS demonstration.	No Action: Five field blanks and 10 method blanks were run, yielding an adequate data set.
Calculation error in original DCL report. Dilution factors applied incorrectly in two samples (SP55 and SP57).	Data Corrected and Included: The correct dilution factors were applied following a teleconference with the DCL analyst.
Sample SP31 failed internal standard recovery limits.	Data Not Included.
The following samples failed one or more surrogate standard recovery limits: SP12, SP16, SP26, SP29, SP33, and SP65.	Data Not Included: SP12; results clearly fall outside of the range of other three replicate samples. Data Included: All others; nearly all target compounds fall within the range of concentration reported for the other three replicate samples.
Hexachlorobutadiene detected as a contaminant in selected blanks and samples.	No Action: This compound was not a target compound for any of the technologies. Its presence as a low-level contaminant does not affect the results of other target compounds.
Chloroethyl vinyl ether was not detected in PE samples known to contain this compound.	No Action: The GC/MS was not calibrated for this compound. None of the technologies included this compound in their target compound lists.
Three sample results (MG20, MG51, and MG59) are from a second withdrawal from the original zero-headspace sample vial.	Data Included: The original volume withdrawn from the vial was 0.05 mL, resulting in an insignificant headspace volume and no expected impact on the composition of the second sample.

Other Data Quality Indicators

The demonstration design incorporated nine PE mixtures of various target compounds at each site that were prepared in the field and submitted in quadruplicate to each technology as well as to the laboratory. Laboratory accuracy and precision checks on these samples were assessed. Precision on replicate analysis of groundwater samples was also evaluated. The results of these assessments are summarized in the following sections.

PE Sample Precision

The relative standard deviation from quadruplicate laboratory analyses of each PE mixture prepared in the field was computed for each target compound in the mixture. As noted in Chapter 3, care was taken to ensure the preparation and distribution of homogeneous samples from each PE mixture. The RSD values represent an overall estimate of precision that takes into account field handling, shipping, storage, and analysis of samples.

The precision data are shown in Figures 4-3 and 4-4 for SRS and Figures 4-5 and 4-6 for MAFB. (See Tables 3-5 and 3-6 for the composition and concentration level of each PE mixture.) The compiled RSDs for all PE sample results had a median value of 7% and a 95th percentile value of 25%. In selected instances, precision in excess of Method 8260A specifications (≤30% RSD) is observed for tetrachloroethene, trichloroethene, *cis*-1,3-dichloropropene, 1,2,3-trichloropropane, and 1,1,2,2-tetrachloroethane. Precision well in excess of method specifications is observed for 1,2-dibromo-3-chloropropane, *trans*-1,3-dichloropropene, and 1,1-dichloropropene. The implications of these results with respect to evaluation of the technology performance are discussed, when applicable, in Chapters 5 or 7.

PE Sample Accuracy

An error propagation analysis was carried out to estimate the degree of uncertainty in the stated "true" concentration level of the PE samples prepared in the field. The sources of uncertainty and their magnitude encountered during PE sample preparation are listed in Table 4-4. These errors are combined using the methodology described by Bevington (1969) to arrive at a combined uncertainty in the PE sample value of ±5%. Thus, for a 100-µg/L PE mix, the true value is known with 99% certainty to be within the range of 95 to 105 µg/L.

Table 4-4. Sources of Uncertainty in PE Sample Preparation

Type of Uncertainty	Magnitude	Source of Estimate
Weight of component in PE mix ampule.	0.5 mg in 1200 mg	Gravimetric balance uncertainty included in PE mix certification documents
Volume of methanol solvent used to dilute neat compounds.	0.2 mL in 600 mL	Published tolerances for volumetric flasks (Fisher Catalog)
Volume of PE solution (from ampule) used in final PE solution.	±5% of microsyringe volume; e.g., 25 µL for a 500-µL syringe	Published tolerances in certificates shipped with microsyringes
Volume of water diluent in final PE solution.	5 ml in 10 L	Published tolerances for volumetric flasks (Fisher Catalog)

The laboratory results for PE samples are compared with the "true" value of the mixture to provide an additional measure of laboratory performance. A mean recovery[3] was computed for each PE compound in each of the four sample splits analyzed from each mixture. The SRS recovery values are shown in Figures 4-7 and 4-8, and MAFB recoveries are shown in Figures 4-9 and 4-10. Acceptable mean percent recovery values, specified in Method 8260A, fall within the range of 70 to 130% with exceptions for a few compounds that pose analytical difficulties. With the following exceptions, all PE compounds at all concentration ranges met the Method 8260A recovery criteria. The exceptions are 1,2,3-trichloropropane, 1,1-dichloropropene, 1,2-dibromo-3-chloropropane,

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[3] Recovery is the ratio of the mean concentration level from analysis of the four sample splits to the reference or "true" concentration levels of the target compounds in each PE mix.

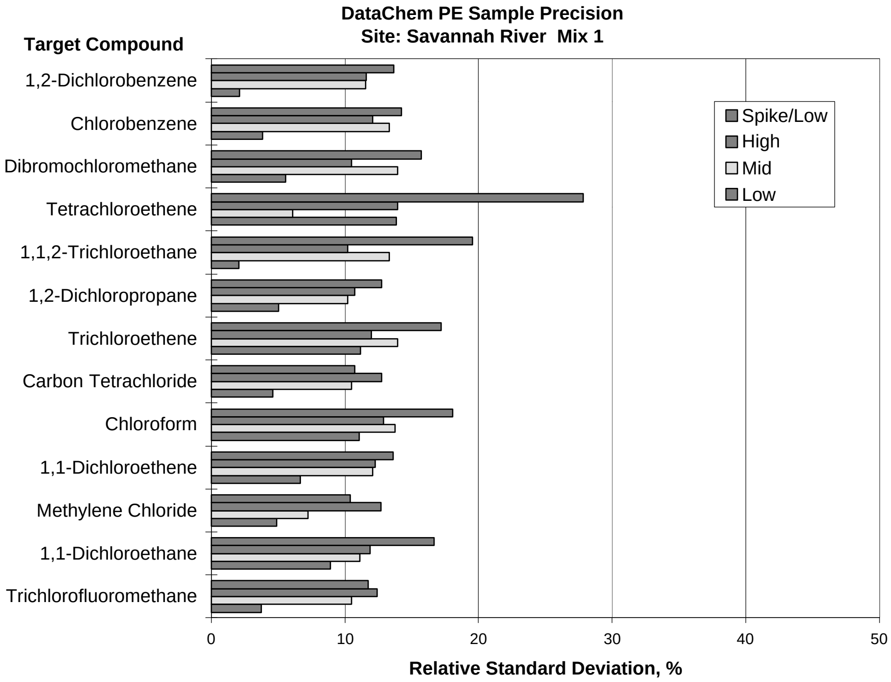

Figure 4-3. Laboratory precision on SRS PE samples containing mix 1. Trichloroethene was spiked into the spike/low samples.

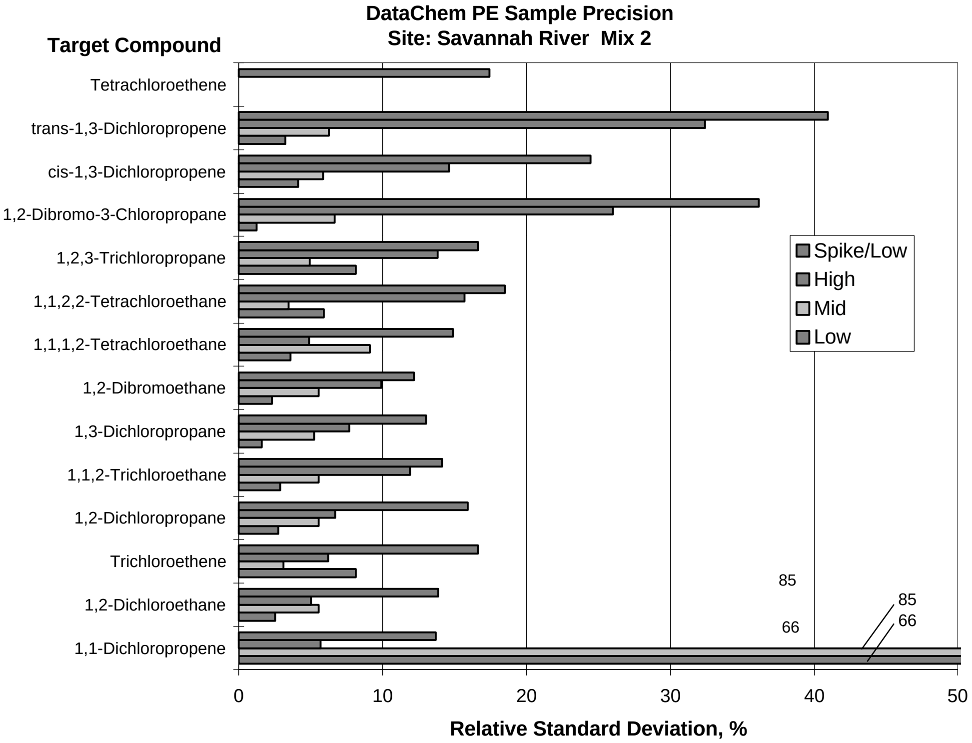

Figure 4-4. Laboratory precision on SRS PE samples containing mix 2. Tetrachloroethene was spiked into the mix 2 samples. Trichloroethene and tetrachloroethene were spiked into the spike/low samples.

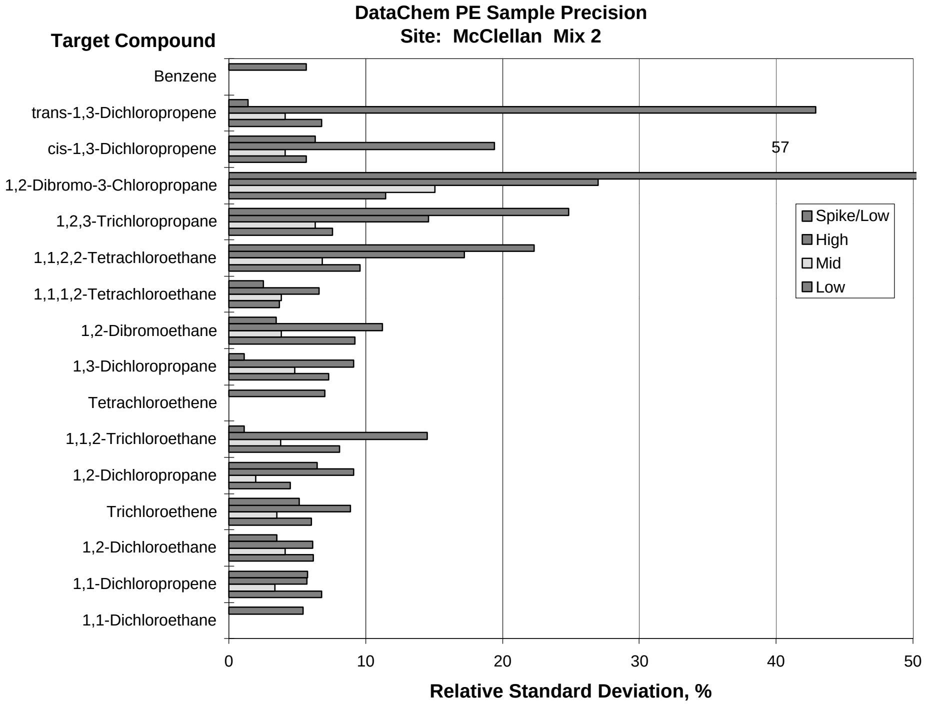

Figure 4-5. Laboratory precision on MAFB PE samples containing mix 2. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

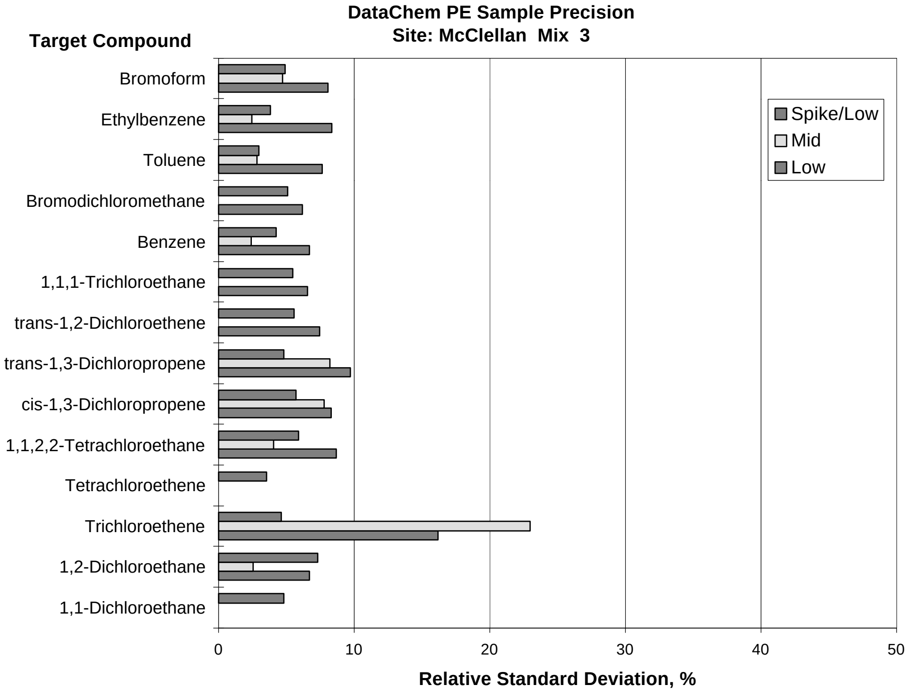

Figure 4-6. Laboratory precision on MAFB PE samples containing mix 3. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

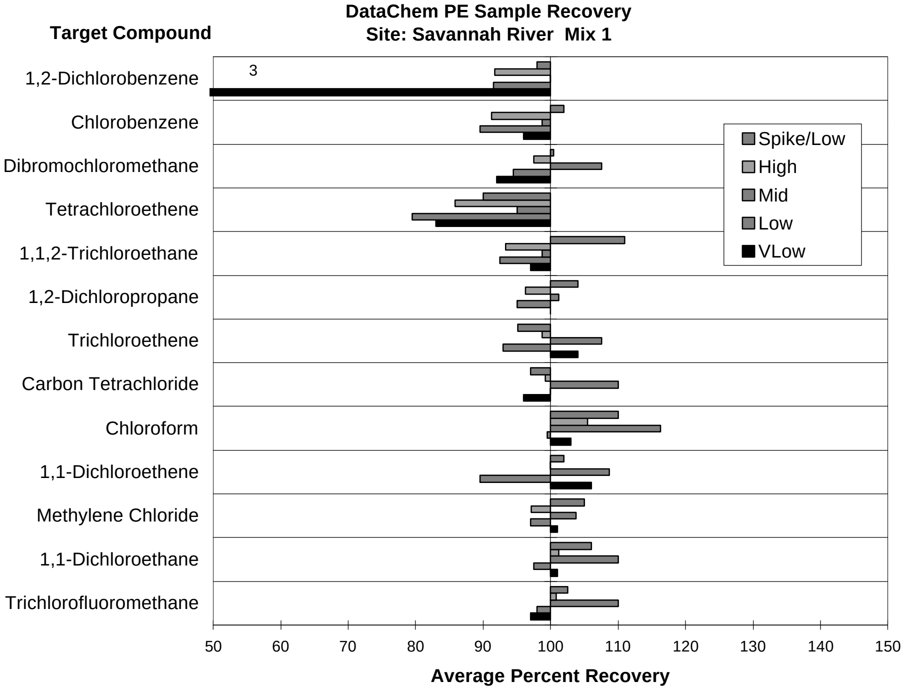

Figure 4-7. Laboratory mean recoveries for SRS PE samples containing mix 1. Trichloroethane was spiked into the spike/low samples.

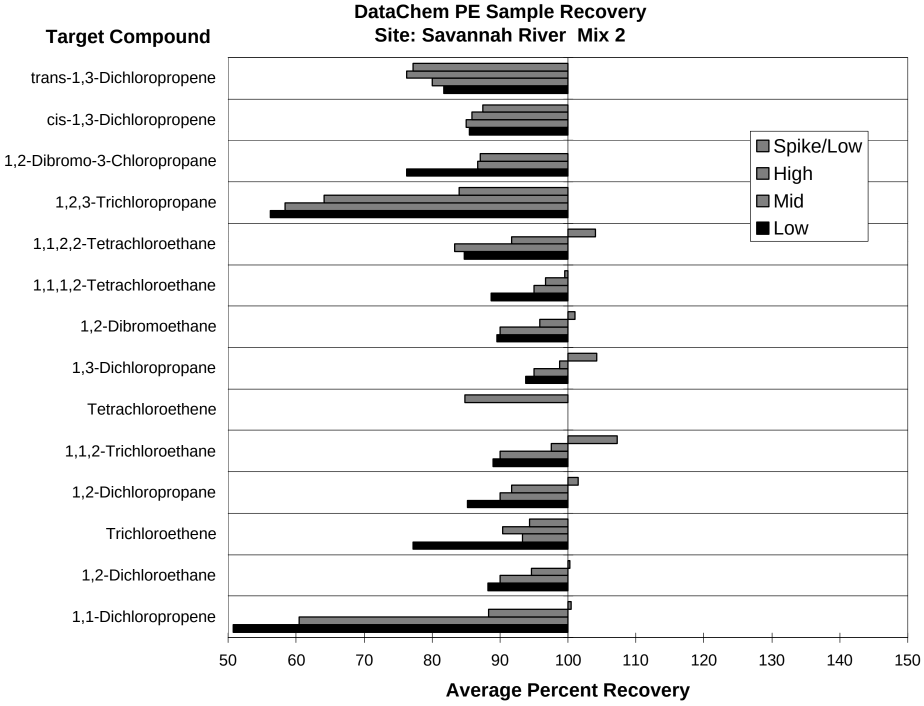

Figure 4-8. Laboratory mean recoveries for SRS PE samples containing mix 2. Trichloroethane and tetrachloroethene were spiked into the spike/low samples.

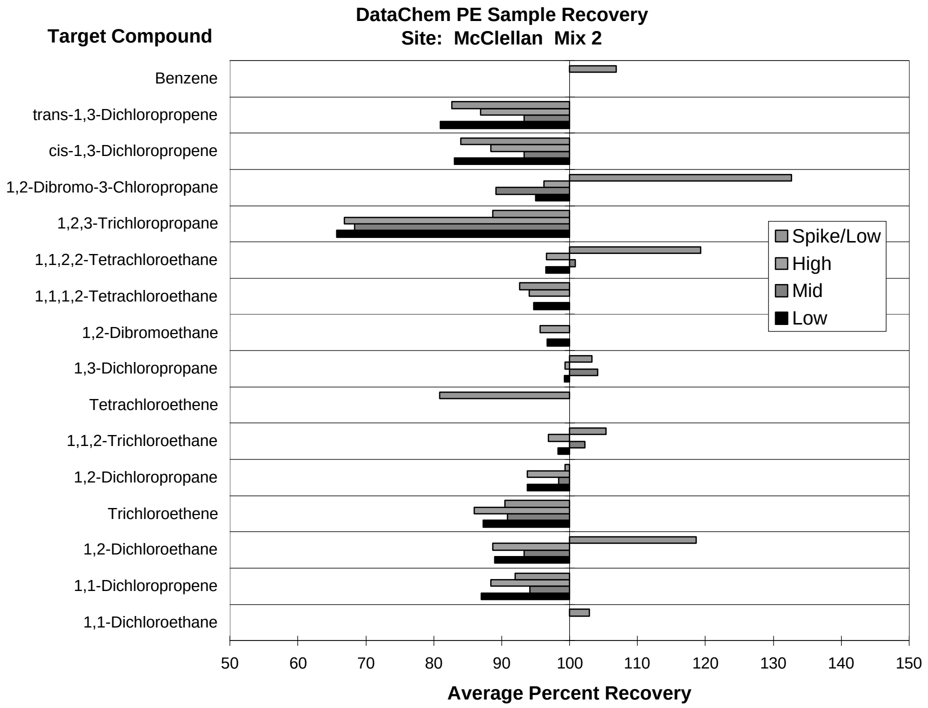

Figure 4-9. Laboratory mean recoveries for MAFB PE samples containing mix 2. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

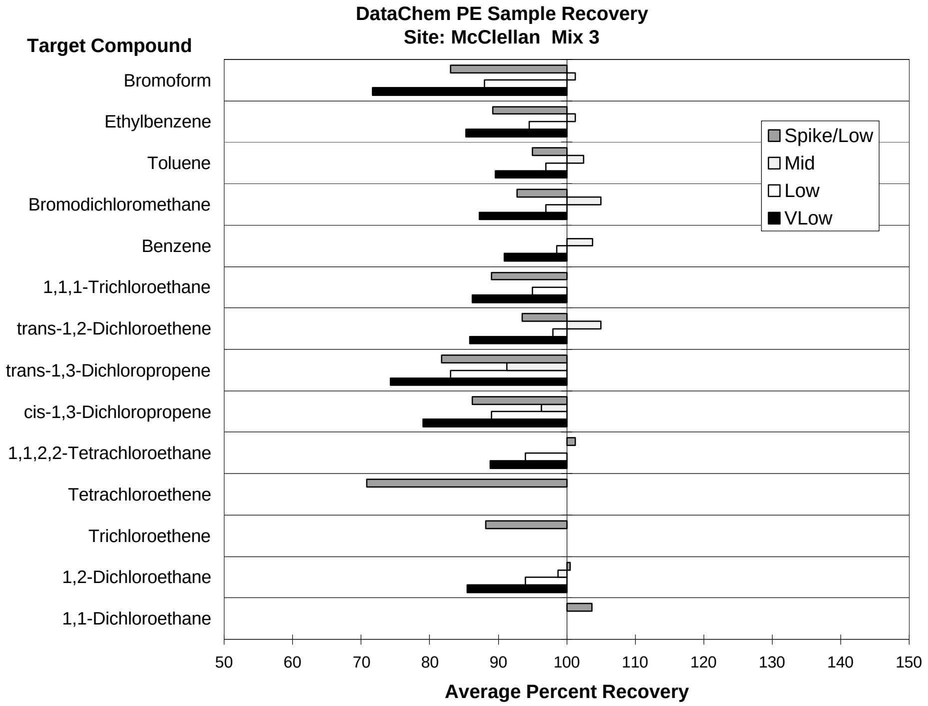

Figure 4-10. Laboratory mean recoveries for MAFB PE samples containing mix 3. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

and 1,2-dichlorobenzene at selected concentration levels. The implications of these exceptions for the technology evaluation are further discussed, if applicable, in Chapter 5. The compiled absolute percent differences (APDs)[4] for all PE sample results had a median value of 7% and a 95th percentile value of 25%.

Groundwater Sample Precision

Relative standard deviations are given in Table 4-5 for compound concentrations in excess of 1 μg/L in groundwater samples from the SRS demonstration. Trichloroethene and tetrachloroethene were the only contaminants detected in SRS groundwater samples. A similar compilation of RSD values from the MAFB groundwater samples is included in Table 4-6. These values are based on analytical results from either three or four replicate samples. With three exceptions, all tabulated values are less than 20%.

Table 4-5. Summary of SRS Groundwater Analysis Precision

Sample Description	Relative Standard Deviation (%)	
	TCE	PCE
Very low 1	10.6	14.3
Very low 2	34.4	12.4
Low 1	5.4	5.7
Low 2	7.1	8.7
Mid 1	9.4	11.6
Mid 2	7.3	4.2
High 1	0.8	1.8
High 2	11.8	7.9
Very high 1	8.4	5.7
Very high 2	6.2	6.3

Table 4-6. Summary of MAFB Groundwater Analysis Precision

Sample Description	Relative Standard Deviation (%)								
	11DCE	TCE	CLFRM	CCL4	PCE	11DCA	c12DCE	t12DCE	BNZN
Very low 1	9.1	5.0							
Very low 2	2.6	<0.1	1.3	4.2	5.7				
Low 1	6.8	3.7	2.0	1.9		<0.1			
Low 2	11.5	5.2		4.0	22.3	4.1	3.8		
Mid 1	12.0	10.5			13.9	9.4	12.6		
Mid 2		3.6	4.9				3.8		
High 1		2.4	20.9				4.1		
High 2		5.3	5.3				5.1	3.8	
Very high 1	2.5	5.4	5.2				6.5		4.9
Very high 2		8.0	6.4	4.9			10.1		

Notes: 11DCE = 1,1-dichloroethene; TCE = trichloroethene; CLFRM = chloroform; CCL4 = carbon tetrachloride; PCE = tetrachloroethene; 11DCA = 1,1-dichloroethane; c12DCE = *cis*-1,2-dichloroethene; t12DCE = *trans*-1,2-dichloroethene; BNZN = benzene.
Blank cells indicate that the compound was not present.

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[4] The absolute percent difference is the absolute value of the percent difference between a measured value and a true value.

Summary of Reference Laboratory Data Quality

With the exceptions noted below, a review of DCL analytical data showed that all Method 8260A QC criteria were met. Internal standard recovery limits were not met for one sample. The results for this sample were markedly different from the other three samples in the replicate set and the sample was omitted from the data set. Six samples failed one or more surrogate standard recovery criteria. These sample results were compared with replicate sample results. Five of the six samples were comparable and were included in the reference data set.

The data for the remaining sample were not comparable and were omitted from the reference data set. Other quality control deviations, which are summarized in Table 4-3, did not significantly affect the quality of the laboratory data.

A review of DCL precision and accuracy on field-prepared PE mixtures corroborates laboratory internal QC results. A similar precision evaluation on groundwater samples from both sites further supports these observations. Overall, the internal and external QC data reveal appropriate application and use of Method 8260A by DataChem Laboratories. The laboratory results for groundwater samples from both sites are considered suitable for use as a reference data set.

Chapter 5
Demonstration Results

HAPSITE Calibrated and Reported Compounds

Prior to the field demonstration, the participants were given a list of all compounds that were to be used in the PE mixtures to facilitate preparations for predemonstration instrument calibration. The HAPSITE GC/MS was calibrated for 32 compounds at SRS, and 6 more were added prior to the MAFB demonstration (Table 5-1). Note that some calibrated compounds were not demonstration PE mixture compounds. A total of 32 chlorinated and nonchlorinated hydrocarbon compounds were included in the PE mixtures noted in Table 3-4. The HAPSITE reported results for 27 of these compounds. It did not report results for 5 PE compounds since it was not calibrated for them. These were dibromochloromethane, 1,2-dichlorobenzene, 2-chloroethyl vinyl ether, 1,2-dibromo-3-chloropropane, and hexachlorobutadiene. Trichlorofluoromethane, another PE compound, was not reported at the SRS but was reported at MAFB.

Preanalysis Sample Information

Groundwater (GW) and PE samples were provided to the HAPSITE team without additional information on the number of compounds in the sample or compound concentration levels.

Table 5-1. HAPSITE Calibrated and Reported Compounds

Calibrated Compounds at Both Demonstrations	
1,1-Dichloroethene	*trans*-1,3-Dichloropropene
Methylene chloride	1,1,2-Trichloroethane
trans-1,2-Dichloroethene	1,3-Dichloropropane
1,1-Dichloroethane	Toluene
Bromochloromethane	Dibromochloromethane
Chloroform	1,2-Dibromoethane
1,2-Dichloroethane	Tetrachloroethene
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane
1,1-Dichloropropene	Chlorobenzene
Benzene	Ethyl benzene
Carbon tetrachloride	*meta*- and *para*-Xylene
Dibromomethane	Bromoform
1,2-Dichloropropane	Styrene
Bromodichloromethane	*ortho*-Xylene
Trichloroethene	1,1,2,2-Tetrachloroethane
cis-1,3-Dichloropropene	1,2,3-Trichloropropane
Additional Calibrated Compounds at MAFB	
Chloromethane	Chloroethane
Vinyl chloride	Trichlorofluoromethane
Bromoethane	*cis*-1,2-Dichloroethene

Sample Completion

All but one of the 166 PE and groundwater samples submitted for analysis to the HAPSITE team were completed at both demonstration sites. The HAPSITE team at the SRS lost a PE sample from the spike/low category of PE mix 1 during sample preparation.

Blank Sample Results

Eight blank samples were provided for analysis at each demonstration site. False positive detects were counted only for compounds reported at concentration levels greater than the HAPSITE MDL for most target compounds (typically 3 µg/L). A listing of false positive detects is given for both sites in Table 5-2.

Table 5-2. False Positive Rates from Blank Sample Analysis

SRS Blank Samples		MAFB Blank Samples	
Compound	**False Positive**	**Compound**	**False Positive**
Dichloromethane	1 of 8 (13%)	1,2-Dichloroethane	1 of 8 (13%)
Trichloroethene	3 of 8 (38%)	Trichloroethene	1 of 8 (13%)
Chlorobenzene	1 of 8 (13%)	*cis*-1,3-Dichloropropene	1 of 8 (13%)
		trans-1,3-Dichloropropene	1 of 8 (13%)
		1,2-Dibromoethane	1 of 8 (13%)

Performance at Instrument Detection Limit

Ten replicate samples of a PE mixture at a concentration level of 10 µg/L (the "very low" concentration level) were provided for analysis at each site. Reported nondetects were compiled and are given as percent false negatives in Table 5-3. Vendor-provided compound detection limits are also shown in the table for comparison.

Table 5-3. False Negative Rates from Very Low-Level PE Sample Analysis

SRS PE Mix 1 (10 µg/L)		MAFB PE Mix 3 (10 µg/L)	
Compound	**False Negative**	**Compound**	**False Negative**
1,1-Dichloroethene (3)	0 of 10	*trans*-1,2-Dichloroethene (3)	0 of 10
Dichloromethane (3)	0 of 10	1,2-Dichloroethane (3)	0 of 10
1,1-Dichloroethane (3)	0 of 10	1,1,1-Trichloroethane (3)	0 of 10
Chloroform (3)	0 of 10	Benzene (3)	0 of 10
Carbon tetrachloride (3)	1 of 10 (10%)	Bromodichloromethane (5)	0 of 10
1,2-Dichloropropane (3)	0 of 10	*cis*-1,3-Dichloropropene (5)	0 of 10
Trichloroethene (3)	0 of 10	*trans*-1,3-Dichloropropene (5)	0 of 10
1,1,2-Trichloroethane (3)	4 of 10 (40%)	Toluene (3)	0 of 10
Dibromochloromethane (3)	2 of 10 (20%)	Ethyl benzene (3)	0 of 10
Tetrachloroethene (3)	4 of 10 (40%)	Bromoform (8)	9 of 10 (90%)
Chlorobenzene (3)	1 of 10 (10%)	1,1,2,2-Tetrachloroethane (10)	3 of 10 (30%)
2-Chloroethyl vinyl ether	No calibration		
Trichlorofluoromethane	No calibration		
1,2-Dichlorobenzene	No calibration		

Notes: Vendor-provided detection limits (in µg/L) are shown in parentheses after each compound.

PE Sample Precision

Precision results from each of the four replicate sample sets provided from eight PE mixtures at SRS and the seven mixtures at MAFB are shown in Figures 5-1 and 5-2 for SRS and Figures 5-3 and 5-4 for MAFB. The figures show the relative standard deviation for each compound in the PE mixtures at the four concentration levels used in the study.[1] (The composition and concentrations of each of these mixtures were given in Table 3-5 for SRS and Table 3-6 for MAFB.) Note that precision and accuracy were not determined for the very low concentration level. Instrument precision data for six target compounds which are all regulated under the Safe Drinking Water Act are shown in Table 5-4. The relative standard deviations are given for each target compound at each of the four concentration levels used in the study. The RSD range for each target compound is given in the last column of the table.

Overall instrument precision is summarized in Table 5-5 for PE mixtures used at each site. For this summary, RSD values from all PE sample analyses for all compounds at each site were pooled and the median and 95th percentile values of the distribution were computed.

Table 5-4. Target Compound Precision for PE Samples at Both Sites

Target Compound	Site	Relative Standard Deviation (%)				
		Low	Mid	High	Spike/Low	Range
Trichloroethene	SRS	7	18	15	16	7 – 18
	MAFB	13	7	13	15	
1,2-Dichloroethane	SRS	6	8	10	2	2 – 12
	MAFB	12	9	8	5	
1,1,2-Trichloroethane	SRS	10	8	9	28	8 – 28
	MAFB	19	15	21	28	
1,2-Dichloropropane	SRS	21	17	8	18	7 – 21
	MAFB	11	7	12	17	
Tetrachloroethene	SRS	22	19	16	14	6 – 22
	MAFB			6	8	
trans-1,3-Dichloropropene	SRS	11	7	18	16	7 – 17
	MAFB	16	17	17	10	

Note: Blank cells indicate that no data were reported.

Table 5-5. Summary of PE Sample Precision and Percent Difference Statistics for SRS and MAFB

Parameter	Percentile	SRS		MAFB		Combined Sites
		PE Mix 1	PE Mix 2	PE Mix 2	PE Mix 3	Combined Mixes
RSD, %	50th	12	12	14	8	12
	95th	24	31	30	19	29
	Number in pool	45	49	52	37	183
Absolute percent difference	50th	11	13	5	4	8
	95th	33	36	26	17	27
	Number in pool	44	49	51	36	180

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[1] Precision data for the PE mix 1 sample set at MAFB are not shown in a figure. Precision results from this mixture were comparable to those obtained from the same mixture at the SRS.

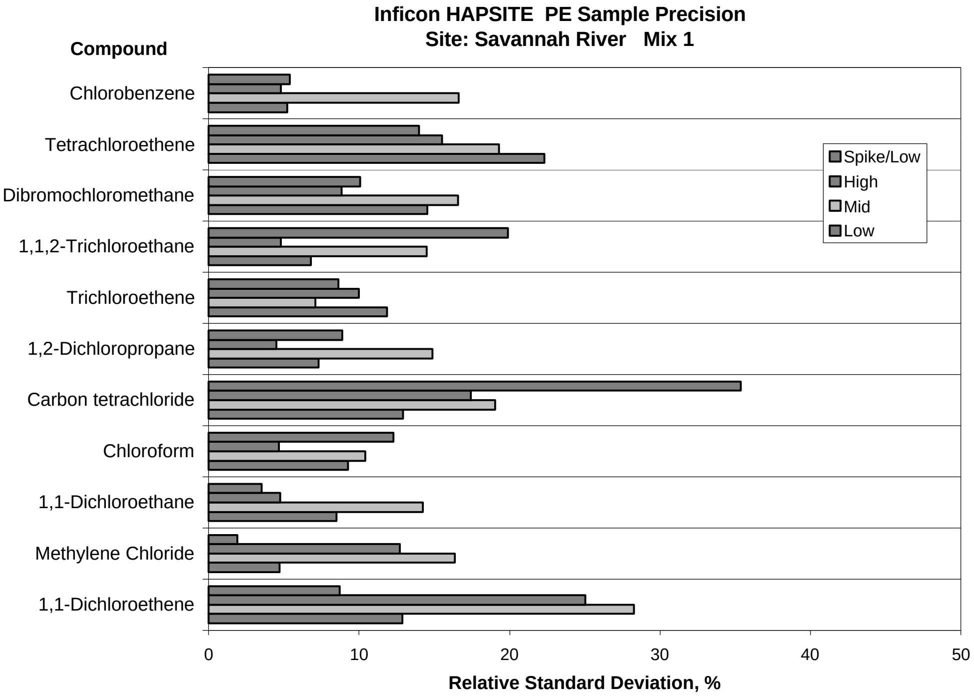

Figure 5-1. HAPSITE precision on PE mix 1 at the SRS. Trichloroethene was spiked into the spike/low samples.

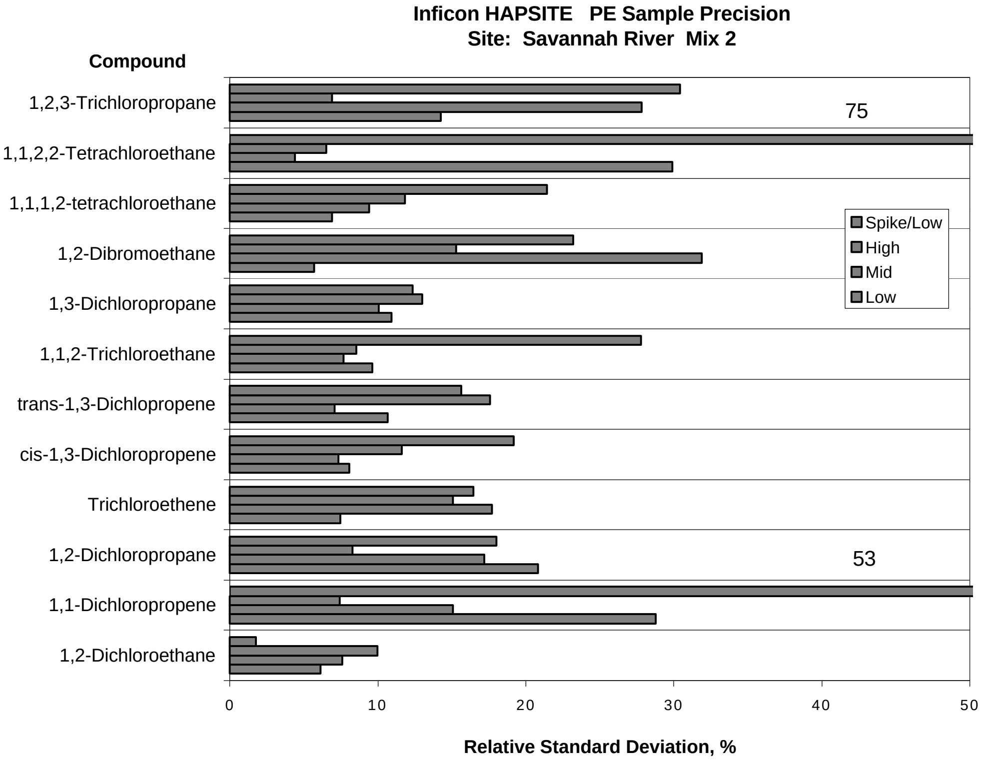

Figure 5-2. HAPSITE precision on PE mix 2 at the SRS. Trichloroethene and tetrachloroethene were spiked into the spike/low samples.

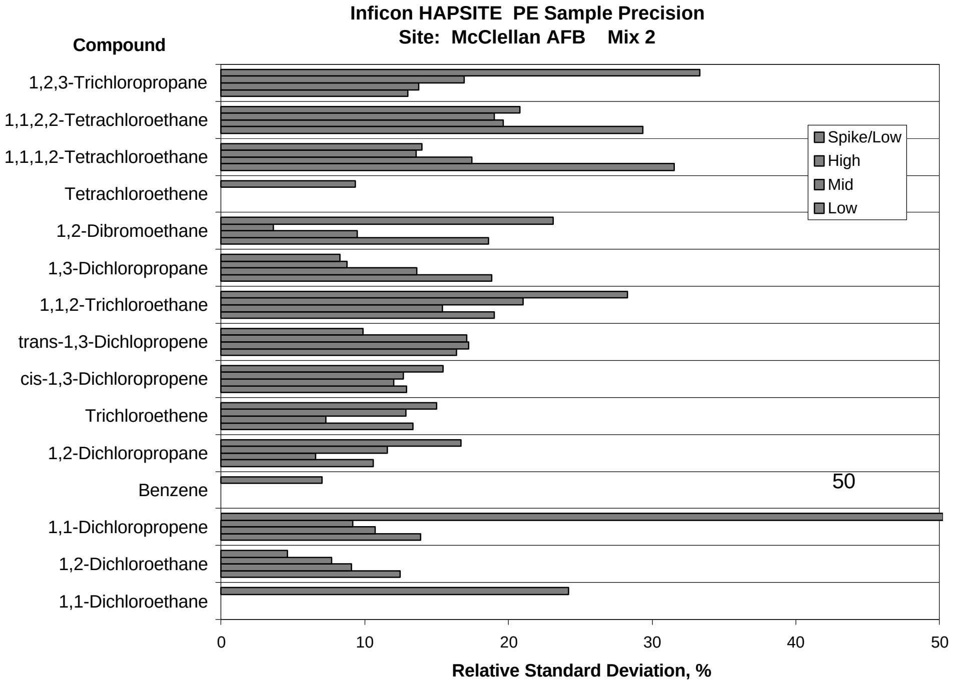

Figure 5-3. HAPSITE precision on PE mix 2 at MAFB. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

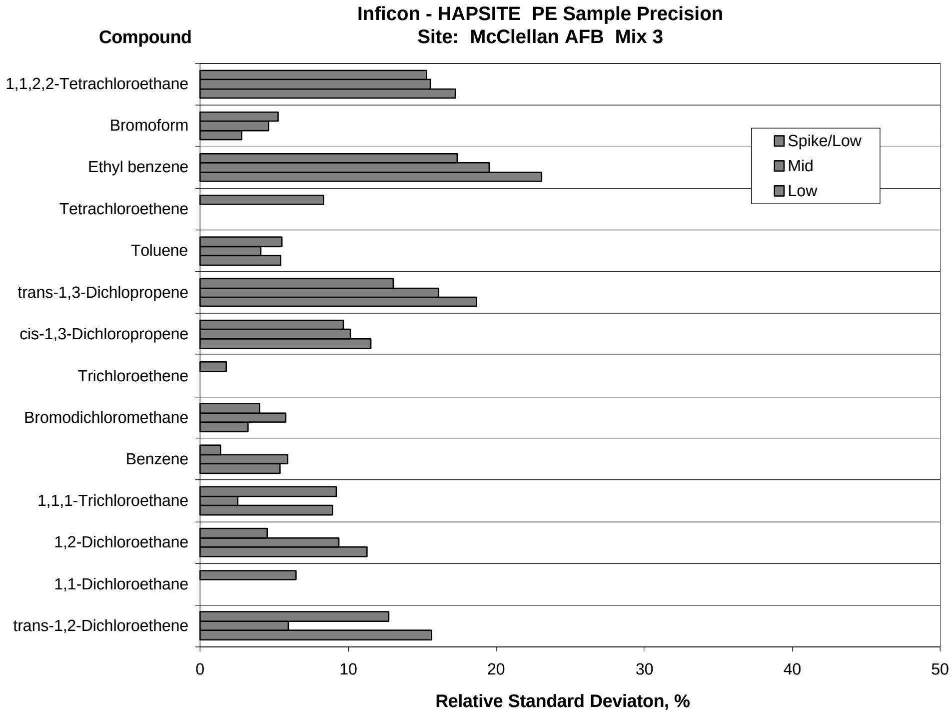

Figure 5-4. HAPSITE precision on PE mix 3 at MAFB. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

PE Sample Accuracy

The HAPSITE accuracy on PE samples was determined by comparing the average value from each of the four-sample replicate sets with the known concentration of the PE mixture (Tables 3-5 and 3-6 for SRS and MAFB, respectively). These comparisons are shown as percent recoveries[2] in Figures 5-5 and 5-6 for SRS and Figures 5-7 and 5-8 for MAFB.[3] To assist in assessment of the sign of the difference, the percent recovery data are plotted as either a positive or negative deviation from the 100% recovery line.

Instrument recovery performance for the six target compounds is shown in Table 5-6, which contains the average percent recoveries and associated ranges for each compound.

Table 5-5 contains a summary of overall HAPSITE differences relative to PE mixture true values for both sites, along with the precision summary. For this summary, percent recoveries were expressed as percent difference (for example, a 90% recovery is equivalent to a –10% difference; 120% recovery is equivalent to a +20% difference) and all data from PE mixtures were pooled. The median and 95th percentiles of the absolute values of these pooled values were computed and are reported under the absolute percent difference (APD) category in Table 5-5.

Table 5-6. Target PE Compound Recovery at Both Sites

Target Compound	Site	Average Recovery (%)				
		Low	Mid	High	Spike/Low	Range
Trichloroethene	SRS	83	103	112	80	80 – 114
	MAFB	113	108	114	101	
1,2-Dichloroethane	SRS	96	93	91	92	91 – 103
	MAFB	102	98	103	80	
1,1,2-Trichloroethane	SRS	120	99	108	84	79 – 120
	MAFB	107	103	101	79	
1,2-Dichloropropane	SRS	79	97	103	113	79 – 113
	MAFB	95	95	113	91	
Tetrachloroethene	SRS	86	94	89	67	67 – 93
	MAFB			93	86	
trans-1,3-Dichloropropene	SRS	90	85	85	85	85 – 101
	MAFB	101	95	96	88	

Note: Blank cells indicate that no data were reported.

Comparison with Laboratory Results

For each demonstration site, a total of 33 samples collected from 10 wells were provided to the participants and to the reference laboratory. Replicate sample sets were composed of either 3 or 4 samples from each well. Average laboratory results from each replicate set were used as the reference values for comparison with technology results. A side-by-side comparison of laboratory and HAPSITE results for all groundwater samples

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[2] Percent recovery is the HAPSITE value divided by the true value, multiplied by 100.

[3] Percent recovery data for the single PE mix 1 sample set at MAFB are not shown in a figure. Recovery results from this mixture were comparable to those obtained from the same mixture at the SRS.

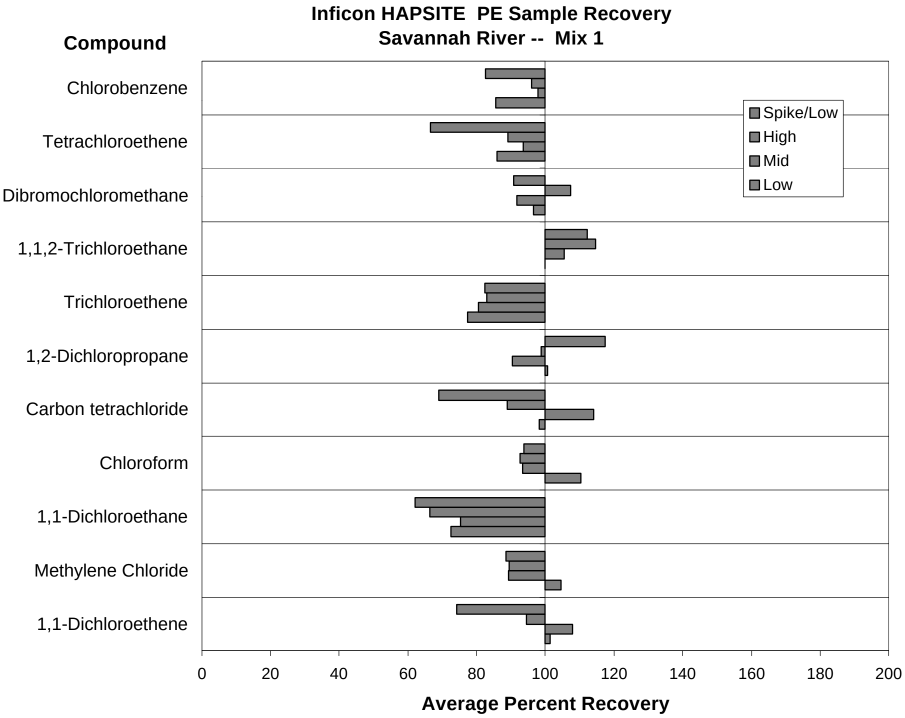

Figure 5-5. HAPSITE recovery on PE mix 1 at the SRS. Trichloroethene was spiked into the spike/low samples.

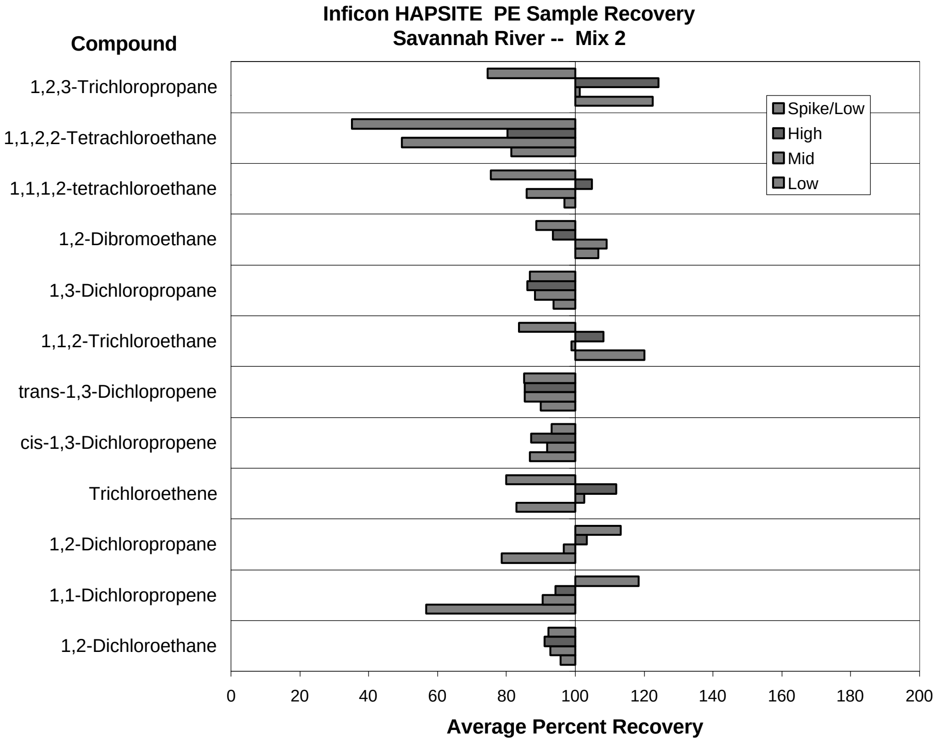

Figure 5-6. HAPSITE recovery on PE mix 2 at the SRS. Trichloroethene and tetrachloroethene were spiked into the spike/low samples.

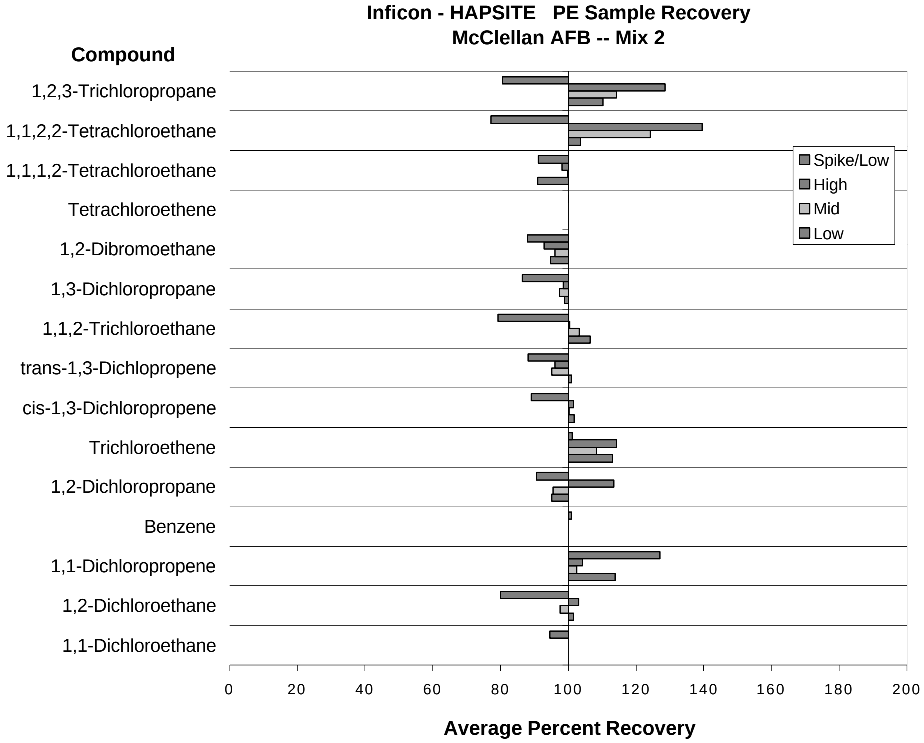

Figure 5-7. HAPSITE recovery on PE mix 2 at MAFB. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

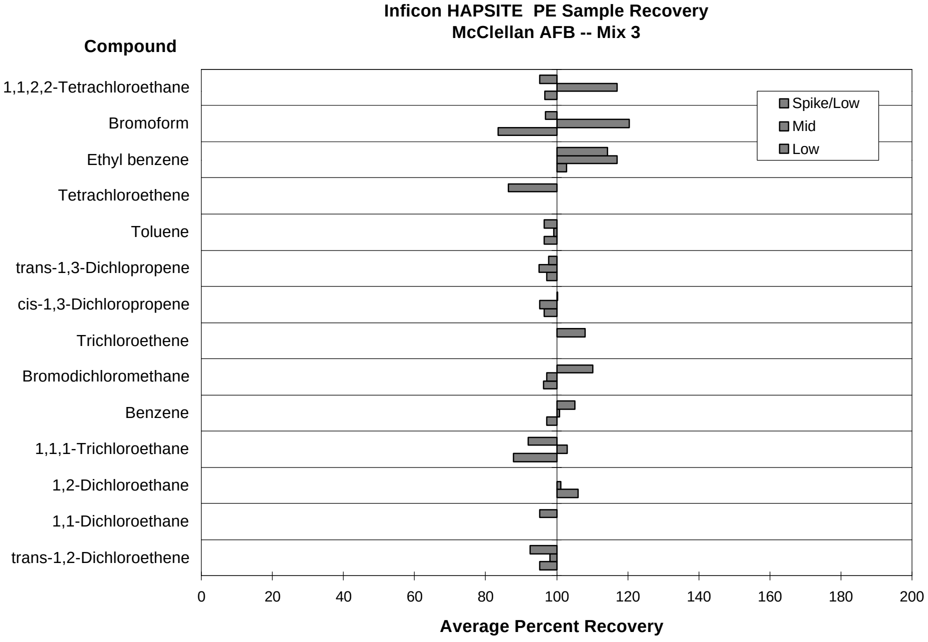

Figure 5-8. HAPSITE recovery on PE mix 3 at MAFB. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

is given in Table 5-7 for the SRS and Table 5-8 for MAFB. The RSD values and their statistical summaries are included in the table. Well designation (very low, low, mid, high, and very high) is based on TCE concentration levels; however, other compounds were present in the groundwater samples at concentration levels noted in the tables. The precision of the HAPSITE on replicate groundwater sample sets is shown as RSDs in the last column of the table.

Table 5-7. HAPSITE and Reference Laboratory Results for SRS Groundwater Samples

Sample Description	Well Number	Compound	Replicates	Lab. Avg. (µg/L)	Lab. RSD (%)	HAPSITE [a] Avg. (µg/L)	HAPSITE [a] RSD (%)
Very low 1	MSB 33B	Trichloroethene Tetrachloroethene	3	9.0 3.5	11 14	8.8 2.5	43 89
Very low 2	MSB 33C	Trichloroethene	3	2.4	34	1.9	32
Low 1	MSB 18B	Trichloroethene Tetrachloroethene	3	11 27	5 6	13 28	11 19
Low 2	MSB 37B	Trichloroethene Tetrachloroethene Chloroform Carbon tetrachloride	4	27 22 1.3 1.0	7 9 0 15	27 19 NR NR	12 17 NR NR
Mid 1	MSB 4D	Trichloroethene Tetrachloroethene	4	150 87	9 12	126 68	7 12
Mid 2	MSB 64C	Trichloroethene Tetrachloroethene 1,1-Dichloroethene	3	35 240 12	7 4 8	29 186 7.3	19 15 19
High 1	MSB 4B	Trichloroethene Tetrachloroethene	3	747 33	1 2	726 37	8 11
High 2	MSB 70C	Trichloroethene Tetrachloroethene 1,1-Dichloroethene	4	1875 520 32	12 8 8	1703 454 26	8 23 42
Very high 1	MSB 14A	Trichloroethene Tetrachloroethene	3	1367 800	8 6	1460 898	6 12
Very high 2	MSB 8C	Trichloroethene Tetrachloroethene	3	4933 3668	6 6	4783 3197	3 12
Range					0 – 34		3 – 89
Median					8		12
95th Percentile					15		43

[a] NR = not reported.

The average percent difference between average HAPSITE and laboratory results for the compounds detected in each set of groundwater samples is shown in Figures 5-9 and 5-10 for SRS and MAFB, respectively. Average laboratory results for groundwater contaminants reported at levels less than 1 µg/L are not included in the comparison. The SRS groundwater comparison in Figure 5-9 includes only TCE and PCE. Two well samples at the SRS were also contaminated with 1,1-dichloroethene and other compounds, as noted in Table 5-7. The groundwater samples at MAFB were more complex, as indicated by the additional compounds shown in Table 5-8 and Figure 5-10. (See the vendor comment in Chapter 7 concerning laboratory results for *cis*-1,2-dichloroethene at MAFB.)

Table 5-8. HAPSITE and Reference Laboratory Results for MAFB Groundwater Samples

Sample Description	Well Number	Replicates	Compound	Lab. Avg. (μg/L)	Lab. RSD (%)	HAPSITE[a] Avg. (μg/L)	HAPSITE[a] RSD (%)
Very low 1	EW-86	3	Trichloroethene	4.6	5	12.8	93
			1,1-Dichloroethene	7.7	9	6.3	7
Very low 2	MW-349	3	Trichloroethene	13	0	15	15
			Tetrachloroethene	2.0	6	NR	NR
			Chloroform	9.0	1	9.0	12
			1,1-Dichloroethene	3.8	3	2.9	20
			Carbon tetrachloride	137	4	140	22
Low 1	MW-331	4	1,1-Dichloroethene	2.5	7	NR	NR
			1,1-Dichloroethane	15	0	13	15
			Carbon tetrachloride	7.5	2	6.0	30
			Chloroform	4.8	2	4.5	4
			Trichloroethene	16	4	17	27
Low 2	MW-351	3	Freon11	20	6	NR	NR
			1,1-Dichloroethene	1.5	12	NR	NR
			1,1-Dichloroethane	5.1	4	4.0	23
			cis-1,2-Dichloroethene	1.5	4	1.9	34
			Carbon tetrachloride	1.4	4	NR	NR
			Trichloroethene	22	5	25.1	11
Mid 1	EW-87	4	1,1-Dichloroethene	180	12	189	23
			1,1-Dichloroethane	3.0	9	NR	NR
			cis-1,2-Dichloroethene	3.3	13	4.0	12
			1,1,1-Trichloroethane	6.8	12	5.3	34
			Trichloroethene	114	11	111	12
			Tetrachloroethene	1.2	14	NR	NR
Mid 2	MW-341	3	*cis*-1,2-Dichloroethene	15	4	24	8
			Chloroform	3.5	5	3.1	16
			Trichloroethene	280	4	264	3
High 1	MW-209	3	*cis*-1,2-Dichloroethene	38	4	56	11
			Chloroform	6.9	21	6.4	5
			Trichloroethene	238	2	240	11
High 2	MW-330	4	*trans*-1,2-Dichloroethene	7.7	4	7.1	10
			cis-1,2-Dichloroethene	66	5	97	12
			Chloroform	42	5	45	5
			1,2-Dibromochloropropane	6.1	6	4.7	19
			Trichloroethene	380	5	398	9
Very high 1	MW-334	3	1,1-Dichloroethene	690	3	1032	33
			cis-1,2-dichloroethene	237	7	426	9
			Chloroform	397	5	418	8
			Benzene	283	5	265	12
			Trichloroethene	10,667	5	11,714	13
			Carbon tetrachloride	350	5	565	44
Very high 2	MW-369	3	*cis*-1,2-Dichloroethene	207	10	299	16
			Chloroform	63	6	67	3
			Carbon tetrachloride	51	5	60	23
			Trichloroethene	6167	8	6821	15
Range					0 – 21		3 – 93
Median					5		13
95th Percentile					14		36

[a] NR = not reported.

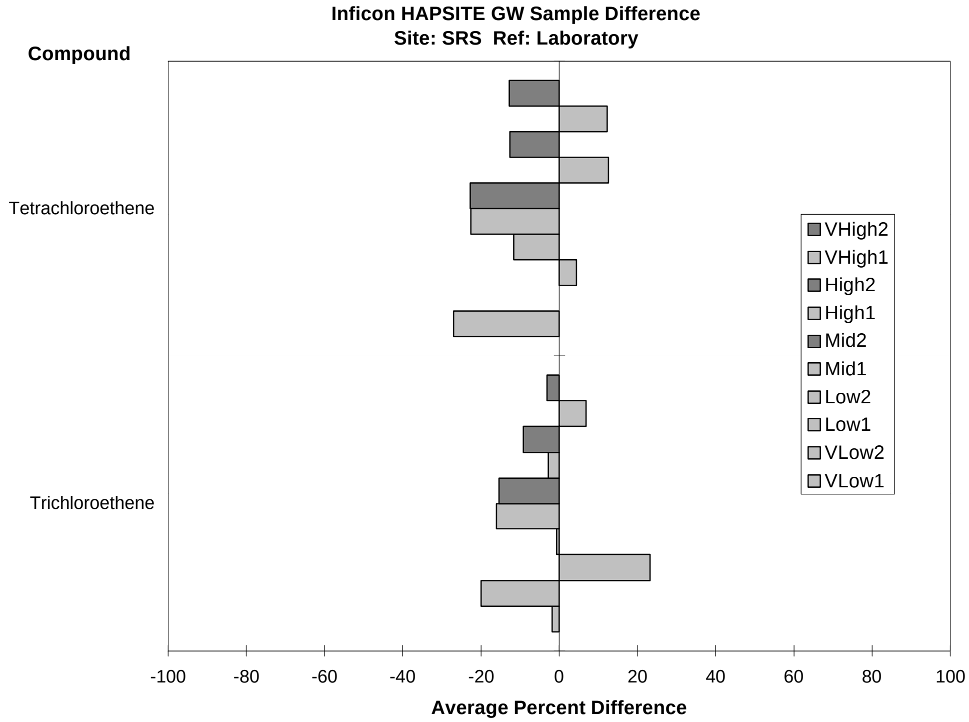

Figure 5-9. HAPSITE groundwater results at the SRS relative to laboratory results.

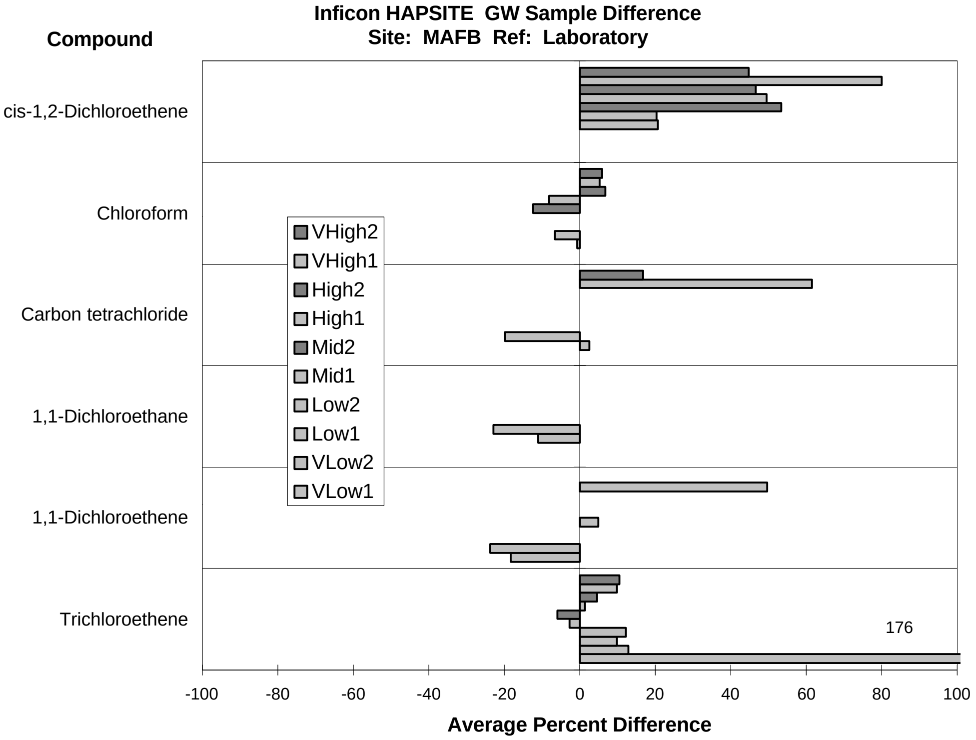

Figure 5-10. HAPSITE groundwater results at MAFB relative to laboratory results.

The median and 95th percentile of the distribution of absolute percent differences between HAPSITE and laboratory results for all groundwater samples are given in Table 5-9.

Table 5-9. HAPSITE Absolute Percent Difference Summary for Pooled Groundwater Results

Percentile	SRS	MAFB	Combined Sites
50th	13	13	12
95th	29	64	43
Number of samples in pool	21	38	59

To assess the degree of linear correlation between the HAPSITE and laboratory groundwater data pairs shown in Tables 5-7 and 5-8, correlation coefficients (*r*) were computed. The data pairs were divided into two subsets for each site to reduce the likelihood of spuriously high *r* values caused by large differences in the data (e.g., concentrations ranging from 1 µg/L to those in excess of 1000 µg/L) (Havlicek and Crain, 1988). One subset contained all data pairs with laboratory results less than or equal to 100 µg/L and the other subset included all data pairs with laboratory values greater than 100 µg/L. The computed correlation coefficients are shown in Table 5-10.

Table 5-10. Correlation Coefficients for Laboratory and HAPSITE Groundwater Analyses

Data Set	Correlation Coefficient	Number of Data Pairs
SRS Laboratory (1 through 100 µg/L)	0.983	13
SRS Laboratory (> 100 µg/L)	0.996	9
MAFB Laboratory (1 through 100 µg/L)	0.978	24
MAFB Laboratory (> 100 µg/L)	1.000	14

Sample Throughput

HAPSITE sample throughput rates ranged from two to three samples per hour. Throughput rates were assessed by using the time lapsed between sample checkout in the morning and delivery of hardcopy results in the afternoon, and the number of samples completed. HAPSITE GC run times were slightly longer at the MAFB demonstration as a result of the additional five compounds in the system calibration file. Sample throughput rates were not significantly influenced by the sample complexity since more complex PE and less complex groundwater samples were run through the same analysis sequence and had the same GC run times.

Performance Summary

Table 5-11 contains a summary of HAPSITE performance characteristics, including important instrument performance parameters and operational features verified in this demonstration. For groundwater samples, the results from the reference laboratory are given alongside HAPSITE performance results to facilitate comparison of the two methodologies.

Table 5-11. Summary of HAPSITE GC/MS Performance

Instrument Feature/Parameter	Performance Summary
Blank sample	False positives detected at low (13 to 38%) rates for 8 compounds
Detection limit sample	False negatives reported at rates between 10 and 90% for 7 of 22 compounds, at concentration levels of 10 µg/L, for which the instrument was calibrated
PE sample precision	Target compounds, RSD range: 2 to 28% All compounds, HAPSITE median RSD: 12%; 95th percentile RSD: 29% All compounds, laboratory median RSD: 7%; 95th percentile RSD: 25% (Target compounds: TCE, 1,2-dichloroethane, 1,1,1-trichloroethane, 1,2-dichloropropane, PCE, and *trans*-1,3-dichloropropene)
PE sample accuracy	Target compounds, absolute percent difference range: 1 to 33% All compounds, HAPSITE median APD: 8%; 95th percentile APD: 27% All compounds, laboratory median APD: 7%; 95th percentile APD: 24% (Target compounds same as those for sample precision)
HAPSITE comparison with laboratory results for groundwater samples	HAPSITE median RSD: 12% Laboratory median RSD: 6% HAPSITE 95th percentile RSD: 43% Laboratory 95th percentile RSD: 14% HAPSITE laboratory median APD: 13%; 95th percentile APD: 60% HAPSITE laboratory correlation: SRS low conc. (≤100 µg/L) $r = 0.983$ SRS high conc. (>100 µg/L) $r = 0.996$ MAFB low conc. (≤100 µg/L) $r = 0.978$ MAFB high conc. (>100 µg/L) $r = 1.000$
Analytical versatility	PE samples: calibrated for 27 of 32 PE compounds (84%) GW samples: HAPSITE reported 59 of 59 compounds detected by the laboratory in all GW samples at or above the 5 µg/L concentration level. A total of 68 compounds at concentration levels ≥ 1 µg/L were detected by the reference laboratory in all groundwater samples.
Sample throughput	2.5 samples per hour
Support requirements	Self-contained carrier gas, batteries, optional printer Internal and surrogate standard solutions and syringes
Operator requirements	Sample processing: technician with 3-day training Data processing and review: B.S. chemist or equivalent
Total system weight	60 pounds
Portability	Transportable—best suited for use in vehicle at the wellhead
Total system cost	$95,000
Shipping requirements	Air freight, hand carry, luggage check Carrier and internal standard gases shipped as hazardous material

Chapter 6
Field Observations and Cost Summary

Introduction

The following subsections summarize the audit findings obtained while observing instrument operation at both field sites. The purpose of the audits was to observe the instrument in operation as well as to verify that the analytical procedures used during the demonstration were consistent with the written procedures submitted to the verification organization prior to the field demonstration. An instrument cost summary and an applications assessment is provided.

Method Summary

The HAPSITE uses a static (equilibrium) headspace method with temperature control. The headspace vapors from a temperature-equilibrated sample are transferred to a gas sampling loop and automatically injected into the GC/MS. Compounds are identified by library spectral matching and quantified by integrating the peak area of a selected quantification ion. Internal and surrogate standards are also incorporated into the method.

Equipment

Without the service module, the HAPSITE GC/MS is 18 inches × 17 inches × 7 inches and weighs 35 pounds. The headspace sampling accessory is 16 inches × 14 inches × 7 inches and weighs 15 pounds; the notebook computer is 8 pounds and the printer weighs 5 pounds. Configured for water sample analysis, the system is transportable and easily fits in the rear luggage storage area of a minivan or station wagon. Equipment weights include batteries and gas cartridges. Nickel-cadmium battery lifetimes are about 2 to 3 hours for the HAPSITE and 4 to 6 hours for the headspace sampling accessory. The system can be connected to a 24-V marine battery for extended remote operation. The system was run with both batteries and ac power during the demonstration. The system is also equipped with a service module with dimensions of 18 inches × 17 inches × 8.5 inches and a weight of 45 pounds. The module is used to pump an initial vacuum on the spectrometer and normally is not taken to the field.

Additional required equipment includes 40-mL screw-cap septa vials (Supelco, graduated with diameter to fit the headspace sampling accessory heater block); 1-mL (with MIN-inert valve) and 2-mL vials for calibration; microliter syringe(s); 50-mL Teflon Luer lock syringes for sample transfer; and commercially available (Supelco or equivalent) internal and surrogate standard mixtures.

The equipment was transported to the SRS by vehicle and to the MAFB site as carry-on luggage. It can also be checked as baggage in its shipping case.

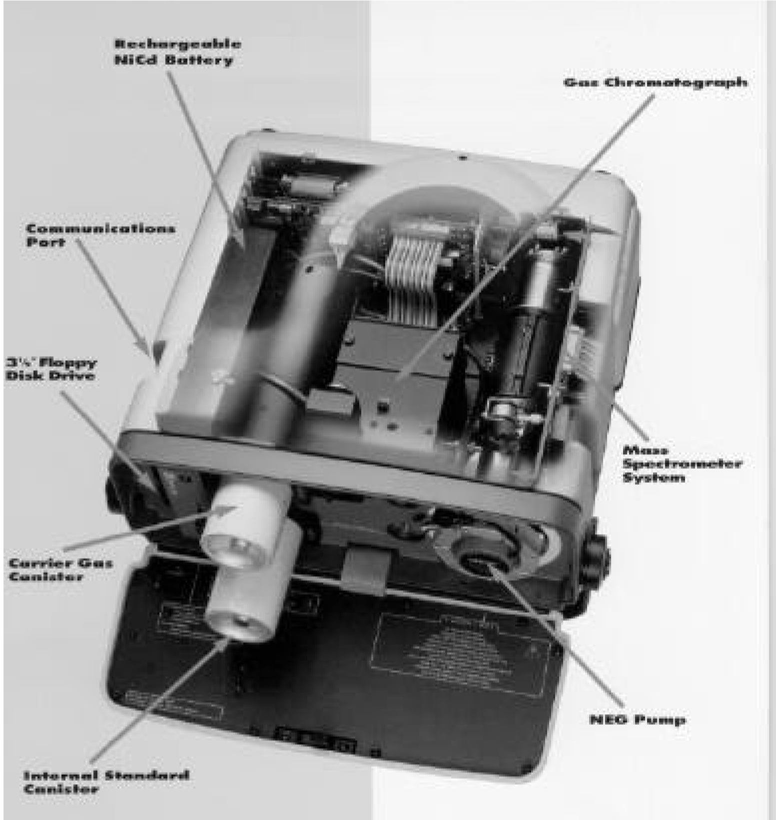

Figure 6-1. The HAPSITE GC/MS.

Sample Preparation and Handling

Sample preparation begins by pouring a cold, zero-headspace, 40-mL volatile organics analysis vial sample into an open 50-mL gas-tight syringe. The syringe plunger is inserted, the syringe inverted, and excess air expelled. Twenty milliliters are ejected as waste. The remaining 20-mL sample is transferred to a headspace vial and capped with a Teflon-lined silicone septum and screw cap. A 5-µL volume of internal or surrogate standard mixture is injected through the septum with a microliter syringe. All samples were prepared in this manner immediately after a sample batch was received. Following preparation, all vials were then stored on ice. Calibration standards were prepared directly in headspace vials. Since the standards were not carried through the sample transfer process, there was little risk for the loss of compounds that may occur during routine sample handling and transfer.

Consumables

Disposable gas bottles are used for the nitrogen carrier gas, the internal standard mixture, and the headspace sampling accessory purge-and-sweep gas. These bottles can be changed out quickly in the field. Standard gas cylinders can also be used for nitrogen carrier and headspace sampling accessory purge and sweep. The system has a chemical getter pump that maintains the system vacuum during operation. The getter pump must be replaced after 240 hours of use.

Historical Use

This is the first demonstration of the HAPSITE GC/MS and the headspace sampling accessory for the analysis of volatile organics in water. The HAPSITE unit alone has been used extensively for vapor analysis applications (exhaust, stack, and soil gas analysis).

Equipment Cost

The HAPSITE and headspace sampling accessory have a combined cost that ranges from $75,000 to $95,000, depending upon options selected. The instrument can be purchased without a service module for $75,000. With this configuration, the instrument must be sent back to the maintenance facility in Syracuse, New York, for periodic replacement of the getter pump. Purchase of the service module allows the user to replace the getter pump in the field. With either option, a notebook computer is included in the package for data processing and instrument control. Daily operating costs are about $150 and include gases, battery packs, and replacement getter pumps (all via maintenance agreement). Instrument costs are summarized in Table 6-1. Laboratory costs were $95 per sample plus overnight Express Mail costs, which were about $30 per batch of 12 samples. HAPSITE sample throughput is in the range of 2 to 3 samples per hour.

Table 6-1. HAPSITE GC/MS Cost Summary

Instrument/Accessory	Cost
Instrument (HAPSITE / headspace sampling accessory / service module (option), notebook computer and startup kit)	$75,000 (without service module) $95,000 (with service module)
Instrument accessories (field-portable printer)	$300 – $500
Sample handling accessories (syringes, vials, standards)	$500 per 100 samples
Maintenance costs	$4500 per 240 hours of use
Carrier gas, internal standard gas, and getter pump replacement	$500 service charge for pump replacement at factory

Operators and Training

One operator is required for GC/MS operation. An additional person is helpful when the headspace sampling accessory is used with a high volume of samples, such as in this demonstration. For wellhead monitoring operations where instrument use would follow a well-sampling team, one operator is probably sufficient since only one or two samples would be provided per hour. The GC/MS operator should be a well-trained technician, preferably with a B.S. in chemistry. Sampling preparation and injection into the instrument could be carried out by a field technician with minimal training; however, instrument response and results would have to be checked daily by a qualified analyst.

Data Processing and Output

The instrument produces a typical GC/MS report with internal and surrogate standard checks directly from operating software. The software operates in a multitasking environment so that a sample analysis can be in progress while the results of completed samples are being reviewed. Data reports were checked and printed within minutes of run completion. The operator's objective of reporting valid results for 90% or more of 20 samples by the end of the day was easily met. The unit can also be operated without the laptop, by use of a liquid crystal display screen and keypad on the instrument. Data collected in this manner can be downloaded to a laptop and reviewed at the end of the day. Total ion chromatograms can be reprocessed (reintegrated) with other parameters and/or calibration responses. Data can be downloaded to a spreadsheet for further evaluation or graphical reporting.

Compounds Detected

With the relatively low column temperature (< 80 °C) in isothermal operation, hexachlorobutadiene and three other higher boiling point compounds (1,2-dichlorobenzene, 2-chloroethyl vinyl ether, and 1,2-dibromochloropropane) listed in the demonstration plan were not included in the GC/MS method. The instrument can detect dichlorobenzenes with a longer run time, but the choice was made to go with shorter run times for this demonstration to increase the sample throughput rate.

Initial and Daily Calibration

Calibration was done as specified in Method 8260A and in the written field method. Linear regression with a forced zero intercept was used to derive a single response factor for each target compound. A full calibration was performed before bringing the instrument to the SRS demonstration. The calibration runs used to generate the method calibration table can be selected by the operator. A continuing calibration check sample was typically run two times per day. These daily calibration checks can be used to update calibration response factors if necessary.

QC Procedures and Corrective Actions

Mass calibration checks and updates were run after 4 hours of operation to account for ambient temperature changes. This procedure is specified in the field method and is required when temperature changes of 10 °C or more are encountered during instrument operation. In addition to periodic calibration checks, internal and surrogate standards were run with nearly every sample. These standards give additional measures of instrument data quality. Blanks were also run at the start of the day. Corrective actions such as calibration rechecks, or sample reruns were taken when surrogate standard recoveries were <60 or >140% of a typical response. See Chapter 7 for additional vendor comments on the use of blank samples following high-concentration samples.

Sample Throughput

Maximum sample throughput is about 25 samples per 10-hour day.

Problems Observed During Audit

No problems were observed during the audit.

Data Availability and Changes

Data from the HAPSITE were obtained at the end of each demonstration day in hardcopy format. Data were provided in spreadsheet format at the conclusion of each demonstration week. Several typographical errors were corrected at the final data review; however, no substantive data changes were made.

Applications Assessment

This demonstration was intended to provide an assessment of the instrument’s suitability for analytical tasks in site characterization and routine site monitoring. Site characterization refers to those instances where subsurface contamination is suspected but information on specific compounds and their concentration levels is not available. The instrument best suited for this application is one that can screen a wide array of compounds in a timely and cost-effective manner. Analytical precision and accuracy requirements may be relaxed in these instances since a general description of the site characteristics is adequate for remediation planning. At the other end of the spectrum is a monitoring application where contaminant compounds and their subsurface concentrations are known with some certainty. Periodic monitoring requirements imposed by local regulatory agencies may specify the performance of analyses for specific contaminant compounds known to be present in the water. Quarterly well-monitoring programs fall into this category.

Based on its performance in this demonstration, the HAPSITE is suitable for both characterization and monitoring applications. Since it is a GC/MS system utilizing data-processing software that includes a mass-spectral library and search routines, it is particularly well suited to characterization applications that require identification of unknowns. The existence of two compound characteristics, GC column retention time and mass spectral data, allows unknown compounds to be identified with a high degree of certainty. The precision and accuracy of the HAPSITE indicate that it is also suited for routine monitoring applications; the results obtained on groundwater samples with the HAPSITE were comparable to those obtained from a reference laboratory.

Chapter 7
Technology Update

Note: The following comments were submitted by the technology developer. They have been edited for format consistency with the rest of the report. The technical content in the following comments has not been verified by the verification organization.

Review of Demonstration and Results

The ETV Program offered an excellent opportunity to test the performance of the HAPSITE with an equilibrium headspace accessory for the determination of VOCs in water. As a developer of new technology, we recognize the benefit of independent verification of system performance as an important step in the acceptance of innovative technologies. The data contained in the report represent a thorough evaluation of important performance attributes, precision, accuracy, and operational parameters of the HAPSITE for field work. We appreciate the commitment of the EPA and the resources contributed by the Agency to make this evaluation possible.

The demonstration has shown that the HAPSITE produces analytical results in the field that are directly comparable to reference laboratory results. The data represented in this report support the use of the instrument in all phases of site investigation and remediation work, including the generation of data for regulatory compliance. Evaluation of the results has resulted in further improvements in the design of the system and analytical method. The following comments address these changes and some minor discrepancies between the HAPSITE and reference laboratory results.

Observed False Positive Results

The initial system design specified a carryover of ≤0.25% from sample to sample. The false positives listed in Table 5-2 indicate that the components are the result of carryover from high-level samples run prior to the blanks. While the carryover was less than the specification of 0.25%, changes have been made to the instrument design to further reduce this specification to ≤0.15%.

Observed False Negative Results

Eight of the 12 false negatives (Table 5-3) from the SRS site were generated on the first day of testing. After the first day, a change was made in the flow rate that transfers the sample from the headspace unit to the GC/MS. This flow rate affects system sensitivity. After the flow rate was reset, only four additional false negatives were generated. These compounds could be seen by manual review of the GC/MS data, but had not been found by the peak detection software. Software settings were modified to correct this. The improved flow rate and software settings were incorporated prior to MAFB testing and resulted in improved performance. The two compounds that did cause false negatives at MAFB (1,1,2,2-tetrachloroethane and bromoform) were tested near the system MDL for these compounds. The performance for bromoform indicates that the reported method detection limit should be raised.

Comparison of HAPSITE and Laboratory Groundwater Sample Results

A comparison of HAPSITE and reference laboratory groundwater results demonstrates a highly linear relationship. The HAPSITE produces results that match laboratory results, within ± 35%, 95% of the time. The absolute percent difference for pooled groundwater samples from the SRS was 29% for the 95th percentile. However, the MAFB absolute percent difference for pooled groundwater samples was 64% for the 95th percentile. We believe this number is unduly high and is a result of an error in the reference laboratory results. Figure 5-10 shows a consistently high percent difference between the HAPSITE and reference laboratory results for the compound *cis*-1,2-dichloroethene. It is our opinion that this systematic bias for *cis*-1,2-dichloroethene is due to an error at the reference laboratory. In the groundwater samples MG-30, MG16, MG17, and MG24 from MAFB, *cis*-1,2-dichloroethene was detected by the HAPSITE at an average value of 42 μg/L with an RSD of 9% for the replicates. The laboratory detected *cis*-1,2-dichloroethene in only one of the replicates at 27 μg/L, resulting in the compound being reported as a nondetect for the average. Manual review of the HAPSITE results confirmed the presence of *cis*-1,2-dichloroethene by retention time and mass spectrum. The last quarterly monitoring result from MAFB supports the HAPSITE data, with *cis*-1,2-dichloroethene present at 41 μg/L for the low, No. 1 sample set (Table 3-3).

Number of Compounds Detected

The HAPSITE detected 59 out of 68 compounds reported by the reference laboratory in the groundwater samples at concentration levels greater than 1 μg/L. Of the 9 compounds not detected, 8 were reported by the reference laboratory at or below the MDL of the HAPSITE for that compound. Trichlorofluoromethane (Freon11) was reported at 20 μg/L by the reference laboratory. The HAPSITE report listed the compound as detected, but no concentration was reported. The concentration was not reported because the standard for the gases failed internal instrument QA/QC.

The HAPSITE detected a number of compounds not reported by the laboratory in the very high-level samples. This is a result of the ability of the HAPSITE to analyze these samples undiluted. The laboratory was informed which samples were expected at a high level and diluted these samples prior to analysis to prevent contamination of the purge-and-trap and stay within the linear range of the system. The dilution caused a number of compounds to go undetected because they were then below the detection limits of the system. The ability to analyze samples without dilution and to run samples of unknown concentration without fear of system contamination is a major advantage when using equilibrium headspace for site work.

Chapter 8
Other Deployments

Monterey Airport (April 1998)
Monterey, CA
Groundwater Investigation
Contractor: U.S. Army Corp of Engineers

Contacts: Pat Cantrell – USACE, Sacramento District
Pam Wehrmann- USACE, Sacramento District

Field-Portable Analytical was contracted by the U.S. Army Corp of Engineers to perform a groundwater plume investigation for benzene, toluene, ethyl benzene, and xylenes. The HAPSITE GC/MS and headspace sampling systems were used to analyze samples during the site investigation. The results were used to decide if further sampling was needed and where the next sample would be located. Immediate, definitive data were required to make these decisions. Results from the HAPSITE were reported within 30 minutes of sample collection.

Field-Portable Analytical, Inc.
6054 Garden Towne Way, Suite G
Orangevale, CA 95662
(916) 989-6200

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