Environmental Technology Verification Report

Field-Portable Gas Chromatograph

Sentex Systems, Inc. Scentograph Plus II

by

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Notice

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

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ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE: **FIELD-PORTABLE GAS CHROMATOGRAPH**

APPLICATION: **MEASUREMENT OF CHLORINATED VOLATILE ORGANIC**
                      COMPOUNDS IN WATER

TECHNOLOGY NAME: **Scentograph Plus II**

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PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification (ETV) Program 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 Sentex Systems, Inc. Scentograph Plus II, field-portable gas chromatograph (GC).

DEMONSTRATION DESCRIPTION

The field demonstration of the Scentograph Plus II portable GC was held in September 1997. The demonstration was designed to assess the ability of the instrument to detect and measure chlorinated VOCs 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 geological 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, Sentex Systems, Inc., Scentograph Plus II.* (EPA/600/R-98/145).

TECHNOLOGY DESCRIPTION

Gas chromatography with electron capture detection is a proven analytical technology that has been used in environmental laboratories for many years. The gas chromatographic column separates the sample into individual components. The electron capture detector measures a change in electron current from a sealed radioactive source as compounds exit the chromatographic column, move through the detector, and capture electrons. The electron capture detector is particularly sensitive to chlorinated compounds. Compound identification is achieved by matching the column retention time of sample components, run under controlled temperature conditions, to those of standard mixtures run under similar conditions. Quantitation is achieved by comparing the detector response intensity of sample component and standard. A GC offers some potential for identification of unknown components in a mixture; however, a confirmational analysis by an alternative method is often advisable. Portable GC is a versatile technique that can be used to provide rapid screening data or routine monitoring of groundwater samples. In many GC systems, the instrument configuration can also be quickly changed to accommodate different sample matrices such as soil, soil gas, water, or air. As with all field analytical studies, it may be necessary to send a portion of the samples to an independent laboratory for confirmatory analyses.

The Scentograph Plus II consists of three modules: a purge-and-trap unit, a GC, and a notebook computer for instrument control and data acquisition. The entire system weighs about 80 pounds and is about the size of a large suitcase. The units can be easily transported and operated in the rear compartment of a minivan or station wagon. Instrument detection levels for most chlorinated VOCs in water range from 0.1 to 50 µg/L. Sample processing and analysis can be accomplished by a chemical technician; however, instrument method development, instrument calibration, and data processing require a higher level of operator experience and training. The recommended training interval for routine sample processing is 1 day for a field technician with limited GC experience. At the time of the demonstration, the baseline cost of the Scentograph Plus II was $35,000. Operational costs, which take into account consumable supplies, are on the order of $25 per 8-hour day.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the Scentograph Plus II were observed:

Sample Throughput: Throughput was about two samples per hour. This rate includes the periodic analysis of blanks and calibration check samples. The sample throughput rate is influenced by the complexity of the sample, with less complex samples yielding higher throughput rates.

Completeness: The Scentograph Plus II reported results for all 165 PE evaluation and groundwater samples provided for analysis at the two demonstration sites.

Analytical Versatility: The Scentograph Plus II was calibrated for and detected 59% (19 of 32) of the PE sample VOC compounds in the PE samples provided for analysis at the demonstration. Three pairs of coeluting compounds were encountered with the GC methods used during this demonstration. For the groundwater contaminant compounds for which it was calibrated, the Scentograph Plus II detected 35 of the 62 compounds reported by the reference laboratory at concentration levels in excess of 1 µg/L. A total of 68 compounds 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 in terms of relative standard deviations (RSD). The RSDs compiled for all reported compounds from both sites had a median value of 8% and a

95th percentile value of 32%. 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 Scentograph Plus II RSD values for specific target compounds were as follows: trichloroethene, 0 to 17%; tetrachloroethene, 3 to 28%; 1,2-dichloropropane, 5 to 12%; 1,1,2-trichloroethane, 6 to 24%; *trans*-1,3-dichloropropene, 4 to 29%; and 1,2-dichloroethane, 6 to 36%.

Accuracy: Instrument accuracy was evaluated by comparing Scentograph Plus II results with the known concentrations of chlorinated VOCs in PE mixtures. Absolute percent difference (APD) values from both sites were calculated for all reported compounds in the PE mixtures. The APDs from both sites had a median value of 10% and a 95th percentile value of 38%. 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 Scentograph Plus II APD values for target compounds were as follows: trichloroethene, 1 to 24%; tetrachloroethene, 0 to 15%; 1,2-dichloropropane, 2 to 22%; 1,1,2-trichloroethane, 3 to 16%; *trans*-1,3-dichloropropene, 0 to 24%; and 1,2-dichloroethane, 3 to 78%.

Comparability: A comparison of Scentograph Plus II and reference laboratory data was based on 33 groundwater samples analyzed at each site. The correlation coefficient (*r*) for all compounds detected by both the Scentograph Plus II and laboratory at or below the 100 µg/L concentration level was 0.974 at Savannah River and 0.959 at McClellan. The *r* values for compounds detected at concentration levels in excess of 100 µg/L were 0.907 for Savannah River and 0.997 for McClellan. These correlation coefficients reveal a highly linear relationship between Scentograph Plus II and laboratory data. The median APD between groundwater compounds mutually detected by the Scentograph Plus II and the reference laboratory was 12% with a 95th percentile value of 194%.

Deployment: The system was ready to analyze samples within 60 minutes of arrival at the site. At both sites, the instrument was transported in a minivan and operated from its folded middle seat. The instrument was powered by line ac or from a small dc-to-ac inverter connected to the vehicle's battery.

The results of the demonstration show that the Sentex Systems, Inc., Scentograph Plus II field-portable GC with electron capture detector 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.

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US EPA ARCHIVE DOCUMENT

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 technologies designed for the analysis of chlorinated volatile organic compounds in groundwater. The developers participating in this demonstration were: Electronic Sensor Technology, Perkin Elmer-Photovac, and Sentex Systems, Inc. (field-portable gas chromatographs); Innova AirTech Instruments (photoacoustic infrared analyzer); and Inficon, Inc. (field-portable gas chromatograph/mass spectrometer). This report documents demonstration activities, presents demonstration data, and verifies the performance of the Sentex Scentograph Plus II field-portable gas chromatograph. 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 designed to evaluate the capabilities of each field-transportable 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 Scentograph Plus II gas chromatograph. Instrument precision and accuracy were determined by an analysis of replicate samples from 16 multicomponent standard mixtures of known composition. The relative standard deviations, obtained from an analysis of 4 replicate samples from each of the 16 standard mixtures, were used as measures of precision. The relative standard deviations from all compounds had a median value of 8% and a 95th percentile value of 32%. Accuracy was expressed as the absolute percent difference between the Scentograph Plus II measured value and the true value component in the standard mixtures. The distribution of absolute percent differences for all reported compounds had a median value of 10% and a 95th percentile value of 38%. A comparison of Scentograph Plus II and reference laboratory results from 33 groundwater samples at each site resulted in a median absolute percent difference of 12% with a 95th percentile value of 194%. A correlation analysis between Scentograph Plus II and laboratory results resulted in correlation coefficients (*r*) greater than 0.96 at low (≤100 µg/L) contaminant concentrations. Correlation coefficients were greater than 0.91 at high (>100 µg/L) contaminant concentrations. The sample throughput rate of the Scentograph Plus II was determined to be two samples per hour. The Scentograph Plus II costs about $35,000 for a single-detector, single-column configuration, and can be operated by a field technician with minimal training in gas chromatography.

Under appropriate applications, the Scentograph Plus II 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 and the project's data quality objectives.

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Acronyms and Abbreviations

ac	alternating current
Ah	ampere hour
Ar	argon
APD	absolute percent difference
BNZN	benzene
°C	degrees centigrade
cc	cubic centimeters
CCC	calibration check compounds
CCL4	carbon tetrachloride
Cl	chlorine
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
ECD	electron capture detector
EPA	Environmental Protection Agency
ETV	Environmental Technology Verification (Program)
e	electron
eV	electron-volt
GC	gas chromatograph
GW	groundwater
GC/MS	gas chromatograph/mass spectrometer
He	helium
^{3}H	tritium
Hz	hertz, cycles per second
i.d.	inside diameter
L	liter
m	meter
mg	milligram
mg/L	milligrams per liter

mL	milliliter
mm	millimeter
MAFB	McClellan Air Force Base
MAID	microargon ionization detector
MCL	maximum concentration level
MDL	method detection limit
MS	mass spectroscopy
NA	not available
ND	not detected
NERL	National Exposure Research Laboratory
ng/L	nanograms per liter
NIST	National Institute of Standards and Technology
NR	not reported
PC	personal computer
PCE	tetrachloroethene (perchloroethene)
PE	performance evaluation
PID	photoionization detector
ppb	parts per billion
ppm	parts per million
PQL	practical quantitation limit
PVC	poly (vinyl chloride)
QA	quality assurance
QC	quality control
R	organic molecule
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
TCD	thermal conductivity detector
V	volts
V ac	volts alternating current
VOA	volatile organics analysis
VOC	volatile organic compound
μg	microgram
μg/L	micrograms per liter
μL	microliter
μm	micrometer

Acknowledgments

For more information on the wellhead monitoring demonstration, contact:

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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 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 Sentex Systems Scentograph Plus II field-portable gas chromatograph. Reports documenting the performance of other 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 Sentex Systems, Inc. Scentograph Plus II is a computer-controlled, field-portable gas chromatograph (GC) designed to provide complete sample analysis, from calibration to interpretation of results. The technology is based on purge-and-trap sample introduction, using a continuous purge-and-trap module in combination with the Scentograph Plus II gas chromatograph. The Scentograph Plus II automatically performs the following functions:

- calibration
- analysis
- sample collection and injection
- chromatographic separation
- compound detection
- peak identification and integration
- data display and storage, including chromatograms, retention times, concentration levels, and operating conditions
- continuous operation
- recalibration at predefined frequencies
- optional remote operation via modem

The instrument consists of a GC module and a detector module. The GC module includes the oven, columns, and detector(s). Packed columns 3 mm in diameter and up to 3 m in length or capillary columns with 0.53-mm i.d. and up to 105 m in length are available. Two columns can be installed in the oven, whose temperature can be adjusted up to 179 °C. The detectors are mounted in a compartment in the oven and heated to operating temperature. The oven is well insulated and maintains the temperature of the column, the on-column injector, and the detector(s). For optimum compound separation, two-stage temperature ramping is available. In this demonstration, a single-column, single-detector configuration was used.

The Scentograph Plus II can be equipped with a variety of detectors, including electron capture (ECD), microargon ionization (MAID), photoionization, and thermal conductivity. In this demonstration an electron

capture detector was used. Both a microargon ionization detector and an electron capture detector are included in one module. Changing from one detection mode to the other is accomplished by electronic switches. This combined detector module is especially suitable for operation with capillary columns, and has the following sensitivity:

- MAID mode: sub-parts-per-billion (ppb, μg/L) levels of volatile hydrocarbons (e.g., benzene, toluene)
- ECD mode: parts-per-trillion (ppt, ng/L) levels of chlorinated hydrocarbons (e.g., carbon tetrachloride, trichloroethene)

Principle of Operation

Volatile organic compounds in water can be analyzed with the Scentograph Plus II by use of an accessory known as the Aquascan, a continuous purge-and-trap device. This module is a fully computerized sampling system that automates the steps required for purge-and-trap analysis of water samples. The operator connects the system tubing and electrical cables and programs appropriate operating parameters. In the Aquascan, samples are automatically drawn into a 10-mL cell and sparged with a carrier gas. The purged vapors are separated by the gas chromatograph and detected by the MAID or ECD. The results and operating conditions are stored in memory for later recall and review.

The purge-and-trap GC method is used to detect low concentrations of VOCs in water. In most cases, concentrations will range from sub-parts-per-billion levels to hundreds of parts per billion. Higher concentrations (i.e., above 200 ppb) are normally detected using headspace analysis by syringe injection of the headspace or by trapping headspace volumes directly.

The purge-and-trap methodology efficiently removes the VOCs from the solution by purging with an inert carrier gas. The VOCs are then carried to a sorbent material (usually Tenax or Carboxen). The adsorbed VOCs are thermally desorbed onto the analytical column for separation. In routine operation, detection is usually accomplished with a microargon ionization detector. The method identifies and measures extremely low levels of VOCs, which are normally undetectable by other methods. Because low levels of VOCs in water may not generally provide sufficient detectable concentrations of vapors when analyzed directly, the purge-and-trap method provides a sample enrichment factor that brings the VOCs into a detectable quantitative range. Higher concentrations of VOCs in the parts-per-million (ppm) range may also be analyzed; however, sample dilution to obtain a concentration within the working range of the instrument may be necessary.

Instrument Description

Operational Mode

The Scentograph Plus II functions in calibration and sample analysis modes. In the calibration mode, a water sample with known VOC composition is introduced into the Scentograph Plus II for chromatographic separation. The software then displays the resulting chromatogram, including the name, concentration level, and retention time of each compound in the calibration mixture. The area under each peak is integrated and the concentration level of the standard is assigned to this peak area. Automatic multipoint calibration is also available.

The instrument is equipped with an internal calibration cylinder that supplies gas directly to the calibration system. Calibration gas from the cylinder flows through a regulator and directly to the sample loop or preconcentrator.

A calibration port is used to calibrate from a sampling bag, the headspace of an external container, or other external source. The analysis port is used to sample air from the environment or from an enclosed source. When a sample bag is utilized for analysis, the bag may be attached to the analysis port with Teflon tubing.

In the sample analysis mode, the Scentograph Plus II is used to analyze field samples. The software displays the analysis chromatogram above the calibration chromatogram and identifies each peak as it appears. The names, concentration levels, and retention times of the compounds that match those identified during calibration are listed. Compounds detected that do not match those identified during calibration are listed as "unknown." Their retention times and concentration levels, compared with the first calibration peak, are also displayed. The "unknown" compounds may be identified by computer-assisted methods. Sample analysis results may be compared with other calibration results stored in the Scentograph Plus II memory, or libraries that contain hundreds of compounds may be scanned for retention time matches. Since calibration and sample analysis modes are operated under the same conditions, and because calibration can be performed as frequently as required, the analysis results obtained with the Scentograph Plus II are highly reliable and accurate.

Detector Systems
For this demonstration, the Scentograph Plus II was configured with a combined MAID/ECD detector module. The following paragraphs discuss this detector system in detail.

Microargon Ionization Detector
This mode is suitable for the detection of most organic compounds. Its simplicity and ruggedness make it ideal for field use. The MAID enables the Scentograph Plus II to detect sub-parts-per-billion levels of many compounds. It operates on the principle that organic compounds with ionization potentials equal to or less than the excitation energy of argon (11.7 eV) can be ionized and detected. When the argon (Ar) carrier gas passes over a tritium (^{3}H) source, some argon atoms are energized to a metastable state and some are ionized. A steady stream of energized atoms (excitons) is produced in the detector cell. When organic molecules (R) enter the detector, they collide with the excitons. During this collision, energy from the excitons is released to the organic molecules. Since the ionization potential of most organic compounds is less than 11.7 eV, they are ionized by the excitons. High voltage applied across the detector produces a current that is amplified, measured, and used to produce the chromatogram. The high energy of the argon excitons can ionize a large number of compounds, including halomethanes and haloethanes, many of which cannot be identified by other detection methods. The following reactions summarize how a MAID works:

$$Ar \xrightarrow{^3H} Ar^* \text{ (energized to the excited state)}$$
$$Ar^* \text{ (exciton)} + R \text{ (organic molecule)} \rightarrow Ar + R^+ + e^- \text{ (electron)}$$
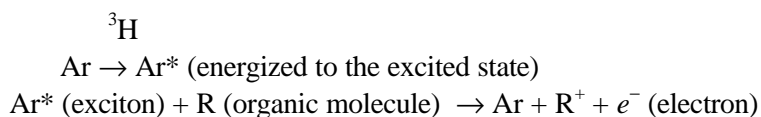
Electron Capture Detector
This mode is highly sensitive and selective for such compounds as halogenated and nitrogenated hydrocarbons capable of capturing electrons. The ECD operates by ionization of the carrier gas by a radioactive source. When argon flows through the detector and over tritium, the following reaction occurs:

$$Ar \xrightarrow{^3H} Ar^+ + e^-$$
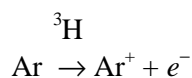
When a low voltage is applied across the detector, a constant current called the standing current is produced. When a compound, such as a halogenated organic, which has an affinity for electrons, enters the detector, the following reaction occurs:

$$R\text{—}Cl \text{ (halogenated compound)} + e^- \rightarrow R\text{—}Cl^-$$
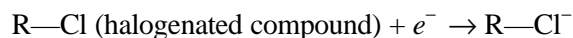
The compound "captures" the electron and becomes a negative ion. The light, fast-moving electrons in the detector are converted to heavy, slow-moving ions. The mobility of the negative charge is decreased, resulting in a decrease in the number of negative charges reaching the electrode. This reduction in current is amplified and measured.

The Scentograph Plus II uses high-purity argon from an internal cylinder for the MAID/ECD configuration and helium carrier gas for the photoionization detector (PID) or thermal conductivity detector (TCD) detection modes.

Sample Injection
Three methods can be used to introduce samples into the Scentograph Plus II: a preconcentrator, a sample loop, or a heated injection port. In this demonstration, a preconcentrator was used.

Preconcentrator
The Scentograph II is normally equipped with a preconcentrator for use when sample concentrations are expected to be 100 parts per million (ppm) or lower. It is packed with an absorbent material such as Tenax and can be varied according to the user's application.

Sample Loop
A sample loop that allows the automatic injection of fixed volumes (usually 0.5 to 1.0 cc sample size) can be installed in place or parallel to the preconcentrator. This loop permits analysis of sample concentrations between 1 ppm and 1000 ppm for most compounds and should be used if concentrations are expected to exceed 10 ppm.

Heated Injection Port
The Scentograph Plus II can be equipped with an optional heated on-column injection port for syringe injection of gas or liquid. Direct injection of gas samples will attain sensitivities similar to using a sample loop. An accessory for an on-column injection to capillary columns is also available. The Scentograph Plus II is equipped with an automatic sampling pump with an intake rate of approximately 80 cc per minute to input sample into the preconcentrator or sampling loop from the internal calibration system, the external calibration port, or the analysis port.

History of the Technology
The Scentograph Plus II has been evaluated for the determination of VOCs in air in three separate field studies. In 1992, its performance was evaluated at a Superfund site under remediation (Berkley et al., 1993). The system was also evaluated in June 1994 for measurement of vinyl chloride emissions at a landfill adjacent to a residential area (Linenberg, 1995). In another study, downwind VOC vapors from an artificial pollution source were analyzed (Berkley et al., 1996).

Applications
The Scentograph Plus II can be used for continuous on-line monitoring of aqueous samples, including drinking water, groundwater, surface water, leachate, and wastewater from hazardous waste sites.

Advantages
Some of the advantages of the Scentograph Plus II are that it:

- *Provides the highest sample integrity* - No sample handling and storage are involved. The Scentograph Plus II pump collects water samples in a 10-mL purge cell. Sample preservation and measurement prior to analysis are eliminated. After each analysis the previous sample is purged with high-purity water to prevent carryover to the next sample.
- *Yields timely and accurate results* - The analysis is done in the field and analytical runs are often completed within 15 minutes. The inherent error due to sample handling and transport is eliminated.
- *Produces off-site laboratory quality results* – Certified standards are used to calibrate the system. The Scentograph Plus II software can accommodate a five-point calibration. In the case where a single-point calibration is used, calibration can be done as often as required. The response factors of the VOCs can be updated daily.
- *Is cost effective* - The availability of results within minutes can guide sample collection for additional off-site laboratory analysis. The Scentograph Plus II can gather large volumes of replicate data that are too expensive to generate using an off-site laboratory.

Limitations
The limiting factor of the Scentograph Plus II is its 179 °C maximum operating temperature. Although it offers two-stage temperature ramping, the absence of a fan to cool the oven makes programming the temperature cumbersome. In some instances, compounds may coelute, making quantitative analysis difficult.

The VOCs are identified by retention time. If the retention time of the sample peak(s) matches the retention time of the standard peak(s), they are assumed to be the same. If any nontarget VOC has the same retention time, it can be misidentified as a target VOC.

Performance Characteristics

Method Detection Limits
The method detection limit (MDL) concentrations for selected VOCs are listed in Table 2-1 and were determined in accordance with 40 CFR Part 136, Appendix B.

Practical Quantitation Limit
With a 50-second purge time, it was determined that the practical quantitation limit (PQL; defined as 10 times the standard deviation of instrument noise) for most compounds is 1 ppb. Increasing the purge time to 200 seconds lowers the PQL of VOCs (e.g., benzene, trichloroethene, and tetrachloroethene) to 0.1 ppb.

Table 2-1. Scentograph Plus II Method Detection Limits for Selected VOCs in Water

Analyte	Retention Time[a] (seconds)	MDL (μg/L)[b]
Chloroform	80	0.08
Benzene	103	0.06
Trichloroethene	124	0.14
Tetrachloroethene	256	0.09
Chlorobenzene	358	0.04

[a] Column condition: 30 m MXT-VOL (Restek) x 0.53 mm i.d. x 3-μm film thickness with argon carrier gas at 23 mL/minute flow rate.
Column temperature held isothermal at 70 °C. Purge time is 50 seconds.
[b] Determined using seven replicates of reagent water spiked with analytes at 1 ppb in accordance with the method outlined in 40 CFR Part 136, Appendix B.

Accuracy
The Scentograph Plus II performs at an accuracy level of ±20% or better over its working range 95% of the time.

Precision
The precision of the Scentograph Plus II, represented by the relative standard deviation (RSD)[1] on replicate measurements, is ≤20% or better over its working range.

Instrument Working Range
At a 50-second purge time, the Scentograph's range is from 1 ppb to 50 ppb. Adjusting the purge time will change the dynamic range.

Comparison with Reference Laboratory Analyses
Prior to this demonstration, no comparative studies had been conducted.

Data Completeness
A total of 20 samples can be analyzed in a 10-hour day. The analytical sequence includes at least one calibration, a blank, a sample duplicate, and a sample spike. This estimate is conservative in order to accommodate sample dilutions and repeat analyses.

Specificity
The MAID can detect compounds with ionization potentials of 11.7 eV or lower. Chlorinated compounds can be confirmed by switching from the MAID to the ECD mode.

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

Other Field Performance Characteristics

Instrument Setup and Disassembly Time
One hour is required to initially assemble and condition the Scentograph Plus II. Disassembly time is about 10 minutes.

Instrument Calibration Frequency
If a multipoint calibration is performed initially, the response factor should be checked daily. If a single-point calibration is used, then a calibration check should be repeated every ten samples.

Ancillary Equipment Requirements

Carrier Gas Cylinder and Regulator
The internal cylinder contains the required carrier gas. The carrier cylinder is easily refilled, and when filled, will provide a minimum of 8 hours of operational time.

Batteries
Lead-acid, 6-V, 6-Ah, rechargeable batteries are used. The batteries must be recharged after each field-portable operation (if applicable) or on a regular basis if the instrument is not in use. For fixed-location operations, the system can be connected to an ac source, using the battery charger supplied with the unit.

Computer and Software
The Scentograph Plus II is equipped with a detachable notebook personal computer (PC) that includes a hard disk drive and a 3.5-inch floppy disk drive. The software program that controls the Scentograph Plus II is contained on the hard drive of the PC. Chromatographic data can be stored on either the removable diskette or the hard drive.

Field Maintenance Requirements
The internal carrier gas cylinder is refilled daily. The lead-acid battery is recharged daily after each field-portable operation. The Scentograph Plus II requires the same preventive maintenance as a bench-top GC.

Sample Throughput Rate
A conservative estimate of sample throughput is 20 samples per 10-hour day, assuming that no significant interferences are encountered during the analyses.

Ease of Operation
The software that controls the GC is user friendly. A few hours of training is sufficient for someone familiar with a GC.

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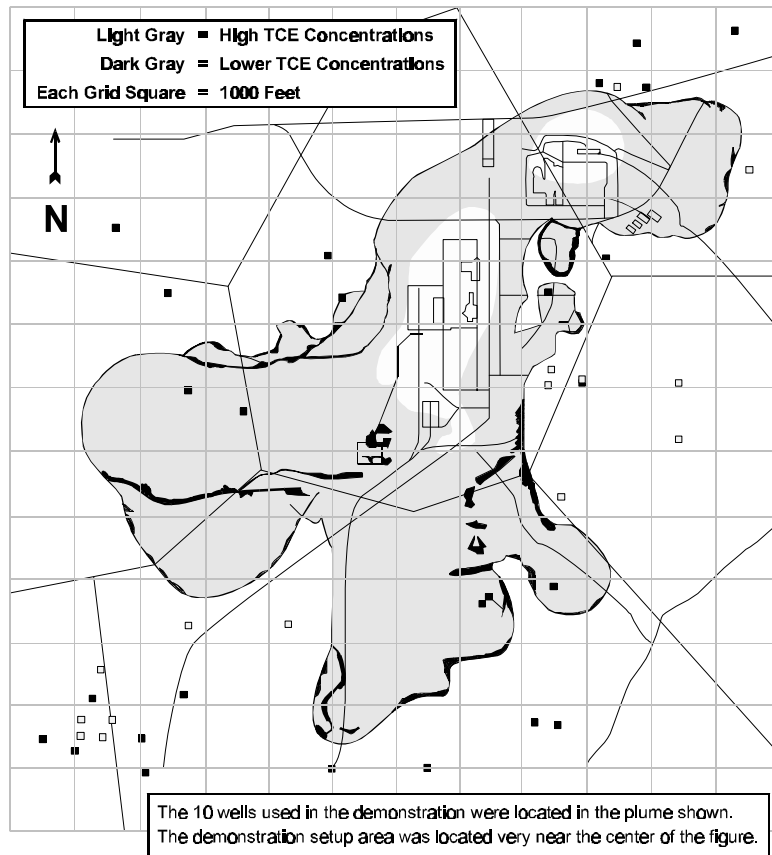
Figure 3-2. A map of the A/M area at the Savannah River Site showing the subsurface TCE plume.

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 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

16
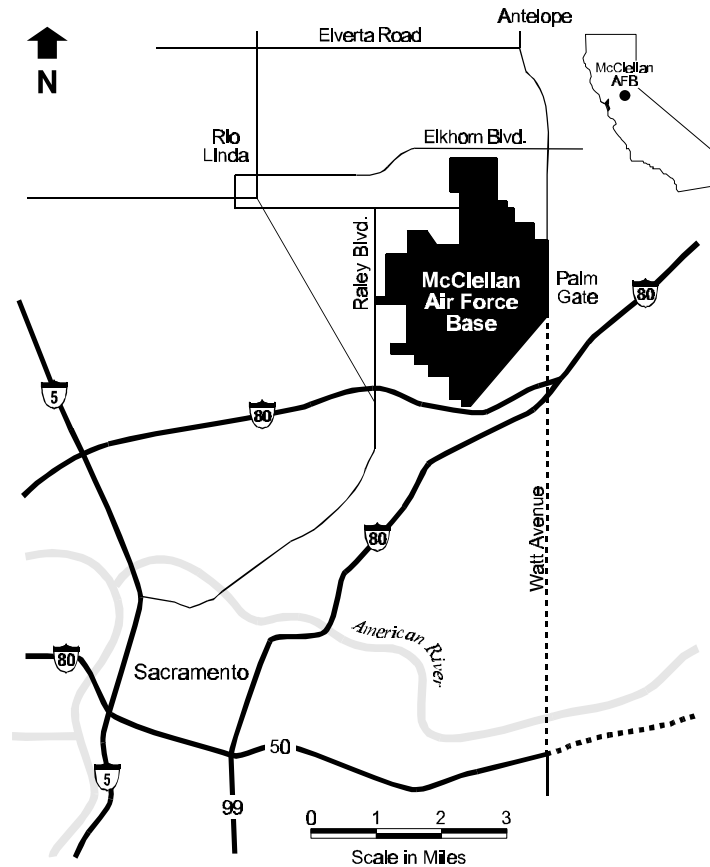
**Figure 3-3. A map of Sacramento and vicinity showing
the location of McClellan Air Force Base.**

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.

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 (MCL) 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.
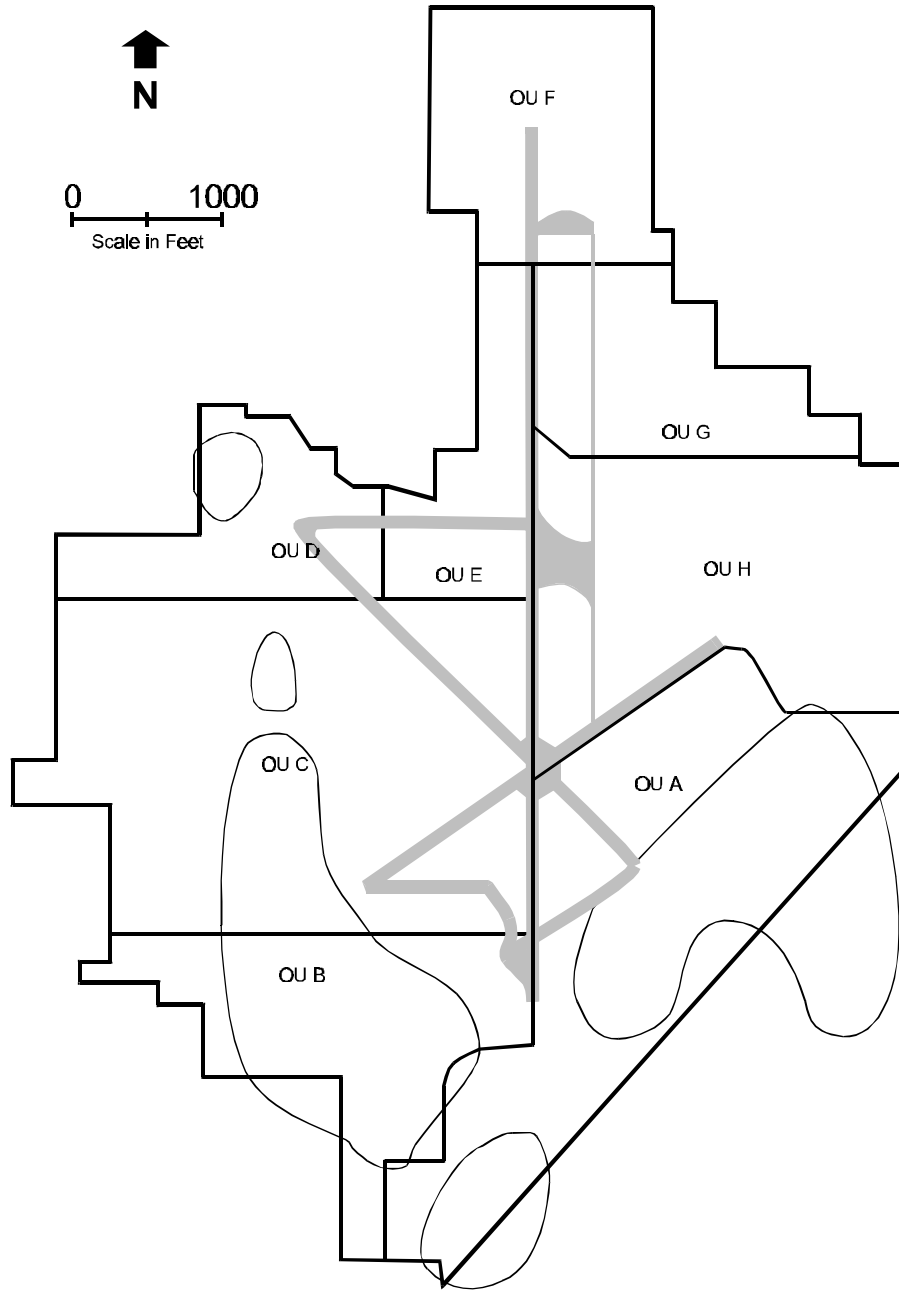
**Figure 3-4. Subsurface TCE plumes at McClellan Air Force Base in the
shallowest (A) aquifer layer. The circular lines enclose plume concentrations in
excess of 5 µg/L TCE. OU refers to operational units. Monitoring wells used in
the demonstration were primarily in OUs A and B. The demonstration setup 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	8
		1,1-Dichloroethene	13
Very low 2	MW-349	Trichloroethene	9
		Tetrachloroethene	5
		Chloroform	8
		Acetone	9
Low 1	MW-331	1,1-Dichloroethane	16
		Carbon tetrachloride	5
		Chloroform	7
		Trichloroethene	19
		cis-1,2-Dichloroethene	41
Low 2	MW-352	1,1-Dichloroethane	6
		Tetrachloroethene	5
		Freon11	115
Mid 1	EW-87	1,1,1-Trichloroethane	17
		1,1-Dichloroethene	334
		Trichloroethene	220
		cis-1,2-Dichloroethene	5
Mid 2	MW-341	Trichloroethene	350
		cis-1,2-Dichloroethene	18
High 1	MW-209	Chloroform	53
		Trichloroethene	586
		cis-1,2-Dichloroethene	80
		trans-1,2-Dichloroethene	13
High 2	MW-330	Chloroform	44
		Trichloroethene	437
		cis-1,2-Dichloroethene	64
		trans-1,2-Dichloroethene	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

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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. Sample Composition and Count for MAFB Demonstration

Sample Concentration Level	PE Mixture - Mixture Concentration[a]	Number 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 ten 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 one month, demonstration samples were run in nine 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.

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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.

DCL Laboratory Control Standard Recoveries
Savannah River Data Set
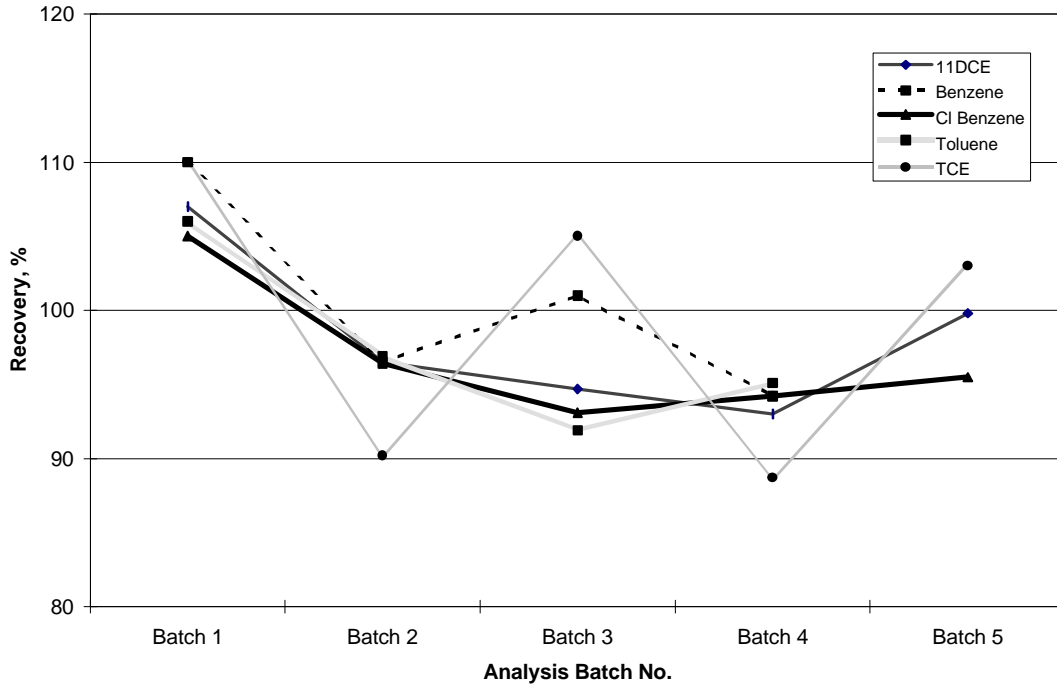
Figure 4-1. Laboratory control standard recovery values for SRS analyses.

DCL Laboratory Control Standard Recoveries
McClellan Data Set
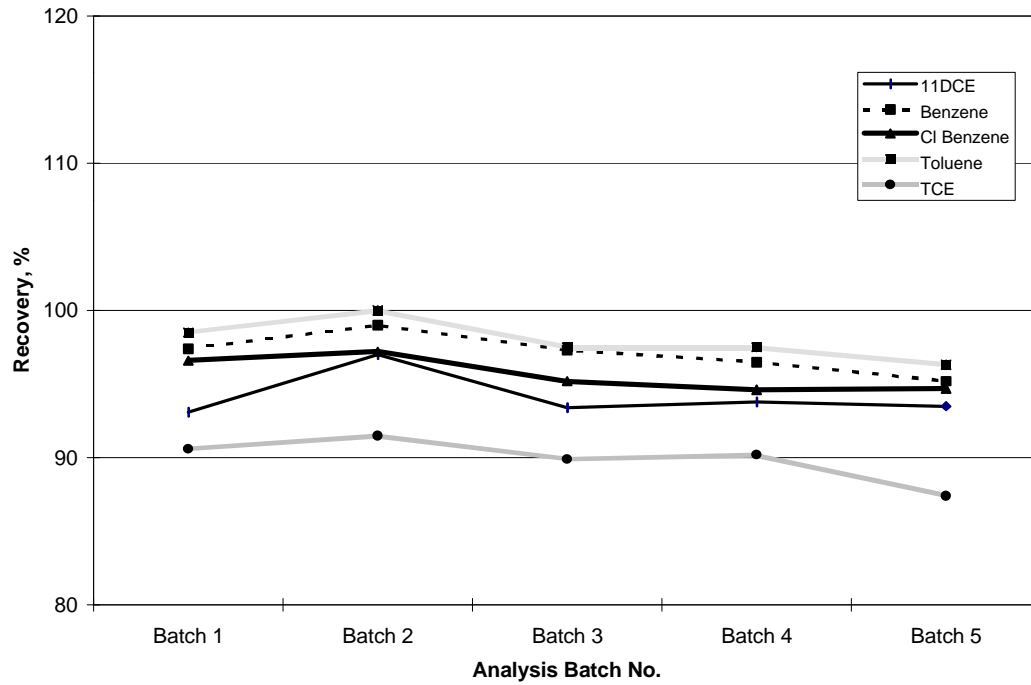
Figure 4-2. Laboratory control standard recovery values for MAFB analyses.

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.

DataChem PE Sample Precision
Site: Savannah River Mix 1

Target Compound

- 1,2-Dichlorobenzene
- Chlorobenzene
- Dibromochloromethane
- Tetrachloroethene
- 1,1,2-Trichloroethane
- 1,2-Dichloropropane
- Trichloroethene
- Carbon Tetrachloride
- Chloroform
- 1,1-Dichloroethene
- Methylene Chloride
- 1,1-Dichloroethane
- Trichlorofluoromethane

Legend:
■ Spike/Low
■ High
□ Mid
■ Low

Relative Standard Deviation, %

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

DataChem PE Sample Precision
Site: Savannah River Mix 2

Target Compound

- Tetrachloroethene
- trans-1,3-Dichloropropene
- cis-1,3-Dichloropropene
- 1,2-Dibromo-3-Chloropropane
- 1,2,3-Trichloropropane
- 1,1,2,2-Tetrachloroethane
- 1,1,1,2-Tetrachloroethane
- 1,2-Dibromoethane
- 1,3-Dichloropropane
- 1,1,2-Trichloroethane
- 1,2-Dichloropropane
- Trichloroethene
- 1,2-Dichloroethane
- 1,1-Dichloropropene

Legend:
■ Spike/Low
■ High
□ Mid
■ Low

Relative Standard Deviation, %

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.

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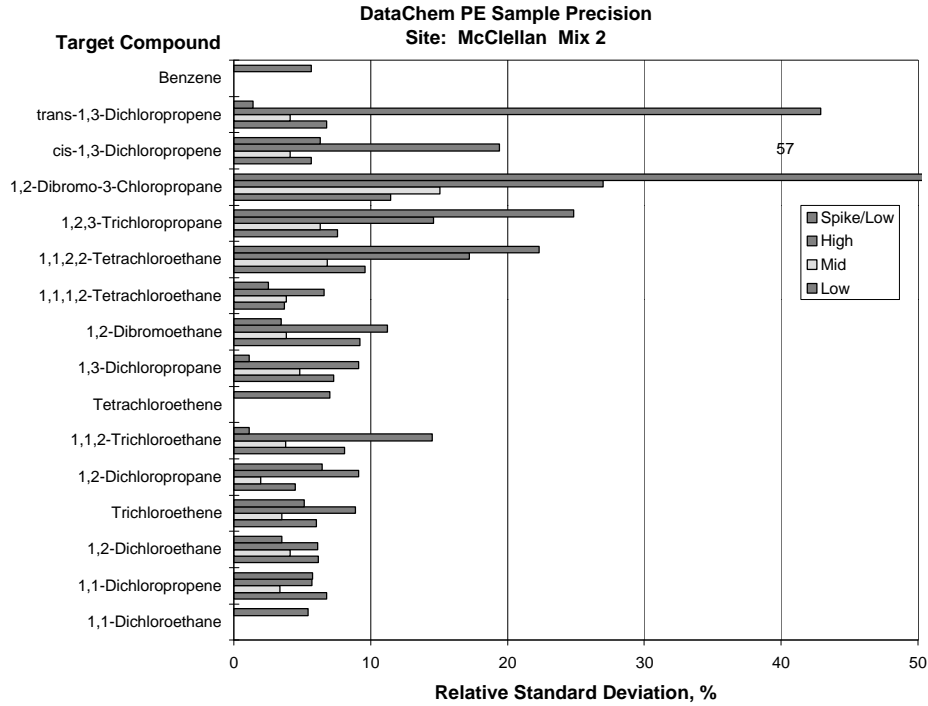
Figure 4-5. Laboratory precision on MAFB PE samples containing mix 2. Trichloroethene, tetrachloroethene, 1,1-dichloroethane and benzene were spiked into the spike/low samples.

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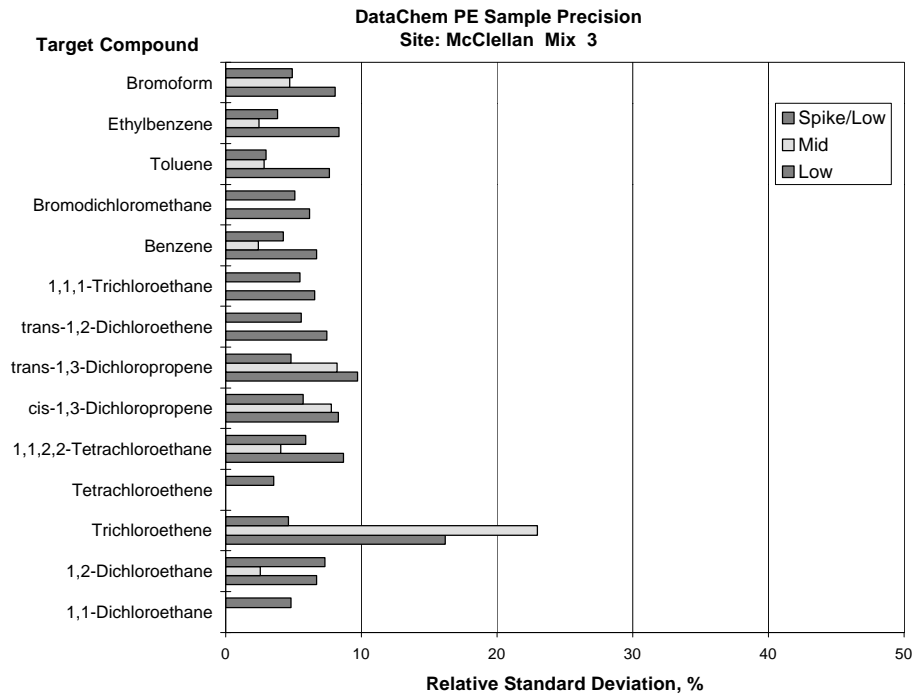
Figure 4-6. Laboratory precision on MAFB PE samples containing mix 3. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

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DataChem PE Sample Recovery
Site: Savannah River Mix 1

Target Compound

Legend: Spike/Low, High, Mid, Low, VLow

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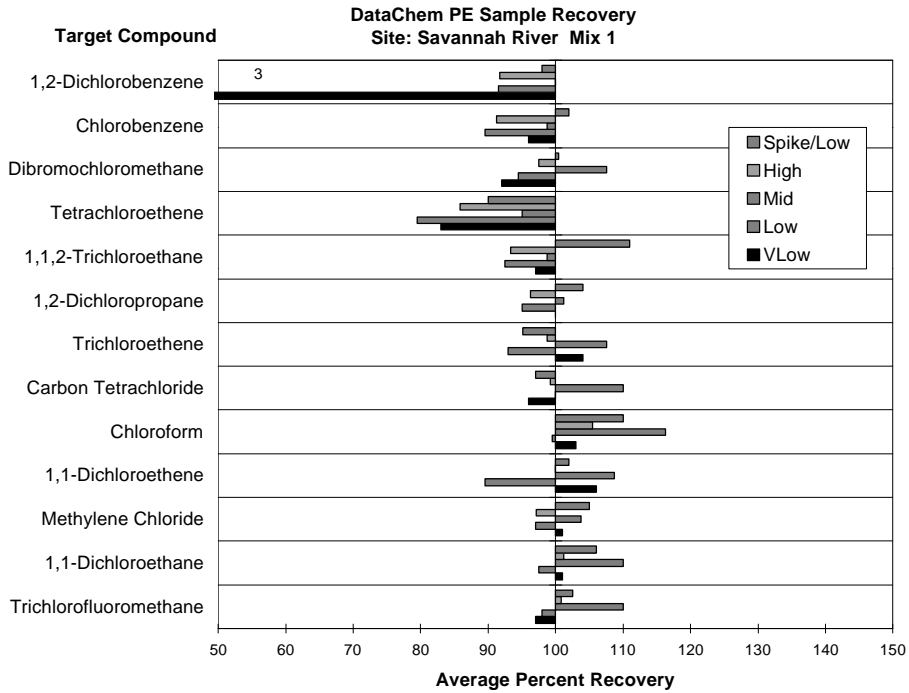
Figure 4-7. Laboratory mean recoveries for SRS PE samples containing mix 1. Trichloroethane was spiked into the spike/low samples.

DataChem PE Sample Recovery
Site: Savannah River Mix 2

Target Compound

Legend: Spike/Low, High, Mid, Low

Average Percent Recovery

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

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DataChem PE Sample Recovery
Site: McClellan Mix 2

Target Compound

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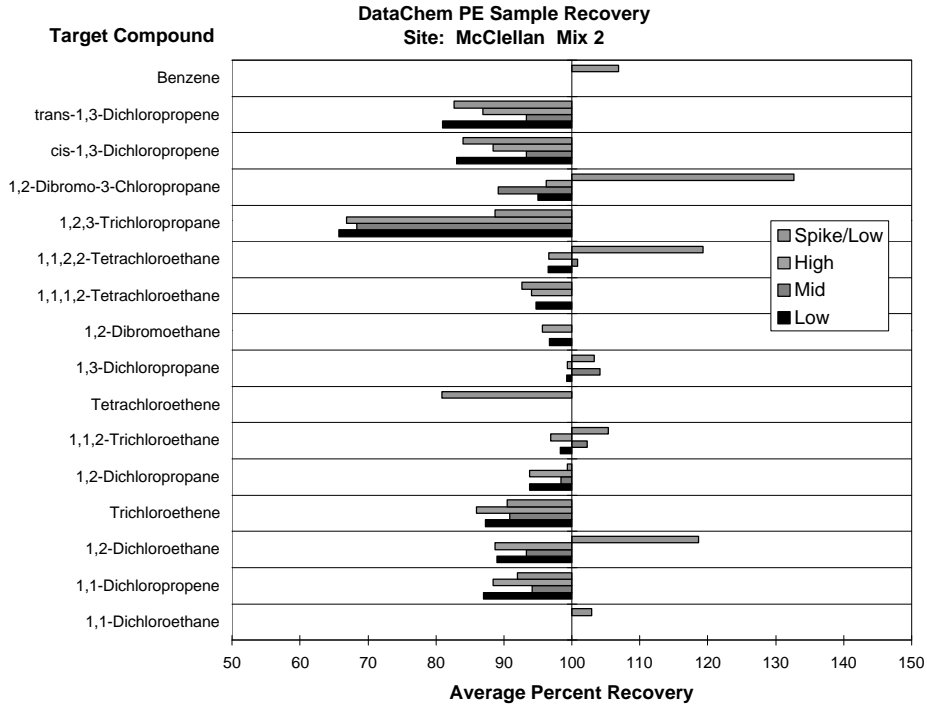
Figure 4-9. Laboratory mean recoveries for MAFB PE samples containing mix 2. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

DataChem PE Sample Recovery
Site: McClellan Mix 3

Target Compound

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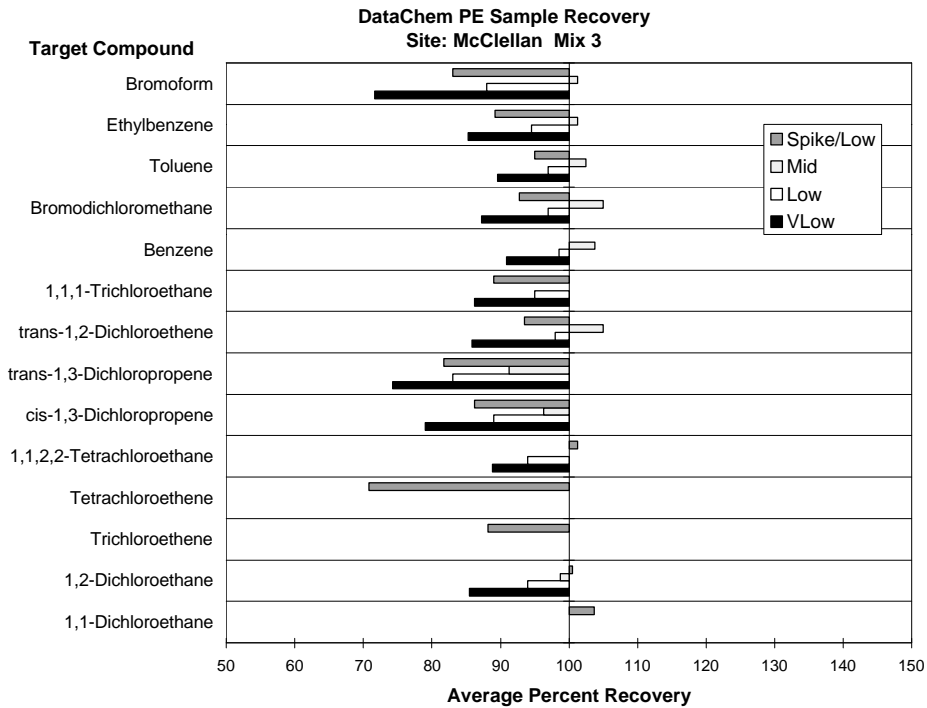
Figure 4-10. Laboratory mean recoveries for MAFB PE samples containing mix 3. Trichloroethene, tetrachloroethene, 1,1-dichloroethane, and benzene were spiked into the spike/low samples.

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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		

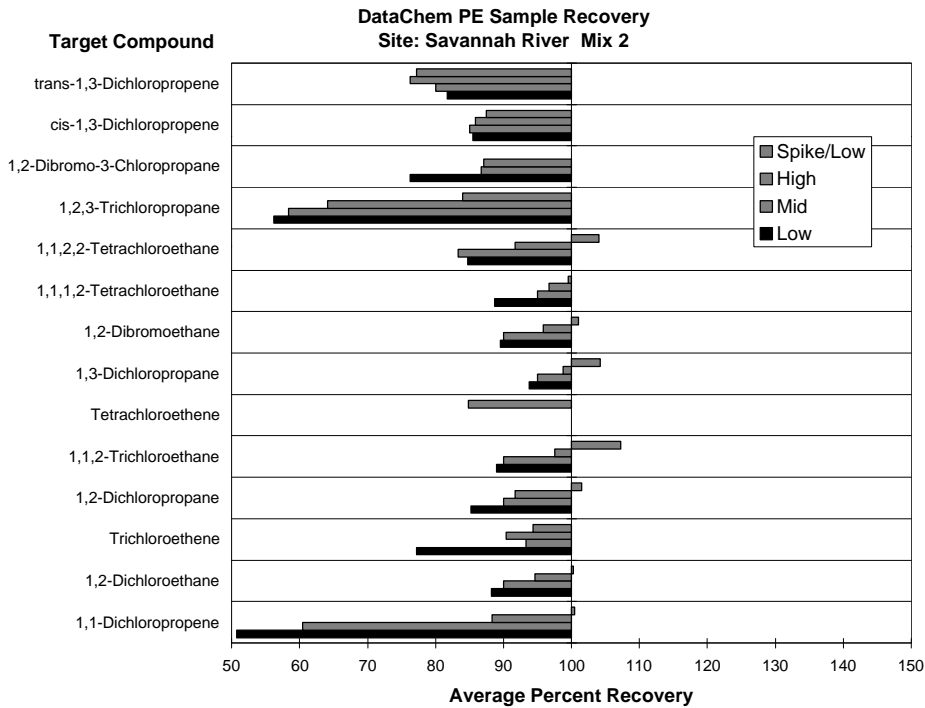Notes: 11DCE = 1,1-dichloroethene; TCE = trichloroethene; CLFRM = chloroform; CCL4 = carbon tetrachloride; PCE = tetrachloroethene; 11DCA = 1,1-dichloroethane; c12DCE = *cis*-1,2-dichloroethene; t12DCE = *trans*-1,2-dichloroethene; BNZN = benzene.
Blank cells indicate that the compound was not present.

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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

Scentograph Plus II 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 performance evaluation (PE) mixtures to facilitate predemonstration instrument calibration. A total of 32 chlorinated and nonchlorinated hydrocarbon compounds were included in the PE mixtures noted in Table 3-4. The Scentograph Plus II system was calibrated for and reported 19 of these compounds at both sites as shown in Table 5-1. One pair of compounds was reported as a coeluting pair. The 13 PE compounds for which the Scentograph Plus II was not calibrated and did not report results are also given in Table 5-1.

Table 5-1. Scentograph Plus II Calibrated and Reported Compounds

PE Compounds Calibrated and Reported	
1,1-Dichloroethene	1,1,2-Trichloroethane
Dichloromethane	Tetrachloroethene
1,1-Dichloroethane	Chlorobenzene
1,2-Dichloropropane	1,1,1,2-Tetrachloroethane
Carbon tetrachloride[a]	cis-1,3-Dichloropropene
1,2-Dichloroethane[a]	trans-1,3-Dichloropropene
Trichloroethene	1,2-Dichloropropane
Toluene	Benzene
Ethyl benzene	trans-1,2-Dichloroethene
1,1-Dichloropropene	
PE Compounds Not Calibrated and Not Reported	
Chloroform	Trichlorofluoromethane
Dibromochloromethane	1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene	1,3-Dichloropropane
Hexachlorobutadiene	1,1,2,2-Tetrachloroethane
Bromodichloromethane	1,2,3-Trichloropropane
2-Chloroethyl vinyl ether	1,2-Dibromoethane
Bromoform	

Note: Superscript denotes coeluting compound pairs (a).

Preanalysis Sample Information

The Sentex team requested that information on concentration accompany each of the samples. Approximate concentration levels of the PE and groundwater sample components were noted on the chain-of-custody forms as

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low (<250 ppb), mid (250 to750 ppb), and high (>750 ppb). The Sentex team used this information to make the appropriate sample dilution prior to GC analysis.

Sample Completion
All 165 PE and groundwater samples submitted for analysis to the Sentex team were completed and reported at both demonstration sites. The demonstration plan specified a total of 166 PE and groundwater samples. As noted in Chapter 3, one of the groundwater replicate samples was inadvertently omitted from the Sentex sample set by the verification organization.

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 1 µg/L. No false positives were reported at either site.

Performance at Method Detection Limit
Ten replicate samples of a PE mixture at a concentration level of 10 µg/L were provided for analysis at each site. Reported nondetects were compiled and are given as percent false negatives in Table 5-2. Vendor-provided compound detection limits, where available, are also shown in the table for comparison.

Table 5-2. 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 Neg.	Compound	False Neg.
1,1-Dichloroethene (NA)	0 of 10	*trans*-1,2-Dichloroethene (NA)	0 of 10
Dichloromethane (NA)	0 of 10	Benzene (0.06)	0 of 10
Carbon tetrachloride (NA)	0 of 10	*cis*-1,3-Dichloropropene (NA)	0 of 10
1,2-Dichloropropane (NA)	3 of 10 (30%)	*trans*-1,3-Dichloropropene (NA)	0 of 10
Trichloroethene (0.14)	0 of 10	Toluene (NA)	0 of 10
1,1,2-Trichloroethane (NA)	0 of 10	Ethyl benzene (NA)	0 of 10
Tetrachloroethene (0.09)	0 of 10	1,1,1-Trichloroethane (NA)	0 of 10
Chlorobenzene (0.04)	0 of 10	1,2-Dichloroethane (NA)	10 of 10
2-Chloroethyl vinyl ether (NA)	No calibration	Bromoform	No calibration
Dibromochloromethane (NA)	No calibration	1,1,2,2-Tetrachloroethane	No calibration
Trichlorofluoromethane (NA)	No calibration	Bromodichloromethane	No calibration
1,1-Dichloroethane (NA)	No calibration		
1,2-Dichlorobenzene (NA)	No calibration		

Notes: Vendor-provided detection limits (in µg/L) are shown in parentheses after each compound.
       NA = not available; vendor did not report method detection limits for these compounds.

PE Sample Precision
Precision results from each of the four replicate sample sets provided to the participant from eight PE mixtures at the SRS and seven mixtures at MAFB are shown in Figures 5-1 and 5-2 for the SRS and Figures 5-3 and 5-4 for

Sentex - Scentograph Plus II PE Precision
SRS Mix 1

Compound
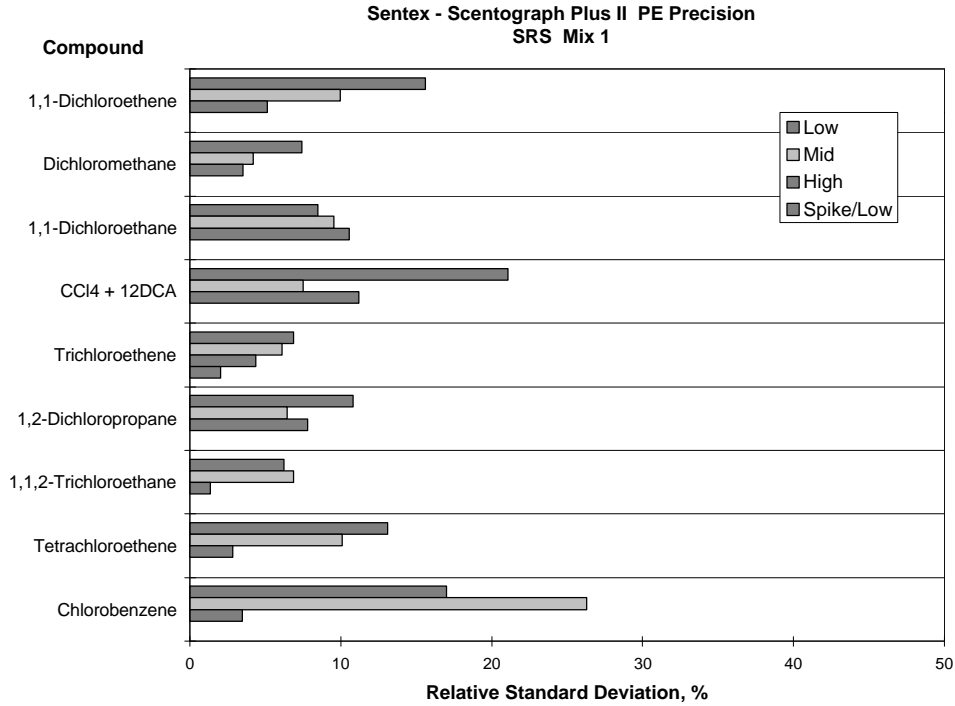
Figure 5-1. Scentograph Plus II precision on PE mix 1 at the SRS.

Sentex - Scentograph Plus II PE Precision
SRS Mix 2

Compound
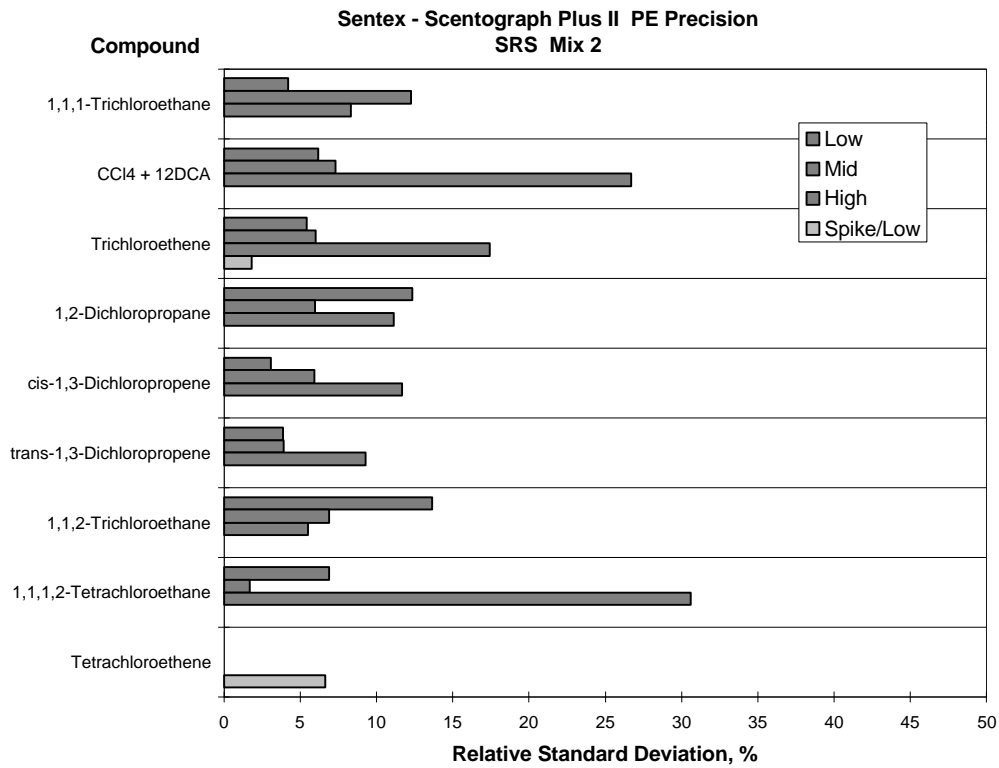
Figure 5-2. Scentograph Plus II precision on PE mix 2 at the SRS.

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Sentex Scentograph Plus II PE Precision
MAFB Mix 2

Compound

1,1-Dichloropropene — 67 — 67

CCl4 + 12DCA

Trichloroethene

1,2-Dichloropropane

cis-1,3-Dichloropropene

trans-1,3-Dichloropropene

1,1,2-Trichloroethane

1,1-Dichloroethane

Benzene

Tetrachloroethene

Legend:
■ Low
□ Mid
■ High
□ Spike/Low

Relative Standard Deviation, % (0, 10, 20, 30, 40, 50)
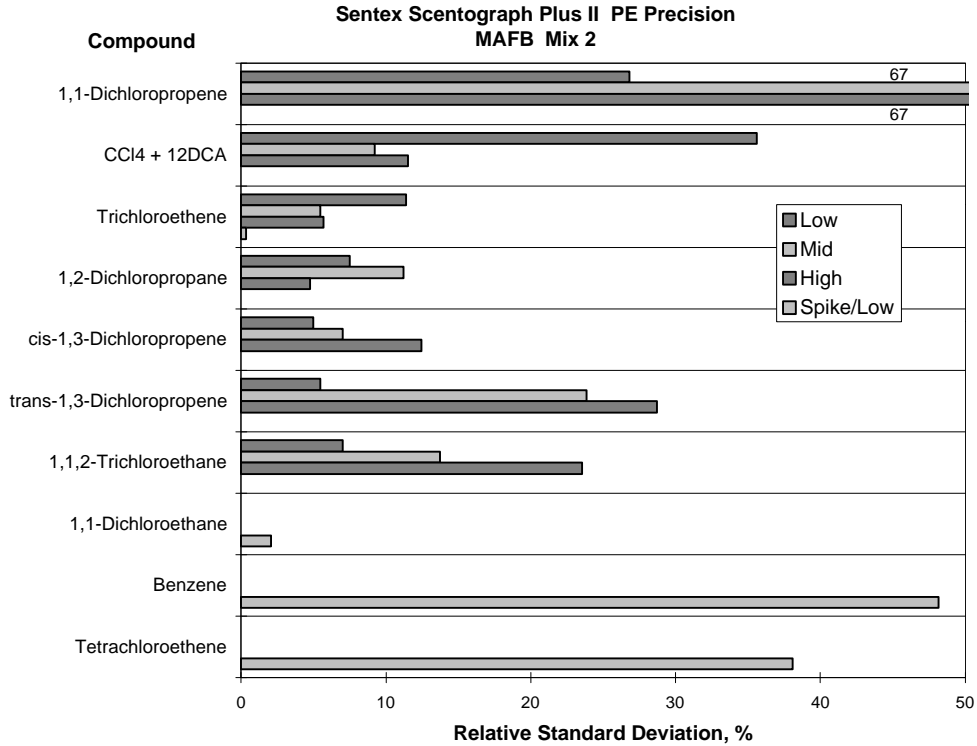
Figure 5-3. Scentograph Plus II precision on PE mix 2 at MAFB.

Sentex Scentograph Plus II PE Precision
MAFB Mix 3

Compound

trans-1,2-Dichloroethene

1,1,1-Trichloroethane

Benzene

cis-1,3-Dichloropropene

Toluene

trans-1,3-Dichloropropene

Ethylbenzene

Trichloroethene

Tetrachloroethene

1,1-Dichloroethane

Legend:
■ Low
□ Mid
■ Spike/Low

Relative Standard Deviation, % (0, 10, 20, 30, 40, 50)
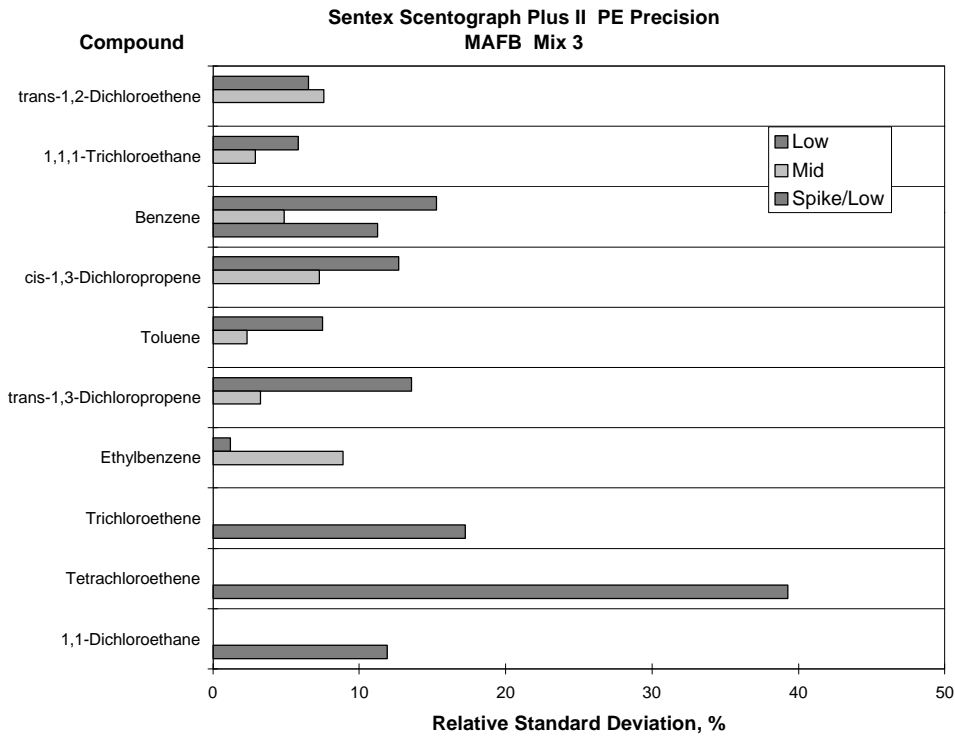
Figure 5-4. Scentograph Plus II precision on PE mix 3 at MAFB.

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MAFB.[1] In instances where no data were reported, no graph bars are shown. The figures show the relative standard deviation for each compound in the PE mixtures at the four concentration levels used in the study. (The compositions and concentrations of each of these mixtures are given in Table 3-5 for SRS and Table 3-6 for MAFB.) Note that precision and accuracy were not determined on the "very low" concentration PE mixes. Relative standard deviations for the coeluting compound pair are also shown in the figures. Instrument precision performance for six target compounds that are all regulated under the Safe Drinking Water act is shown in Table 5-3. The RSDs 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 also given in the last column of the table.

Table 5-3. Target Compound Precision for PE Samples at Both Sites

Target Compound	Site	Relative Standard Deviation (%)				
		Low	Mid	High	Spike/Low	Range
Trichloroethene	SRS	5	6	17	2	0 – 17
	MAFB	11	5	6	0	
1,2-Dichloroethane	SRS	6	7	27		6 – 36
	MAFB	36	9	12		
1,2-Dichloropropane	SRS	12	6	11		5 – 12
	MAFB	8	11	5		
1,1,2-Trichloroethane	SRS	14	7	6		6 – 24
	MAFB	7	14	24		
Tetrachloroethene	SRS	13	10	3		3 – 28
	MAFB			3	28	
trans-1,3-Dichloropropene	SRS	4	4	9		4 – 29
	MAFB	5	24	29		

Note: Blank cells indicate that no data were reported.

A summary of overall instrument precision is given in Table 5-4 for the PE mixtures used at both sites. 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.

PE Sample Accuracy
The Scentograph Plus II accuracy for PE sample analyses was determined by comparing the average value from each of the four-sample replicate sets with the known concentration of the PE mixture (given in 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] In instances where no data were reported, no graph bars are shown. To assist in assessing the sign of the difference, the percent recovery data are plotted as either a positive or negative difference from the 100% recovery line. Instrument recovery performance for the six target

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

[2] Percent recovery is the Scentograph Plus II 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.

Table 5-4. Summary PE Sample Precision and Percent Difference Statistics for the 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	7	7	11	8	8
	95th	20	26	63	21	32
	Number in pool	28	23	25	18	94
Absolute percent difference	50th	6	13	12	8	10
	95th	25	24	66	24	38
	Number in pool	28	23	25	18	94

compounds is shown in Table 5-5. The average percent recoveries and associated ranges are given in the table for each compound.

A summary of overall Scentograph Plus II differences relative to PE mixture true values is given for both sites, alongside the precision summary in Table 5-4. For this summary, percent recoveries were expressed as percent difference (e.g., a 90% recovery is equivalent to a −10% difference; a 120% recovery is equivalent to a +20% difference) and all data from PE mixtures were pooled. The median and 95th percentile of the absolute values of these pooled values were computed and are reported under the absolute percent difference (APD) category in Table 5-4.[4]

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 Scentograph Plus II results for all groundwater samples is given in Tables 5-6 for SRS and 5-7 for MAFB. Well designation (very low, low, mid, high, very high) is based on TCE concentration levels; however, other compounds were present in the groundwater samples at the concentration levels noted in the tables. Average laboratory results for groundwater contaminants reported at levels less than 1 µg/L are not included in the comparisons. The precision of the Scentograph Plus II on replicate groundwater sample sets is also shown as percent RSD in the last column of the tables.

The average percent difference between average Scentograph Plus II and laboratory results for the compounds detected in each set of groundwater samples is shown in Figures 5-9 and 5-10 for the SRS and MAFB, respectively. The SRS groundwater comparison in Figure 5-9 includes only TCE and PCE. Two well samples were also contaminated with 1,1-dichloroethene, as noted in Table 5-6. More complex water samples were selected at MFB, as indicated by the additional compounds shown in Table 5-7 and Figure 5-10.

The median and 95th percentiles of the distribution of absolute percent differences between Scentograph Plus II and laboratory results for all groundwater samples are given in Table 5-8.

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[4] The absolute percent difference is the absolute value of the percent difference between a field and reference (in this case reference laboratory) measurement. As an example, the percent difference between a field measurement of 85 and a laboratory measurement of 110 is –22.7% and the absolute percent difference is 22.7%.
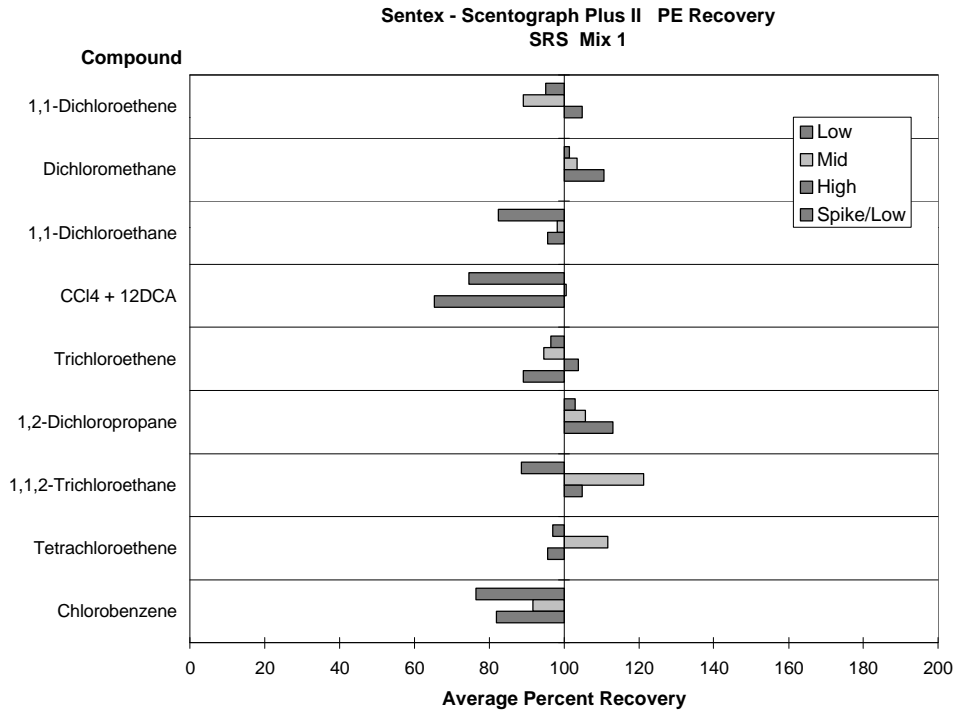
Figure 5-5. Scentograph Plus II recovery on PE mix 1 at the SRS.
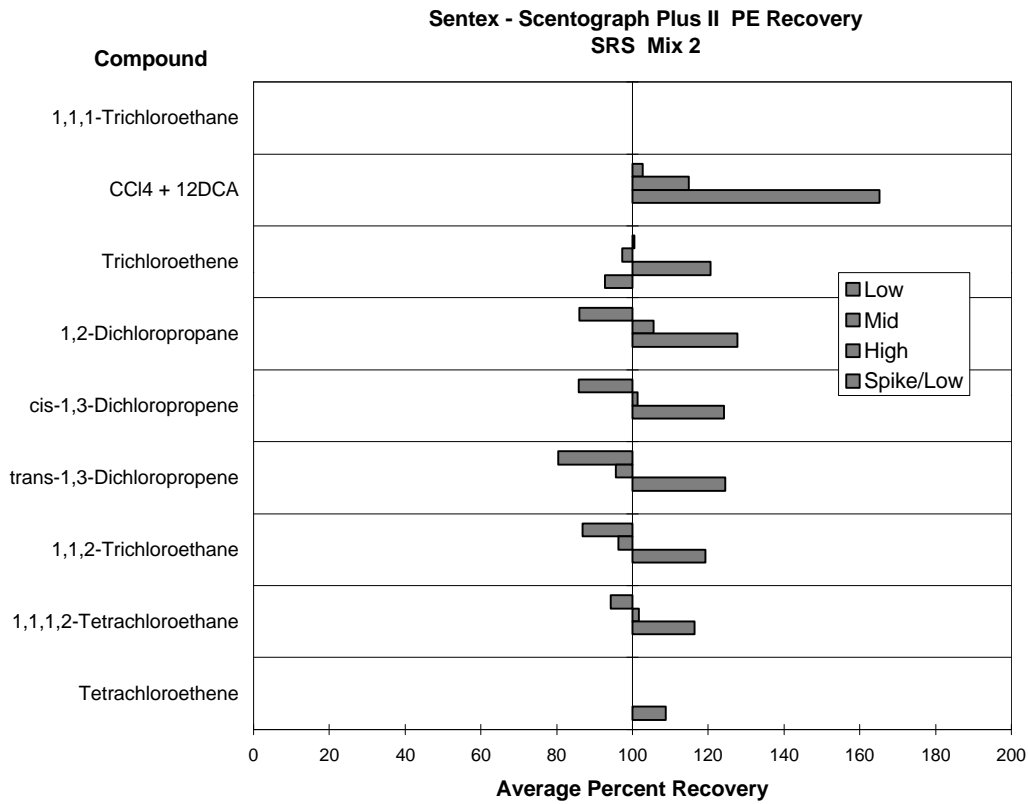
Figure 5-6. Scentograph Plus II recovery on PE mix 2 at the SRS.

Sentex Scentograph Plus II PE Recovery
MAFB Mix 2
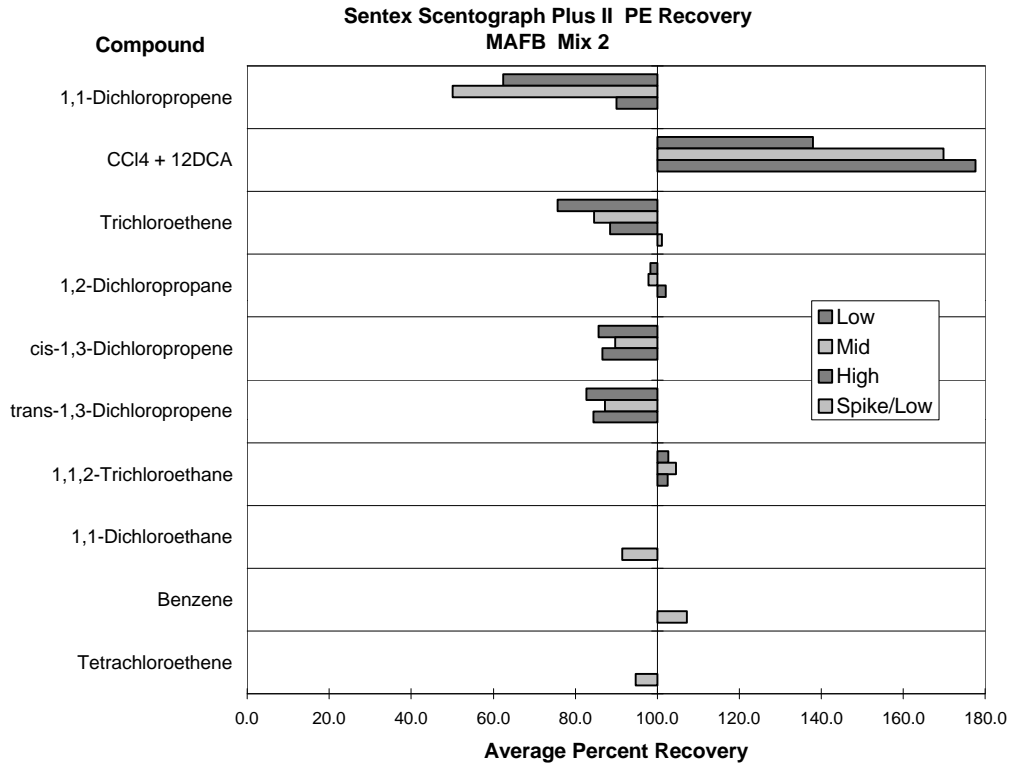
Compound

Figure 5-7. Scentograph Plus II recovery on PE mix 2 at MAFB.

Sentex Scentograph Plus II PE Recovery
MAFB Mix 3
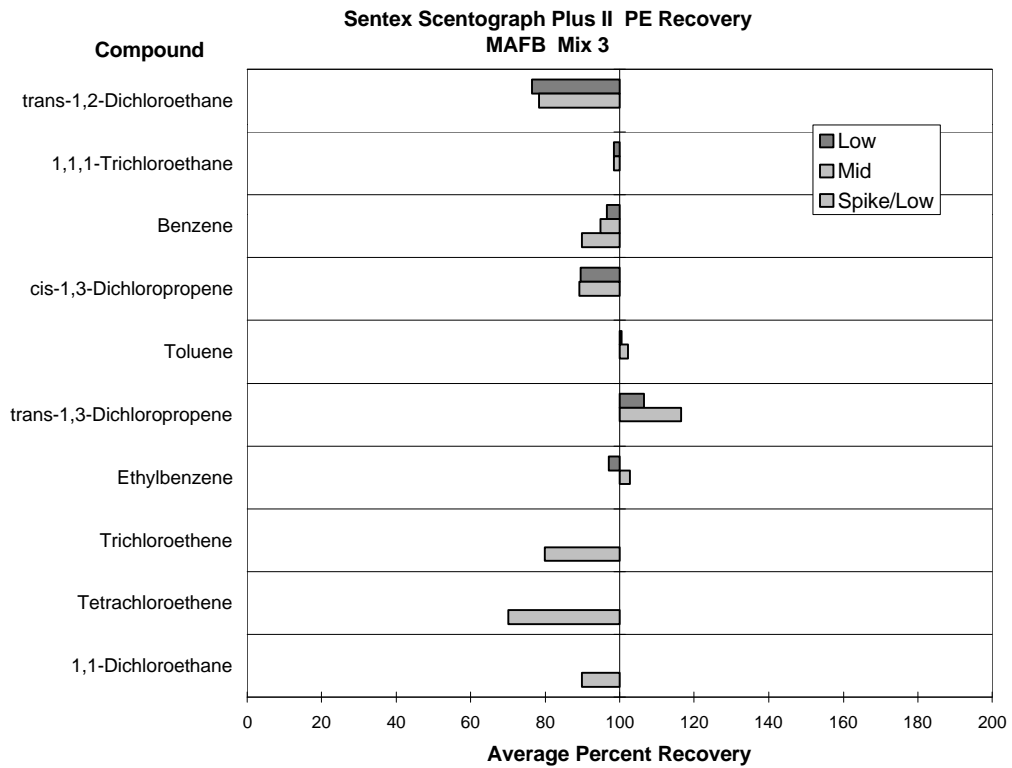
Compound

Figure 5-8. Scentograph Plus II recovery on PE mix 3 at MAFB.

Table 5-5. Scentograph Plus II Target Compound Recovery for PE Mix 2 at Both Sites

Target Compound	Site	Average Recovery (%)				
		Low	Mid	High	Spike/Low	Range
Trichloroethene	SRS	101	97	117	92	76 – 117
	MAFB	76	85	89	101	
1,2-Dichloroethane	SRS	103	113	140		103 – 178
	MAFB	138	170	178		
1,2-Dichloropropane	SRS	84	105	122		84 – 122
	MAFB	98	98	102		
1,1,2-Trichloroethane	SRS	85	96	116		85 – 116
	MAFB	103	105	103		
Tetrachloroethene	SRS	97	112	96		96 – 115
	MAFB			100	115	
trans-1,3-Dichloropropene	SRS	124	100	120		83 – 124
	MAFB	83	87	84		

Note: Blank cells indicate that no data were reported.

Table 5-6. Scentograph Plus II and Reference Laboratory Results for SRS Groundwater Samples

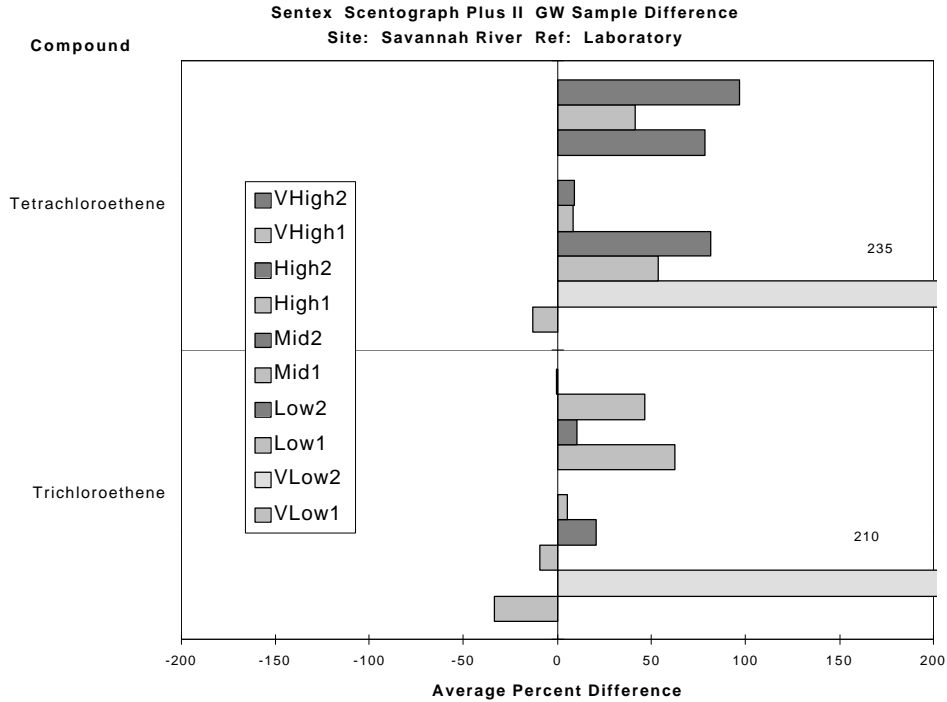| Sample Description	Well Number	Compound	Replicates	Lab. Avg. (µg/L)	Lab. RSD (%)	Plus II[a] Avg. (µg/L)	Plus II[a] RSD (%)
Very low 1	MSB 33B	Trichloroethene	3	9.0	11	6.0	0
		Tetrachloroethene		3.5	14	3.0	0
Very low 2	MSB 33C	Trichloroethene	3	2.4	34	7.3	87
Low 1	MSB 18B	Trichloroethene	3	11	5	9.7	16
		Tetrachloroethene		27	6	41	19
Low 2	MSB 37B	Trichloroethene	4	27	7	32	6
		Tetrachloroethene		22	9	40	17
		Carbon tetrachloride		1.0	15	NR	NR
		Chloroform		1.3	0	NR	NR
Mid 1	MSB 4D	Trichloroethene	4	150	9	158	12
		Tetrachloroethene		87	12	95	20
Mid 2	MSB 64C	Trichloroethene	3	35	7	NR	NR
		Tetrachloroethene		240	4	263	10
		1,1-Dichloroethene		12	8	NR	NR
High 1	MSB 4B	Trichloroethene	3	747	1	1214	7
		Tetrachloroethene		33	2	NR	NR
High 2	MSB 70C	Trichloroethene	4	1875	12	2073	5
		Tetrachloroethene		520	8	927	15
		1,1-Dichloroethene		32	8	NR	NR
Very high 1	MSB 14A	Trichloroethene	3	1367	8	1335	87
		Tetrachloroethene		800	6	754	87
Very high 2	MSB 8C	Trichloroethene	3	4933	6	4911	1
		Tetrachloroethene		3668	6	7215	4
Range					0 – 34		0 – 87
Median					8		12
95th Percentile					15		87

[a] NR = not reported.

Table 5-7. Scentograph Plus II and Reference Laboratory Results for MAFB Groundwater Samples

| Sample Description	Well Number	Replicates	Compound	Lab. Avg. (µg/L)	Lab. RSD (%)	Plus II[a] Avg. (µg/L)	Plus II[a] RSD (%)
Very low 1	EW-86	3	Trichloroethene	4.6	5	4.3	13
			1,1-Dichloroethene	7.7	9	7.7	8
Very low 2	MW-349	3	Trichloroethene	13	0	33	8
			Tetrachloroethene	2.0	6	NR	NR
			Chloroform	9.0	1	26	10
			1,1-Dichloroethene	3.8	3	NR	NR
			Carbon tetrachloride	137	4	NR	NR
Low 1	MW-331	4	1,1-Dichloroethene	2.5	7	NR	NR
			1,1-Dichloroethane	15	0	14	0
			cis-1,2-Dichloroethene	NR	NR	31	2
			Carbon tetrachloride	7.5	2	6.0	0
			Chloroform	4.8	2	NR	NR
			Trichloroethene	16	4	17	0
Low 2	MW-352	3	Freon11	20	6	NR	NR
			1,1-Dichloroethene	1.5	12	NR	NR
			1,1-Dichloroethane	5.1	4	NR	NR
			cis-1,2-Dichloroethene	1.5	4	NR	NR
			Carbon tetrachloride	1.4	4	NR	NR
			Trichloroethene	22	5	21	10
Mid 1	EW-87	4	1,1-Dichloroethene	180	12	159	6
			1,1-Dichloroethane	3.0	9	NR	NR
			cis-1,2-Dichloroethene	3.3	13	NR	NR
			1,1,1-Trichloroethane	6.8	12	NR	NR
			Trichloroethene	114	11	99	9
			Tetrachloroethene	1.2	14	NR	NR
Mid 2	MW-341	3	*cis*-1,2-Dichloroethene	15	4	NR	NR
			Chloroform	3.5	5	NR	NR
			Trichloroethene	280	4	248	4
High 1	MW-209	3	*cis*-1,2-Dichloroethene	38	4	195	4
			Chloroform	6.9	21	NR	NR
			Trichloroethene	238	2	199	0
High 2	MW-330	4	*trans*-1,2-Dichloroethene	7.7	4	NR	NR
			cis-1,2-Dichloroethene	66	5	52	67
			Chloroform	42	5	NR	NR
			1,2-Dibromochloropropane	6.1	6	NR	NR
			Trichloroethene	380	5	325	7
Very high 1	MW-334	3	1,1-Dichloroethene	690	3	NR	NR
			cis-1,2-dichloroethene	237	7	NR	NR
			Chloroform	397	5	NR	NR
			Benzene	283	5	NR	NR
			Trichloroethene	10,667	5	11,031	1
			Carbon tetrachloride	350	5	NR	NR
Very high 2	MW-369	3	*cis*-1,2-Dichloroethene	207	10	NR	NR
			Chloroform	63	6	NR	NR
			Carbon tetrachloride	51	5	NR	NR
			Trichloroethene	6167	8	5486	1
Range					0 – 21		0 – 67
Median					5		6
95th Percentile					13		24

[a] NR = not reported.

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Sentex Scentograph Plus II GW Sample Difference
Site: Savannah River   Ref: Laboratory

Compound

Figure 5-9. Scentograph Plus II groundwater results at the SRS relative to laboratory results.

Sentex Scentograph Plus II GW Sample Difference
MAFB Ref: Laboratory

Compound
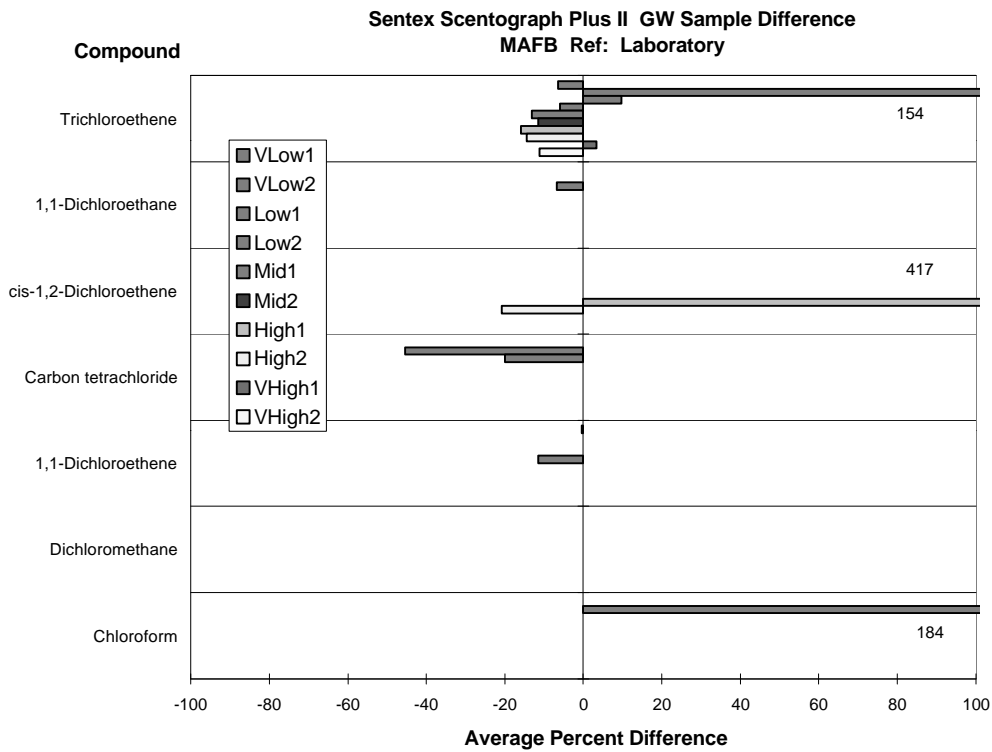
Figure 5-10. Scentograph Plus II groundwater results at MAFB relative to laboratory results.

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Table 5-8. Scentograph Plus II Absolute Percent Difference Summary for Pooled Groundwater Results

Percentile	SRS	MAFB	Combined Sites
50th	14	12	12
95th	118	234	194
Number of samples in pool	17	17	34

To assess the degree of linear correlation between the Scentograph Plus II and laboratory groundwater data pairs shown in Tables 5-6 and 5-7, 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 5-9.

Table 5-9. Correlation Coefficients for Laboratory and Scentograph Plus II Groundwater Analyses

Data Set	Correlation Coefficient	Number of Data Pairs
SRS Laboratory (1 through 100 µg/L)	0.974	8
SRS Laboratory (> 100 µg/L)	0.907	10
MAFB Laboratory (1 through 100 µg/L)	0.959	10
MAFB Laboratory (> 100 µg/L)	0.997	7

Sample Throughput

The Scentograph Plus II throughput was about 2 samples per hour or 16 samples per 8-hour day. Throughput rates were assessed by using the time lapsed between sample checkout in the morning and delivery of hardcopy results in the afternoon or the following day, and the number of samples completed. This throughput estimate includes periodic instrument calibration checks, sample reruns, and data processing tasks. Samples with only one or two known contaminants could be processed faster.

Performance Summary

Table 5-10 contains a summary of Scentograph Plus II 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 Scentograph Plus II performance results to facilitate comparison of the two methodologies.

Table 5-10. Scentograph Plus II Performance Summary

Instrument Feature/Parameter	Performance Summary
Blank sample	No false positives were reported for 19 calibrated compounds.
Detection limit sample	False negatives reported for two compounds at a rate of 30% (1,2-dichloropropane) and 100% (1,2-dichloroethane) out of 18 compounds at 10 μg/L concentration levels. There were no other false negatives.
PE sample precision	Target compounds, Scentograph Plus II RSD range: 4 to 103% All compounds, median RSD: 8%; 95th percentile RSD: 32% All compounds, laboratory median RSD: 7%; 95th percentile RSD: 25% (Target compounds: TCE, PCE, 1,2-dichloroethane, 1,2-dichloropropane, 1,1,2-trichloroethane, *trans*-1,3-dichloropropene)
PE sample accuracy	Target compounds: absolute percent difference range: 3 to 78% All compounds, Scentograph Plus II median APD: 10%; 95th percentile APD: 38% All compounds, laboratory median APD: 7%; 95th percentile APD: 24% (Target compounds same as those for sample precision)
Scentograph Plus II comparison with laboratory results for groundwater samples	Scentograph Plus II median RSD: 8% Laboratory median RSD: 6% Scentograph Plus II 95th percentile RSD: 87% Laboratory 95th percentile RSD: 14% Scentograph Plus II laboratory median APD: 12% 95th percentile APD: 194% Scentograph Plus II: laboratory correlation: SRS low conc. ($\leq$100 μg/L) r = 0.974 SRS high conc. (>100 μg/L) r = 0.907 MAFB low conc. ($\leq$100 μg/L) r = 0.959 MAFB high conc. (>100 μg/L) r = 0.997
Analytical versatility	PE samples: calibrated for 19 of 32 PE compounds (59%) One pair of coeluting compounds was reported (carbon tetrachloride and 1,2-dichloropropane). GW samples: The reference laboratory detected 68 compounds at concentration levels of 1 μg/L or greater in all groundwater samples. The Scentograph Plus II was calibrated to report 62 of these compounds. The Scentograph Plus II reported values for 35 of the 62 compounds.
Sample throughput	2 samples per hour
Support requirements	Tritium in the detector requires state permit or license
Operator requirements	Sample processing: field technician Data processing and review: experienced GC chemist
Total system weight	80 pounds
Portability	System is transportable
Total system cost	$35,000 (single column, single MAID/ECD detector)
Shipping requirements	Air freight, luggage check (no compressed gas via commercial flight) Recharge carrier gas cylinder requires drop shipment

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 analytical procedures used during the demonstration were consistent with written procedures submitted to the verification organization prior to the field demonstration. An instrument cost summary and an applications assessment is also provided.

Method Summary

The Scentograph Plus II GC (Figure 6-1) incorporates a purge-and-trap GC method for the analysis of VOCs in water. A room-temperature water sample is sparged with a volume of argon carrier gas and the entrained VOCs are transferred to an adsorbent trap. The vapors are subsequently thermally desorbed and injected onto the column of the Scentograph Plus II. For this demonstration, the instrument was configured with a single GC column, programmable temperature control, and a microargon ionization/electron capture detector with tritium as the ionization source. The detector was operated in the electron capture mode. Compounds eluting from the column capture free electrons in the detector and thereby reduce the detector's standing current. This current reduction is amplified and is used to generate the chromatogram. Compounds are identified by column retention time, and quantitation is achieved by using detector response intensity.

Figure 6-1. The Scentograph Plus II GC.

Equipment

The Scentograph Plus II system consists of three separate units: the Aquascan—a field-portable sample purge system (7 inches × 15 inches × 20 inches, 25 pounds); the Scentograph Plus II—field-portable gas chromatograph (6 inches × 21 inches × 20 inches, 48 pounds); and a laptop computer (2 inches × 12 inches × 10 inches, 7 pounds), as shown in Figure 6-1. At the demonstrations, the unit was used on the folded-down middle seat of a minivan. The three units are field-portable, but they are best suited for use in a vehicle and not outdoors at a wellhead. The sample purge and GC units are contained in steel cases suitable for shipping; however, they are too large to be hand carried during air travel.

Equipment weight includes an argon carrier gas cylinder inside the instrument. A small cylinder of compressed high-purity argon gas is used for periodic recharge of the internal cylinder. The system was powered by 110 V ac for this demonstration. It can also be powered by a dc-to-ac inverter connected to the vehicle's battery. Additional equipment used at the demonstration included 250-mL, screw-cap septa sample vials, standards mixtures, microliter syringes and needles, and tubing for sample transfer.

Sample Preparation and Handling

Samples were handled in the same manner at both sites: Chilled (~ 4 °C) water samples were supplied in 250-mL amber bottles with zero headspace. The sample was analyzed either diluted or undiluted, based upon the anticipated concentration level of the target compounds. Dilutions of either 1:5, 1:20, or 1:50 were obtained by pouring off a portion of a 250-mL commercial bottled water sample and making up the volume (via syringe transfer) with cold sample from the original 250-mL sample container. (Sentex field experience indicates that commercially available bottled water is very low in VOC content and is suitable as a low-cost, readily available diluent.) Following dilution, if required, an extraction tube was immersed in the sample bottle and a water sample withdrawn at a rate of 150 mL per minute for 60 seconds and flushed through a 10-mL sparge cell using a motorized impeller pump in the purge unit. An argon gas stream was bubbled through the 10-mL sample volume and the entrained vapors routed through tubing to a sorbent trap in the GC module. Following the adsorption cycle, the sorbent trap was heated and carrier gas flow through the trap was used to move the vapors onto the head of the column for separation and quantitation. All purge-and-trap and GC analysis functions were computer controlled and were initiated by the instrument operator with keyboard commands.

Consumables

An internal gas bottle contains argon carrier gas. The internal gas supply lasts for about 8 hours and then requires refilling. An external cylinder is used to periodically refill the internal cylinder. For long- term use, the system can be operated from an external carrier gas cylinder.

Historical Use

The Scentograph Plus II GC has been used for monitoring industrial wastewater, sewage treatment plant effluent, and groundwater. The GC module can also be used to monitor soil gas or air as well. The GC unit can be equipped with a gas sampling loop instead of a sorbent trap for gas analysis applications.

Equipment Cost

The Scentograph Plus II, as equipped for this demonstration, has a purchase price of about $35,000. This includes proprietary software, a laptop computer, and connection cables for data processing and instrument control.

Instrument costs are summarized in Table 6-1. Reference laboratory costs were $95 per sample plus Express Mail shipping, which was about $30 for a batch of 12 samples. The Scentograph Plus II throughput is 2 samples per hour.

Table 6-1. Scentograph Plus II Cost Summary

Instrument/Accessory	Cost
Instrument (Scentograph Plus II GC, field-portable Aquascan sample unit, laptop computer, software)	$35,000
Instrument accessories (field-portable printer)	$500
Sample handling accessories (carrier gas, syringes, vials, standards)	$25 per day
Maintenance costs	Not determined

Operators and Training
The Scentograph Plus II analysis team consisted of three persons: a chemical technician, who operated the instrument; a Ph. D. chemist, who assisted in instrument calibration and setup at each site; and an electronics technician, who carried out sample handling and dilution. Only one person is required to operate the instrument. With a half day of training, an experienced chemical technician could learn how to operate the system. A novice technician operator would require additional training. Experience and additional training in GC data processing are required to do method development and analysis of complex mixtures.

Data Processing and Output
The instrument uses proprietary software to control all run events (e.g., purge time, valve switching, temperature ramps, and acquisition time). The chromatograms are stored as text files for further analysis using spreadsheet software. A real-time, color-monitor display is available. It includes current analysis results, including compound identification, concentration, retention time, and a chromatogram superimposed on the latest calibration chromatogram.

Hardcopy data were not available immediately after a sample run. The system is operated under a Microsoft disk operating system that is not capable of multitasking. To retrieve hardcopy data, the analytical software must be shut down and the data output software started. Only analysis screen output or hardcopy output, but not both, were available at any given time. No hardcopy data were presented or available during the audit. Final data were provided to the verification organization in spreadsheet format on disk.

Compounds Detected
The system was calibrated for a total of 19 compounds at both sites. The calibrated compounds are listed in Table 5-1. The system is capable of detecting organic compounds with ionization potentials of 11.7 eV or less. The analytical method used at this demonstration resulted in one coeluting compound pair (carbon tetrachloride and 1,2-dichloroethane). The possibility of coeluting pairs requires that some information about sample content be available so that the methods can be adjusted to minimize or avoid compound coelution.

The microargon ionization/electron capture detector is susceptible to overloading, and thus care must be taken to appropriately dilute the samples to the proper range prior to analysis. Detector overload requires about an hour for complete column and detector re-equilibration, during which time samples cannot be run.

Initial and Daily Calibration

A three-point calibration was conducted daily prior to analysis of field samples. The software automatically chooses the best curve fit routine for calibration points and stores the information in a calibration subroutine. Choices for curve fitting are linear, second-degree, and third-degree equations. The latest calibrations are used by the analysis routine to identify and quantify unknown analytes during the chromatographic run. During the demonstration, the calibration was checked at the end of the day with a control standard solution. If the results from the control standard deviated significantly (>20%) from the three-point calibration curve, a recalibration at two points was initiated.

QC Procedures and Corrective Actions

Internal standards were not used. Blank sample analyses or corrective actions for false positives in blank samples were not specified in the field analysis procedure. An acceptable accuracy window of ±20% was used as an acceptance criterion for daily calibration check samples. Failure of a calibration check sample required a rerun of the calibration standards.

Sample Throughput

Gas chromatograph analysis time was about 30 minutes per sample. Additional time was required to review and process data. In many cases analytical results were available in real time; however, as noted earlier, hardcopy printout could not be obtained without exiting the analysis software. Preliminary handwritten hardcopy data were generally available the day following analysis. Sample throughput is on the order of two samples per hour. This includes periodic instrument calibration checks, sample reruns, and all data processing tasks. Samples with only one or two known contaminants could be processed faster.

Problems Observed During Audit

Some hardware problems were observed during the MAFB demonstration period. Toward the end of the week, problems were encountered with the computer-GC interface. The problem was eventually determined to be a faulty computer, and the computer was replaced. Problems were also encountered with the interface between the GC and the Aquascan (the unit containing the purge cell, pump, and valves). A spare Aquascan unit was deployed and the field sample analysis was completed. A complete calibration rerun was required following equipment changeout.

Data Availability and Changes

Preliminary results from each daily set of samples from the Scentograph Plus II were obtained on the following day in handwritten format. Data were provided on disk in spreadsheet format at the conclusion of each demonstration week. A final change to TCE and PCE values for seven samples was made following a data review by the vendor: The field analyst noted that incorrect information on groundwater sample dilution was entered by the Sentex team during the field analysis.

Applications Assessment

This demonstration was designed to assess 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 level 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 usually 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 that analyses be carried out 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 Scentograph Plus II is most applicable to routine monitoring situations where the sample composition is known and not particularly complex. The system could also be successfully used in sample screening situations where target contaminants are known. The instrument is able to detect most chlorinated VOCs at submicrogram-per-liter concentration levels; thus it is well suited for monitoring groundwater compounds at or near regulatory action levels. As with most GC systems, care must also be taken to avoid compound coelution, and manual data processing methods may be required when coeluting compounds are encountered in the analysis.

The observed precision and accuracy of the Scentograph Plus II is expected to be adequate for use of this instrument in routine monitoring or screening situations. When selecting a technology for use at a site, the analyst or site manager must take instrument performance into account, along with the project's data quality objectives.

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.

Comments on Demonstration Design

The field demonstration was a good learning experience for the participants. There was frustration over the extreme weather conditions, coupled with a few surprises and even instrument breakdown.

Suggestions for future demonstrations:

1. A specific list of target analytes should be made available. Existing laboratory methodologies give a list of target analytes.
2. A method detection limit must be established for similar technologies. If the MDL cannot be established, at least a prescribed practical quantitation limit must be determined.
3. An initial accuracy study must be conducted at a specific concentration level instead of determining accuracy at different concentration levels.
4. Accuracy limits must be established by analyzing spiked samples at a given concentration and frequency.
5. An initial precision study must be conducted at a specific concentration instead of determining precision at different concentration levels.
6. Precision limits must be established by determining the relative percent standard deviations of the percent recovery of the spiked samples.

If a field protocol is established that is similar to the off-site laboratory protocol, fewer PE samples will be required and more groundwater samples can be analyzed. This will more accurately reflect the throughput capability of the GC.

The results of groundwater analysis from both sites should be combined and presented in a graph that plots the reference laboratory data against the field data.

Additional Comments on Gas Chromatograph Performance

The drawbacks of using a field-portable gas chromatograph are the need to resolve early coeluting peaks and extended analysis time when late-eluting peaks are present. This is primarily the case for isothermal runs.

Although temperature ramping is available, the absence of a cooling fan makes temperature-ramped runs impractical. The alternative to temperature ramping is the use of multiple columns. A Sentex field-portable GC system has been developed that uses two dissimilar columns sharing the same detector and operated at a constant temperature. A sample is taken and separation occurs in the primary column. The primary column is usually a short, packed column that will separate early-eluting compounds (e.g., the gases, dichloroethenes, and dichloroethanes). After elution of the early-eluting compounds, the run is stopped and the system is backflushed to eliminate late-eluting compounds that have been introduced in the column together with early-eluting compounds. Another sample is taken at the end of the backflush and a second column is used to separate the late-eluting compounds (e.g., xylenes and dichlorobenzenes). The second column is usually a 30-m capillary column.

Chapter 8
Previous Deployments

ACME Project (1993) New Jersey

The ACME project was conducted under the auspices of the New Jersey Department of Environmental Cleanup. The investigation included approximately 800 soil borings, along with collection and on-site analyses and quantitation of approximately 800 soil samples with the Scentograph Plus II. The objective of the on-site analytical component of the investigation was to characterize petroleum contamination in soil for use in developing a comprehensive cleanup plan. The analytes of interest were the purgeable aromatic hydrocarbons (benzene, toluene, ethyl benzene, and xylenes).

DOE Remediation Site (1995), Pinellas Plant, Florida

The Aquascan was evaluated as an in-field, automated monitoring system for contaminated groundwater at an active DOE remediation site in Pinnelas, Florida. The instrument measured concentrations of methylene chloride, trichloroethene, and toluene in the parts per million level in the groundwater. Reported values for the three compounds were within 20% of reference laboratory values.

Superfund Site (1996), North Carolina

A Sentex field-portable gas chromatograph was used to define a shallow and top-of-rock groundwater contamination plume at a site in North Carolina. The analytes of interest were tetrachloroethene, trichloroethene, and *cis*-1,2-dichloroethene.

Toluene Monitoring (1996 – Present), New Jersey

A rack-type Aquascan has been installed at an industrial facility to monitor toluene levels in discharge water. It operates in a continuous mode and sets off an alarm when the toluene level exceeds the set threshold level.

Trihalomethane Monitoring in Drinking Water (1997 – Present), Italy

A rack-type Aquascan was installed to monitor trihalomethanes in the drinking water supply.

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