

**A Dynamic Site Investigation
Adaptive Sampling and Analysis Program
for Operable Unit 1 at Hanscom Air Force Base
Bedford, Massachusetts**

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Abstract

In this study, field analytical instrumentation and methods were used to support a risk assessment study at the airfield at Hanscom Air Force Base (HAFB, Bedford, Massachusetts). The site investigation is part of an ongoing effort to determine contaminant risk to ground water from soil. The field tools supported a dynamic workplan/adaptive sampling and analysis demonstration. Over a two-week period, 601 soil samples were screened (30-sec/sample) for volatile organic compounds by direct measuring thermal desorption gas chromatography/mass spectrometry (TDGC/MS). From these results, 158 soil samples were selected for quantitative analysis by purge and trap GC/MS. In addition, quantitative analysis of 68 soil samples by TDGC/MS was made for polychlorinated biphenyls and polycyclic aromatic hydrocarbons. Quantitative analysis times were 10-min/sample for PCBs and PAHs and 15-min/sample for VOCs. A field-practical microwave digestion procedure and an inductively coupled plasma/optical emission spectrometry method were used to analyze 121 samples for metals. Results of the dynamic site investigation and field method performance are presented. Finally, HAFB staff modified the ground water collection system into the treatment plant increasing the influent concentration. This project was funded through the U.S. Environmental Protection Agency and the Environmental Technology Initiative.

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1.0 Introduction - Environmental Technology Initiative

In 1995, the U.S. Environmental Protection Agency (EPA) issued a Request for Proposals in support of President Clinton's efforts to promote innovative environmental technologies and to address the many factors that might pose barriers toward their commercialization. The President's Environmental Technology Initiative (ETI) is aimed at accelerating environmental protection, strengthening America's industrial base, and increasing exports of U.S. technologies and expertise. Through a cooperative agreement with EPA Region I and supported by ETI funding, Tufts University's Center for Field Analytical Studies and Technology (CFAST) addressed two key objectives identified in the FY95 strategic plan; namely, strengthening the capacity of technology developers and users to succeed in environmental innovation *and* strategically investing EPA funds in the development and commercialization of promising new environmental monitoring, control, and remediation technologies.

The U.S. Environmental Protection Agency *and* the Departments of Defense (DoD) and Energy (DoE) have sponsored programs to support research and to validate field analytical technologies.^{1,2} Despite EPA's efforts to encourage the use of field analytics,³ they have not played a significant role in either hazardous waste site investigations or the verification of hazardous waste site cleanup programs. The ETI project, in part, supported an ongoing soil investigation at Hanscom Air Force Base (HAFB, Bedford, MA). HAFB is in the process of conducting Human Health and Ecological Risk Assessments and Feasibility Studies for the airfield. The core technical team included staff from HAFB, EPA Region I, their respective contractors, CH2MHill and Camp Dresser & McKee (CDM), the Massachusetts Department of Environmental Protection (MA DEP) *and* Tufts University. The team developed and carried out a dynamic site investigation at Operable Unit 1. Dynamic site investigations depend on an adaptive sampling and analysis plan. The objective was to demonstrate the ability of field analytics to produce data of quality to support risk assessments. The HAFB investigation relied on data produced in the field to make decisions as to the location of samples collected and the types of analysis performed. Field instruments and methods were developed by the principal investigator (PI) in cooperation with several analytical instrument companies. The premise being that if analytical data can be produced in the field with known quality to support risk assessments then the perceived *and/or* institutional barriers impeding their usage should be greatly reduced.

With these objectives in mind, the technical team collaborated to produce an eighteen minute videotape illustrating the dynamic site investigation process.⁴ To complement the videotape, a dynamic workplan guideline was produced.⁵ The dynamic workplan provides a framework for changes in direction based on what is learned in the field during the site investigation or cleanup verification process. The guideline illustrates the many factors that should be considered when incorporating field analytical instrumentation and methods into an adaptive sampling and analysis program. The videotape and guideline are aimed at helping federal and state regulators, siteowners and their consulting engineers, and remediation companies understand what is involved in developing and carrying out a dynamic site investigation or cleanup verification program where the decisions made rely on field data. When compared with the traditional site characterization process, dynamic workplan/adaptive sampling and analysis projects should result in faster, better, and hopefully cheaper site investigations and cleanup.

Results of the HAFB soil investigation and the performance of the field instruments are presented. Direct measuring thermal desorption gas chromatography/mass spectrometry (TDGC/MS) was used to screen 601 soil samples for volatile organic compounds (VOCs). Based on these findings, 158 soil samples were quantitatively analyzed for VOCs by purge and trap GC/MS. Analysis times were 30-sec/sample and 15-min/sample for TDGC/MS screening and quantitative purge and trap GC/MS measurements, respectively.

Soil samples were selected for polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) analysis based on site history, the presence of petroleum contaminated soils observed in the field, and VOC screening results. Quantitative PCB and PAH analyses were made for 70 soil samples by TDGC/MS. Simultaneous analysis of these contaminants was accomplished in 10-minutes per sample. On-site analysis of 121 soil samples for the EPA target analyte list metals was made by a field-practical inductively coupled plasma/optical emission spectrometry (ICP/OES) method. Site contamination maps were produced to facilitate the on-site decision making process. The adaptive sampling and analysis program was completed in ten days. The data produced will be used in risk assessments to determine the need for future vadose zone soil actions. HAFB has already modified the ground water collection system. VOC influent concentrations into the treatment plant have increased from 500-ppb (August 1996) to 900-ppb (August 1997).

1.1 Hazardous Waste Site Investigation and Cleanup Costs

The EPA estimates that the cost for hazardous waste site cleanups will exceed \$300 billion over the next 10 years.⁶ In another study, Russel and coworkers⁷ project cleanup costs between \$480 billion and \$1 trillion over the next 30 years. These estimates exclude administration and transaction fees. Since 1980 the cost for Superfund alone has exceeded \$26 billion for both government and industry. More than 100 sites are now considered “cleaned” by the EPA, with another 275 sites expected to be in remedial design and construction by the year 2005.⁸ The U.S. General Accounting Office estimates that \$32 billion will be spent on these sites in long-term operation and maintenance monitoring costs through fiscal year 2040. Yet, another estimate suggests total remediation costs will range between \$44 billion (1,350 sites) to as high as \$138 billion (3,000 sites) for private non-Federal facility Superfund sites through the year 2020.⁹

The following questions can be posed: Do inadequate site investigations and, therefore, a lack of understanding with respect to the chemical and physical dynamics affecting the cleanup contribute to the costs? Can field-based analytical instrumentation and methods give on-site project engineers the kind of data needed that will lead to faster, better, and cheaper cleanups? Hanscom Air Force Base is an example where traditional site investigations have led to the construction of a ground water collection and treatment facility. Over the past 5-years, VOC concentrations into the plant have remained the same. These results have suggested the need for another field investigation aimed at determining whether plant operating conditions can be further optimized.

1.2 Hanscom Air Force Base Background

HAFB is in the towns of Bedford, Concord, Lexington, and Lincoln, Massachusetts. HAFB is approximately fourteen miles northwest of downtown Boston. From 1941 to 1973 HAFB's primary mission was the support of fighter aircraft operations and maintenance and the support of Air Force Research and Development (R&D). Thereafter, HAFB no longer provided fighter aircraft maintenance and began to support Air Force Command, Control, Communications, and Intelligence activities and R&D. Massachusetts obtained control of the airfield in 1974 and renamed it the L.G. Hanscom Field. The airfield is currently operated by the Massachusetts Port Authority as a civilian airport. Except for the airfield, the remainder of the base was retained by the Air Force.

HAFB and L.G. Hanscom Field were added to the National Priorities List in 1994. More than \$25 million has been spent on traditional hazardous waste site investigations and cleanups at Hanscom. Hazardous waste site investigations for Operational Unit 1 (Sites 1, 2, and 3) began at Hanscom Field in 1982, see Figure 1. Site 1 was used as a fire training area where waste oils, flammables, aircraft wreckage and fuselages were burned. Sites 2 and 3 are where fifty gallon drums containing waste solvents, fuels, and paints were buried. All visibly contaminated soils and drums were removed from these sites in 1987 and 1988.

In 1991, integrated ground water collection, recharge, and treatment systems were put into operation to remediate the three sites and to contain the plumes of contaminated ground water within the airfield. The ground water collection and recharge system and the 200-gal/min VOC treatment plant were built at a cost of \$6 million. Initial ground water influent concentrations were 10,000-ppb total VOCs. After six months of operation total VOC levels were 500-ppb. From late 1990 through July 1996 influent concentrations have remained constant, while yearly treatment facility operation and maintenance costs have increased to \$600,000/year.¹⁰

1.3 Traditional versus Dynamic Workplans

The ability to rapidly assess the disposition of environmental contaminants at purported or existing hazardous waste sites are an essential component of the nation's environmental restoration program. Each site, whether owned by the public or private sector, must be evaluated to decide whether risk to human health or the environment exists. If the data obtained supports the notion that either no risk or an acceptable level of risk exists for the intended land usage, then no further action may be required. If, on the other hand, sufficient risk has been determined to warrant a full site characterization, the site investigation effort must delineate the nature, extent, direction, concentration and rate of movement of the contamination along with the physical and chemical site attributes.

Sampling and analysis programs are a significant part of the environmental restoration process. From the initial site investigation on through to the design and completion of remedial actions, sampling and analysis programs play a key role. By design, traditional sampling programs collect soil or ground water samples based on pre-specified grid patterns in an attempt to maximize data while reducing chemical analysis costs, see Figure 2. They rely on

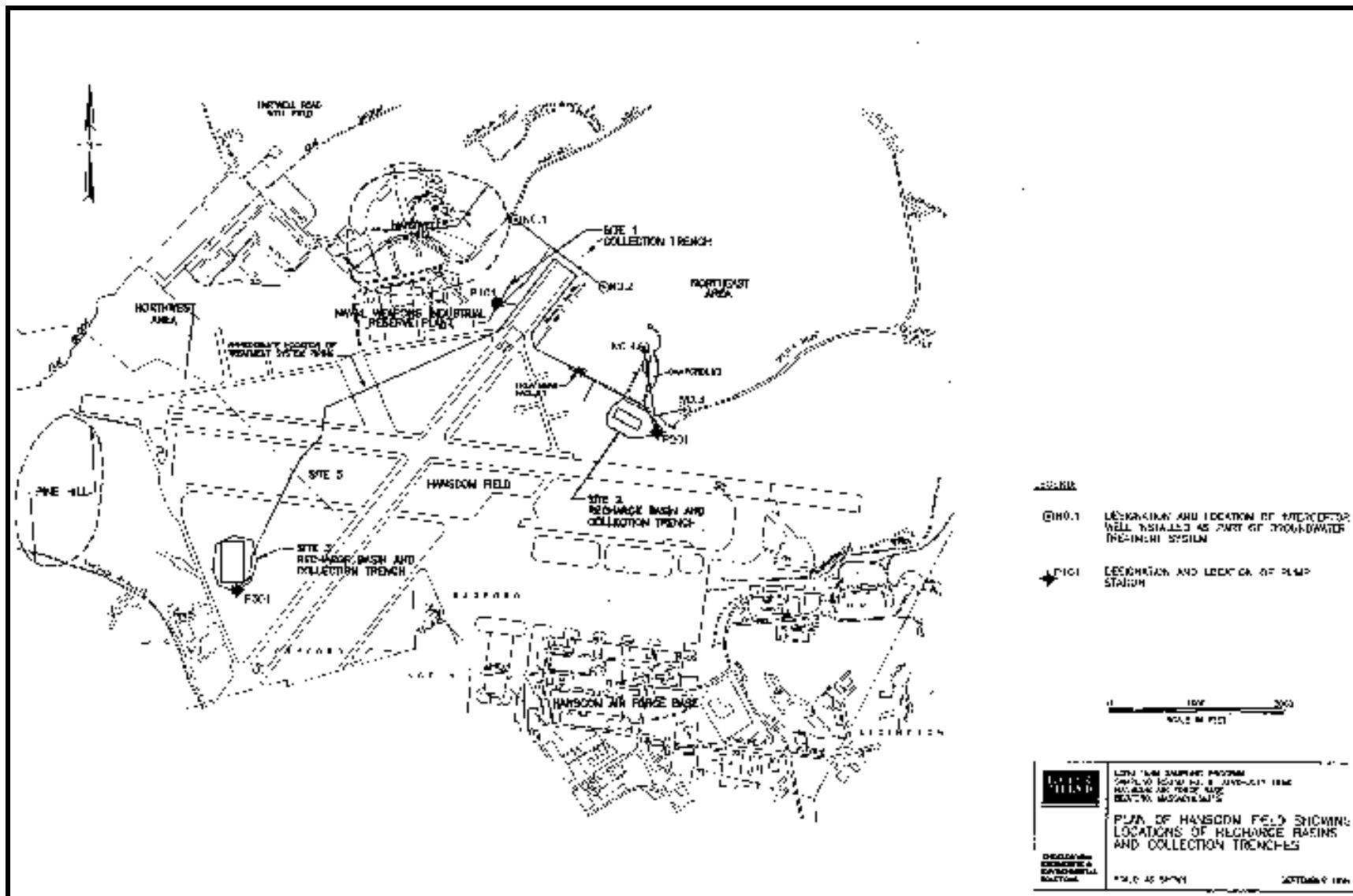


Figure 1. Hanscom Field

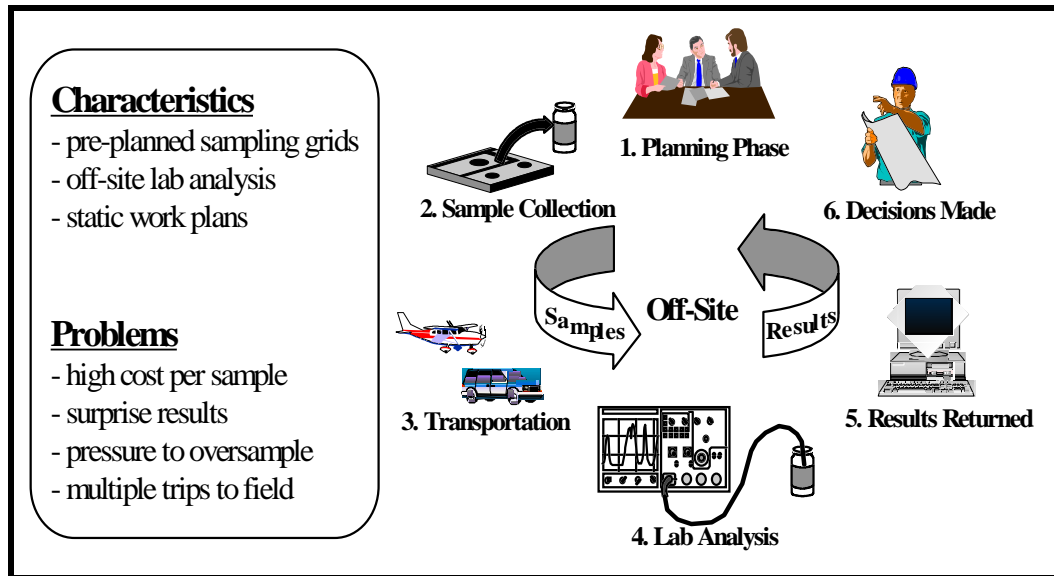


Figure 2. Traditional Site Investigation

predetermined specifications for the location and number of samples to be collected and the type of analyses to be conducted. Traditional site investigations are generally based on a phased engineering approach, which does not provide the framework for obtaining analytical data in the field nor for making changes in direction while in the field. Samples are collected, packaged and typically sent off-site for analysis. Because data turnaround times can range from a few days to several weeks, data “surprises” or concerns must be addressed in subsequent field studies. Each successive investigation continues to add cost to the overall restoration effort. Improvements in the way sampling and analysis programs are designed and executed should lead to faster, better, and more cost-effective site assessments and cleanups.

In contrast, adaptive sampling and analysis programs are based on a dynamic workplan where the program itself relies on field analytical instrumentation and methods to generate near real-time information on the nature, extent, direction, concentration and rate of movement of the contamination present at the site. Figure 3 illustrates the dynamic nature of this approach. Rather than dictate the details of the sample analyses to be performed, the numbers of samples to be collected, and the location of each sample; dynamic workplans specify the decision making logic that will be used in the field to determine what analyses will be performed, where the samples will be collected, and when sampling stops. Adaptive sampling and analysis programs change or adapt based on the analytical results produced in the field.

Successful hazardous waste site investigations should be focused with goals and objectives clearly defined. A dynamic workplan provides an alternative to the traditional approach. It relies, in part, on an adaptive sampling and analysis strategy.¹¹ An adaptive sampling and analysis program requires analytical methods and instrumentation that are field-practical and can produce data fast enough to support the dynamic workplan process. Past studies have shown that the dynamic site investigation process employing field analytics can result in significant time and cost savings.^{12,13} A properly developed dynamic workplan includes the following six steps summarized below:⁵

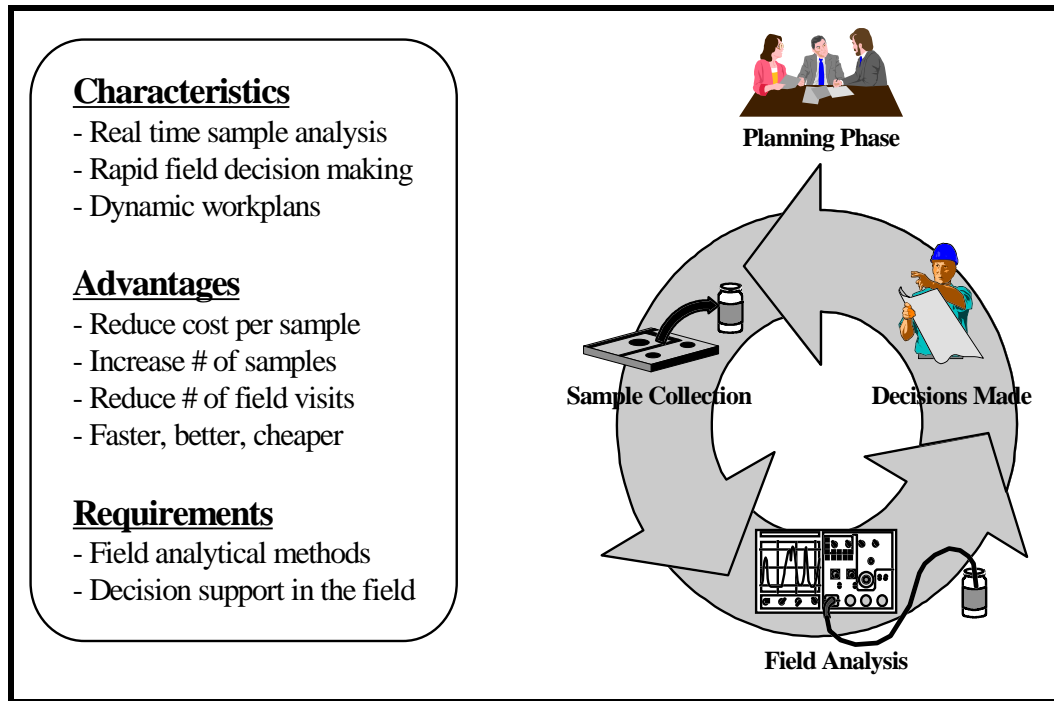


Figure 3. Adaptive Sampling and Analysis Program

- Step 1: Select the core technical team whose responsibility it will be to prepare and carry out the dynamic workplan. The core technical team must understand the scientific and engineering questions under investigation and the quality of data needed to answer these questions. One member of the team should have the authority to make field decisions.
- Step 2: Develop the Initial Conceptual Model and Decision Making Framework. The model contains the best-available information at the start of the project and evolves as field data is produced. It depicts three-dimensional site profiles based on vadose zone and ground water flow systems that can exert influence on contaminant movement. The model is based on site-specific Data Quality Objectives (DQO's). DQO's ensure that the type, quantity, and quality of field data used in decision making are appropriate for the intended application.
- Step 3: Develop Standard Operating Procedures. SOPs for sample collection and analysis should be produced by the core technical team and approved by the appropriate regulatory body before initiating field activities. The field methods should be "performance based" and provide data of sufficient quality to achieve site-specific DQO's, with sample analysis rates that can support the dynamic site investigation process.
- Step 4: Develop Data Management Plan. The ability to manage and easily use all of the data (chemical, physical, geological, hydrological) produced in the field is critical to the success of the dynamic process. Data integration, sampling, and analysis protocols should be incorporated into an overall data management plan.

Step 5: Develop Quality Assurance Project Plan. Quality assurance/quality control (QA/QC) defines the responsibility of the technical team and regulators. It describes the procedures to be used to monitor conformance with, or documentation and justification of departure from the SOPs.

Step 6: Prepare Health and Safety Plan. A health and safety plan is produced with DQO's established for the field analytical tools used to monitor worker and community safety.

2.0 Site Investigation Purpose and Objectives

HAFB is completing its Human Health and Ecological Risk Assessments and Feasibility Study for Operable Unit 1. The effectiveness of the 1987/88 drum and soil removal actions and five-year operation of the ground water collection, recharge and treatment systems must be assessed. The core technical team's primary objective was to show that the field analytical instrumentation and methods can support a dynamic workplan/adaptive sampling and analysis program. The quality of data produced in the field will support its use in the risk analysis *and* in assessing the effectiveness of past removal actions. Another goal was to videotape the development of the workplan and execution of the site investigation. A third goal was to produce a dynamic workplan/field analytics guideline. The videotape and guideline should provide a framework for making decisions in the field and assist in technology transfer. Finally, interviews were conducted with technology developers and manufacturers, field instrumentation purchasers, and data users to identify barriers that pose impediments to the use of field analytics.¹⁴

3.0 Methodology

When the ETI proposal was submitted for funding, several facilities were identified as potential locations for the dynamic site investigation program. The premise being that an ideal site is one in which the soil investigation program was in the planning stage. This would allow ETI funds to be used to demonstrate field analytics and the dynamic investigation process. At the time ETI funding was received, HAFB was the best available site in Region I.

Just before mobilization the core technical team held its final field investigation planning session at Tufts University. Details of the site investigation objectives, sample collection process, field analyses to be performed, and the framework for making decisions in the field were finalized.¹⁵ The planning session was videotaped, with staff from each organization immediately interviewed after the meeting to gain perspective. Creative Video (Medfield, MA) developed the interview questions and videotape format with the assistance of the PI. HAFB staff assumed primary responsibility for directing the sample collection effort. When questions were raised concerning measured contaminant concentrations at the action level, EPA provided guidance to determine whether additional sampling was required. The work performed was conducted under an EPA approved workplan. The Agency conducted laboratory audits, reviewed SOP's and method detection limit (MDL) studies, *and* verified the data. Staff from Tufts and CH2Mhill prepared chain-of-custody forms and logged information about the samples. Tufts prepared

samples for field and off-site laboratory analysis, while CH2MHill was responsible for shipping samples to the off-site laboratory. Field analysis for organics and metals was provided by Tufts, while Spectrum Analytical (Agawam, MA) performed the off-site laboratory analysis. Soil samples were collected in 4-ft plastic tubes using a Geoprobe™ operated by Kestral (Agawam, MA). Project milestones including site selection, dynamic workplan preparation, field mobilization and investigation concomitant with videotape production, data and final report submission are shown in Appendix II.

3.1 Adaptive Sampling and Analysis Strategy

The site-specific action levels (i.e. 20DAF), quantitation limits (QL), and method detection limits (MDL) for the compounds of interest are shown in Table 1. 20DAF is derived from EPA's Soil Screening Levels (SSL)¹⁶ and was established as the action level for determining risk to ground water. 20DAF is the dilution-attenuation factor (DAF) of 20, which takes into account the natural attenuation processes for the soil to ground water migration pathway. For Operable Unit 1, one-half the action level (i.e. $\frac{1}{2} \times 20\text{DAF} = 10\text{DAF}$) was established as the measurement quantitation limit to insure that site-specific action concentrations were achieved. For convenience, the 10DAF values were used to produce the site maps.

Adaptive sampling and analysis programs require that on-site chemical analyses be fast enough to support the sample collection and decision making process. For Sites 1, 2, and 3, VOCs, PCBs, PAHs, cadmium, and lead were the contaminants of concern. Field instruments, their corresponding rate of analysis, SW 846 reference method, and analysis type are shown below for each field method used in this investigation.

<u>Field Methods</u>	<u>Sample Introduction and Instruments</u>	<u>Analysis Type</u>	<u>Sample Analysis Rates</u>
Metals Modified SW 846 Method 6010	Hildebrand nebulizer Leeman ICP/OES	Quantitative	8-min
PCBs and PAHs Modified SW 846 Method 8270B	Tufts TD & Hewlett Packard GC/MS	Quantitative	10-min
VOCs Modified SW 846 Method 8260A	Tekmar purge and trap & Hewlett Packard GC/MS	Quantitative	15-min
VOCs Tufts method	Bruker TD & Bruker GC/MS	Screening	< 30-sec

Table 1. Site-specific Action Levels, Quantitation Limits, and Method Detection Limits

Compound	Action Level	QL	MDL (mg/kg)
	20DAF (mg/kg)	10DAF (mg/kg)	
Acenaphthene	570	285	0.1
Acenaphthylene	570	285	0.1
¹ Anthracene	12,000	6,000	0.2
Benzene	0.03	0.015	0.003
² Benz(a)anthracene	2	1	0.2
³ Benzo(b)fluoranthene	5	2.5	0.3
³ Benzo(k)fluoranthene	490	245	0.3
Benzo(a)pyrene	8	4	0.1
Cadmium	8	4	0.11
Carbon Tetrachloride	0.07	0.035	0.004
Chlorobenzene	1	0.5	0.008
Chloroform	0.6	0.3	0.008
² Chrysene	1600	800	0.2
Dibenz(a,h)anthracene	2	1	0.2
1,1-Dichloroethane	23	11.5	0.006
1,2-Dichloroethane	0.02	0.01	0.013
1,1-Dichloroethene	0.06	0.03	0.003
cis-1,2-Dichloroethene	0.4	0.2	0.005
trans-1,2-Dichloroethene	0.7	0.35	0.006
Ethylbenzene	13	6.5	0.006
Fluoranthene	43,000	21,500	0.1
Indeno(1,2,3-cd)pyrene	14	7	0.2
Lead	400	200	1.65
Naphthalene	84	42	0.4
¹ Phenanthrene	NA	280	0.2
Pyrene	42,000	21,000	0.1
Total PCBs	NA	0.5	0.2
Styrene	4	2	0.006
Tetrachloroethene	0.06	0.03	0.006
Toluene	12	6	0.010
1,1,1-Trichloroethane	2	1	0.008
Vinyl Chloride	0.01	0.005	0.033
⁴ m-Xylene	210	105	0.016
o-Xylene	190	95	0.003
⁴ p-Xylene	200	100	0.016

Notes: Organics with the same superscript co-elute. EPA has not established a 20DAF for total PCBs, therefore, the site-specific quantitation limit was set as 0.5-mg/kg. No 20DAF concentration was available for lead. One-half of the screening level of 400-mg/kg for ingestion was used based on the Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities (U.S. EPA 1994).

Geoprobe™ push technology was used to collect subsurface soils in 4-ft plastic sleeves. VOC screening analysis was made at 1-ft intervals by making a small incision along the sleeve, with the TDGC/MS sampling probe head placed immediately over the hole. Volatile vapors present in the soil were instantaneously detected by the MS, which was operated in the continuous direct measuring mode. The Bruker TDGC/MS simultaneously monitored eleven of the eighteen targeted VOCs. To insure compound identity, three ions per compound were monitored. Soil samples were collected by rounds at each site as follows:

- Round 1- Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water at the center of each fire training and drum burial pit.
- Round 2 - Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water outside the fire training and drum burial pits. Soil samples were also collected near the upper aquifer collection trenches to assess contamination migration toward the trenches.
- Round 3 - Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water further out from the conceptualized fire training and drum burial pit boundary whenever GC/MS screening and quantitative data from Round 2 produced detectable levels above the 10DAF quantitation limit. Note that the site-specific action level for all three sites was set at 20DAF.

Rounds 1 and 2 were prespecified in the dynamic workplan and collected as a group from each site. Both screening and quantitative data dictated whether additional samples from Rounds 1, 2, or 3 required further analysis, for example:

- If GC/MS screening results indicated non-detectable VOC levels within the 4-ft sleeve for all sleeves from a particular boring, a soil sample for quantitative GC/MS analysis was selected from the 2-ft section of the sleeve nearest to ground water.
- If only one 4-ft sleeve from a boring produced screening level concentration at detectable levels, a soil sample was selected for quantitative analysis from the 2-ft section of the sleeve within the area of highest concentration. An additional soil sample was selected for quantitative analysis from the 2-ft section of the sleeve nearest ground water whenever the sample selected by screening was not from the sleeve nearest the ground water level.
- If target compounds were present in multiple 4-ft sleeves within the same boring above ground water, a soil sample was selected for quantitative analysis from the 2-ft section of the sleeve shown to be the area of highest concentration. Additional soil samples were selected for quantitative

analysis from the 2-ft section of the sleeve nearest ground water whenever the sample selected by screening was not from the sleeve nearest the ground water level. Supplementary samples were selected for quantitative analysis to determine extent of contamination from these boring locations.

3.2 Field Instruments, Sample Preparation and Analysis Procedures

All soil samples were prepared and analyzed in the field. Detailed descriptions of the sample preparation methods, analyte quantitation procedures, and quality control and assurance criteria can be found in the VOC, semi-VOC, and metals standard operating procedures (SOPs), see Appendix III. Data reduction for quantitative VOC and PCB/PAH GC/MS analysis was accomplished in the data management trailer. Off-line data analysis maximized the sample throughput rates of the quantitative methods. Screening results were instantaneous, with instrument response visible on the monitor and recorded manually. Data reduction for metals was made in the metals trailer using the Leeman Laboratory data analysis software. Several software mapping programs were proposed as part of the ETI project. Once enough organics and metals data were generated, site maps were produced using SitePlanner™ (Consolve, MA) to visualize site contamination profiles. Daily inspection of the maps (versus tables of data) facilitated the on-site decision making process. Plume™ (a geostatistical sampling tool) was not used in the investigation. Site dimensions at each site in Operable Unit 1 were too small to use the program.

3.2.1 Organics Analysis

A Bruker Instruments (Billerica, MA) GC/MS was used to provide direct measuring screening data of VOCs in soil. Each 4-ft sleeve was marked at 1-ft intervals along the length of the tube as described earlier. A hole was cut at the center of each 1-ft section. The 3-ft TD sampling probe head was held directly over the hole and the signal response for each target analyte recorded.¹⁷ If no response was noted after 1-min the section was considered blank. This procedure was repeated for each 1-ft interval of the sleeve, where possible. The data generated using this screening method was used to determine which, if any, 2-ft section was to be sampled for quantitative GC/MS analysis.

Two different VOC purge and trap devices were used in the ETI study. The Tekmar 3000 sample introduction system was used with a Hewlett Packard (HP, Palo Alto, CA) GC/MS to provide quantitative analysis of all soils in the HAFB soil screening investigation/adaptive sampling and analysis program. The quantitative VOC measurements were used to confirm the screening data, while both screening and quantitative data were used to delineate the extent of contamination. The Tufts VOC purge and trap/thermal desorption (TD) GC sample introduction system, designed as a lower cost alternative (\$5,000) to the Tekmar (\$15,000) for field application, was initially intended to be used in the field investigation. Because of carrier gas line leakage and the start of the sampling program, the Tekmar sample introduction system became the primary means of producing VOC data. Purge and trap GC/MS is the standard laboratory method for quantitative VOC analysis. Sample introduction by Tekmar provided a

common baseline when comparisons were made between the HP GC/MS data analysis software (EnviroQuant) and the mathematical algorithms (Ion Fingerprint Detection™ software) developed at Tufts. The HP model 5890 II/5972 GC/MS was used with the Tekmar. The HP GC/MS model GCD was used with the Tufts purge and trap system. The field method for VOCs was modified from the EPA standardized SW 846 method 8260A, see Appendix III.

Unlike the Bruker TD, the Tufts thermal desorber was not used to measure organics from soil directly. For PCB (by homologs) and PAH (compound-specific) analysis, 2-g soil was extracted with 2-ml methylene chloride, with known aliquots (50- μ l) of the extract injected into a disposable glass sleeve in the TD. The thermal desorber was installed into the GC injection port of the HP GC/MS model GCD. Standard syringe injection techniques (typically 1 to 2- μ l) cannot provide the detection limits achieved by the TD without preconcentrating the soil extract. Extensive sample cleanup and/or fractionation may also be needed depending on the complexity of matrix interferences. PCBs and PAHs were measured in the same 10-min analysis. The PCB/PAH field method was modified from previously published studies using the Bruker TDGC/MS^{18,19,20,21} and from reference method SW 846, 8270B, see Appendix III. In this project, HP's mass spectrometry detection system and data acquisition software were used to provide quantitative data.

All data analysis software comparisons were made using the same MS data files, with either Tufts' IFD or HP's EnviroQuant software. All computer systems were linked to a central data processing work station in the data management trailer through a local area network. All of the GC/MS data was backed up on removable storage media (Iomega Zip100 drives).

3.2.2 Metals Analysis

A Leeman Laboratory (Lowell, MA) model PS-1000 ICP/OES was modified to provide on-site analysis of metals. Typical ICP/OES instruments require highly temperature controlled laboratory environments, ± 5 °C, or optical components drift quickly out of calibration. This temperature requirement is costly to achieve in field laboratories especially during the extreme summer and winter months. The Leeman ICP/OES is a scanning sequential spectrometer. Modifications were made by pneumatically locking in place the optical system's movable components including the optical plate and Photo Multiplier (PMT) sled. The chassis was also ruggedized to increase instrument stability during transport and field operation. These design changes from traditional laboratory instruments to a more field-practical instrument were made by the Leeman and Tufts staff. We also modified the standard ICP/OES spray chamber with a Hildebrand grid nebulizer. This system can handle samples whose total dissolved solids content is greater than 1,000-ppm and digestate acid concentrations greater than 25% by volume. The spectrometer and Argon plasma torch were powered in the field by two different electrical services, 110-V 20-Amp and 240-V 30-Amp. The field ICP/OES instrument has been used at site investigation projects conducted with the Departments of Defense²² and Energy.²³

The ICP/OES was calibrated by a standard ratio method between the signals from blank and standard solutions. A laboratory control standard, prepared from an ERA Waste WatR™ solution, was diluted to approximately the instrument limit of quantitation. Analysis of this standard was used to verify instrument response. Instrumental QA/QC solutions consisted of

calibration blanks, calibration verification check standards, interferant A/B solutions, and laboratory control solutions. Instrument standards were purchased from Leeman, laboratory control standards from ERA (Arvada, CO) and Plasma-Chem (Farmingdale, NJ). Calibration responses were made for each metal and then verified with check standards. After the analysis of ten samples, instrument stability was checked by analyzing a check standard and performing a continuing calibration verification check with the concentration falling within a $\pm 20\%$ range. Calibration blanks were run before calibration verification standards. Interferant solutions A and B were run before site samples were analyzed. Details of the Tufts microwave digestion sample preparation procedure and ICP/OES analysis can be found in Appendix III.

Metals data was also produced from an energy dispersive x-ray fluorescence (EDXRF) instrument. The Spectrace QuanX differs from other field XRF instruments by employing an x-ray tube as the source rather than multiple radionuclides such as ^{55}Fe , ^{109}Cd , and ^{241}Am . It is tabletop in size and employs a thermoelectrically cooled Li drifted silicon semiconductor detector as opposed to liquid nitrogen cooled detectors typically found in laboratory instruments. This instrument provides increased sensitivity over radionuclide source instruments and has the same sensitivity as the much larger x-ray tube-based XRF's.

Soil samples were placed into a 32-mm mylar windowed sample cell. Samples were analyzed for 120 livetime seconds each for lead and cadmium. Fundamental parameter calculations were made to determine lead and cadmium concentrations in addition to producing the Pb ($L\beta$) and Cd ($K\alpha$) emission signal versus concentration plots based on standard reference materials (SRM) 2704, 2709, and 2710 obtained from National Institute of Standards and Testing. HAFB soil samples were analyzed with the field XRF after the completion of the soil screening investigation. The data from this instrument was produced as part of the ETI technology transfer project to determine instrument performance, see SOP in Appendix III for method details.

3.2.3 Quality Assurance/Quality Control Procedures

As was established in the dynamic workplan/adaptive sampling and analysis strategy, the HAFB site-specific data quality objectives were established to provide data of sufficient quality to support a risk analysis and the effectiveness of prior removal actions. Tables 2 and 3 list the data quality objectives for quantitative analysis of organics and metals.

3.2.4 Qualitative Identification and Quantitation Methods

Organics The Bruker mass spectrometer was operated in the selected ion monitoring mode to provide rapid detection of VOCs. Compound identity was made when the selected ions (three per compound) were normalized to 100% at the peak maxima and on either side at the half-peak maxima on three consecutive scans through the chromatographic peak. Signal amount, i.e., the logarithmic value of the SIM signal, was recorded when the above criteria was met. Concentrations were not calculated, relative signal response was used to guide sample location selection and the selection of samples analyzed quantitatively in the field.

Table 2. HAFB Data Quality Objectives for Quantitative Analysis of Organics

<u>Initial Calibration:</u> 5-point calibration	<u>Requirements</u> ave RF %RSD +/- 30% for 2/3 and +/- 40% for remaining 1/3 target compounds	<u>Corrective Action</u> check standards, recalibrate, check instrument
<u>Continuing RF Calibration:</u> begin and end of day	+/- 30% difference between ave RF and daily RF (CCV) for 2/3 and +/- 40% difference for remaining 1/3 of target compounds	check standards, recalibrate, check instrument
<u>Method Blank:</u> beginning and end of day or after analysis of highly contaminated sample	No more than 4 target compounds, concentration < 3 times QL	use new source of deionized water
<u>Measurement Precision:</u> duplicate or triplicates every 20 th sample	concentrations > 5 x QL: RPD < 60% concentrations < 5 x QL: RPD < 100%	review lab QC to determine whether in control, if out of control flag data
<u>Measurement Accuracy:</u> 1) surrogate fortified samples 2) field versus laboratory comparison	1) between 30% and 200% 2) concentrations > 5 x QL: RPD < 60% concentrations < 5 x QL: RPD < 100%	1) flag data 2) not applicable

The HP mass spectrometers were operated in the total ion current (TIC) mode for both VOC and semivolatile analyses. The software (IFD) extracted selected ions and their intensity for each compound. It utilized a set of mathematical algorithms and predetermined retention windows to identify and quantify each compound. The retention windows were determined from the 5-point linear calibration separation for each compound. Positive identification required the detection of the primary ion and at least two secondary ions for each target compound. Target compound quantitation was based on the integrated abundance of the primary ion and was calculated as follows:

$$\text{Concentration (ng/g)} = K(A_x)(C_{is})/(A_{is})(RF)(W_s)(D)$$

where, A_x = integrated area of primary ion, C_{is} = concentration of internal standard, A_{is} = integrated area of internal standard, $RF = (A_{std}) / (C_{is}) / (A_{is}) / (C_{std})$, K = dilution factor, W_s = weight of the sample (g), and $D = (100 - \% \text{moisture in the sample}) / 100$. The average response factor, RF, for each compound was determined over the 5-point linear calibration curve.

Table 3. HAFB Data Quality Objectives for Metal Analytes

<u>Initial Calibration:</u> 2-point calibration, a blank and one known high level concentration	<u>Requirement</u> average of three solutions	<u>Corrective Action</u> check standards, recalibrate, check instrument
<u>Continuing Calibration Verification:</u> every 10 th sample	percent recovery +/- 20%	check standards, recalibrate, check instrument
<u>Instrument Blank:</u> every 10 th sample	concentration below reporting limit	scan selected wavelengths, increase rinse time, reanalyze acid solution
<u>Method Blank:</u> every 20 th sample	concentration below reporting limit	scan selected wavelengths, increase rinse time, prepare new samples and reanalyze
<u>Measurement Precision:</u> duplicate every 20 th sample	+/- 25% RPD	review lab QC, determine whether in control, if in control flag data
<u>Measurement Accuracy:</u> 1) laboratory control check samples (ERA soil and solution) analyzed every 20 th sample 2) field versus laboratory	percent recovery +/- 20% a) +/- 60% RPD and b) $50\% < R < 200\%$ where $R = 100 \times C_{\text{on-site}}/C_{\text{off-site}}$	1) review lab QC, determine whether in control, if in control flag data 2) site-specific

Metals Metals were identified by their characteristic atomic emission wavelengths. Each metal has a unique wavelength at which spectral interferences were minimal. The area count was obtained by integrating the plot of emission intensity versus wavelength. The metal was quantified by calculating the concentration from a 2-point calibration plot (matrix blank and one known high level concentration) as follows:

$$\text{Concentration (ng/g)} = (C_s)(A_x)/(A_s)$$

where C_s = concentration of calibration standard, A_x = sample area count, and A_s = calibration standard area count.

4.0 Results and Discussion

The quality of data produced in the dynamic site investigation will support its use in the HAFB risk assessment *and* in deciding if additional actions are needed in the vadose zone soil. Described below are the results of the adaptive sampling and analysis program *and* the performance of the field instruments and methods.

4.1 Adaptive Sampling and Analysis Results

The lowest calibration standard from the 5-point curve established the quantitative GC/MS reporting limits (RL). The RL's were supported by method detection limit studies for the targeted organics and metals of concern. Table 1 lists the action level, the corresponding quantitation limit, and MDL for each analyte, see Sections 4.2.2 and 4.2.3 for results. Site contamination maps are expressed at each target compounds' 10DAF concentration (the QL) for ease of visual inspection, see Figure 4 for map key.

Figures 5 and 6 depict surface to bedrock cross sectional profiles for Sites 1, 2, and 3.^{15,24} The maps also show the initial conceptual model for each site used to help guide the sample collection process and locate wells found to contain high levels of VOCs. The depths to critical horizons below the ground surface were:

- depth from surface to pit bottoms (3-ft Site 1, 6-ft Sites 2 and 3),
- pit bottoms to top of recharge basin (~ 12-ft Sites 2 and 3),
- depth from surface to top of lacustrine unit (Site 1 not available, Site 2 ~ 15-ft, Site 3 ~ 18-ft),
- depth from surface to bottom lacustrine unit (Site 2 21-23-ft, Site 3 23-ft to 26-ft),
- depth from surface to bedrock (Site 1 15-20-ft, Site 2 40-ft, Site 3, 90-ft),
- depth to bedrock from top of recharge basin (Site 1 not applicable, Site 2 46-ft, Site 3 95-ft)

An average of 75 soil samples was screened for VOCs per day by TDGC/MS over a two-week period for a total of 601 samples analyzed. Quantitative VOC analysis of 158 soil samples by purge and trap GC/MS was made to confirm the screening results and to delineate the extent of contamination. Quantitative analyses of 70 soil samples for PCBs and PAHs and 121 soil samples for metals were made. Described below are our findings for Sites 1-3 *and* the collection and analysis decisions made in the field.

Icons of Samples:

△ Qualitative ND

The sample comes from a boring that was ND for all depths during screening and confirmed by one quantitative analysis for the boring. For example in [Site 2](#) the samples relating to sample location [S2-B02-1A](#) are [light blue](#) for samples [above 8 feet](#), and [dark blue](#) for samples [below 8 feet](#). This means that each boring contained non detectable levels of target compounds as analyzed by qualitative fast-GC/MS. Confirmation analysis by quantitative GC/MS and Tufts data interpretation software for one sample showed the same result. In this case the confirmation sample was from a depth below 8 feet.

□ Quantitative, concentration less than 10DAF

The sample was quantitated using GC/MS and Tufts data interpretation software and was either an ND or was found to contain contaminants in lower concentration than 10DAF.

○ Quantitative, concentration more than 10DAF

The sample was quantitated using GC/MS and Tufts data interpretation software and was found to contain contaminants at higher concentration than 10DAF.

Note: To normalize* the action levels, all target compound concentrations have been divided by their corresponding 10DAF value (≡ 1/2 of the 20DAF value). The maps indicate contaminants above the 10DAF.

* Calculation example, Site 2 - Chlorinated VOCs below 8ft, sample S2-B13-(14-16):
 380 ppb cis-1,2-dichloroethene, 10DAF = 200 pbb, $380/200 = 1.9$ -10DAF
 42 ppb tetrachloroethene, 10DAF = 30 ppb, $42/30 = 1.4$ -10DAF

Compound numbering and DAF values:

#	Compound	10DAF
1	Vinyl Chloride	5 ppb
2	Methylene Chloride	10 ppb
3	1,1-Dichloroethene	30 ppb
4	1,1-Dichloroethane	10 ppb
5	cis-1,2-Dichloroethene	200 ppb
6	trans-1,2-Dichloroethene	300 ppb
7	Chloroform	300 ppb
8	1,2-Dichloroethane	10 ppb
9	1,1,1-Trichloroethane	1000 ppb
10	Carbon tetrachloride	35 ppb
11	Trichloroethene	30 ppb
12	Benzene	15 ppb
13	Tetrachloroethene	30 ppb
14	Toluene	6000 ppb
15	Chlorobenzene	500 ppb
16	Ethylbenzene	6500 ppb
17	Styrene	2000 ppb
18	o-Xylene	95000 ppb
19	m/p-Xylene	105000 ppb

Figure 4. Map Key

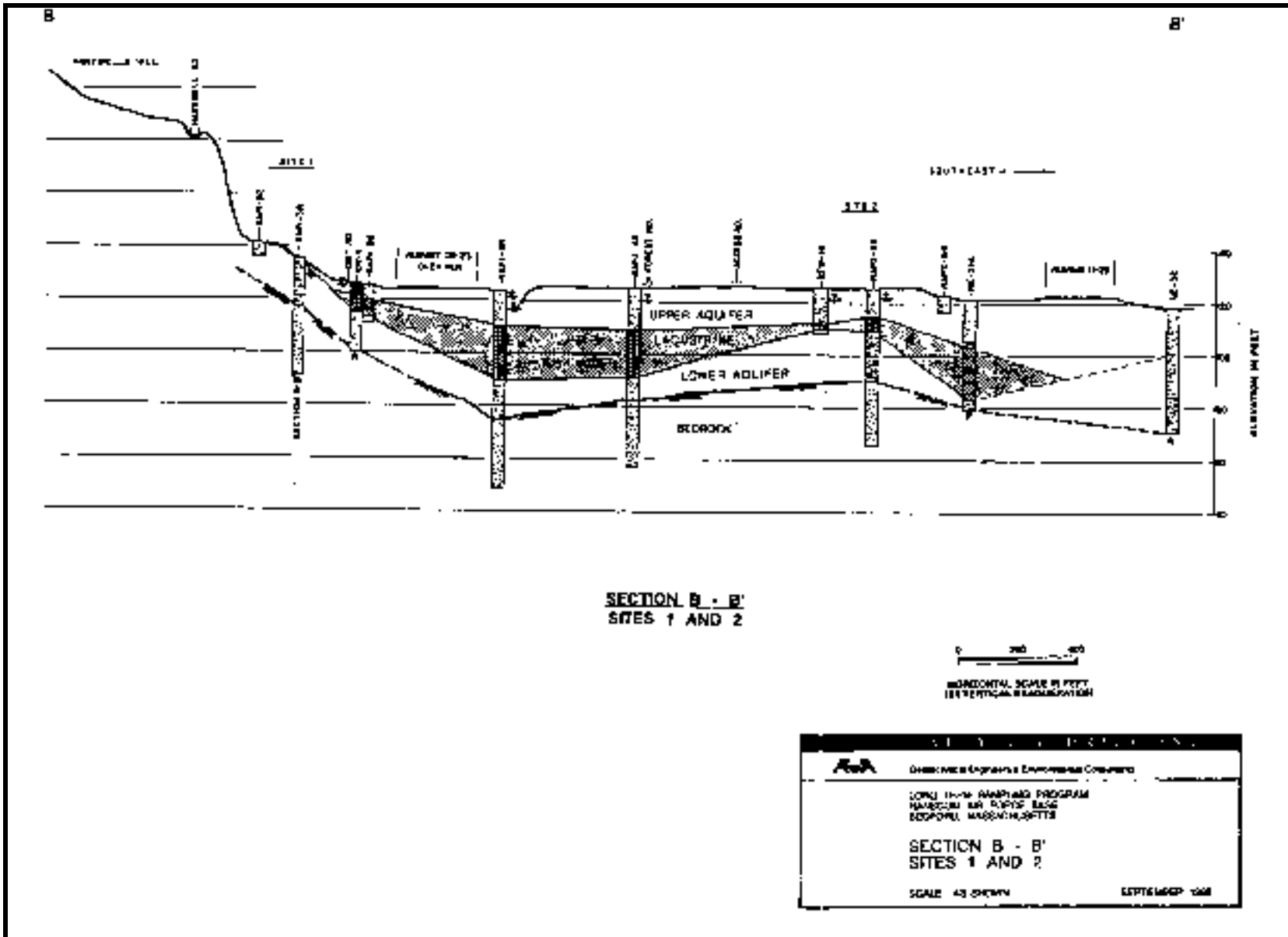


Figure 5. HAFB Sites 1 and 2

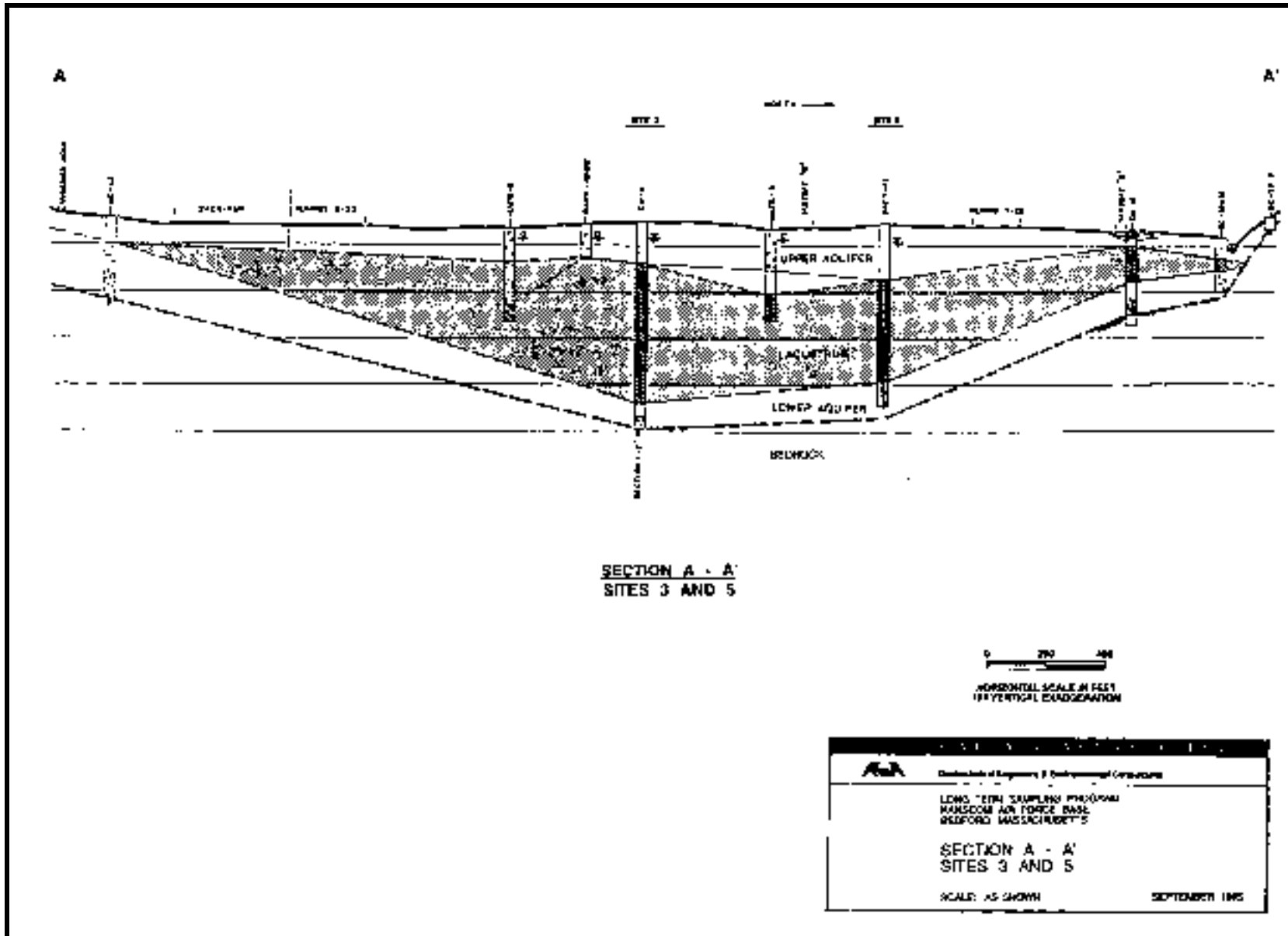


Figure 6. HAFB Site 3

SITE 1

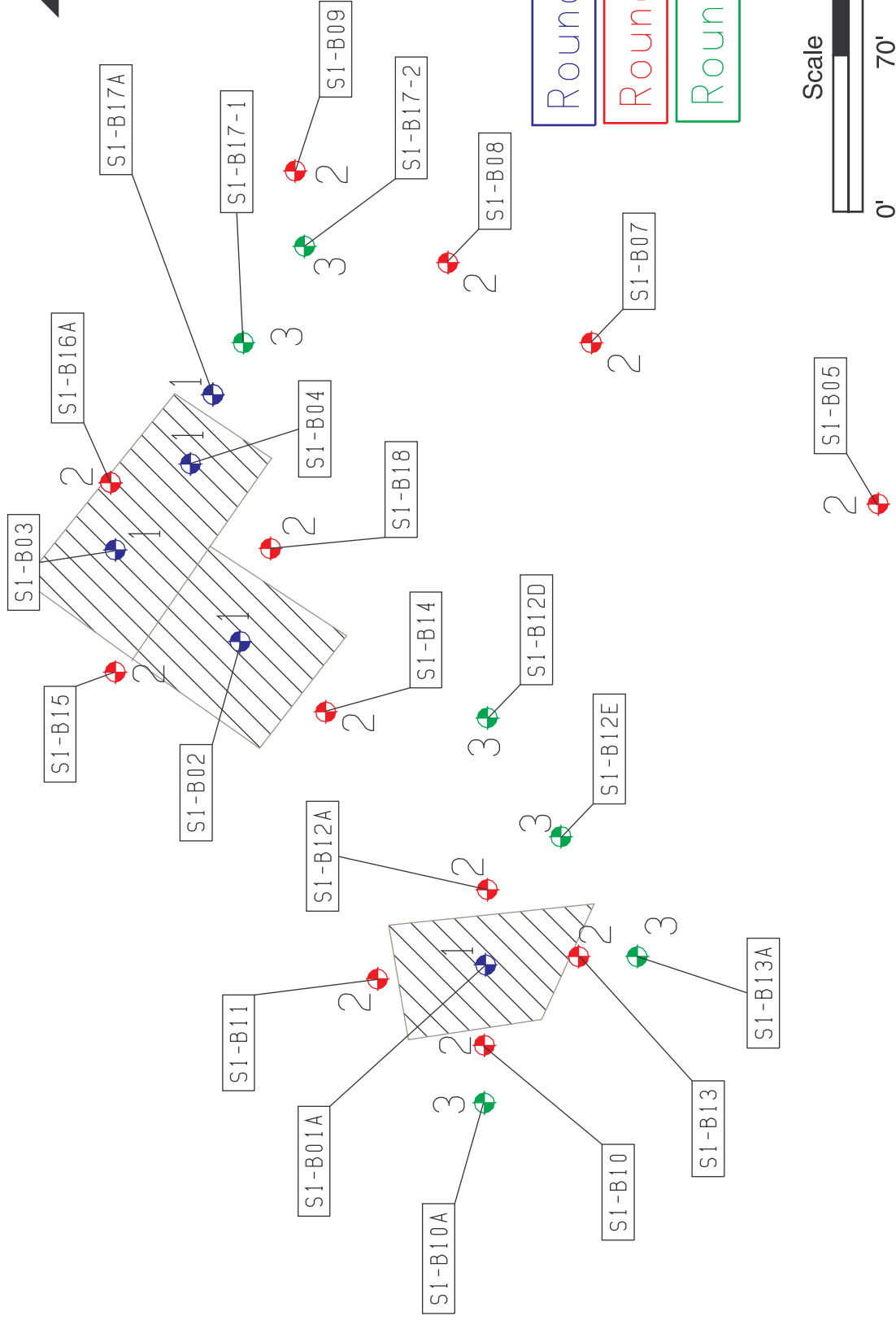
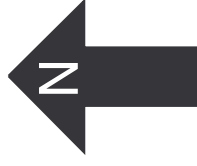
VOCs. Figure 7a shows the location of samples collected during Rounds 1, 2, and 3. Figures 7b-7e depict the chlorinated VOCs, benzene, toluene, ethylbenzene and xylenes (BTEX) contamination at depths above and below the 4-ft fire training burn pit bottoms. Contaminant concentrations are shown with respect to their 10DAF concentrations as measured by quantitative GC/MS. For example, cis-1,2-dichloroethene is compound #5 in the Figure 4 table. Figure 7d shows that cis-1,2-dichloroethene was detected in soil from boring S1-B17A at depths between 5.75 and 8-ft. It is listed as S1-B17A-(5.75-08): #5 4-10DAF. The 4-10DAF shows that this target compound was present at four times the 10DAF concentration or 800-ppb. This notation was used to facilitate the decisions made in the field and to quickly visualize the contamination at the quantitation limit. Recall that the action level for Operable Unit 1 was set at the 20DAF soil screening level for evaluating contaminant risk to ground water.

Whenever a triangle is shown in the map, it indicates non detectable (ND) target compound concentrations by rapid screening GC/MS at the specified interval. In every instance for Site 1 another soil sample from the same boring at another depth was analyzed by quantitative GC/MS and found to contain no detectable VOCs. As an example, samples from S1-B05, S1-B07, S1-B08, S1-B17-2, and S1-B09 were found to contain no detectable VOCs above the burn pits by screening GC/MS, see Figures 7b and 7c. Subsequent confirmation was made by quantitative GC/MS below the pit bottom, see Figures 7d and 7e. A square indicates target compound concentrations less than 10DAF or at non-detectable levels as determined by quantitative GC/MS. In contrast, circles show samples where quantitative GC/MS measured target compound concentrations were greater than the corresponding 10DAF level.

Although samples were collected from 23 boring locations, only five samples from three borings (S1-B04, S1-B10A, and S1-B17A) contained detectable target compounds above their 10DAF concentration. A total of 210 soil samples was screened from which 51 samples were analyzed by quantitative GC/MS. All areas positively identified to be contaminated were encircled by soil samples from borings shown to contain non detectable target compound concentration except S1-B10A-(4.5-6) which contained cis-1,2-dichloroethene and trichloroethene at levels between 10DAF and 20DAF. Therefore, the core technical team decided that no additional Round 3 samples required analysis to bound this location.

Semi-VOCs No maps were produced for PCBs and PAHs. A total of 46 samples was collected and analyzed by quantitative TDGC/MS. Only one sample, S1-B04-(6.2-8), contained detectable levels of PAH: acenaphthene 1-ppm, acenaphthylene 0.7-ppm, fluorene 2-ppm, anthracene/phenanthrene 5-ppm, pyrene 1-ppm, fluoranthene 0.8-ppm, and benzo(a)anthracene/chrysene 2-ppm. No samples contained detectable levels of PCBs. Since the reporting limits for PAH were well below the 20DAF concentrations and the fact that only one burn pit sample contained any appreciable PAH, no additional samples were collected or analyzed.

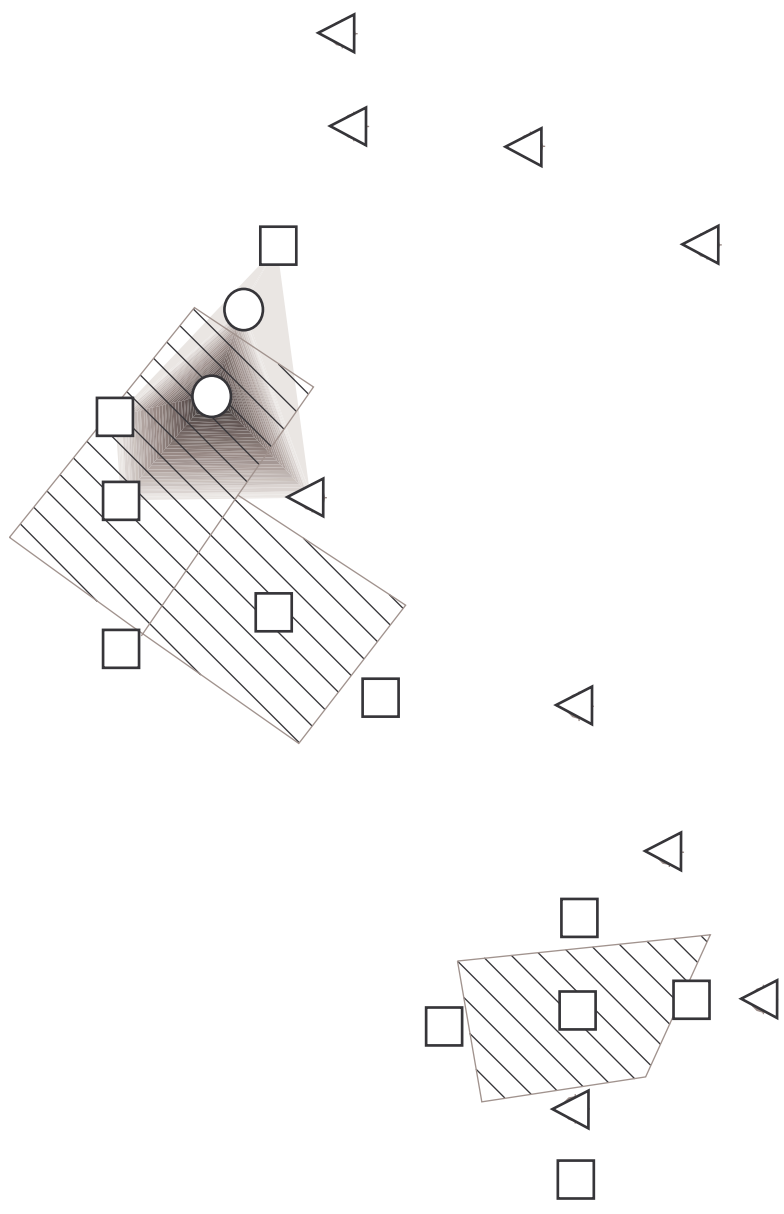
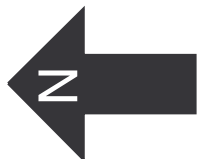
Site 1



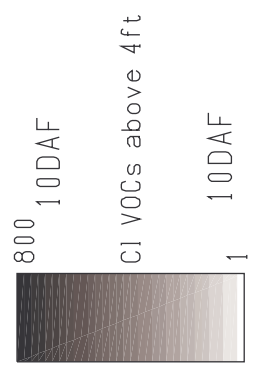
2.5x vertical exaggeration

Figure 7a.

HAFB: Site 1 - Chlorinated VOCs above 4ft



S1-B04-(02-04): #5 800-10DAF, #11 150-10DAF, #4 15-10DAF, #8 12-10DAF, #6 5-10DAF, #13 4-10DAF, #12 1-10DAF
 S1-B17A-(02-04): #5 24-10DAF, #11 3-10DAF, #13 2-10DAF



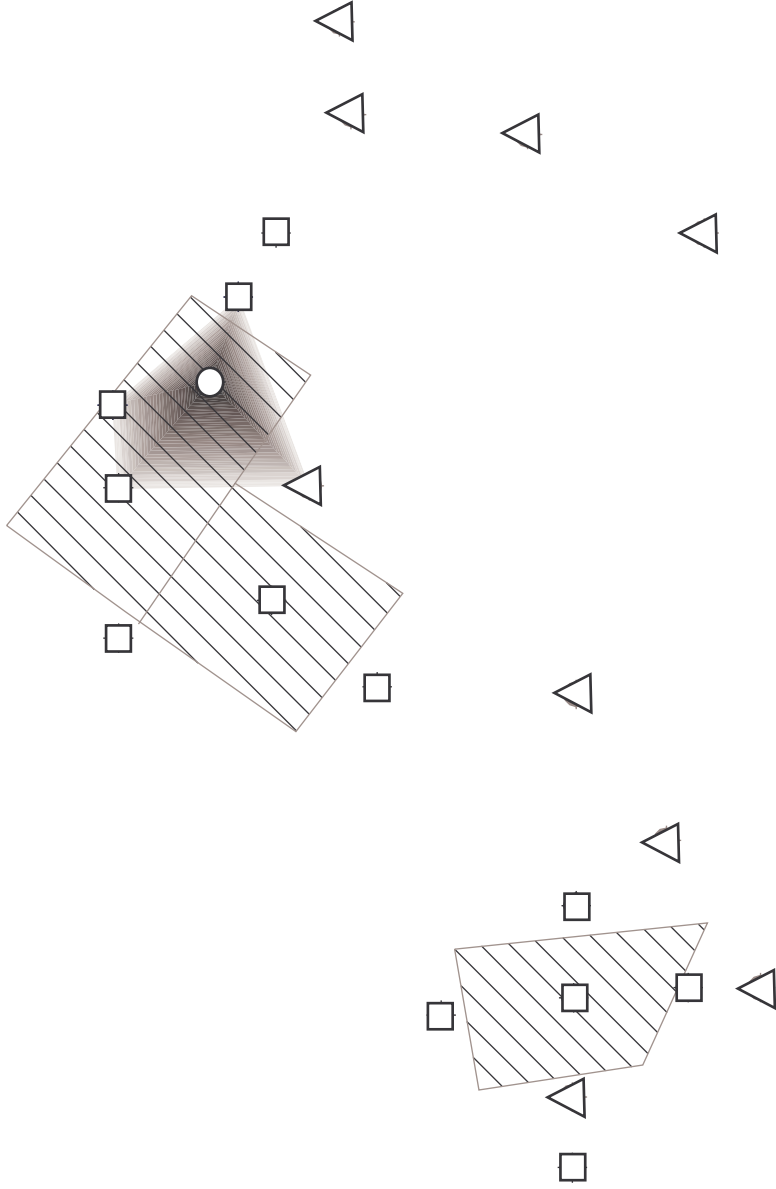
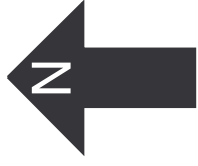
Scale



2.5x vertical exaggeration

Figure 7b.

HAFB: Site 1 - BTEX above 4ft



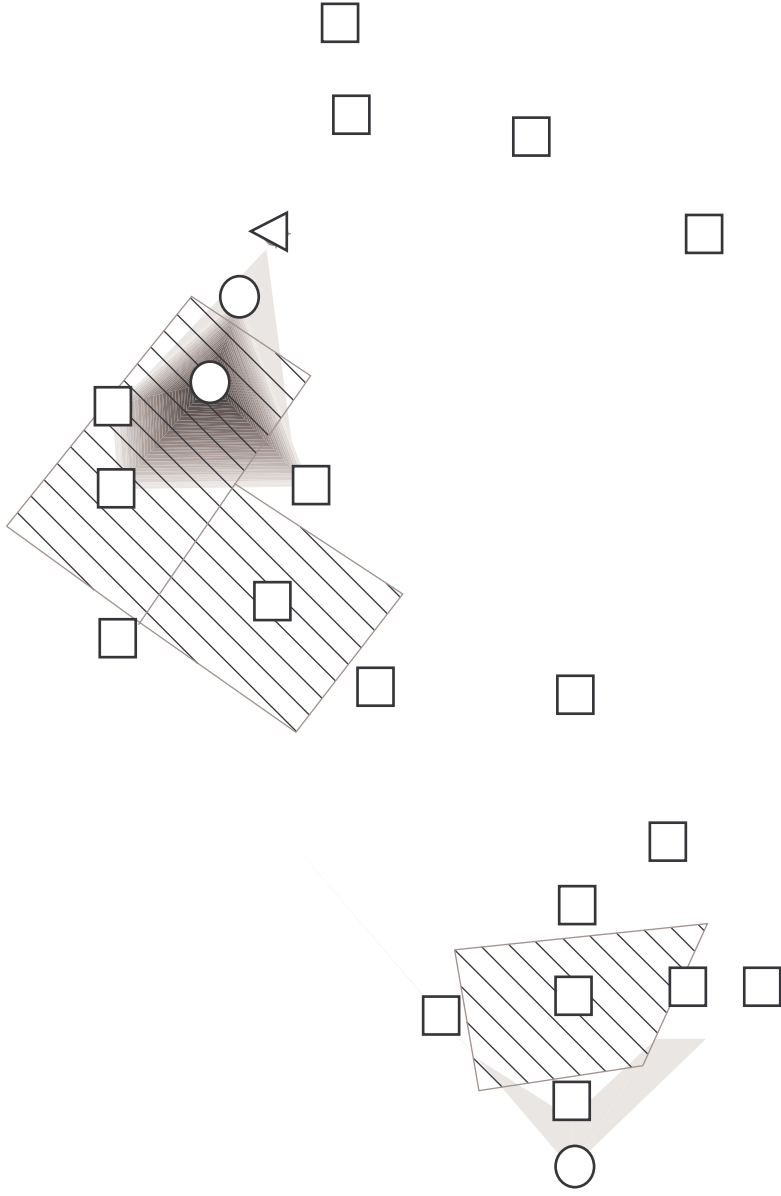
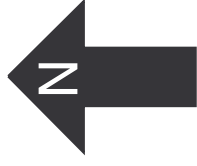
S1-B04-(02-04): #5 800-10DAF, #11 150-10DAF, #4 15-10DAF, #8 12-10DAF, #6 5-10DAF, #13 4-10DAF, #12 1-10DAF
 S1-B17A-(02-04): #5 24-10DAF, #11 3-10DAF, #13 2-10DAF



2.5x vertical exaggeration

Figure 7c.

HAFB: Site 1 - Chlorinated VOCs below 4ft



S1-B04-(6.2-8): #11 330-10DAF, #5 200-10DAF, #4 13-10DAF, #13 11-10DAF, #8 10-10DAF, #6 5-10DAF, #12 1.1-10DAF

S1-B10A-(4.5-06): #11 2-10DAF, #5 1.5-10DAF

S1-B17A-(5.75-08): #5 4-10DAF

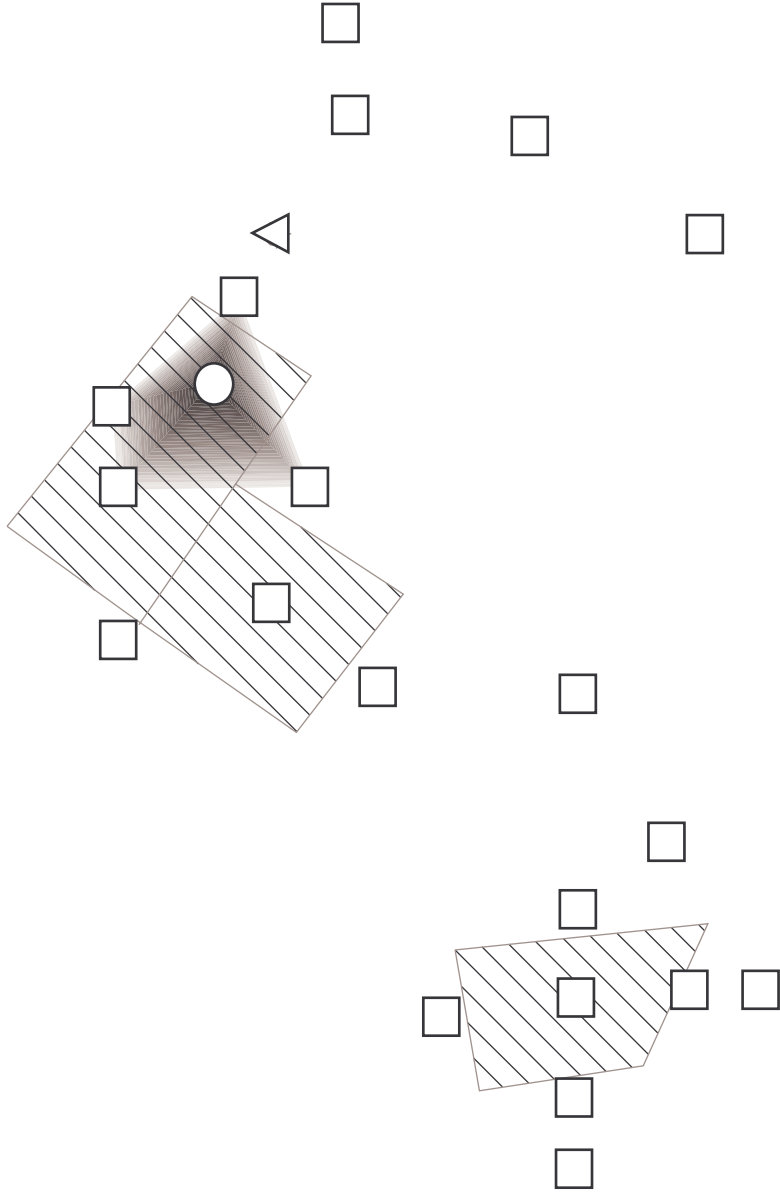
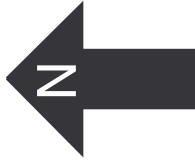
Scale



2.5x vertical exaggeration

Figure 7d.

HAFB: Site 1 - BTEX below 4ft



S1-B04-(6.2-8): #11 330-10DAF, #5 200-10DAF, #4 13-10DAF, #13 11-10DAF, #8 10-10DAF, #6 5-10DAF, #12 1.1-10DAF

S1-B10A-(4.5-06): #11 2-10DAF, #5 1.5-10DAF

S1-B17A-(5.75-08): #5 4-10DAF

Scale



2.5x vertical exaggeration

Figure 7e.

Metals No maps were produced for metals. Soil samples were analyzed above and below the pit bottoms. Field ICP/OES analysis yielded no detectable lead or cadmium concentrations above their respective 10DAF concentrations; namely, 200-ppm and 4-ppm respectively. The total number of soil samples analyzed was twenty-two, fourteen samples had concentrations between 10 and 40-ppm lead while twelve samples produced concentrations between 0.35-ppm and 0.71-ppm cadmium. After analyzing seven of the 38 samples collected in Round 2, EPA, Air Force and Tufts staff agreed that none of the remaining samples required analysis.

SITE 2

Situated on this site is a recharge basin whose elevation is 6-ft above ground level. Sample IDs within the recharge basin include the 6-ft elevation depth, while those samples outside the recharge basin depict depth from ground level.

VOCs Soil samples were collected from 18 borings to subsurface depths of 16-ft, Figure 8a. A total of 177 sample locations was screened from these borings. Samples were collected for quantitative GC/MS analysis from one-half of these locations yielding 58 soil samples. Of the eighteen borings seven borings (S2-B01, S2-B02, S2-B03, S2-B04, S2-B13, S2-B15, S2-B15A) were contaminated at twelve different sample locations. Target compound concentrations were greater than 10DAF. Tetrachloroethene is present in the soil between 42 and 540-ppb in three borings (S2-B01, S2-B02, S2-B04) above 8-ft (i.e., depth from ground level), see Figure 8b. In contrast, eight chlorinated solvents were found below 8-ft, see Figure 8d. The three solvents with the highest concentrations were tetrachloroethene at percent levels (estimated, S2-B15), 1,1,1-trichloroethane at 15-ppm (S2-B02) and cis-1,2-dichloroethene at 15-ppm (S2-B13). Figures 8c and 8e reveal toluene as the only BTEX contaminant at levels above 10DAF in the pits: 24 to 42-ppm between the depths of 7-ft and 12-ft. Benzene was found in only one boring (S2-B15) at 8 to 10-ft. No detectable amounts of benzene were found in borings S2-B15A or S2-B04, two very closely sampled locations. All pit locations shown to contain target compounds in Round 1 greater than 10DAF were encircled by the collection of soil samples in Rounds 2 and 3. Two samples were collected away from the recharge basin to assess if contaminant migration through soil may be occurring. No detectable levels of VOCs were found by either screening or quantitative GC/MS down to subsurface depths of 16-ft.

Semi-VOCs No maps were made for PCBs and PAHs. Historical information at this site indicated that PCB/PAH analysis was not necessary. During the investigation, it became evident that some soil locations were contaminated by a highly viscous petroleum product. Soils were initially selected for analysis based on visible contamination. Positive identification was made in three Round 1 samples: drum pit S2-B04 at two depths (13 to 15-ft and 19 to 21-ft) and S2-B15 (14 to 16-ft). Positive identification was made in one second round sample S2-B13 at a depth of 14 to 16-ft. The highest PAH concentrations found in all soil samples analyzed and their action levels (20DAF) were benzo(a)anthracene 0.3-ppm (2-ppm), benzo(b)fluoranthene 0.5-ppm (5-ppm), benzo(a)pyrene 1-ppm (8-ppm), and dibenzo(a,h)anthracene 0.3-ppm (2-ppm). All PAH detected were well below their 20DAF concentrations. Based on results from 12 of the 24 samples collected, a decision was made not to analyze the remaining samples.

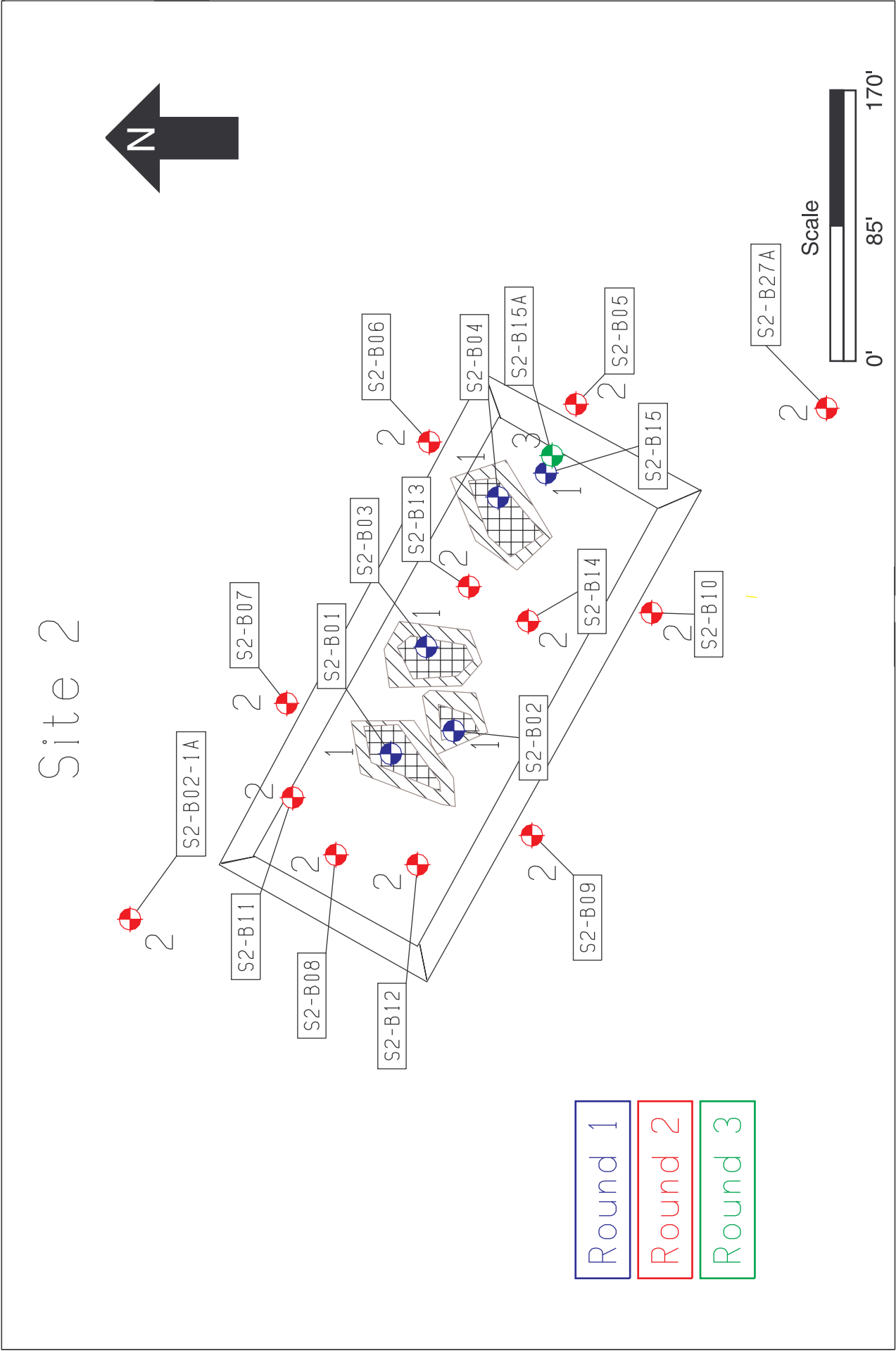
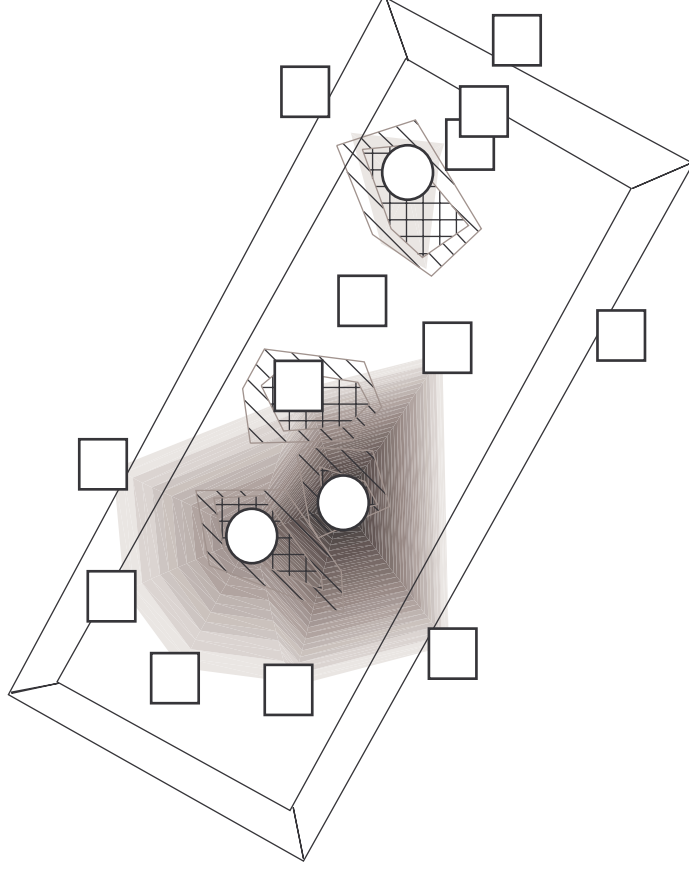
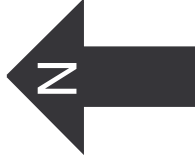
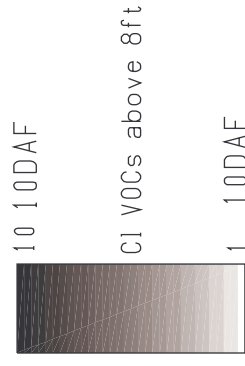


Figure 8a.

HAFB: Site 2 - Chlorinated VOCs above 8ft



- S2-B01-(12-14): #13 5-10DAF
- S2-B02-(13-15): #13 18-10DAF, #14 1.5-10DAF
- S2-B03-(13-15): #14 4-10DAF
- S2-B04-(13-15): #13 1.4-10DAF

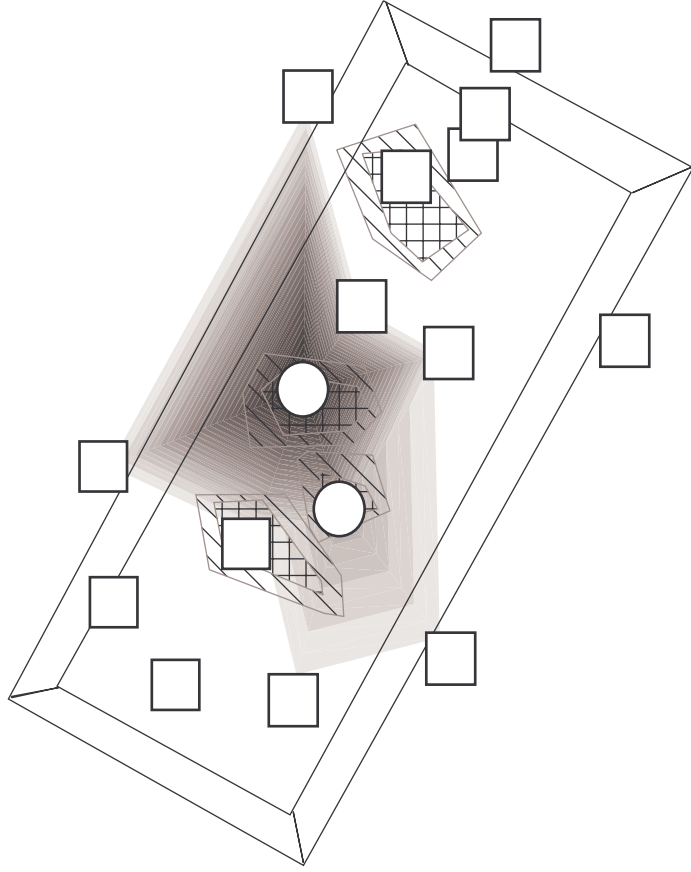
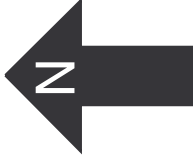


Scale

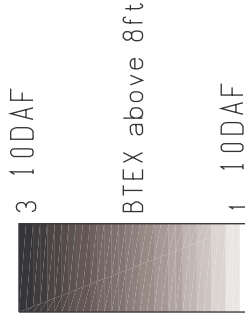


Figure 8b.

HAFB: Site 2 - BTEX above 8ft



- S2-B01-(12-14); #13 5-10DAF
- S2-B02-(13-15); #13 18-10DAF, #14 1.5-10DAF
- S2-B03-(13-15); #14 4-10DAF
- S2-B04-(13-15); #13 1.4-10DAF

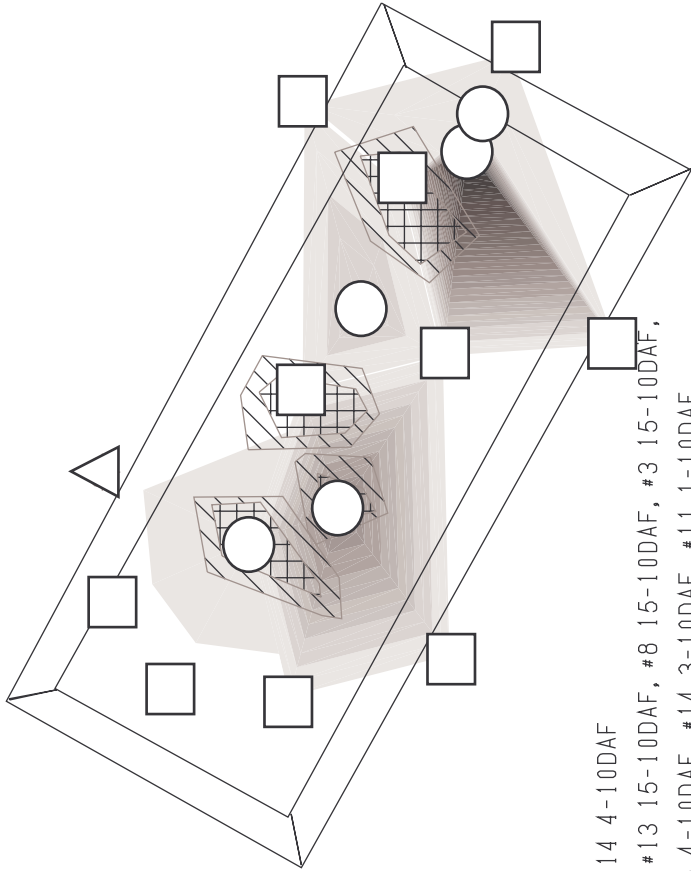
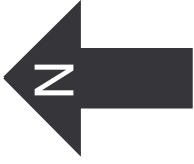


Scale

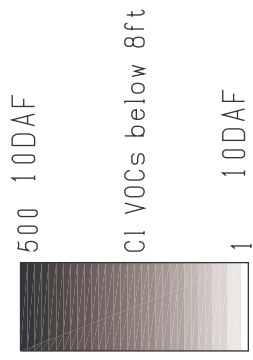


Figure 8c.

HAFB: Site 2 - Chlorinated VOCs below 8ft



- S2-B01-(20-22): #13 4-10DAF, #14 4-10DAF
- S2-B02-(16-18): #4 160-10DAF, #13 15-10DAF, #8 15-10DAF, #3 15-10DAF,
#9 7-10DAF, #5 4-10DAF, #14 3-10DAF, #11 1-10DAF
- S2-B02-(20-22): #14 7-10DAF, #4 4-10DAF, #13 4-10DAF, #5 3-10DAF, #3 1-10DAF
- S2-B03-(16-18): #14 1.6-10DAF
- S2-B13-(14-16): #5 1.9-10DAF, #13 1.4-10DAF
- S2-B13-(18-20): #5 75-10DAF, #3 1.7-10DAF, #4 1.5-10DAF
- S2-B15-(14-16): #11 27000-10DAF, #12 9-10DAF, #13 9-10DAF, #5 2-10DAF, #6 1.5-10DAF
- S2-B15A-(18-20): #5 33-10DAF, #3 1.1-10DAF

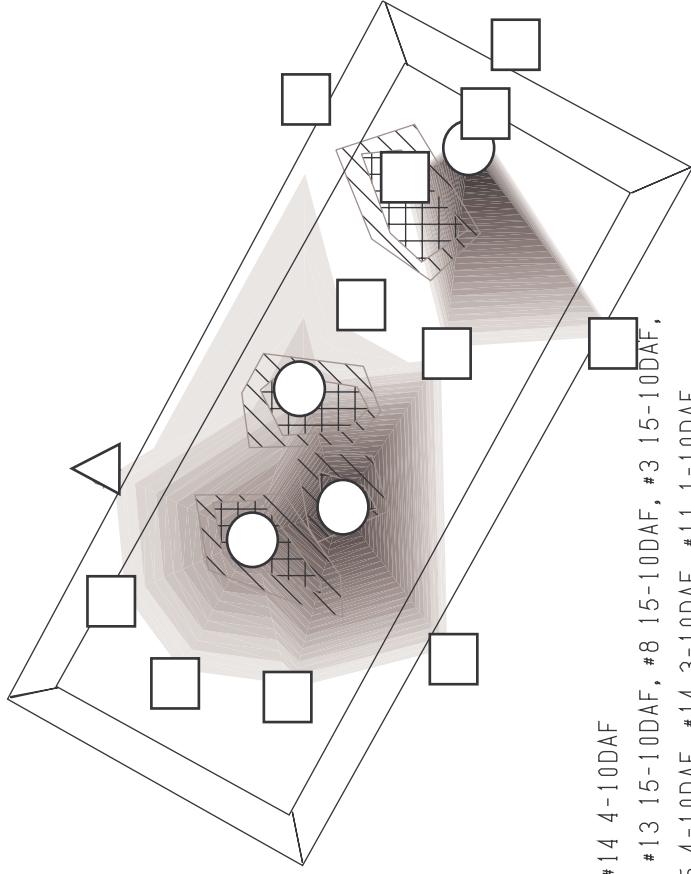
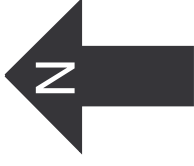


Scale



Figure 8d.

HAFB: Site 2 - BTEX below 8ft



- S2-B01-(20-22): #13 4-10DAF, #14 4-10DAF
- S2-B02-(16-18): #4 160-10DAF, #13 15-10DAF, #8 15-10DAF, #3 15-10DAF,
#9 7-10DAF, #5 4-10DAF, #14 3-10DAF, #11 1-10DAF
- S2-B02-(20-22): #14 7-10DAF, #4 4-10DAF, #13 4-10DAF, #5 3-10DAF, #3 1-10DAF
- S2-B03-(16-18): #14 1.6-10DAF
- S2-B13-(14-16): #5 1.9-10DAF, #13 1.4-10DAF
- S2-B13-(18-20): #5 75-10DAF, #3 1.7-10DAF, #4 1.5-10DAF
- S2-B15-(14-16): #11 27000-10DAF, #12 9-10DAF, #13 9-10DAF, #5 2-10DAF, #6 1.5-10DAF
- S2-B15A-(18-20): #5 33-10DAF, #3 1.1-10DAF

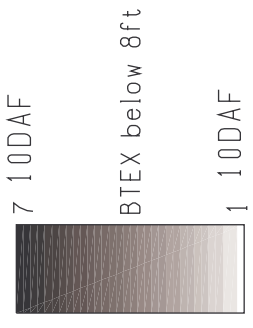
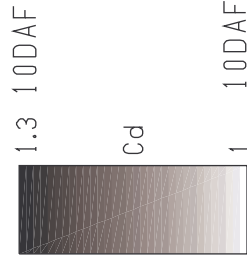
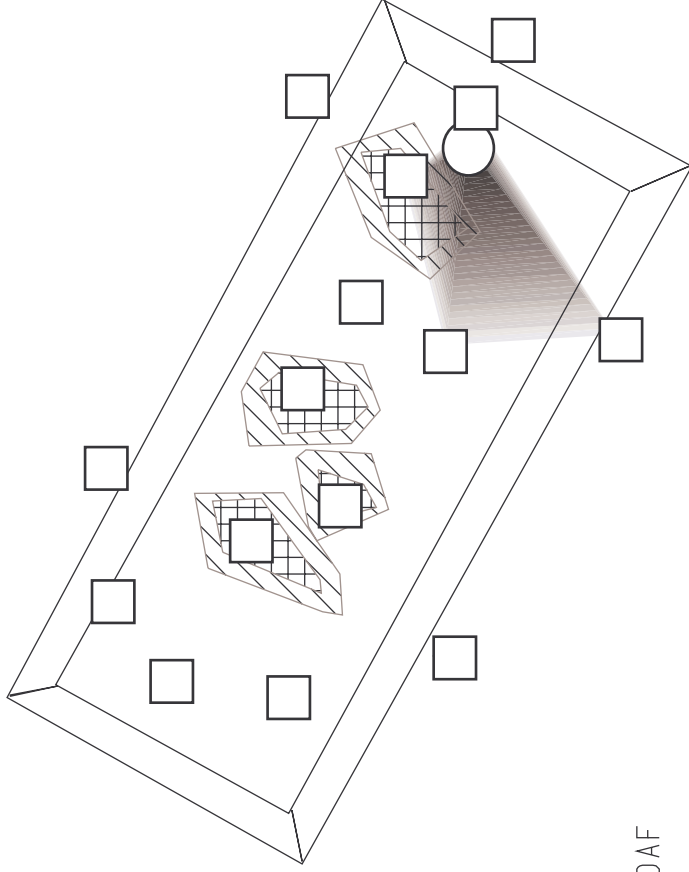
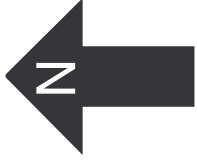


Figure 8e.
31

HAFB: Site 2 - Cadmium



- S2-B01-(12-14): Pb 1.3-10DAF
- S2-B04-(13-15): Pb 1.1-10DAF
- S2-B15-(14-16): Pb 1.8-10DAF, Cd 1.6-10DAF

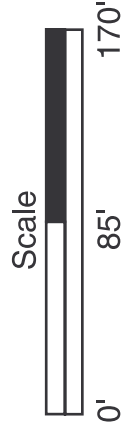
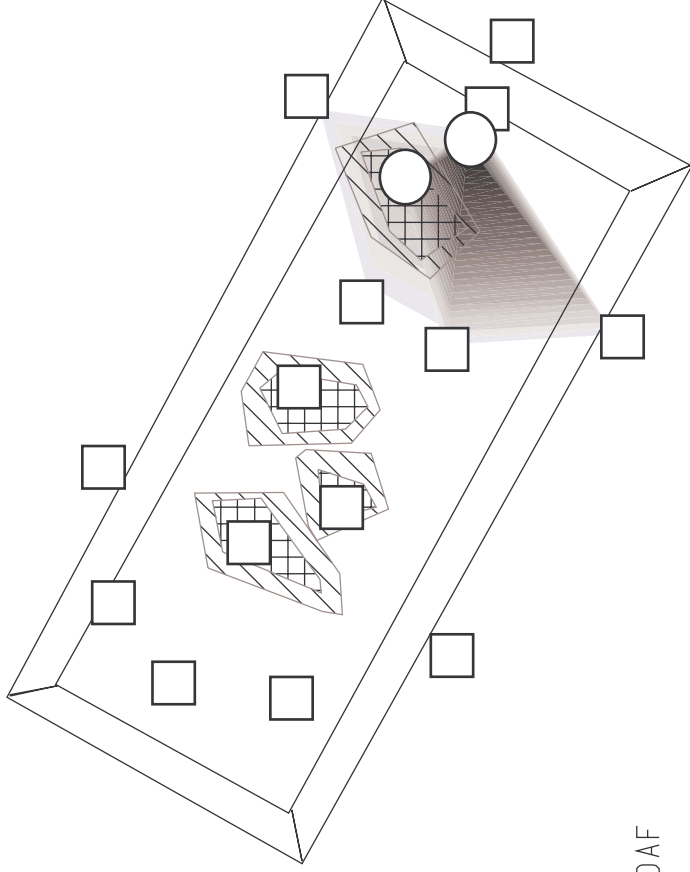


Figure 8f.

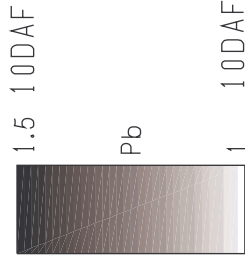
HAFB: Site 2 - Lead



S2-B01-(12-14): Pb 1.3-10DAF

S2-B04-(13-15): Pb 1.1-10DAF

S2-B15-(14-16): Pb 1.8-10DAF, Cd 1.6-10DAF



Scale



Figure 8g.

Metals Figures 8f and 8g depict the cadmium and lead concentrations for Site 2. The pit samples (S2-B01, S2-B02, S2-B03, S2-B04) had the highest concentrations of lead and cadmium. For lead the concentrations ranged between 10-ppm and 370-ppm at or near the pit bottoms while cadmium concentrations were between 0.35-ppm and 1-ppm. Outside the pits the concentrations dropped dramatically to levels between ND and 50-ppm for lead and non detectable levels for cadmium. Boring sample S2-B15 (14-16) was the exception with 360-ppm lead and 6-ppm cadmium. All samples were below the site-specific 20DAF levels of concern. After reviewing data from 54 of the 68 samples collected, it was decided that no additional sample analysis was needed.

SITE 3

Situated on this site is a recharge basin whose elevation is 6-ft above ground level. Sample IDs within the recharge basin include the 6-ft elevation depth, while those samples outside the recharge basin depict depth from ground level.

VOCs Figure 9a shows the boring locations sampled during Rounds 1, 2, and 3. Soil from 25 boring locations was analyzed to subsurface depths of approximately 16-ft. Screening analyses were performed on 214 samples from these borings with 49 analyses performed by quantitative GC/MS. Figures 9b-9c illustrate the results for samples collected and analyzed above 8-ft (i.e., depth from ground level). All of the BTEX compounds were found in soil samples collected from boring S3-B01; 1.3-ppm, 3,100-ppm, 71-ppm, and 210-ppm, respectively. At the same location cis-1,2-dichloroethene was also detected at 2.8-ppm. Contamination was found in samples collected from S3-B06 (tetrachloroethene at 60-ppb) and S3-B08 (cis-1,2-dichloroethene at 800-ppb). Analysis of Round 2 samples from all elevations surrounding the two borings revealed no detectable VOCs. No chlorinated solvent contamination was found above the 20DAF level at depths below 8-ft. Toluene was the only BTEX constituent detected (18-ppm in S3-B05) below 8-ft and above the 20DAF (12-ppm) concentration. The analysis of soil samples surrounding all four pits where contamination was found yielded non detectable signals.

Semi-VOCs Based on historical information, no samples for this site were preselected for PAH and PCB analysis in the dynamic workplan. Soil samples were analyzed from borings where BTEX constituents were found and from pits where drums were buried. Ten samples were selected to verify the absence of PAHs and PCBs. Of the eight drum burial pits only two pits contained measurable contaminants. Total PCB concentrations in S3-B01-(13-15) and S3-B06-(10-12) were 3-ppm and 2-ppm, respectively. PAHs were also detected in S3-B06 below the 20DAF levels. No PAHs or PCBs were found in any other sample. Although total PCBs were found in two samples above the action level, the core technical team determined that no additional analyses were required to decide future vadose soil actions.

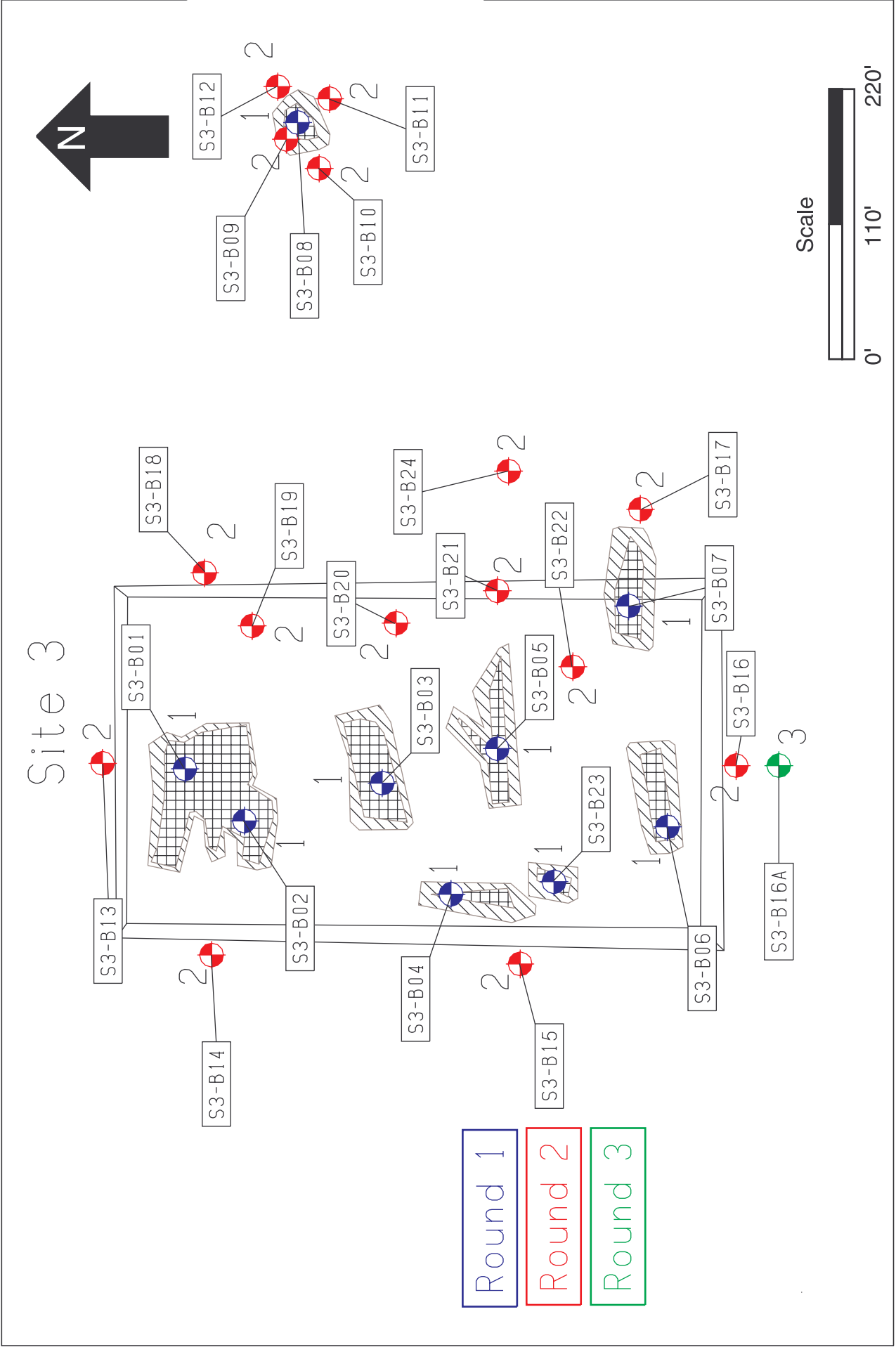
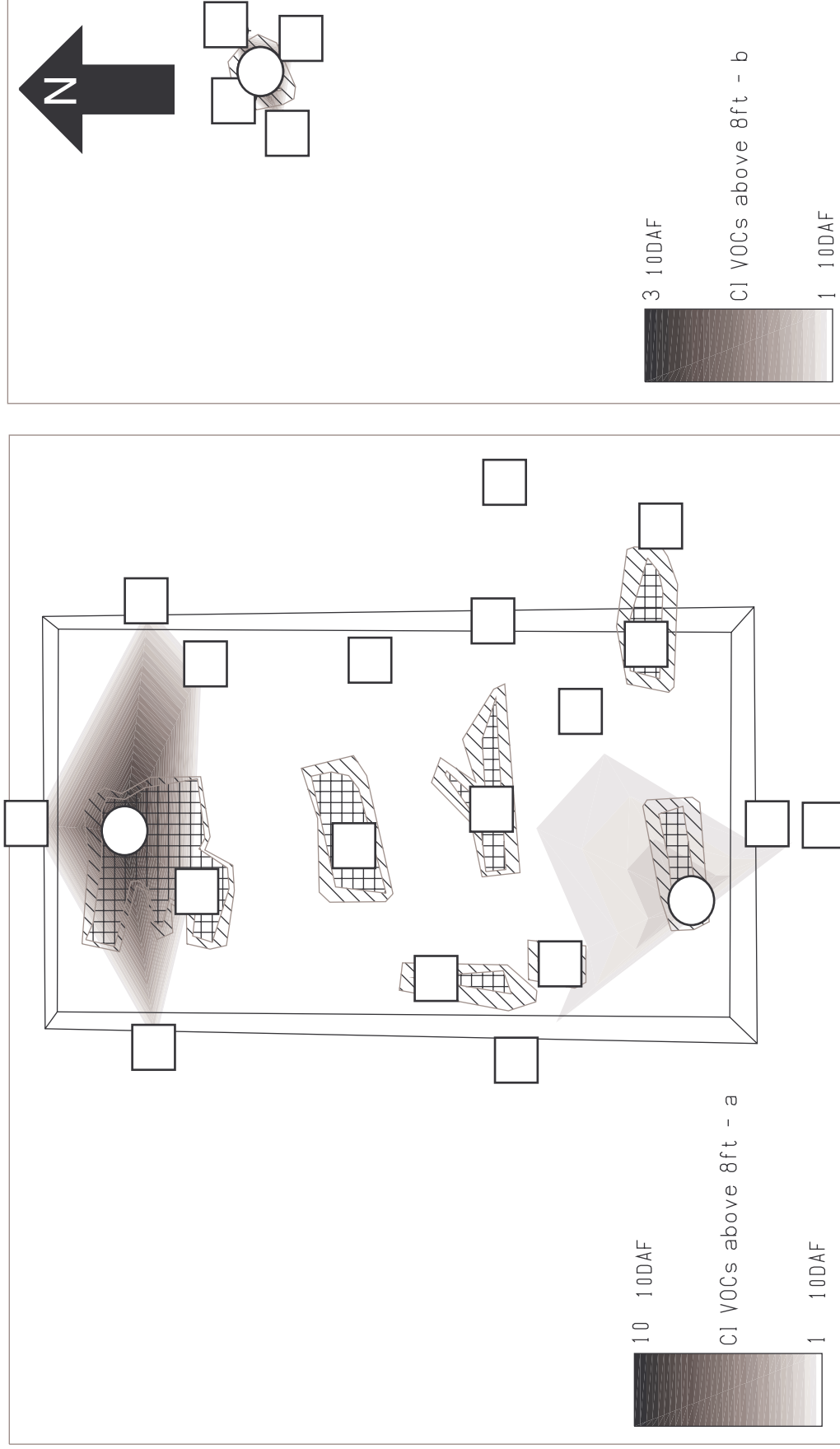


Figure 9a.

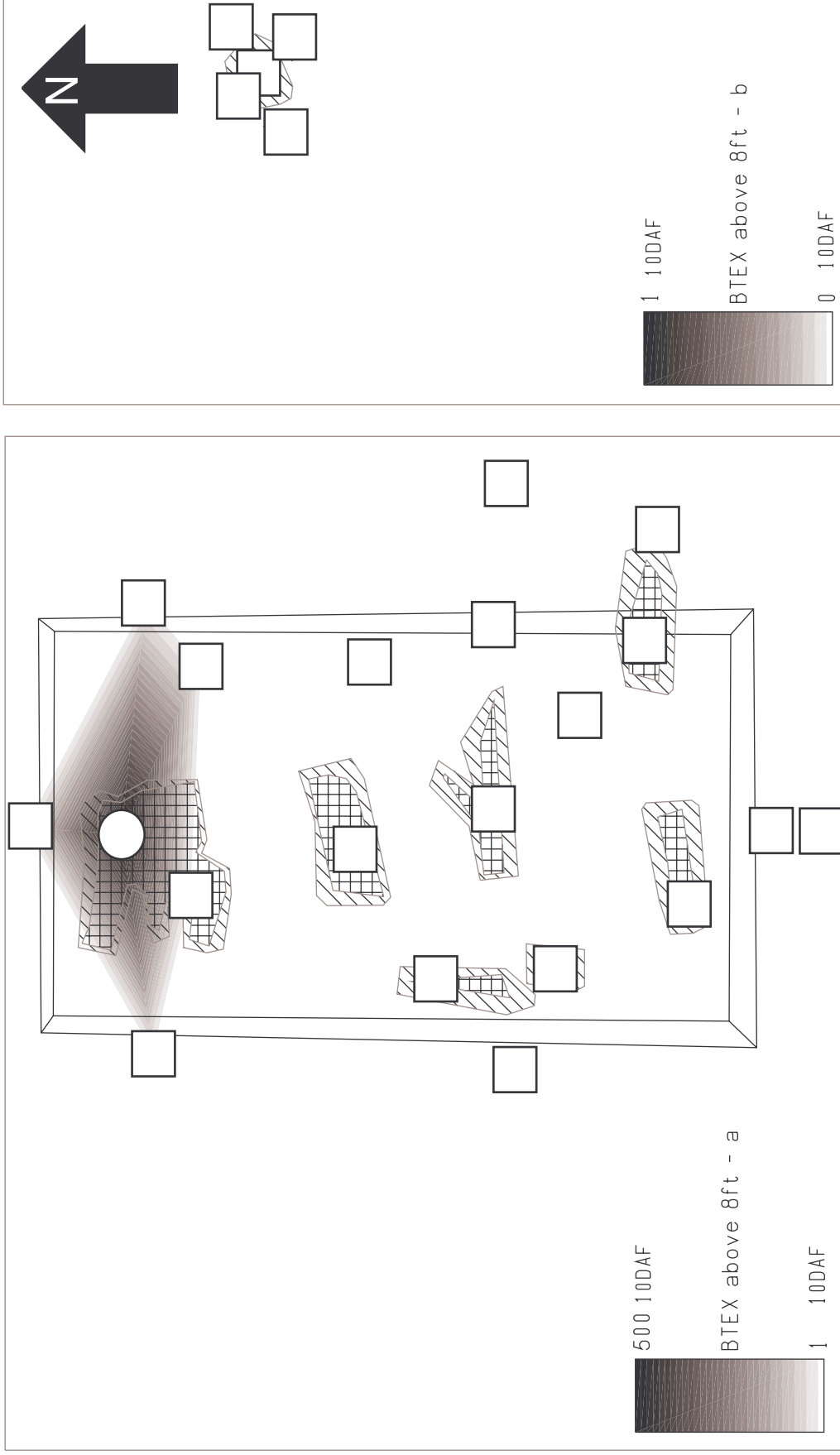
HAFB: Site 3 - Chlorinated VOCs above 8ft



S3-B01-(13-15): #14 520-10DAF, #12 87-10DAF, #5 14-10DAF, #16 11-10DAF, #19 2-10DAF
 S3-B06-(13-15): #13 2-10DAF
 S3-B08-(06-08): #5 4-10DAF

Figure 9b.

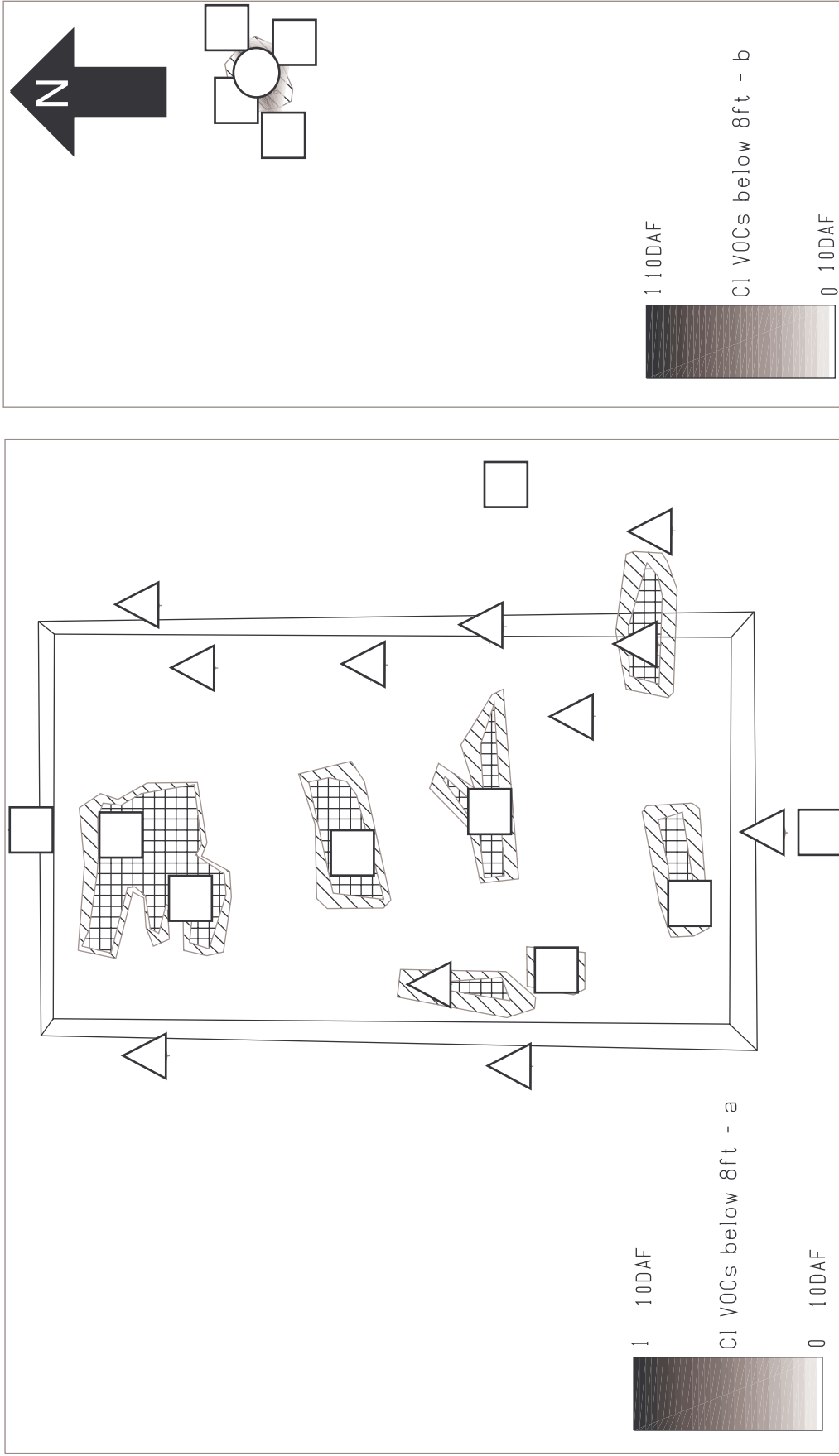
HAFB: Site 3 - BTEX above 8ft



S3-B01-(13-15): #14 520-10DAF, #12 87-10DAF, #5 14-10DAF, #16 11-10DAF, #19 2-10DAF
 S3-B06-(13-15): #13 2-10DAF
 S3-B08-(06-08): #5 4-10DAF

Figure 9c.

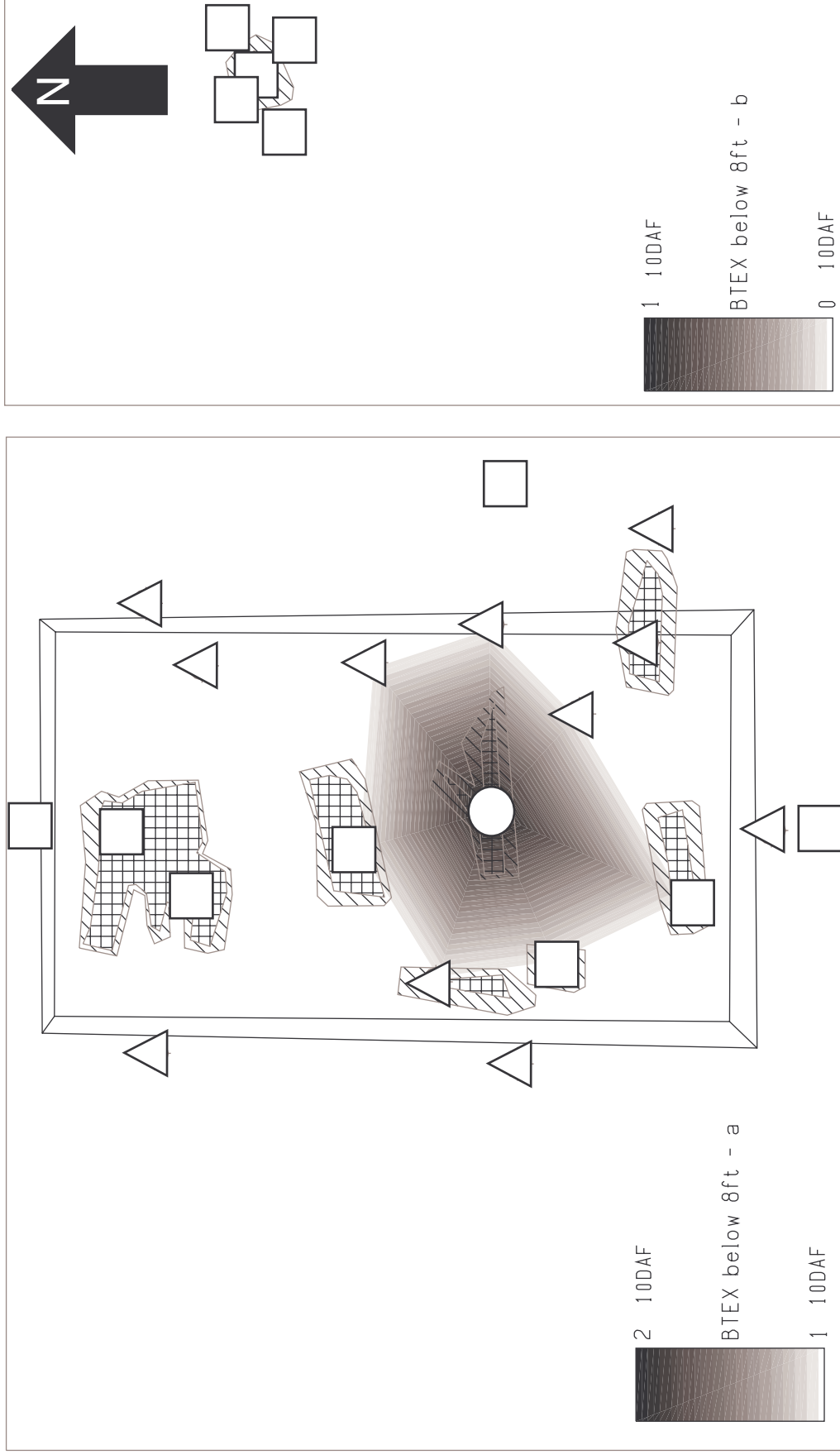
HAFB: Site 3 - Chlorinated VOCs below 8ft



S3-B05-(14-16): #14 3-10DAF
S3-B08-(10-12): #5 1.2-10DAF

Figure 9d.

HAFB: Site 3 - BTEX below 8ft



S3-B05-(14-16): #14 3-100DAF
 S3-B08-(10-12): #5 1.2-100DAF

Figure 9e.

HAFB: Site 3 - Cadmium

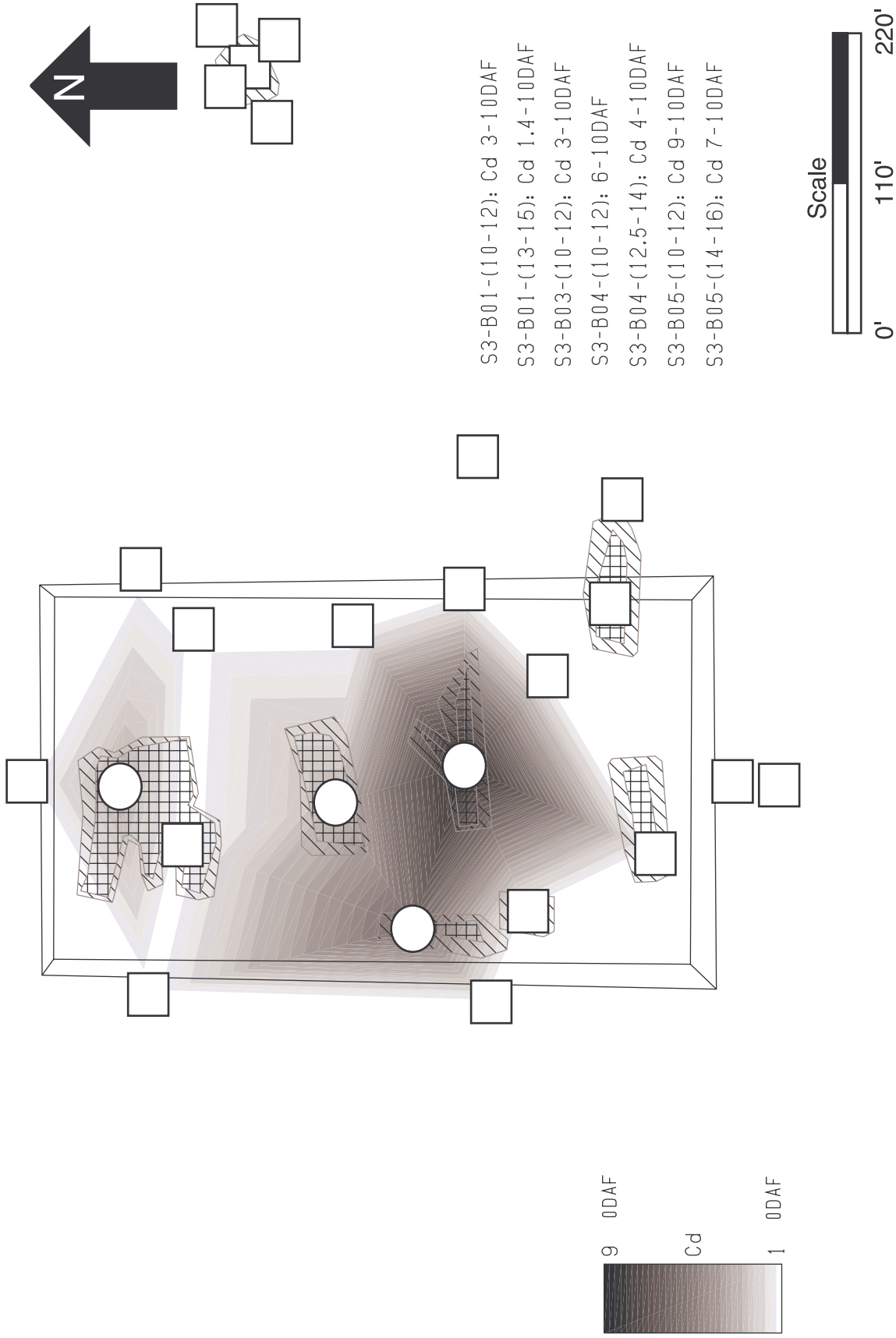


Figure 9f.

Metals Initially 53 samples were collected for this site. ICP/OES analysis was performed on 45 of these samples. Pit bottom concentrations for lead were less than 65-ppm and much less outside the pits. Cadmium concentrations at pit borings S3-B01, S3-B03, S3-B04, and S3-B05 were between 11-ppm and 36-ppm. Multiple samples at different depths above and below the pit bottoms were analyzed from each boring. Only at the pit bottoms were concentrations greater than 20DAF. To learn whether cadmium migration had occurred outside the pits at least one sample per boring was analyzed at the same depth cadmium was found inside the pit. The results of these analyses were ND, see Figure 9f. It appears that contamination is confined to the pit since non detectable levels were found outside the pit.

4.1.1 Summary of Findings

The adaptive sampling and analysis program provided information on a “real-time” basis to support on-site decision making. Both the screening and quantitative data met the data quality objectives of the project. Contaminated soil volumes were conservatively estimated for VOCs at each site. Quantitative data was used to determine which borings contained contamination above or below the action level. For each boring, the screening data were used to estimate the vertical distance between points of contamination and non measurable levels. (Recall that screening analyses were made at 1-ft intervals, where possible.) Thus, the x-z and x-y coordinates were determined by using a combination of quantitative and screening data. From this, contaminated soil volumes were estimated by linearly interpolating between soil concentrations above the action level and non measurable levels for each x-z and x-y coordinate. Approximately 28,000-ft³, 243,000-ft³, and 66,000-ft³ of soil are estimated to be contaminated for Sites 1, 2, and 3; see Appendix IV.

Screening data showed the presence of target compounds in three of 23 borings in Site 1 with quantitative data verifying the same three borings. Moreover, both screening and quantitative data for borings S1-B4 and S1-B17A revealed the same contamination profiles. Namely, the presence of some chlorinated solvents at high concentrations with low levels of some BTEX constituents. No detectable VOCs were found between the surface and 4-ft by either analysis for boring S1-B10A where as both techniques identified low levels of trichloroethene at the 20DAF concentration below 4-ft. No additional quantitative analysis was done since the screening GC/MS response indicated no detectable organics at depths below 6-ft.

Contaminants above 10DAF were found in seven of the eighteen borings in Site 2. Overall, BTEX was found at or near the water table in medium to high concentrations, with chlorinated solvents more evenly distributed but also more concentrated at the lower depths. For instance, for boring S2-B02 both screening and quantitative data indicated increasingly higher levels of chlorinated solvents from 14-ft and down as well as fairly high levels of toluene at 19-ft and deeper. For boring S2-B15 screening data for many of the 4-ft tube sections was not possible. Soil concentrations overloaded the Bruker MS, requiring a 15 to 30-min bakeout period before analyzing the next sample. Screening data, given to the quantitative field laboratory staff, provided a good estimate for sample dilution. This reduced the number of samples requiring re-analysis by quantitative GC/MS and, at the same time, protected the MS. Quantitative GC/MS analysis indicated percentage levels of trichloroethene in the soil. Data for boring S2-B15A, only a few feet away, showed elevated levels of cis-1,2-dichloroethene at 18 to 20-ft and non detectable levels of trichloroethene at all depths by both screening and quantitative analysis.

Of the twenty-five borings in Site 3, only four of them contained target compounds greater than their 10DAF concentration. BTEX compounds were found in borings S3-B01 and S3-B05, while chlorinated solvents were found in S3-B06 and S3-B08. The quantitative data confirmed the screening data.

4.1.2 Dynamic versus Traditional Site Investigation Costs

The rationale for conducting an adaptive sampling and analysis program is to obtain more information about the site while the technical team is in the field. This should lead to faster and better site characterizations at cheaper cost than the traditional approach. At Hanscom Field, VOCs are the primary contaminants that drive the risk assessment and operation of the ground water treatment facility. The dynamic investigation process resulted in 601 soil samples screened and 164 (158 soil and 6 ground water) site and QC samples quantitatively analyzed for VOCs. In addition, 69 and 121 site and QC soil samples were analyzed for PCBs/PAHs and metals, respectively. Soil samples were selectively chosen to test the initial conceptual model for each site in Operable Unit 1. The models were refined in the field as additional data were gathered.

The data produced in the field will be used to evaluate contaminant risk to ground water and what, if any, future action may be taken. HAFB also used the field data (soil, ground and well water) to help optimize VOC ground water collection into the treatment plant. The location of soil contaminated areas adjacent to wells containing high concentrations of VOCs (in some cases pure product) were identified. At Sites 2 and 3, larger capacity pumps were installed during December 1996 in selected wells to increase the flow rate (to 320-gal/min) into the treatment plant. Based on these modifications, influent concentrations increased from 500-ppb in August 1996 to 900-ppb one year later. The ground water recharge system was reactivated in July 1997. Initial checks at some of the wells now show VOC levels as high as 1,200-ppb. It is hoped that influent concentrations will reach levels of 2,000 to 3,000-ppb.²⁵

Comparisons of traditional and dynamic site investigation costs are shown in Table 4. Assume that in the traditional approach, cost estimate 1, the number of samples collected and the types of analyses performed are the same as in the dynamic site investigation. To reach the same end point, consider that the traditional field investigation is conducted in two phases: 1) a screening phase to determine extent and movement of contamination and 2) a more quantitative phase to determine risk to ground water. Off-site laboratory sample analysis costs are based on local laboratory pricing, with data turnaround times typically 2-4 weeks. Higher project costs can occur if samples are analyzed by laboratories with more national reach, presumably due to increased sales and marketing costs and the overhead associated with the wide range of state and federal certification programs (see Table 5).⁵ For phase 1, a total of 661 site and quality control samples are collected over the same 8-day period site samples were collected and screened in the dynamic investigation. Phase 2 begins after the screening data have returned from the laboratory and a new workplan has been prepared. Total project costs, \$142,176, include laboratory analysis sample charges for VOCs, PCBs, PAHs, and metals for site and QC samples as well as sample shipping charges (1,062 sample jars), field team remobilization, and sample collection. The cost for consulting services has been excluded in this analysis.

Table 4. Cost Comparison between Traditional and Dynamic Field Investigations

Estimate 1 Traditional Investigation Off-Site Analysis Data Turnaround 2-4 Weeks		Estimate 2 Traditional Investigation Off-Site Analysis Data Turnaround Two Days		Estimate 3 Dynamic Investigation On-Site Analysis Data Turnaround Next Day	
Predetermined VOC Screening Analysis 601 Site Samples 60 QC Samples	Cost 39,065 3,900	Directed VOC Screening Analysis 601 Site Samples 60 QC Samples	Cost 58,598 5,850	Directed VOC Screening Analysis 601 Site Samples	Cost 19,833
VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 19,750 2,000	VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 29,625 3,000	VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 15,800 1,600
PCB Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 6,800 700	PCB Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 10,200 1,050	PCB and PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 6,800 700
PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 9,860 1,015	PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 14,790 1,523		
Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 36,300 3,600	Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 54,450 5,400	Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 33,275 3,300
Analytical Cost Mobilization Cost Remob to Collect Quant Samples 3 Additional Field Days Sample Shipping Charge	\$122,990 \$5,000 \$5,000 \$6,000 \$3,186	Analytical Cost Mobilization Cost (50% surcharge) 11 Additional Field Days Sample Shipping Charge	\$184,486 \$5,000 \$22,000 \$3,186	Analytical Cost Field Laboratory/Instrument Mobilization Cost	\$81,308 \$10,000
Total Project Cost	\$142,176	Total Project Cost	\$214,672	Total Project Cost	\$91,308

See Table 5 for laboratory and field methods and per analyte group sample analysis cost.

Table 5. Traditional versus Laboratory Sample Charges and Data Turnaround Times

Regional Laboratory	National Laboratory Contract Laboratory Program	Field TDGC/MS Data Turnaround: Quantitative Next Day Screening Same Day	Analyte
<p>Data Turnaround: 14 Calendar Days</p>	<p>Data Turnaround: 35 Calendar Days</p>		
<p><u>\$125/sample</u> SW 846 method 8260A 35-min/sample analysis</p> <p><u>\$65/sample</u> SW 846 method 3810 20-min/sample analysis</p>	<p><u>\$165/sample</u> SW 846 method 8260A 35-min/sample analysis</p> <p><u>\$65/sample</u> SW 846 method 3810 20-min/sample analysis</p>	<p><u>\$100/sample</u> modified SW 846 method 8260A 20-min/sample</p> <p><u>\$33/sample</u> rapid screen TDGC/MS 30-sec/sample analysis</p>	VOCs
<p><u>\$100/sample</u> SW 846 method 8080 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>	<p><u>\$150/sample</u> SW 846 method 8080 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>	<p><u>\$100/sample</u> modified SW 846 method 8270B 10-min per analysis; sample preparation 1-hr/batch of 20 samples</p>	PCBs
<p><u>\$145/sample</u> SW 846 method 8100/8310 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>	<p><u>\$255/sample</u> SW 846 method 8100/8310 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>		PAHs
<p><u>\$300/sample</u> SW 846 method 6010</p> <p>8-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>	<p><u>\$325/sample</u> SW 846 method 6010</p> <p>8-min/sample analysis; sample preparation 2-hr/batch of 20 samples</p>	<p><u>\$275/sample</u> modified SW 846 method 6010 8-min/sample analysis; sample preparation 1.5-hr/batch of 20 samples</p>	Metals

Notes: Laboratory costs can vary greatly. The volume of samples analyzed, whether they are received in bulk or over an extended period, the type of QC and documentation required, and the current workload of the laboratory greatly influences the sample analysis charge. Sample charges are based on quotes from commercial laboratories for the analysis of 30 samples delivered in one shipment.

The second cost estimate, \$214,672, is based on expediting the traditional site investigation process. A 50% surcharge is applied to obtain data 24-hours after the off-site laboratory receives the samples. With overnight shipping, data turnaround time is two days. To accomplish the adaptive sampling and analysis investigation in one field mobilization, 21-days will be needed to complete both the screening and quantitative analysis. When compared to the dynamic investigation (10-days), eleven additional days will be needed and has been included in the cost. Using a more conservative sample surcharge of 100% results in total project costs of \$276,166.

Cost estimate 3 is based on the dynamic workplan/adaptive sampling and analysis program. Total project costs, \$91,308, include charges for on-site analysis of VOCs, PCBs, PAHs, and metals, as well as field team, instruments, and laboratory mobilization. The dynamic site investigation takes advantage of the fact that any data surprises will be addressed by decisions made in the field. For instance, no PAH or PCB samples were initially scheduled for analysis at Sites 2 and 3, see Table 6. During VOC screening, it was noticed that the soil was contaminated by petroleum products. Samples from Sites 2 and 3 were subsequently analyzed for PCB/PAH until sufficient data suggested that these contaminants were generally well-below the site-specific action levels.

Total project cost savings between estimates 1 and 3 *and* between 2 and 3 are \$50,868 (36%) *and* \$123,364 (57%), respectively. On the one hand, on-site screening yields the greatest savings as a percent of the total project cost. On the other hand, the analysis of semivolatile organics by TDGC/MS with IFD data analysis produced the greatest per sample cost savings; namely, \$100/sample for PCB/PAH versus \$245/sample. Based on these assumptions, savings are accrued by implementing field analytics when on-site mobilization costs are compared against traditional costs for sample collection, sample shipping charges and the need for multiple mobilizations. It should be reemphasized that these costs exclude all additional consulting costs associated with multiple field investigations.

The IFD data analysis algorithm greatly increases sample analysis productivity when compared against other MS data analysis systems. Unlike standard methods that obtain compound selectivity by adjusting the chromatography to separate organics, selectivity is obtained through the software. IFD reduces the per sample analysis costs in two significant ways. First, the time required to prepare complex environmental samples for analysis is reduced. Second, the time of analysis is reduced by minimizing the reliance on chromatographic separation. In this project, PCBs and PAHs were detected in petroleum contaminated soils in ten minutes without the need for sample cleanup. These productivity gains result in more samples analyzed per day per instrument, while measurement sensitivity was obtained through TD sample introduction. Similarly, the field-practical microwave digestion method reduces sample preparation times over current procedures, while the modification to the ICP optical bench provided stable instrument response under adverse field conditions.

Finally, the on-site adaptive sampling and analysis program can better target areas shown to be contaminated. For example, target compounds were found in 121/158 samples (77%) selected for quantitative analysis. Although 37 samples did not contain measurable contaminants, they were selected to determine the extent of VOC contamination at each site. In this context, several points should be made. First, field analysis produced contaminants in five of fourteen (36%) samples while the off-site confirmatory samples only contained VOCs in two of the fourteen

Table 6. Dynamic Workplan Projected and Actual Number of Samples Analyzed

Type of Analysis	Site 1 Samples		Site 2 Samples		Site 3 Samples		Total Samples Analyzed	
	Projected	Actual	Projected	Actual	Projected	Actual	Projected	Actual
VOC Samples Screened	162	210	135	177	288	214	585	601
VOC Samples Quantified	42	51	36	58	59	49	137	158
PCB/PAH Samples Quantified	42	46	0	12	0	10	42	68
Metals Samples quantified	51	22	44	54	36	45	131	121

Sample number includes field duplicates.

samples (14%). Second, off-site laboratory concentrations were much lower than field results for the two samples where both laboratories did detect VOCs. This VOC loss by off-site laboratories is consistent with findings reported in the literature.^{17,26,27} Third, the adaptive sampling and analysis program can better address the scientific and engineering questions under investigation. For example, HAFB staff have modified the ground water collection system based on the data produced in the investigation. These improvements have increased the VOC influent concentrations into the treatment plant.

4.2 Field Analytical Results

The method data quality objectives listed in Tables 2 and 3 were established to demonstrate that field analytics can support the site-specific HAFB Operable Unit 1 field investigation objectives; namely, to determine future vadose zone soil action (risk-analysis) and to decide whether improvements to the ground water collection system can be made for these sites. Field measurement performance including method selectivity, sensitivity, precision, and accuracy is presented below. Table 7 summarizes these findings, with all supporting data reported in Appendix V.

4.2.1 Evaluation of Screening TDGC/MS Data

The method used in the HAFB dynamic site investigation was developed at Tufts University. The mass spectrometer was operated in the direct measuring mode, with three fragment ions per target compound monitored simultaneously for identification purposes. A total of eleven compounds was screened in less than 30-sec. The TDGC probe head was placed over a small hole made in the Geoprobe™ sleeve at 1-ft intervals. The relative target compound signal heights were recorded at each hole. At the beginning of each day, and when the analyst determined it was needed, a known standard of VOCs was injected into the sleeve to approximate a 10-ppb/compound standard. Samples were not analyzed unless signal responses were ± 2 log units. The direct measuring TDGC/MS, operated in this manner, provided instantaneous and selective detection of each target compound. Whenever a highly contaminated soil was analyzed, the analyst waited until all MS targeted fragment ion signals returned to baseline before analyzing the next sample. This practice insured no carry-over between samples.

Over an eight-day period, 601 individual soil samples were screened at approximately 1-ft intervals. Based on these results, quantitative VOC measurements were made on 144 2-ft composite soil samples. Each 2-ft section of sleeve was cut lengthwise, with soil collected over the tube length and placed into sample collection jars. Samples for quantitative VOC analysis were collected first and were not homogenized. The difference between 158 total soil samples analyzed and the 144 site samples analyzed is due to the number of QC samples. Once the VOC sample had been collected, the remaining sleeve soil was put into a cleaned mixing pan and homogenized for PCB/PAH and metals analysis.

Whenever the screening results indicated the presence or absence of VOCs, quantitative GC/MS confirmed this finding at the measurement reporting limit at a confirmation rate of 90% (142/158 samples). For six of the samples, screening results indicated a positive response while

Table 7. Performance Measurements Obtained in the Field for HAFB

Performance Measurement	VOC Screening by TDGC/MS	Quantitative VOC Analysis by Purge & Trap GC/MS Modified Method 8260A	Quantitative PCB/PAH Analysis by TDGC/MS Modified Method 8270B	Quantitative Metals Analysis by ICP/OES Modified Method 6010
Sensitivity	± 2 log units, daily standard at 10-ppb	3-ppb to 33-ppb, see Table 12 MDL study	total PCBs 200-ppb; PAHs 100-ppb to 400-ppb, see Table 13 MDL study	0.3 to 40-ppm, see Tables 31 and 32 for MDL study
Selectivity	11 target compounds, 3 ions/compound monitored simultaneously	18 target compounds, preselected retention windows and extraction of 3 ions/compound	PCB detection by homologs, 16 target PAHs speciated, preselected retention windows and extraction of 3 ions/compound	22 metals, preselected wavelength for each metal
Precision	± 2 log units, no duplicate analyses performed	average RPD 40 ± 25%, see Table 14	samples selected for replicate analysis contained no measurable concentrations	ERA WastWatR #9967 10% RSD, Table 18; ERA T/CLP Soil #228 3% to 15% RSD, Table 19; site triplicate samples ave RSD 16±15%, Table 22
Accuracy	no sample fortification	surrogate, 83% within DQO, Section 4.2.2; field vs. lab comparison, field measured concentrations greater than off-site analysis, see Table 15	surrogates, 83% within DQO, Section 4.2.2; field vs. lab comparison, only one sample selected for analysis contained reportable concentrations	ERA T/CLP Soil #228 metal dependent average recovery 103±37%, Table 19; field vs laboratory average percent difference 32 ± 15%, Table 21
Other	carryover minimized by on screen baseline monitoring	method blanks made after analysis of high concentration samples	method blanks made after analysis of high concentration samples	method blanks and laboratory control check samples run every 20 th sample

quantitative GC/MS indicated no measurable concentrations at the reporting limit (4% false positive) and the converse (6% false negative) for the remaining ten samples. At the 10-ppb level, the concentration for which the screening TDGC/MS was calibrated, the confirmation rate was 86% (136/158) with 3% false positive and 11% false negative. Finally, if yes/no detection is the benchmark for comparison, a 76% (120/158) confirmation rate is obtained, with 1% false positive and 23% false negative responses. This trend is what should be expected; namely, that quantitative GC/MS is more sensitive than screening MS. Moreover, rapid screening GC/MS measurements were made over a hole the size of a nickel as compared to the composite sampling where samples were collected over a 2-ft section of the sleeve. From the composite sample, a 5-g quantity of soil was analyzed. Comparing measurement accuracy other than by false positives or negatives for discrete and composite soils is nonsensical. It is evident that the rapid screening TDGC/MS method provided an excellent guide to determine where samples should be collected, what VOCs were present, and their relative concentrations in the sample.

4.2.2 Evaluation of Quantitative GC/MS Data

The VOC site maps were produced in the field from data obtained by a standard laboratory purge and trap GC/MS system. The semivolatile data were produced by a Tufts modified TDGC/MS system. All samples were analyzed by full scan mass spectrometry detection, with the data analyzed by the IFD software. To determine sources of deviation, the IFD data generation software was evaluated. Results were consistent with HP's EnviroQuant data analysis software for standard solutions and site samples where the matrix posed little or no data interpretation problems. When high level matrix interferences were present, IFD detected target compounds and internal standards more easily than did EnviroQuant, see Section 5. Based on the results presented below, deviations from the DQO's were in the data generation process, i.e., instrument and human error, and not in the IFD data analysis software.

Initial and Continuing Calibration Comparison Study IFD was used throughout the study to identify and calculate the line parameters of each compound from the total ion current chromatography data. Table 8 lists each VOC's initial 5-point calibration linear regression parameters as well as the average response factor (RF_{cal}) and relative standard deviation (%RSD). The %RSD's were well within the field criterion of $\leq 30\%$ for two-thirds and $\leq 40\%$ for the remaining one-third of the target compounds. VOCs were typically within the DQO's required of fixed-based laboratory data. Shown in Table 9 are the initial and continuing calibration response factor percent differences for the VOCs. The field data meets the data quality objective established for this study with one exception. Vinyl chloride yielded $> 40\%$ differences one-half of the time. It was the only target compound that exists as a gas at ambient temperature. Despite handling the VOC standards with care, we were unable to meet the DQO for this compound in the field.

The DQO's were met for both the initial and continuing PCB/PAH RF calibrations, see Tables 10 and 11. The TD initially installed for semivolatile analysis broke during the first day of the investigation. Carrier gas flow lines developed a leak at one of the solder joints resulting in loss of signal detection. A second TD unit was installed which also developed the same type of leak. Carrier gas line problems were identified by the decreasing internal standard MS signal. This is evident by the poor instrument performance demonstrated during the last RF calibration

of each initial calibration. Nine site samples were not bracketed by an end of day continuing RF calibration verification. Although the TDGC/MS produced lower than normal internal standard responses for six of the nine site samples, none of them showed measurable PAH/PCB signals above the site-specific reporting limits. At the conclusion of the site investigation, the TD was redesigned to minimize manual operation of the sample introduction valve, see Section 5.

Method Detection Limit Study MDLs for VOCs were produced using the purge and trap GC/MS. VOC concentrations were based on an attempt (trade off) to meet the quantitation limit. The lowest concentration where vinyl chloride was observable in the field was 20-ppb. Seven different aliquots (n=7) were prepared and analyzed at this concentration. Table 12 lists the results along with the QL (10DAF) and reporting limit. Each sample measurement, average concentration, standard deviation, and %RSD are shown in the table. All compounds were detected at concentrations below the action level (2 x QL) except for vinyl chloride, methylene chloride, and 1,2-dichloroethane. The poor vinyl chloride measurement precision, 33% RSD, yielded MDL concentrations that exceeded the 5-ppb quantitation limit.

The short timeframe between mobile laboratory setup, the rate of site sample collection, and problems with the TD resulted in the MDL study being performed at the end of the field investigation. By that time, the air in the GC/MS trailer had become contaminated with methylene chloride from the soil/solvent extraction procedure used in the PCB/PAH method. Near constant methylene chloride concentrations were found in the sample data over time. We informed EPA's Office of Environmental Measurement and Evaluation (OEME) staff that the methylene chloride data had become compromised. After discussion, it was agreed that methylene chloride should be taken out of the target compound list.

One lesson learned is that better laboratory ventilation is needed for future field work to reduce solvent contamination of the mobile laboratory from the sample preparation procedure. Another lesson learned is that obtaining such low limits of detection for the VOC gases in the field may not be achievable. Therefore, it is better to insure that the DQO's are met for all other VOCs rather than compromising on data quality. For example, conducting the MDL study at 10-ppb would have resulted in meeting the QL for 1,2-dichloroethane. Visual inspection of the 1,2-dichloroethane peak signal showed that under the experimental conditions employed, lower measured concentrations could have been achieved.

For PAH and PCBs, the MDL study was conducted prior to the initial calibration. MDL's were determined by taking 2-g of ERA soil previously analyzed and shown to contain no detectable organics. The soil was added to a 7-ml vial and fortified to contain 300-ppb of each PAH and 300-ppb of Aroclor 1248. The soil was extracted by hand-shaking with 2-ml methylene chloride. The soil/extract was centrifuged for 3-min and the extract placed into a 2-ml sealed vial. Seven soil samples were extracted and analyzed by TDGC/MS. Table 13 lists the peak areas, average area, standard deviation, and %RSD as well as the MDL and QL.

Benzo(a)pyrene and dibenzo(a,h)anthracene have the lowest PAH action level (20DAF) concentrations, viz., 2-ppm each. All other PAH 20DAF concentrations are \geq 5-ppm, with quantitation limits \geq 2.5-ppm. The site-specific QL for total PCBs was set at 0.5-ppm. The measured MDL's for PAH were between 87-ppb and 374-ppb. The MDL for total PCB was 153-ppb. TDGC/MS measurements easily met the MDL DQO's for this project. Moreover, PCB and

PAH were detected in the presence of each other in one 10-min analysis, with measured MDL concentrations below typical laboratory detection limits. Standard laboratory methods generally require two different analyses; GC/MS for PAHs (method 8270B) and GC with electron capture detection (ECD) for PCBs (method 8080A). The estimated quantitation limit for these methods with gel permeation chromatography cleanup of soil extracts is ~ 660-ppb in soil. Because the TD can introduce volumes of up to 500-uL of extract, standard syringe injection of 1 to 2-uL volumes require 10-g of sample compared to the 2-grams used in this method.

The IFD software allows the mass spectrometer to operate as a universal detector and, at the same time, provide selective data analysis capability. Although MS instruments can provide increased sensitivity when operated in the selective ion monitoring (SIM) mode, the universality of full total ion current mass spectrometry is lost when library matching (compound identification) of nontargeted compounds is needed. Sample concentrations were reported and flagged (J) whenever the measured concentrations were between the MDL and RL (the low calibration standard).

Measurement Precision The sampling and analysis plan required field duplicate (FD) samples to be collected every twentieth sample. Seven samples were collected and analyzed as field duplicates. These samples were obtained by taking soil from the same 2-ft section of sleeve as the initial site sample. One site sample was analyzed as a triplicate. Initial and field duplicate samples were not typically analyzed on the same day. The goal was to analyze as many unique site samples as quickly as possible to confirm the screening results and to direct the final round of sample collection. Measurement precision results are shown in Table 14. Field replicate samples were run either as duplicates or triplicates. All positive responses are shown. (Benzene produced low level background signals at the MDL.) Relative percent differences (RPD) and RSD are reported when multiple samples from the same container were analyzed two or three times, respectively.

Unfortunately, all but two samples analyzed multiple times contained compounds at trace levels. The DQO measurement precision was $< 5 \times \text{QL}$, $\text{RPD} < 100\%$ and at $> 5 \times \text{QL}$, $\text{RPD} < 60\%$, see Table 2. For samples where target compounds were found in all replicates, the measurement precision DQO was met in every case. In four instances where measurable quantities were obtained in one replicate but not the other, concentrations were well below each target compound's quantitation limit. These results are consistent with past data where concentration levels are at the detection limit.

Measurement precision is highly dependent on the variance in sample homogeneity. Screening data showed that there can be great differences in concentration from one foot to the next within the same 4-ft sleeve. Since initial and field duplicate samples are collected by scooping soil over a 2-ft section, the differences in the initial and field duplicate VOC concentrations may be attributable to sampling and the loss of analyte by the time FD samples were analyzed. Another lesson learned was that preselecting the samples for determining measurement precision and accuracy falls in much the same category as prespecifying the samples to be collected in the traditional investigation process, i.e., many data are generated with little information value produced.

Measurement Accuracy During the planning session it was agreed that matrix spike experiments were not needed as part of the site-specific DQO's. Surrogates were added to VOC and semivolatile site samples. Known and measured surrogate concentrations were compared. For VOCs, 158 site samples were analyzed. The average surrogate recovery, either 4-bromofluorobenzene or 1,4-difluorobenzene, was $132 \pm 44\%$, with 82% of the analyses within the data quality objective, see Table 2. After data review, it appears that 4-bromofluorobenzene yielded recoveries ($87 \pm 40\%$) closer to the fortified value than did 1,4-difluorobenzene. The 4-bromofluorobenzene measured concentration was within the DQO 92% of the time. The average semivolatile surrogate (octachloronaphthalene) recovery was $94 \pm 49\%$, with 92% of the analysis within the data quality objective. The mass range limitation of the HP GCD mass spectrometer to 425 amu resulted in the selection of secondary ions used to quantitate octachloronaphthalene resulting in poor signal sensitivity.

Field versus Laboratory Comparison Study The dynamic workplan for Operable Unit 1 called for the fifth sample and every tenth subsequent sample to be sent off-site for laboratory analysis. A total of fourteen samples was analyzed by the two laboratories for VOCs and five samples for PCB/PAH. Analysis of the samples listed below produced no reportable VOC levels for the target compounds by both laboratories: S1-B2-(2-4), S1-B8-(6-8), S1-B17-2-(10-12); S2-B6-(2-4), S2-B10-(10-12), S2-B12-(12-14), S2-B15A-(10-12); S3-B6-(10-12), S3-B14-(6-8). Field and laboratory results where one of the laboratories obtained reportable data for VOCs are listed in Table 15.

In no instance did the off-site laboratory find target compounds where field analysis did not. For samples where either the matrix (sample interferences) or target compounds were low in concentration, i.e., no dilution of sample was required, field analysis found low levels of VOCs where the off-site laboratory did not. It has been well documented in the literature that VOCs are lost in transport and/or storage by the time off-site laboratories analyze the sample.^{25, 26}

In addition to potential VOC losses, samples S2-B2-(20-22) and S3-B1-(13-15) required 5:1 and 50:1 dilutions, respectively, before analysis by the off-site laboratory. The IFD software minimizes the masking of low concentration target compounds by high concentrations of matrix interferences and/or other target compounds. The data analysis software can "look-through" non target ion signals and determine compound identity unambiguously. Field analyzed samples were only diluted when target compounds overloaded the MS detector. The project objectives allowed for samples to be quantitated outside the initial calibration range for the field analyses. This is best illustrated by the field and laboratory results for S2-B2-(20-22) where 1,1-dichloroethene and 1,1-dichloroethane were diluted by the laboratory below the MDL and where cis-1,2-dichloroethene was masked by a matrix interferant. Although there is very little positive response data to compare, whenever field analysis produced a positive identification at high target compound concentrations so too did the off-site laboratory.

4.2.3 Metals Analysis by ICP/OES

ICP/OES Initial and Continuing Calibration Comparison Study ICP/OES instrument manufacturers typically require laboratory environments of ± 5 °C to insure instrument performance. This is sometimes difficult to achieve in the field. Therefore, a site-specific

instrument performance DQO of $\pm 20\%$ was established. Although lead and cadmium were the target analytes of concern for the HAFB field investigation, initial calibration plots were made on a daily basis for 22 inorganics. Tables 16 and 17 show the average percent recoveries and their %RSD's obtained over a 7-day period for both the initial and continuing calibrations, respectively. The average percent recoveries for both the initial and continuing calibrations were well-within $\pm 20\%$ for all elements including silver and antimony. The 25% 3:2 HNO₃:HCl mixture produces a more stable environment for these two metals than the standard EPA method using 5-10% HNO₃ solution.

Measurement Accuracy Table 18 lists the ICP/OES limit of quantitation (LOQ), certified value, and percent recoveries (%R) for an ERA, WasteWatR #9967, laboratory control check standard. This sample was analyzed at the beginning and end of each day. The MDL for lead (5-ppm) and cadmium (0.33-ppm) is the LOQ x 43.28, i.e., the weight factor of the acid digestion solution. The percent recovery data for this sample are shown in Table 18. The average percent recovery (%R) between the measured and certified values was within $\pm 20\%$, with the %RSD generally $< 10\%$. A second laboratory control check sample, PriorityPollutnT/CLP Soil # 228 also purchased from ERA, was analyzed every 20th sample. The average percent recovery for nine measurements over a 7-day period is shown in Table 19. Measurement precision was excellent with recoveries closer to the made-to concentrations than the certified value. The probable difference in accuracy is in the extraction procedure used to determine the certified value. EPA method 3050 is an open hot plate acid digestion procedure as opposed to the field microwave digestion method, see Section 5.

Matrix Interferant Test Solution A standard solution (A) containing calcium, magnesium, aluminum, and iron was used as the interferant solution. A second solution (Sol AB) containing these inorganics as well as twelve other constituents were analyzed. The average percent recoveries for the combined standard were within $\pm 10\%$ of the certified value, see Table 20.

Field versus Laboratory Results Comparison of field versus laboratory data for lead and cadmium has been tabulated in Table 21. The percent difference was calculated by dividing the difference between the field and laboratory concentrations by the mean value of the two. Excellent agreement was found between the field and laboratory results. Soil from sample S2-B2-(20-22) was prepared for analysis three times and found to contain lead at 16-ppm $\pm 4\%$ RSD. Replicate analysis for this sample was consistent with measurement precision results produced at the conclusion of the HAFB investigation, see Section 5.

Measurement Precision In addition to S2-B2-(20-22), nine other samples collected from the site were prepared and analyzed three times. These samples were selected randomly from each batch of twenty samples. Table 22 lists the results for cadmium and lead as well as the other inorganics. In general, the average concentration %RSD's were less than 30% for each element except for sample S2-B4-(19-21), which produced values as high as 70% for six of the seventeen inorganics present in the sample. These results are remarkable given the heterogeneous nature of the soil and the high degree of petroleum contamination in some samples; 128 of the 135 detectable responses (95%) produced RSD's $< 30\%$.

4.2.4 Summary of Findings: Field Analytical Instrument Performance

The data produced in the field met the DQO's established for this project. Where site geology is amenable, the Geoprobe™ can collect soil samples much faster than the time required to screen and process each 4-ft sleeve, including:

- tube preparation and TDGC/MS screening time
- the preparation of samples for subsequent quantitative field and off-site (laboratory) analysis for VOCs, PCBs, PAHs, and metals
- cleanup of the work area prior to analyzing the next tube
- completion of chain-of-custody forms, and
- geological logging of soil sample.

Nonetheless, one analyst processed 75 samples per day. The total cycle time for each 4-ft sleeve was approximately 20-min. Focusing only on sample screening analysis rates without careful planning and on-site decision making can lead to over collection of samples and the loss of VOCs before quantitative measurements can be made. Based on the chain-of-custody, 66% of the sleeves were screened for VOCs the same day they were collected, with the remaining sleeves analyzed within a 24-hr period. Samples were selected for quantitative VOC analysis on a daily basis to delineate contamination based on the screening data. All soil samples were quantitatively analyzed within the 14-day holding period. Most samples were analyzed within 5-days of collection or in the first week of the investigation. These were generally pit and surrounding samples. Longer holding time samples were typically collected from the boundary of the contaminated areas. These samples contained no measurable VOCs as confirmed by quantitative GC/MS at reporting level concentrations.

<u>From Date of collection</u>	<u>Cumulative Daily Analysis</u>	<u>From Date of Collection</u>	<u>Cumulative Daily Analysis</u>
same day	14%		
1	35%	6	87%
2	48%	7	91%
3	59%	8	96%
4	74%	9	98%
5	82%	10	100%

PCB, PAH, and metal samples were also analyzed within sample holding times. The combination of TD sample introduction and the mass spectral algorithms developed for both the Bruker and HP mass spectrometers provided the necessary sensitivity and selectivity for compound speciation under fast GC operating conditions. The modification made to the ICP/OES resulted in a stable instrument capable of providing quantitative data in the field. The data produced in this project was in conformance with the site-specific sensitivity, precision, and accuracy DQO's.

Although enzyme immunoassay kits were originally proposed, field analysis found PAH in only six samples and PCBs in only three samples. All sample concentrations were below typical enzyme kit detection limits. The original proposal also included in situ organics detection using a cone penetrometer. Studies performed by Applied Research Associates (ARA), Fugro, and CFAST staff showed that the HAFB site was not amenable to cone penetrometer detection. For example, ARA snapped the cone tip and bent the pipe in an attempt to collect ground water during an Air Force/Tufts University demonstration at HAFB.

Table 23. Screening VOC Analysis of Soil Samples

QC Parameters	Field Method TDGC/MS Screening Analysis	Field Method Heated Headspace GC Screening Analysis Method 3810
Sensitivity	±2 log units daily standard at 10 ppb	1 ppm standard adjust to > 2 times background
Selectivity	48 ions can be monitored simultaneously, 16 target compounds can be preselected if 3 ions/compound are monitored	adjust chromatography to separate VOCs of interest
Precision	no duplicate analyses performed	no data available
Accuracy	no fortified analyses performed	no data available
Other	carryover minimized by on screen baseline monitoring	carryover monitored by analysis of blanks, watch baseline on chromatograms

5.0 Technology Transfer

Over the past decade, research has led to field-practical TDGC/MS methods of analysis for organic compounds. In addition, field-practical sample preparation procedures for the digestion of metals from soils have also been developed, with modifications made to the ICP/OES instrument for field application. Tables 23-26 were developed as an outgrowth of this project to illustrate current SW 846 QC requirements for volatile and semivolatile organics and metals analysis and their corresponding field methods. The purpose of these tables is to show that field analytical technologies can produce data quality equal to standard laboratory instrumentation and methods.

Table 24. VOC Analysis of Soil Samples by Purge and Trap GC/MS

QC Parameters	Field Method SW-846 Modified Method 8260A	Laboratory Analysis SW-846 Method 8260A
Instrument Performance Tests for MS Tuning	perform check as per instrumental method, minimum requirement once to initiate shift	perform check as per instrumental method, minimum requirement once to initiate 12-hr shift
Initial Calibration 5-point	DQO dependent; can match SW 846 or all RF %RSDs \leq 40% and no more than $\frac{1}{3}$ $>$ 30% or all RF %RSDs \leq 30%	calibration check compounds (CCC) %RSD's must be $<$ 30%, if all RF %RSD \leq 15% then use Ave. RF else use linear regression
Laboratory Control Standard	sample throughput dependent, can match SW 846	after each initial calibration; percent accuracy within 80% to 120%
Continuing Calibration Verification	DQO dependent; can match SW 846 or begin & end of day, percent difference for all compounds \leq 40% and no more than $\frac{1}{3}$ $>$ 30%	one per 12-hr shift; (calibration check compound) CCCs $<$ 20%. All analytes within \pm 25% of expected value
Method Blank	once per day and after highly contaminated sample; all target compound concentrations $<$ PQL	one per analytical batch; all target compound concentrations $<$ PQL
Surrogate Spike Analysis	DQO sample throughput dependent; for each sample, blank, standard or other QC run	for each sample, blank, standard or other QC run, laboratory established recovery limits (e.g. 80-130 %)
Sensitivity	5-2500 ppb levels, matrix dependent	5-2500 ppb levels, matrix dependent
Selectivity	can do up to 97 VOCs with 2-6 ions per compound; minimal chromatographic separation employed, selectivity is achieved by data analysis algorithms	can do up to 97 VOCs with 1-6 ions per compound; adjust chromatography to separate VOCs of interest
Precision	replicate analysis QC acceptance criteria	replicate analysis QC acceptance criteria
Accuracy	sample throughput dependent; can match SW 846; laboratory control check sample (LCS) once per day	surrogate dependent recovery within 70-120%; laboratory control check sample (LCS) once per 12-hr shift
Other	carryover monitored by analysis of blanks, watch baseline on chromatograms	carryover monitored by analysis of blanks, watch baseline on chromatograms

Table 25. SVOC Analysis of Soil Samples by Thermal Desorption GC/MS

QC Parameters	Field Method SW-846 Modified Method 8270A	Laboratory Method SW-846 Method 8270B
Instrument Performance Tests for MS Tuning	perform check as per instrumental method, minimum requirement once to initiate shift	perform check as per instrumental method, minimum requirement once to initiate 12-hr shift
Initial Calibration 5-point	DQO dependent; can match SW 846 or all RF %RSDs \leq 40% and no more than $\frac{1}{3}$ $>$ 30%	calibration check compounds (CCC) %RSD's must be $<$ 30%, if all RF %RSD \leq 15% then use Ave. RF else use linear regression
Laboratory Control Standard	DQO sample throughput dependent; after each initial calibration, percent accuracy within 80% to 120%	after each initial calibration; percent accuracy within 80% to 120%
Continuing Calibration Verification	DQO dependent; can match SW 846 or begin & end of day, % difference for all compounds \leq 40% and no more than $\frac{1}{3}$ $>$ 30%	one per 12-hr shift; %D for all compounds \leq 20%
Method Blank	once per extraction batch; all target compound concentrations $<$ PQL	one per extraction batch; all target compound concentrations $<$ PQL
Surrogate Spike Analysis	sample throughput dependent; for each sample, blank, standard or other QC run	for each sample, blank, standard or other QC run, laboratory established recovery limits (e.g. 20-130 %)
Sensitivity	100-ppb to 1000-ppb	660-ppb to 3300-ppb
Selectivity	can do up to 350 SVOC with 2-6 ions per compound; minimal chromatographic separation employed, selectivity is achieved by data analysis algorithms	can do up to 350 SVOC with 2-5 ions per compound; adjust chromatography to separate SVOC of interest
Precision	replicate analysis QC acceptance criteria	replicate analysis QC acceptance criteria
Accuracy	sample throughput dependent; can match SW 846 for surrogate and MS/MSD recoveries	surrogate recovery compound dependent; MS/MSD per extraction batch
Other	carryover monitored by analysis of blanks, watch baseline on chromatograms	carryover monitored by analysis of blanks, watch baseline on chromatograms

Table 26. Metals Analysis of Soil Samples by ICP/OES

QC Parameter	Field Method SW 846 Modified Method 6010	Laboratory Method Superfund Hazardous Waste Analysis CLP SOW ILMO1.0
Initial Calibration	1 standard and a blank, daily, or every 24-hrs	1 standard and a blank, daily, or every 24-hrs
Calibration Verification	mid-range standard begin, end, and every 10 samples; recovery 80-120%	mid-range standard begin, end, and every 10 samples, or every 2-hrs; recovery 90-110%
Interference Check Sample	beginning of each run; recovery 80-120%	beginning and end of each run or 2 every 8-hrs; recovery 80-120%
Calibration Blanks	begin, end, and every 10 samples; all analytes \leq LOQ	begin, end, and every 10 samples or every 2-hrs; all analytes \leq CRDL
Preparation Blank	1 per SDG or digestion batch; all analytes \leq LOQ except Fe & Al	1 per SDG or digestion batch; all analytes $<$ CRDL
Laboratory Control Standard	Beginning and end of each run; recovery 80-120%	1 per SDG, digestion batch, or matrix; recovery 80-120%
Duplicate Samples	1 per SDG or every 20 samples; $<30\%$ RPD for values \geq LOQ flag value if out	5% or 1 per SDG per matrix per level ; $<20\%$ RPD for values $\geq 5 \times$ CRDL $\pm 1 \times$ CRDL for values $< 5 \times$ CRDL
Sensitivity	100-ppb to 10,000-ppm levels, matrix dependent	100-ppb to 10,000-ppm levels, matrix dependent
Selectivity	22 elements spectrally resolved with no need for interelement correction factors	22 elements mostly resolved with provisions for interelement correction (instrument dependent)
Precision	triplicate analysis 1 per SDG or every 20 samples; RSD $< 30\%$	replicate analysis QC acceptance
Accuracy	soil and standard 1 per SDG or every 20 samples; recovery 70-130% except Al, Fe, Na	no data available
Other	carryover monitored by sequential scans of rinse solution; rinse time between samples 3-min	no data available

5.1 Organics Analysis

Field methods have evolved from an instrument specifically designed for chemical warfare detection (Bruker Instruments) to more typical instruments found in the laboratory (Hewlett Packard). The Bruker GC/MS weighs about 400-lbs and has a footprint of 2-ft x 2-ft x 3-ft. It is field-rugged and was used in the Gulf war. In contrast, typical laboratory instruments are about one-half the size and weigh approximately 50-lbs. Two key technologies provide the core breakthrough for fast GC/MS analysis. First is the mass spectral data analysis software. The U.S. Patent Office (April 1997) issued a Notice of Allowance for all claims related to the mass spectral data interpretation patent. The software provides the unique capability of extracting between two and ten characteristic fragment ions produced in mass spectrometry from targeted organic compounds. Based on a set of mathematical algorithms, compound identity and concentration are determined. Although all MS systems provide MS ion extraction, they cannot determine compound presence using current statistical or library matching routines. The Tufts approach provides compound identification in complex environmental samples without the need for extensive sample cleanup. It is hoped that the underlying mathematical algorithms and process are amenable to other analytical instruments that produce characteristic signatures for targeted analytes like optical emission spectroscopy.

The second technology breakthrough is the thermal desorber (patent pending). Although we had some plumbing (gas leak) problems in the field during the investigation, these problems were fixed and the TD capability was demonstrated. Unlike other commercially available units, it can be ballistically heated from subambient temperatures to 320 °C in 8-sec. The TD uses a standard Tenax tube for VOCs. An empty glass tube is used for direct desorption of organics from solid materials or organic extracts. The latter sample introduction method was used in the HAFB field investigation for PCBs and PAHs.

To test the data generation software, comparisons were made between the EnviroQuant and IFD software systems. Also shown in Tables 8-11 are the initial and continuing calibration line parameters and %RSD RFcal *and* the percent difference as determined by the EnviroQuant software. Note that all GC/MS total ion current chromatograms were acquired by HP's data acquisition system. The same data files, therefore, were analyzed by both IFD and EnviroQuant. The tables reveal no statistical differences in the information obtained. Moreover, the %RSD's for the eighteen VOC RFcal's were well within the field criterion of $\leq 30\%$ for two-thirds of the target compounds and $\leq 40\%$ for the remaining one-third of the organics established for this field study. Good agreement was also found for PCBs and PAH except for naphthalene. EnviroQuant produced a 65% RSD RFcal in comparison to the 37% RSD calculated by IFD. Excellent agreement was also obtained between the two data analysis systems for the continuing calibration data. The VOC and semivolatiles data were typically within the 30% required of fixed-based laboratory data.

To determine instrument and method stability in the field, each sample's internal standard signal was compared against the corresponding average internal standard signal calculated over the first two calibration curves. The internal standard signal was inside the -50 to 150 percent difference HAFB DQO for 134 of 157 samples (85% success rate) analyzed by IFD. EnviroQuant fell outside of the DQO for 32 of the same samples, while missing the internal standard identity in two samples completely for a 78% success rate. Both data analysis systems fell out of QC because of coeluting matrix constituent(s) that affected the internal standard integration.

Table 27 shows IFD and EnviroQuant site sample data comparisons for the same calibration files where at least one of the data analysis systems identified a target compound above the reporting limit. Differences between the two calculated concentrations were due to each data system's peak identification and integration routines. For example, IFD provided better integration where matrix constituents coeluted with either target compounds or internal standards. EnviroQuant cut peaks in half depending upon where peak separation was determined by the software. Target compound concentrations in file #'s 100.D, 178.d, 179.D, and 194.D were reported by IFD whereas, EnviroQuant estimated their concentration below the reporting limit. In addition, EnviroQuant missed compounds in file #'s 102.D, 143.D, and 178.D that were well above the reporting limit, with IFD missing one compound in file # 031.D. Of the 205 reported compounds by both software systems, 85% had RPD's $\leq 50\%$ and 65% had RPD's $\leq 20\%$. This is remarkable given that both data systems identified peaks and integrated signals very differently.

The TD is temperature controlled to minimize introduction of matrix interferences into the GC/MS. It must be continually purged by an inert carrier gas such as helium to exclude oxygen from entering the GC/MS. The purpose is to minimize GC stationary phase degradation at elevated temperatures and to avoid filament burnout in the ionization chamber of the MS. Because of problems with the TD/manual injection valve construction (leaks), an electronically controlled and ballistically heated (ambient to 320 °C in 8-sec) TD unit was built at the conclusion of the HAFB field investigation. Reducing manual manipulation of the TD unit should result in less strain on the TD carrier gas flow lines. An evaluation of the purge and trap TD sample introduction system was made for VOCs. It included a Supelco 15-ml sparging vessel connected on one end to an inert gas supply and on the other end to a Tenax tube supplied by SKC, Inc. (Canonsburg, PA). A 5-min purge time for VOCs was used. The Tenax tube was placed in the TD, with VOCs desorbed onto the GC column. Table 28 shows the initial calibration results for the standard laboratory Tekmar and Tufts systems. The calculated Rfcal %RSD's were well below the DQO criteria. Table 29 shows the MDL results. The MDLs for all target compounds were below the quantitation limit, note that the lowest standard was 6.7-ppb as compared to 20-ppb in the HAFB field investigation.

Tekmar and Tufts purge and trap data comparison shown in Table 30 differed in the purge conditions employed and in the GC operating temperature. The same MS was used to acquire VOC data. HAFB site samples initially contained a wide range of target compound concentrations when analyzed in the field. Evident from these results was the dramatic loss of VOCs from the sample. RPDs were within the DQO's when both measurement techniques found contaminants. Consistent with the MDL study, it appears that the Tufts purge and trap/TD can provide lower measurement detection for VOCs conspicuous by the twelve detectable contaminants found by Tufts as opposed to Tekmar.

5.2 Metals Analysis

Table 31 lists the ICP/OES instrument detection limit study for the Target Analyte Metals (excluding mercury). Individual standards were prepared and analyzed seven times according to standard EPA procedures. Table 32 compares the calculated MDL and 10DAF concentrations for these metals. Note that the field instrument can achieve lower MDL's than required for the Hanscom project.

Table 16 lists the daily recoveries for the target analyte metals that can be measured by ICP/OES, while Table 17 shows the daily continuing calibration data. Percent recoveries were excellent for all metals, with 9% the largest deviation from 100% recovery. Table 19 illustrates the percent recovery for a standard reference soil (ERA Priority/PollutnT/CLP). The ERA sample, Soil #228, was analyzed every 20th sample. A total of nine samples was analyzed producing average percent recoveries much greater than 100% when compared against the certified value. The certified value represents the concentration obtained if the soil sample is prepared for analysis by EPA method 3051. The extraction procedure employed a 50% 3:2 HNO₃:HCl as opposed to concentrated HNO₃. Comparison of the data reveals that the average percent recoveries fall much closer to the theoretical (made-to) concentrations as prepared by ERA than they do to the certified values. The results show that greater recoveries are obtained for Na, Ag, Al, Fe, and Sb employing 50% 3:2 HNO₃:HCl as the digestate than in the standard EPA microwave digestion method. Importantly, the stability of all the metals in a single acid mixture (6-months) reduces the number of sample preparation steps to reconstitute antimony and silver from the standard EPA acid digestion solution (stability < 1-day).

Replicate analysis of the site samples shown below produced data consistent with measurement precision results found in the HAFB investigation when experiments were performed to evaluate the performance of the field sample preparation method (50% 3:2 HNO₃:HCl) and standardized EPA method 3501 (50% HNO₃).

	50% 3:2 HNO ₃ :HCl		50% HNO ₃		Number of Digestions
	Ave Pb (mg/kg)	% RSD	Ave Pb (mg/kg)	%RSD	
S2-B06-(2-4)	41	7	39	27	3
S2-B01-(16-18)	181	12	153	11	9
S3-B01-(13-15)	61	19	43	24	3
S3-B06-(10-12)	20	10	18	14	6

The two different microwave extraction procedures produced comparable data for lead. Recall that for S2-B06-(2-4), the commercial laboratory produced a concentration of 168-mg/kg as compared to 46-mg/kg (n=1). The off-site laboratory prepared soil samples by EPA Method 3050. In this method, the sample is refluxed in concentrated nitric acid and hydrochloric acid in an open vessel. It is a total digestion method that can produce greater analyte matrix dependent recovery for some metals over others. For lead, field versus laboratory percent differences were < 60%. Nonetheless, much debate exists in the literature over the benefits of microwave versus open vessel digestion.^{28,29}

X-ray Fluorescence (XRF) and ICP/OES Data Comparison Study At the conclusion of the field investigation, the Spectrace tabletop energy dispersive XRF was evaluated. Soil samples from the site were homogenized and directly placed into the XRF sample cup. Each sample was analyzed three times for lead. EDXRF results are compared against field and laboratory ICP/OES data for four samples.

	XRF (mg/kg) Ave Pb (%RSD)	field ICP/OES Pb (mg/kg)	off-site Lab Pb (mg/kg)	Total Ave Pb (%RSD)
S2-B01-(16-18)	128 (12)	194	222	181 (27)
S2-B06-(2-4)	39 (13)	46	168	84 (86)
S3-B01-(13-15)	57 (13)	65	76	66 (14)
S3-B06-(10-12)	24 (13)	19	29	24 (21)

Excellent measurement precision was obtained by the Spectrace instrument. In addition, the intermethod measurement accuracy was good for all samples except S2-B06-(2-4). Evident from the XRF and ICP/OES replicate analysis data is that the true lead concentration in the sample lies closer to 40-ppm than 170-ppm. The plot of EDXRF and ICP/OES data for 53 site samples is shown in Figure 10. Good correlation was obtained, with somewhat higher concentrations measured by EDXRF; $r^2 = 0.95$, slope 0.96 ± 0.03 and intercept 11 ± 2 . Only twelve site samples contained cadmium. The correlation of EDXRF with ICP/OES data was much poorer; $r^2 = 0.81$, slope 3.1 ± 0.5 and intercept -3 ± 8 . The Spectrace EDXRF with 200 livetime seconds can produce quantitative data in the field for lead and useful screening quality data for cadmium. At 120 livetime seconds the instrument continues to produce quantitative data for lead and screening data for cadmium. The shorter analysis time per element results in higher sample throughput rates. A 10-sample batch can be analyzed in 40-min for lead and cadmium. When choosing a field method, the microwave extraction and ICP/OES can quantitatively analyze a batch of ten samples for Target Analyte List metals except mercury in 125-min.

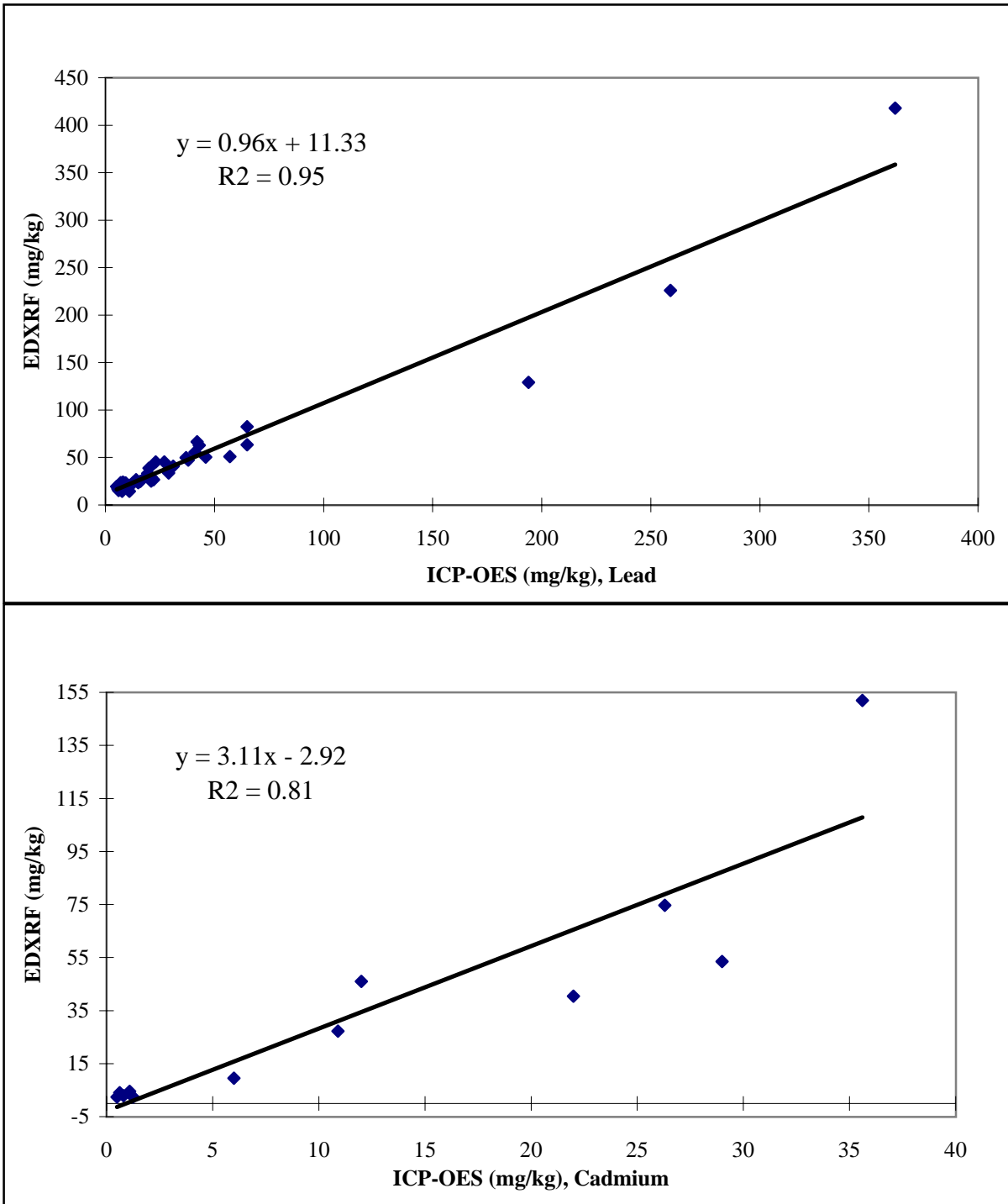


Figure 10 Plot of ICP/OES vs EDXRF for Lead and Cadmium

5.3 Lessons Learned

Summarized below are key findings learned while conducting the HAFB investigation.

Dynamic Workplans and Field Analytics

1. Field analytics employing performance-based methods can produce data of equal quality to fixed-based laboratories employing standardized EPA methods.
2. Field analytics can support a dynamic workplan/adaptive sampling and analysis program.
3. Field analytics can support risk assessments and cleanup verification programs.
4. Cost effectiveness is maximized when site DQO's, analytical throughput rates, data turnaround times, sample collection rates, and sample analysis costs are evaluated and optimized to meet the site-specific scientific and engineering questions under investigation prior to the beginning of the field work.
5. The technical team should be in daily communication, with one person assigned the responsibility for making all final field decisions.
6. Field analytics can result in a higher percentage of samples collected and analyzed containing target compounds when an adaptive sampling and analysis program is implemented compared to the phase engineering approach.
7. Preselecting samples to determine measurement precision and accuracy as in the traditional workplan concept provides minimum information value. The field team should select 5% to 10% of the site samples analyzed in the field. Based on actual results, 70% of these samples should contain target compounds. The remainder should be selected to insure that the boundary of contamination has been established.
8. VOC loss is less when field analytics is used to support the field investigation or cleanup verification program. The longer the holding time and lower the VOC concentration, the more accentuated the difference between on and off-site results.

Field Instrument and Method Performance

9. TDGC/MS and the mass spectrometry data analysis algorithms allow more samples to be analyzed per day than current MS vendor data analysis systems, probabilistic library sample identification matching routines, forward/backward regression search routines, or compound identification through the standard EPA/NIST library matching data systems.
10. The software algorithms can be used to obtain compound selectivity rather than adjusting the gas chromatography operating conditions. This decreases the per sample analysis time and increases the number of samples that can be analyzed per day per instrument over standard GC/MS instruments.

11. Software algorithms can “look through” non target MS ion signals and unambiguously determine compound identity, minimizing masking of low concentration target compounds by high concentration matrix interferents.
12. In this context, low level target compounds are not lost because of the need to dilute sample. Software makes sample dilution less necessary.
13. TDGC/MS provides increased method detection limits over standard syringe sample introduction techniques for GC/MS and comparable detection limits with GC/ECD without the need for a sample preconcentration step.
14. TDGC/MS and the mass spectrometry data analysis algorithms allow PCB/PAH analyses to be performed at the same time without the need for sample cleanup and fractionation time.
15. DQO’s can be met for all target compounds except vinyl chloride. A trade-off may need to be considered between achieving low limits of detection for VOC gases and meeting DQO’s for all other VOC target compounds.
16. ICP/OES modifications resulted in a stable instrument during field operation.
17. A 25% 3:2 HNO₃:HCL mixture produced a more stable environment for the digestion of all Target Analyte Metals with the exception of mercury for quantitative ICP/OES analysis.
18. A 25% 3:2 HNO₃:HCL produced a stable acid mixture (6-months) and high recoveries for silver and antimony as compared to the standard EPA digestion methods (1-2-days).
19. Microwave digestion is field-practical when compared to open vessel acid digestion, with recoveries comparable to what can be obtained in the laboratory.
20. The microwave digestion procedure reduces the number of sample preparation steps and thus time, compared to the standard EPA microwave digestion method.
21. Increased XRF sensitivity can be achieved with an x-ray tube source as opposed to radionuclide sources.

Mobile Laboratory Set-up and Operation

22. A minimum of one week is required to install and calibrate all field instruments. MDL study should be performed prior to beginning field work.
23. Depending on the number of field instruments, separate electrical services should be provided per instrument compliment.
24. Line voltage regulators are recommended to protect instruments and computers from line voltage surges or brownouts.

25. Instrument backup or service repair plan should be incorporated into the workplan. For example, the TD carrier gas leakage problem was addressed by using the Tekmar purge and trap system for the HAFB investigation. The new electrically controlled injection valve system was found to be more rugged than the manual valve unit.
26. Sample pretreatment for semivolatile samples should be separated from the VOC sample analysis area to eliminate sample cross-contamination during the sample extraction process.
27. Good ventilation is critical to prevent sample cross-contamination.
28. All instruments can be electronically linked to a data management computer system for ease of data review and site map generation.

6.0 Summary of ETI Goals and Objectives

The funding provided by the U.S. Environmental Protection Agency as part of President Clinton's goal to strengthen the capacity of technology developers and users to succeed in environmental innovation enabled Tufts University and its manufacturing partners, Hewlett Packard, Bruker Analytical Instruments, Leeman Laboratories, TN Spectrace, CEM, and SiteWorks, to demonstrate their innovative field analytical instrumentation and methods during an ongoing site investigation at Hanscom Air Force Base. More than 800 analyses were made during the investigation for VOCs, PCBs, PAHs, and metals. EPA verified the quality of data produced in this project by conducting field and laboratory audits. The quality of field data produced in this project will allow its use in determining contaminant risk to ground water. The data produced in this investigation has resulted in design changes to the ground water collection system. These changes have already increased the concentration of contaminated ground water into the treatment facility.

The HAFB site investigation employed a dynamic on-site decision making framework. The core technical team included staff from EPA Region I, Massachusetts Department of Environmental Protection, Hanscom Air Force Base, CH2MHill, CDM, and Tufts. The technical team participated in the creation of the dynamic workplan and in carrying out the field investigation. This process, from planning to implementation, was videotaped and provided the basis for a dynamic workplan guideline produced by Tufts University in cooperation with EPA Region I, Office of Site Remediation and Restoration *and* Office of Environmental Measurement and Evaluation.

The guideline, videotape, and HAFB report should provide users of environmental data with confidence that screening and quantitative data can be produced in the field and that the data generated will be accepted by the regulatory community. The success at HAFB should reduce regulator uncertainty as to the quality of data that can be produced with these innovative technologies and their cost competitiveness. Without regulatory acceptance, consulting and remediation companies will continue to be reluctant to purchase field analytical services for site investigation and cleanup projects. Analytical service providers will not purchase new instrumentation or technologies or adopt innovative methods of analysis unless their customers

(environmental consulting and remediation companies as well as federal and state regulators) request their usage. This will result in continued reluctance by instrument manufacturers to develop, license, manufacture, or market innovative field analytical instrumentation.

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Appendix I

This section contains the data that support the information provided in Table 7 as well as Sections 4 and 5 of this report.

TABLE 8 VOC 5-point Calibration Linear Regression Analysis and Average RF

Calibration Date: 8/21/1996

Compound	IFD Algorithm			EnviroQuant Software		
	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 101	RFcal ± %RSD	RFcal ± %RSD	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 101
1,1,1-Trichloroethane	11.3 ± 0.3	-2.41 ± 1.43	1.01 ± 8	1.05 ± 8	11.2 ± 0.3	-1.89 ± 1.28
1,1,-Dichloroethane	8.38 ± 0.31	-2.08 ± 1.29	0.73 ± 11	0.76 ± 9	8.27 ± 0.28	-1.72 ± 1.20
1,1-Dichloroethene	1.87 ± 0.03	-0.326 ± 0.120	0.17 ± 12	0.18 ± 5	1.86 ± 0.03	-0.26 ± 0.15
1,2-Dichloroethane	4.40 ± 0.14	-0.475 ± 0.580	0.43 ± 24	0.45 ± 27	4.21 ± 0.11	-0.06 ± 0.48
Benzene	24.9 ± 1.3	-10.1 ± 4.8	1.93 ± 16	1.99 ± 15	24.6 ± 1.2	-9.29 ± 4.72
Carbon Tetrachloride	13.7 ± 0.6	-4.91 ± 2.32	1.11 ± 13	1.12 ± 13	13.5 ± 0.6	-4.65 ± 2.30
Chlorobenzene	15.9 ± 0.7	-3.77 ± 2.98	1.41 ± 14	1.45 ± 15	15.7 ± 0.7	-3.27 ± 2.83
Chloroform	12.0 ± 0.4	-1.59 ± 1.49	1.16 ± 12	1.16 ± 12	11.9 ± 0.3	-1.43 ± 1.35
cis-1,2-dichloroethene	4.90 ± 0.19	-1.02 ± 0.80	0.46 ± 14	0.46 ± 13	4.87 ± 0.18	-0.955 ± 0.766
Ethylbenzene	20.8 ± 1.3	-8.29 ± 5.37	1.71 ± 18	1.70 ± 17	20.7 ± 1.2	-8.04 ± 5.14
m/p-Xylene	18.3 ± 1.2	-17.4 ± 10.1	1.42 ± 18	1.41 ± 18	18.0 ± 1.2	-16.8 ± 9.7
o-Xylene	20.3 ± 1.4	-12.4 ± 6.4	1.53 ± 22	1.53 ± 21	20.1 ± 1.4	-12.0 ± 6.1
Styrene	18.6 ± 1.3	-8.80 ± 5.66	1.49 ± 20	1.49 ± 19	18.4 ± 1.2	-8.44 ± 5.45
Tetrachloroethene	9.15 ± 0.37	-1.46 ± 1.55	0.86 ± 10	0.88 ± 13	9.07 ± 0.36	-1.24 ± 1.51
Toluene	16.9 ± 1.0	-9.00 ± 4.32	1.31 ± 20	1.40 ± 13	16.3 ± 0.8	-5.28 ± 3.49
trans-1,2-dichloroethene	2.30 ± 0.05	-0.176 ± 0.220	0.22 ± 6	0.22 ± 5	2.29 ± 0.06	-0.194 ± 0.245
Trichloroethene	6.56 ± 0.26	-1.33 ± 1.10	0.59 ± 9	0.61 ± 12	6.49 ± 0.26	-1.07 ± 1.07
Vinyl Chloride	0.812 ± 0.226	-0.492 ± 1.090	0.09 ± 29	0.10 ± 19	0.679 ± 0.233	1.51 ± 1.21
Summary:						
	≤ 30%		18	18		
	≤ 40%		0	0		
	> 40%		0	0		

TABLE 8 continued. VOC 5-point Calibration Linear Regression Analysis and Average RF

Calibration Date: 8/26/1996

Compound	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 101	RFcal ± %RSD
1,1,1-Trichloroethane	19.3 ± 1.2	-5.23 ± 4.97	1.73 ± 11
1,1,-Dichloroethane	15.4 ± 0.9	-3.87 ± 3.61	1.40 ± 13
1,1-Dichloroethene	2.74 ± 0.15	-0.630 ± 0.637	0.26 ± 22
1,2-Dichloroethane	8.22 ± 0.60	-1.39 ± 2.47	0.81 ± 21
Benzene	19.5 ± 5.6	25.6 ± 26.0	2.59 ± 24
Carbon Tetrachloride	10.2 ± 1.8	6.11 ± 8.92	1.15 ± 25
Chlorobenzene	20.9 ± 1.2	-5.28 ± 4.10	1.78 ± 12
Chloroform	23.9 ± 1.8	-7.88 ± 7.73	2.14 ± 13
cis-1,2-dichloroethene	8.64 ± 0.53	-1.78 ± 2.20	0.82 ± 13
Ethylbenzene	25.6 ± 1.5	-7.57 ± 5.08	2.10 ± 15
m/p-Xylene	22.0 ± 1.5	-15.3 ± 10.3	1.74 ± 17
o-Xylene	27.3 ± 2.4	-11.0 ± 7.9	1.96 ± 24
Styrene	31.3 ± 2.2	-18.5 ± 9.1	2.18 ± 25
Tetrachloroethene	8.03 ± 0.35	-2.12 ± 1.45	0.71 ± 11
Toluene	18.2 ± 0.9	-5.13 ± 3.15	1.48 ± 15
trans-1,2-dichloroethene	3.05 ± 0.16	-0.360 ± 0.655	0.30 ± 15
Trichloroethene	7.23 ± 0.30	-2.24 ± 1.24	0.60 ± 13
Vinyl Chloride	0.624 ± 0.093	-0.750 ± 0.448	0.03 ± 59
Summary:			
≤ 30%			17
≤ 40%			0
> 40%			1

TABLE 8 continued. VOC 5-point Calibration Linear Regression Analysis and Average RF

Calibration Date: 8/29/1996

Compound	(Slope \pm Std.Dev.) x 103	(Intercept \pm Error) x 101	RFcal \pm %RSD
1,1,1-Trichloroethane	12.8 \pm 0.3	-2.61 \pm 0.94	1.03 \pm 12
1,1,-Dichloroethane	10.5 \pm 0.3	-2.20 \pm 1.00	0.85 \pm 12
1,1-Dichloroethene	9.93 \pm 0.31	-2.30 \pm 1.01	0.78 \pm 12
1,2-Dichloroethane	4.20 \pm 0.04	-0.225 \pm 0.144	0.40 \pm 8
Benzene	38.5 \pm 2.1	-15.6 \pm 6.9	2.42 \pm 27
Carbon Tetrachloride	15.4 \pm 0.7	-2.89 \pm 1.17	1.18 \pm 14
Chlorobenzene	15.4 \pm 0.7	-5.84 \pm 2.30	0.99 \pm 26
Chloroform	11.5 \pm 0.2	-1.80 \pm 0.76	0.98 \pm 9
cis-1,2-dichloroethene	7.80 \pm 0.22	-1.56 \pm 0.72	0.64 \pm 11
Ethylbenzene	19.0 \pm 1.2	-5.00 \pm 2.14	1.28 \pm 21
m/p-Xylene	17.5 \pm 1.3	-13.2 \pm 5.1	1.08 \pm 27
o-Xylene	16.7 \pm 1.2	-6.18 \pm 2.30	1.03 \pm 27
Styrene	14.4 \pm 0.9	-4.10 \pm 1.54	0.92 \pm 25
Tetrachloroethene	12.4 \pm 0.6	-2.58 \pm 1.02	0.91 \pm 16
Toluene	15.9 \pm 0.8	-5.06 \pm 1.55	1.06 \pm 23
trans-1,2-dichloroethene	9.40 \pm 0.33	-2.13 \pm 1.09	0.76 \pm 11
Trichloroethene	9.20 \pm 0.29	-2.56 \pm 0.94	0.64 \pm 25
Vinyl Chloride	16.9 \pm 0.9	-4.03 \pm 1.52	1.15 \pm 25
Summary:			
	\leq 30%		18
	\leq 40%		0
	$>$ 40%		0

TABLE 9. VOC Comparison of Initial and Continuing Calibration RF Values

Calib. Date: 8/21/96

CCV Date:	8/21/96		08/22/96		08/22/96		08/23/96		08/24/96	
	IFD	ENVQ	IFD	ENVQ	IFD	ENVQ	IFD	ENVQ	IFD	ENVQ
Compound	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)
1,1,1-Trichloroethane	1.24 (23)	1.27 (26)	1.22 (21)	1.25 (24)	1.29 (28)	1.32 (31)	1.14 (13)	1.18 (16)	1.15 (15)	1.27 (26)
1,1-Dichloroethane	0.97 (33)	0.98 (34)	0.93 (27)	0.95 (30)	0.96 (32)	0.98 (34)	0.89 (22)	0.90 (23)	1.00 (37)	1.00 (38)
1,1-Dichloroethene	0.21 (29)	0.21 (24)	0.21 (27)	0.21 (24)	0.20 (22)	0.21 (24)	0.19 (17)	0.20 (16)	0.19 (18)	0.20 (17)
1,2-Dichloroethane	0.54 (25)	0.54 (26)	0.54 (25)	0.54 (26)	0.51 (17)	0.51 (19)	0.48 (10)	0.47 (10)	0.55 (27)	0.52 (21)
Benzene	2.52 (30)	2.53 (31)	2.42 (25)	2.43 (26)	2.37 (23)	2.38 (23)	2.22 (15)	2.24 (16)	2.39 (24)	2.38 (23)
Carbon Tetrachloride	1.37 (24)	1.38 (24)	1.28 (16)	1.30 (17)	1.24 (12)	1.24 (12)	1.17 (6)	1.18 (6)	1.24 (12)	1.24 (12)
Chlorobenzene	1.49 (5)	1.51 (7)	1.53 (8)	1.54 (9)	1.38 (3)	1.38 (2)	1.58 (12)	1.61 (14)	1.58 (12)	1.58 (12)
Chloroform	1.32 (14)	1.43 (23)	1.35 (16)	1.46 (26)	1.36 (18)	1.49 (28)	1.20 (4)	1.32 (14)	1.02 (12)	1.47 (27)
cis-1,2-Dichloroethene	0.56 (23)	0.57 (24)	0.54 (18)	0.54 (17)	0.57 (24)	0.57 (24)	0.51 (12)	0.52 (12)	0.54 (19)	0.56 (22)
Ethylbenzene	1.87 (9)	1.88 (10)	1.88 (10)	1.88 (10)	1.65 (3)	1.66 (3)	1.97 (15)	1.99 (16)	2.02 (18)	2.02 (18)
m/p-Xylene	1.58 (11)	1.58 (11)	1.57 (11)	1.57 (11)	1.33 (6)	1.33 (6)	1.60 (13)	1.61 (14)	1.71 (21)	1.70 (20)
o-Xylene	1.81 (19)	1.83 (20)	1.76 (15)	1.76 (15)	1.32 (13)	1.30 (15)	1.80 (18)	1.78 (17)	2.01 (32)	1.99 (30)
Styrene	1.74 (17)	1.75 (17)	1.72 (15)	1.73 (16)	1.29 (14)	1.29 (13)	1.77 (19)	1.79 (20)	1.91 (28)	1.91 (28)
Tetrachloroethene	0.87 (2)	0.88 (2)	0.81 (5)	0.82 (5)	0.81 (6)	0.81 (6)	0.87 (2)	0.88 (2)	0.85 (0)	0.85 (1)
Toluene	1.43 (9)	1.45 (11)	1.44 (10)	1.47 (12)	1.46 (11)	1.47 (12)	1.61 (23)	1.62 (24)	1.50 (14)	1.50 (15)
trans-1,2-Dichloroethene	0.27 (21)	0.27 (23)	0.25 (14)	0.25 (14)	0.27 (23)	0.27 (23)	0.22 (2)	0.23 (3)	0.22 (1)	0.23 (4)
Trichloroethene	0.61 (4)	0.61 (3)	0.65 (12)	0.66 (12)	0.64 (9)	0.65 (10)	0.64 (9)	0.65 (10)	0.59 (1)	0.59 (1)
Vinyl Chloride	0.05 (31)	0.06 (33)	0.11 (55)	0.11 (22)	0.02 (73)	0.02 (78)	0.09 (30)	0.10 (8)	0.04 (41)	0.06 (34)
Summary :										
≤ 30%	18	17	19	20	18	17	20	20	17	18
≤ 40%	2	3	0	0	1	2	0	0	2	2
> 40%	0	0	1	0	1	1	0	0	1	0

TABLE 9 continued. VOC Comparison of Initial and Continuing Calibration RF Values

Calibration Date: 8/26/96				Calibration Date: 8/29/96	
CCV Date: Compound	8/27/96 RF (%Diff.)	08/28/96 RF (%Diff.)	8/28/96 RF (%Diff.)	8/29/96 RF (%Diff.)	8/30/96 RF (%Diff.)
1,1,1-Trichloroethane	1.41 (19)	1.74 (0)	1.72 (1)	0.98 (4)	1.36 (32)
1,1-Dichloroethane	1.19 (15)	1.40 (0)	1.66 (18)	0.84 (1)	1.01 (19)
1,1-Dichloroethene	0.20 (23)	0.22 (15)	0.25 (6)	0.80 (2)	0.94 (21)
1,2-Dichloroethane	0.57 (29)	0.73 (10)	0.89 (9)	0.40 (0)	0.50 (23)
Benzene	2.67 (3)	3.06 (18)	3.41 (31)	2.53 (5)	3.13 (29)
Carbon Tetrachloride	1.36 (18)	1.57 (36)	1.56 (36)	1.37 (15)	1.52 (28)
Chlorobenzene	1.93 (8)	2.10 (18)	2.18 (22)	1.12 (14)	1.19 (21)
Chloroform	1.77 (17)	2.04 (5)	2.32 (9)	0.99 (1)	1.22 (24)
cis-1,2-Dichloroethene	0.61 (25)	0.82 (1)	0.89 (9)	0.62 (3)	0.77 (19)
Ethylbenzene	2.41 (15)	2.84 (35)	2.82 (35)	1.67 (30)	1.79 (39)
m/p-Xylene	2.02 (16)	2.26 (29)	2.35 (35)	1.46 (36)	1.49 (38)
o-Xylene	2.30 (17)	2.70 (38)	2.53 (29)	1.41 (37)	1.28 (23)
Styrene	2.18 (0)	2.59 (19)	2.77 (27)	1.28 (38)	1.27 (38)
Tetrachloroethene	0.78 (10)	0.95 (33)	0.87 (22)	1.16 (28)	1.17 (29)
Toluene	1.77 (20)	1.97 (33)	1.86 (25)	1.41 (33)	1.47 (39)
trans-1,2-Dichloroethene	0.25 (18)	0.24 (20)	0.27 (9)	0.75 (2)	0.95 (24)
Trichloroethene	0.69 (15)	0.74 (23)	1.32 (118)	0.69 (7)	0.76 (18)
Vinyl Chloride	0.02 (43)	0.05 (49)	0.08 (120)	1.46 (27)	1.10 (4)
Summary :					
≤ 30%	17	12	12	14	13
≤ 40%	0	5	4	4	5
> 40%	1	1	2	0	0

TABLE 10. SVOC 5-point Calibration Linear Regression Analysis and Average RF
Calibration Date: 9/4/1996

Compound	IFD Algorithm			EnviroQuant Software		
	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 101	RFcal ± %RSD	RFcal ± %RSD	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 101
Acenaphthene	4.16 ± 0.18	-0.537 ± 0.385	0.84 ± 14	0.84 ± 15	4.21 ± 0.18	-0.551 ± 0.387
Acenaphthylene	7.68 ± 0.53	-1.39 ± 1.15	1.55 ± 24	1.39 ± 22	7.81 ± 0.53	-1.50 ± 1.15
Anthracene/Phenanthrene	3.36 ± 0.13	-0.347 ± 0.549	0.81 ± 9	1.79 ± 27	5.58 ± 0.13	0.954 ± 0.577
Benzo(a)pyrene	6.01 ± 0.00	-0.654 ± 0.271	1.31 ± 11	1.36 ± 11	6.05 ± 0.13	-0.641 ± 0.326
Benzo(g,h,l)perylene	5.38 ± 0.23	-0.630 ± 0.502	1.14 ± 16	1.18 ± 19	5.41 ± 0.24	-0.623 ± 0.523
Benzo[a]anthracene/Chrysene	5.14 ± 0.05	-0.471 ± 0.200	1.55 ± 21	1.57 ± 18	5.20 ± 0.05	0.613 ± 0.197
Benzo[b]/(k)fluoranthene	11.6 ± 0.1	-0.053 ± 0.282	2.83 ± 10	1.41 ± 10	11.8 ± 0.1	-0.102 ± 0.263
Dibenz(a,h)anthracene	2.50 ± 0.57	1.16 ± 1.24	1.03 ± 27	1.17 ± 18	5.48 ± 0.15	-0.520 ± 0.335
Fluoranthene	5.77 ± 0.14	0.566 ± 0.304	1.94 ± 31	2.01 ± 40	5.83 ± 0.13	0.538 ± 0.276
Fluorene	4.24 ± 0.09	-0.102 ± 0.197	1.05 ± 9	1.03 ± 6	4.30 ± 0.08	-0.120 ± 0.182
Indeno(1,2,3-c,d)pyrene	3.56 ± 0.44	0.761 ± 0.948	1.18 ± 22	1.41 ± 19	6.63 ± 0.28	-0.788 ± 0.597
Naphthalene	2.63 ± 0.33	-0.194 ± 0.377	0.53 ± 37	0.58 ± 65	2.69 ± 0.32	-0.242 ± 0.366
Pyrene	6.04 ± 0.09	0.701 ± 0.207	1.89 ± 21	1.85 ± 19	6.00 ± 0.09	0.668 ± 0.217
Cl-1	1.65 ± 0.20	-0.063 ± 0.069	0.32 ± 30	0.39 ± 32	2.15 ± 0.13	-0.072 ± 0.046
Cl-2	1.88 ± 0.15	-0.037 ± 0.055	0.47 ± 27	0.37 ± 16	1.63 ± 0.11	-0.042 ± 0.040
Cl-3	1.49 ± 0.10	0.111 ± 0.036	0.58 ± 36	0.33 ± 12	1.30 ± 0.06	-0.013 ± 0.021
Cl-4	0.946 ± 0.033	-0.043 ± 0.025	0.20 ± 15	0.20 ± 10	0.845 ± 0.009	-0.019 ± 0.008
Cl-5	0.798 ± 0.018	-0.025 ± 0.013	0.17 ± 13	0.18 ± 11	0.695 ± 0.005	-0.004 ± 0.004
Cl-6	0.745 ± 0.012	-0.003 ± 0.014	0.17 ± 18	0.17 ± 8	0.644 ± 0.007	0.002 ± 0.006
Cl-7	0.659 ± 0.026	0.002 ± 0.028	0.16 ± 12	0.17 ± 8	0.608 ± 0.017	0.019 ± 0.018
Cl-8	0.338 ± 0.003	-0.012 ± 0.003	0.07 ± 16	0.07 ± 8	0.294 ± 0.005	-0.001 ± 0.005
Cl-10	0.352 ± 0.008	-0.037 ± 0.015	0.07 ± 21	0.06 ± 19	0.304 ± 0.008	-0.024 ± 0.014
Summary:						
	≤ 30%		19	19		
	≤ 40%		3	2		
	> 40%		0	1		

TABLE 10 continued. SVOC 5-point Calibration Linear Regression Analysis and Average RF
Calibration Date: 9/17/1996

Compound	(Slope ± Std.Dev.) x 103	(Intercept ± Error) x 102	RFcal ± %RSD
Acenaphthene	2.04 ± 0.19	-2.29 ± 3.18	0.45 ± 26
Acenaphthylene	4.14 ± 0.16	-1.09 ± 2.59	1.03 ± 12
Anthracene/Phenanthrene	8.04 ± 0.39	-10.9 ± 6.6	1.64 ± 18
Benzo(a)pyrene	1.41 ± 0.09	-0.406 ± 1.760	0.32 ± 26
Benzo(g,h,I)perylene	0.840 ± 0.057	0.200 ± 1.170	0.20 ± 25
Benzo[a]anthracene/Chrysene	3.29 ± 0.10	-9.35 ± 3.54	0.60 ± 28
Benzo[b]/(k)fluoranthene	1.92 ± 0.12	0.119 ± 4.520	0.45 ± 27
Dibenz(a,h)anthracene	1.23 ± 0.04	0.400 ± 0.696	0.31 ± 22
Fluoranthene	2.74 ± 0.11	-1.73 ± 1.06	0.59 ± 14
Fluorene	2.92 ± 0.14	-4.64 ± 2.44	0.55 ± 25
Indeno(1,2,3-c,d)pyrene	1.31 ± 0.06	0.426 ± 1.300	0.30 ± 30
Naphthalene	2.95 ± 0.12	-2.62 ± 2.27	0.66 ± 8
Pyrene	5.32 ± 0.26	-11.0 ± 4.9	0.93 ± 23
Cl-1	2.44 ± 0.16	-0.055 ± 0.267	0.59 ± 26
Cl-2	1.95 ± 0.18	-0.857 ± 0.689	0.34 ± 31
Cl-3	1.18 ± 0.06	-0.382 ± 0.195	0.21 ± 23
Cl-4	0.640 ± 0.035	-0.331 ± 0.125	0.11 ± 36
Cl-5	0.821 ± 0.100	-1.39 ± 0.77	0.12 ± 39
Cl-6	0.765 ± 0.088	-1.12 ± 0.76	0.12 ± 36
Cl-7	0.577 ± 0.034	-0.562 ± 0.173	0.08 ± 36
Cl-8	0.335 ± 0.020	-0.251 ± 0.118	0.06 ± 34
Cl-10	0.240 ± 0.013	-0.226 ± 0.254	0.05 ± 34
Summary:			
≤ 30%			15
≤ 40%			7
> 40%			0

TABLE 11. SVOC Comparison of Initial and Continuing Calibration RF Values

Calibration Date: 9/4/96

CCV Date:	9/4/96		9/5/96		9/5/96	
	IFD	ENV	IFD	ENV	IFD	ENV
Compound	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)
Acenaphthene	0.91 (9)	0.92 (10)	1.01 (20)	1.02 (22)	0.74 (12)	0.59 (29)
Acenaphthalene	1.45 (6)	1.47 (6)	1.62 (5)	1.64 (18)	1.02 (34)	1.20 (14)
Phenanthrene/Anthracene	1.58 (2)	1.59 (11)	1.71 (6)	1.72 (4)	1.28 (58)	1.83 (2)
Benzo[a]pyrene	1.38 (5)	1.38 (1)	1.18 (9)	1.23 (10)	1.05 (20)	0.84 (38)
Benzo[g,h,i]perylene	0.87 (24)	0.87 (26)	0.91 (20)	0.91 (23)	0.33 (71)	0.26 (78)
Benzo[a]anthracene/Chrysene	1.46 (6)	1.47 (6)	1.42 (8)	1.43 (9)	1.95 (26)	1.55 (1)
Benzo[b]/(k)fluoranthene	1.48 (5)	1.49 (6)	1.1 (22)	1.38 (2)	1.14 (20)	0.91 (35)
Dibenz(a,h)anthracene	1.00 (3)	1.01 (14)	1.01 (2)	1.02 (13)	0.56 (46)	0.44 (62)
Fluoranthene	1.59 (18)	1.59 (21)	1.81 (7)	1.82 (9)	2.12 (9)	1.7 (15)
Fluorene	1.08 (3)	1.09 (6)	1.19 (14)	1.21 (17)	1.17 (12)	0.94 (9)
Indeno(1,2,3-c,d)pyrene	0.98 (17)	1.08 (23)	0.92 (22)	1.14 (19)	0.48 (59)	0.38 (73)
Naphthalene	1.31 (148)	1.11 (91)	0.68 (29)	0.58 (0)	0.37 (30)	0.30 (48)
Pyrene	1.95 (3)	1.94 (5)	1.74 (8)	1.73 (6)	2.50 (32)	2.07 (12)
Cl-1	0.36 (12)	0.49 (26)	0.38 (19)	0.55 (41)	0.35 (9)	0.36 (7)
Cl-2	0.33 (29)	0.32 (12)	0.35 (27)	0.36 (1)	0.26 (45)	0.23 (37)
Cl-3	0.27 (33)	0.24 (26)	0.29 (28)	0.25 (23)	0.26 (36)	0.18 (45)
Cl-4	0.17 (16)	0.18 (11)	0.17 (15)	0.18 (11)	0.16 (21)	0.17 (16)
Cl-5	0.16 (7)	0.16 (10)	0.14 (18)	0.14 (21)	0.12 (28)	0.12 (32)
Cl-6	0.13 (25)	0.15 (10)	0.22 (31)	0.14 (16)	0.13 (22)	0.12 (28)
Cl-7	0.19 (21)	0.16 (5)	0.13 (18)	0.14 (17)	0.15 (5)	0.16 (5)
Cl-8	0.08 (10)	0.07 (5)	0.06 (12)	0.06 (19)	0.05 (23)	0.06 (19)
Cl-10	0.08 (29)	0.08 (27)	0.06 (8)	0.06 (5)	0.04 (40)	0.03 (52)
Summary :						
	≤ 30%	20	21	21	13	12
	≤ 40%	1	0	1	4	4
	> 40%	1	1	0	5	6

TABLE 11 continued. SVOC Comparison of Initial and Continuing Calibration RF Values

Calibration Date: 9/16/96

CCV Date:	9/16/96	9/17/96	9/17/96
Compound	RF (%Diff.)	RF (%Diff.)	RF (%Diff.)
Acenaphthene	0.41 (9)	0.42 (6)	0.60 (35)
Acenaphthalene	0.69 (33)	0.77 (25)	1.15 (13)
Phenanthrene/Anthracene	0.74 (10)	0.70 (15)	1.13 (37)
Benzo[a]pyrene	0.35 (10)	0.38 (18)	0.24 (26)
Benzo[g,h,I]perylene	0.21 (4)	0.25 (23)	0.14 (30)
Benzo[a]anthracene/Chrysene	0.53 (11)	0.69 (16)	0.59 (1)
Benzo[b]/(k)fluoranthene	0.45 (0)	0.57 (27)	0.29 (34)
Dibenz(a,h)anthracene	0.29 (7)	0.33 (7)	0.21 (30)
Fluoranthene	0.48 (18)	0.66 (12)	0.97 (64)
Fluorene	0.45 (18)	0.49 (9)	0.79 (45)
Indeno(1,2,3-c,d)pyrene	0.31 (4)	0.37 (25)	0.33 (11)
Naphthalene	0.36 (46)	0.67 (1)	0.64 (3)
Pyrene	0.87 (6)	0.83 (11)	1.30 (40)
Cl-1	0.50 (16)	0.48 (19)	0.54 (8)
Cl-2	0.32 (6)	0.27 (21)	0.27 (20)
Cl-3	0.23 (8)	0.23 (7)	0.27 (25)
Cl-4	0.11 (3)	0.11 (5)	0.14 (34)
Cl-5	0.09 (27)	0.08 (35)	0.13 (11)
Cl-6	0.08 (36)	0.07 (44)	0.09 (24)
Cl-7	0.08 (9)	0.10 (17)	0.10 (18)
Cl-8	0.05 (17)	0.05 (6)	0.06 (0)
Cl-10	0.05 (5)	0.06 (26)	0.03 (45)
Summary :			
≤ 30%	19	20	14
≤ 40%	2	1	5
> 40%	1	1	3

TABLE 12 VOC Purge and Trap GC/MS MDL Study, 20ppb Measured

Repetitions	n=1	2	3	4	5	6	7	Conc. Average ppb	STDEV	%RSD	MDL ppb	1/2 20DAF (QL) ppb	RL ppb
Compounds													
1,1,1-Trichloroethane	17.2	13.0	15.3	21.5	15.5	18.2	15.6	16.6	2.70	16	8	1000	20
1,1-Dichloroethane	19.6	18.8	18.2	22.1	16.4	20.2	21.4	19.5	1.94	10	6	10	20
1,1-Dichloroethene	23.5	20.1	20.5	21.3	21.0	21.3	20.9	21.2	1.10	5	3	30	20
1,2-Dichloroethane	19.7	13.8	21.0	27.2	20.8	15.9	18.9	19.6	4.26	22	13	10	20
Benzene	17.8	16.9	18.5	19.7	18.0	16.4	17.3	17.8	1.09	6	3	15	20
Carbon Tetrachloride	22.3	19.3	20.0	20.1	19.1	19.7	18.2	19.8	1.27	6	4	35	20
Chlorobenzene	9.8	13.5	14.7	18.5	12.6	15.8	13.9	14.1	2.70	19	8	500	20
Chloroform	22.8	22.1	24.1	24.7	18.7	23.3	26.8	23.2	2.50	11	8	300	20
cis-1,2-Dichloroethene	20.4	19.4	23.6	22.8	19.6	19.4	22.1	21.1	1.74	8	5	200	20
Ethylbenzene	17.7	19.3	15.3	18.8	16.5	21.1	18.0	18.1	1.90	10	6	6500	20
m/p-Xylene	69.9	69.3	61.4	70.1	60.5	74.9	64.8	67.3	5.22	8	16	105000	80
Methylene Chloride	112.4	93.1	91.2	102.2	92.6	100.8	101.1	99.1	7.47	8	23	10	20
o-Xylene	20.2	21.0	20.3	20.7	19.2	21.0	18.7	20.1	0.91	4	3	95000	20
Styrene	17.0	13.8	12.0	14.7	15.6	15.9	17.2	15.2	1.82	12	6	2000	20
Tetrachloroethene	20.0	21.4	17.3	20.8	16.8	20.1	18.2	19.2	1.77	9	6	30	20
Toluene	9.6	16.2	16.6	13.7	13.8	19.6	13.8	14.7	3.11	21	10	6000	40
trans-1,2-Dichloroethene	21.3	19.2	22.1	25.9	23.2	21.9	22.1	22.2	2.03	9	6	300	20
Trichloroethene	15.6	30.1	14.3	19.7	11.9	18.3	18.3	18.3	5.84	32	18	30	20
Vinyl Chloride	33.7	29.1	22.9	53.9	25.1	29.0	26.9	31.5	10.45	33	33	5	20

Notes

- MDL was calculated based on seven repetitions of the lowest concentration point on the calibration curve
- MDL was calculated using 3.14 times the Std. Deviation
- Project RL shown as lowest calibration standard

TABLE 13 PAH and PCB TD GC/MS MDL Study

Repetitions	n=1	2	3	4	5	6	7	Average Area	STDEV	%RSD	MDL ppb	1/2 20DAF (QL) ppb
Compound												
Acenaphthene	6649403	7851807	8191401	7377699	8438145	7063528	6058243	7375747	854546	12	87	285
Acenaphthylene	14563063	17842996	17246170	16310802	15325549	11898542	12502504	15098518	2270521	15	113	285
Benzo(a)pyrene	8413426	9666725	6531195	8494624	7549711	9732398	9077397	8495068	1154703	14	102	4
Benzo(g,h,i)perylene	5142449	9884367	10925683	6537324	8415502	7071490	8446701	8060502	1984279	25	186	NA
Benzo[a]anthracene /Chrysene	20432464	25734266	23100056	21369574	15665514	25460688	23522470	22183576	3468755	16	236	1
Benzo[b]/(k)fluoranthene	19447460	22131472	18448872	17972126	15064513	22207020	20813890	19440765	2551794	13	297	2.5
Dibenz(a,h)anthracene	6100368	9427042	5158919	7058936	8385818	7205351	8998377	7476401	1552438	21	156	1
Fluoranthene	10345314	12216560	11324217	14004980	10300769	14506519	10800127	11928355	1724108	14	109	2150
Fluorene	5924988	8511248	8256192	11185856	7468333	7163530	7861336	8053069	1620917	20	152	280
Indeno(1,2,3-c,d)pyrene	6130939	10688591	5935142	7760468	9561425	7682154	10045352	8257724	1884167	23	172	7
Naphthalene	7283304	6927502	6000463	3327857	7377352	1434547	2601105	4993161	2478049	50	374	42
Phenanthrene /Anthracene	23240630	36364432	29846948	32649398	30643870	30505696	31870770	30731678	3950958	13	194	280
Pyrene	10468125	15433087	13827595	14585089	8808344	13354526	13720097	12885266	2368184	18	139	2100
Total PCB	31782678	53055163	45648091	35342905	30341274	41428176	43905398	40214812	9705925	20	153	0.5

Notes

- MDL was run prior to the initial calibration and calculated based on seven repetitions of a fortified soil sample containing 300-ppb total PCB and 300-ppb each PAH
- 40uL injected, normally inject 50 uL (4/5 coefficient used when MDL calculated)
- MDL was calculated using 3.14 times the product of the %RSD and the concentration injected

Table 14. VOC Measurement Precision

Sample ID: S1-B1A-(02-04)

<u>Compounds</u>	<u>MDL</u>	<u>8/26/96</u> <u>Initial (ppb)</u>	<u>8/27/96</u> <u>FD1 (ppb)</u>	<u>FD2 (ppb)</u>	<u>RPD</u>
benzene	3	1	4	5	11

Sample ID: S1-B17-1-(02-04)

<u>Compounds</u>	<u>MDL</u>	<u>8/28/96</u> <u>Initial (ppb)</u>	<u>8/28/96</u> <u>FD1 (ppb)</u>	<u>FD2(ppb)</u>	<u>RPD</u>
benzene	3	10	8	2	60

Sample ID: S3-B12-(09-11)

<u>Compounds</u>	<u>MDL</u>	<u>8/26/96</u> <u>Initial (ppb)</u>	<u>8/26/96</u> <u>FD1 (ppb)</u>	<u>FD2(ppb)</u>	<u>RPD</u>
benzene	3	1	3	5	25

Sample ID: S2-B12-(18-20)

<u>Compounds</u>	<u>MDL</u>	<u>8/21/96</u> <u>Initial (ppb)</u>	<u>8/28/96</u> <u>FD1 (ppb)</u>	<u>FD2 (ppb)</u>	<u>RPD</u>
benzene	3	ND	9	5	29

Sample ID: S3-B02-(12-14)

<u>Compounds</u>	<u>MDL</u>	<u>8/26/96</u> <u>Initial (ppb)</u>	<u>8/27/96</u> <u>FD1 (ppb)</u>	<u>FD2 (ppb)</u>	<u>RPD</u>
benzene	3	5	4	ND	
carbon tetrachl.	4	ND	7	7	0
cis-1,2-DCE	5	ND	5	ND	
ethylbenzene	6	22	ND	ND	
m/p-xylene	16	180	ND	ND	
o-xylene	3	40	17	4	62

Sample ID: S1-B09-(09-11)

<u>Compounds</u>	<u>MDL</u>	<u>8/27/96</u> <u>Initial (ppb)</u>	<u>8/27/96</u> <u>FD1 (ppb)</u>	<u>FD2 (ppb)</u>	<u>FD3(ppb)</u>	<u>Ave ± RSD</u>
benzene	3	2	4	8	7	6 ± 33%

Table 14 continued. VOC Measurement Precision

Sample ID: S2-B02-(20-22)

Compounds	<u>MDL</u>	8/26/96		8/27/96		<u>RPD</u>
		<u>Initial (ppb)</u>	<u>FD1 (ppb)</u>	<u>FD2 (ppb)</u>		
1,1,1-TCA	8	300	ND	ND		
1,1-DCA	6	42	ND	ND		
1,1-DCE	3	31	ND	ND		
chloroform	8	8	ND	ND		
cis-1,2-DCE	5	580	ND	ND		
ethylbenzene	6	1,000	26	ND		
m/p-xylene	16	7,700	92	ND		
o-xylene	3	2,400	81	6	82	
tetrachloroethene	6	120	ND	ND		
toluene	12	39,000	120	58	35	

Sample ID: S1-B1A-(09-11)

Compounds	<u>MDL</u>	8/27/96			<u>Ave ± RSD</u>
		<u>Triplicate Run 1 (ppb)</u>	<u>Run 2 (ppb)</u>	<u>Run 3 (ppb)</u>	
1,1,1-TCA	8	8	41	33	23 ± 74%
1,1-DCE	3	2	20	151	12 ± 75%
chloroform	8	2	7	8	6 ± 56%
cis-1,2-DCE	5	5	4	6	4 ± 25%
ethylbenzene	6	61	39	40	47 ± 25%
m/p-xylene	16	280	150	229	220 ± 30%
o-xylene	3	69	81	85	78 ± 10%
styrene	6	6	7	9	7 ± 20%
trans-1,2-DCE	6	6	12	12	10 ± 35%

Table 15. Field versus Laboratory VOC Data Comparison

Sample ID	Compounds	Field (ppb)	Laboratory (ppb)
S2-B2-(20-22)	1,1-dichloroethene	30	< 50
	1,1-dichloroethane	41	< 50
	cis-1,2-dichloroethene	560	< 50
	1,1,1-trichloroethene	300	250
	toluene	37,000	2,000
	tetrachloroethane	120	< 50
	ethylbenzene	990	240
	m/p-xylene	7,400	1,200
	o-xylene	2,200	480
	S3-B1-(13-15)	toluene	280,000
ethylbenzene		3,000	14,500
m/p-xylene		320,000	58,700
o-xylene		83,000	25,500
S3-B23-(13-15)	1,1-dichloroethene	15	< 10
	carbon tetrachloride	6	< 10
	tetrachloroethane	23	< 10
	ethylbenzene	7	< 10
	o-xylene	17	< 10

Table 16 Target Analyte Metals Percent Recovery for Initial Calibration Verification

Date:		8/16	8/20	8/21	8/22	8/25	8/26	8/28	Average ²	%
(mg/kg)		% R	% R	% R	% R	% R	% R	% R	% R	RSD
Element	TRUE ¹									
K	150.9	98	100	102	99	101	91	103	99	4
Ca	151.0	101	101	99	99	95	101	95	99	3
Mg	148.7	99	100	100	102	94	104	95	99	3
Na	149.4	103	103	103	103	104	95	110	103	4
Ag	6.00	98	96	100	100	99	92	104	98	4
Mn	0.452	96	101	99	100	100	97	100	99	2
Zn	0.600	96	101	102	101	96	107	92	99	5
Cr	0.300	99	100	99	101	101	100	103	100	1
Ni	1.200	96	101	101	98	99	93	99	98	3
Al	1.801	99	102	101	96	99	97	100	99	2
Be	0.150	96	98	98	97	97	93	96	96	2
Cu	0.751	100	100	101	99	102	91	102	99	4
V	1.503	100	99	101	101	98	95	99	99	2
Ba	6.00	99	97	99	100	103	90	106	99	5
Co	1.50	97	101	101	101	99	106	98	100	3
Fe	3.12	97	93	94	95	94	98	96	95	2
As	3.00	98	98	98	106	100	101	101	100	3
Cd	0.60	98	102	101	101	103	103	103	101	2
Pb	3.01	96	100	98	97	103	100	102	100	2
Se	3.04	97	94	97	101	99	105	98	99	3
Tl	3.06	92	87	94	95	101	98	100	95	5
Sb	1.845	96	99	100	107	93	107	100	100	5

¹ Sample concentration shown prepared by gravimetric dilution of a standard stock solution obtained from Leeman Labs ² Average percent recovery (%R) is based upon n = 7 initial calibration verification percent recoveries.

Table 17 Target Analyte Metals Percent Recovery for Continuing Calibration Verification

Date:	8/16	8/16	8/20	8/20	8/21	8/21	8/22	8/22	8/22	8/22	8/25	8/25	8/25	8/25	8/26	8/28	8/28	Avg ¹	%
# of CCV (mg/kg)	(1) % R	(2) % R	(1) % R	(2) % R	(1) % R	(2) % R	(1) % R	(2) % R	(3) % R	(1) % R	(2) % R	(3) % R	(4) % R	(1) % R	(1) % R	(2) % R	Avg ¹	%	
Element	TRUE																	% R	RSD
K	150.9	100	100	93	91	102	93	87	85	85	98	92	91	93	92	93	93	93	6
Ca	151.0	100	99	102	104	105	104	94	96	94	102	103	103	103	112	92	96	101	4
Mg	148.7	98	96	100	107	113	110	95	99	98	103	106	107	106	115	92	103	103	6
Na	149.4	103	103	97	92	100	93	95	90	89	101	93	92	100	95	98	95	96	5
Ag	6.00	100	98	91	90	99	93	92	90	89	98	92	88	94	92	99	96	93	4
Mn	0.452	99	97	95	97	104	97	91	92	92	99	100	98	94	103	96	98	96	4
Zn	0.600	90	94	103	109	116	115	96	96	99	102	109	107	106	118	93	101	103	8
Cr	0.300	99	97	99	100	104	99	93	91	91	102	104	104	101	109	97	103	99	5
Ni	1.200	99	98	100	103	109	105	92	96	98	91	95	92	92	102	99	104	98	6
Al	1.801	101	100	102	103	105	97	92	94	93	102	101	97	99	102	96	96	99	4
Be	0.150	96	95	95	94	100	91	90	87	88	98	93	92	95	94	89	90	93	4
Cu	0.751	101	100	92	86	97	91	91	89	89	99	91	88	92	88	93	91	93	5
V	1.503	103	99	94	90	101	95	94	93	91	97	98	95	96	99	93	96	96	4
Ba	6.00	100	99	89	86	94	85	87	85	82	99	91	90	95	91	96	90	91	7
Co	1.50	97	95	95	97	107	102	94	93	96	103	108	108	105	115	102	104	100	6
Fe	3.12	101	100	92	92	97	94	90	89	91	98	100	97	96	105	93	96	95	4
As	3.00	95	95	91	97	100	100	93	93	96	100	97	98	100	105	96	100	96	3
Cd	0.60	97	97	102	110	111	111	99	102	105	112	114	118	117	113	101	114	107	7
Pb	3.01	99	99	101	107	110	109	96	97	101	111	114	117	115	110	98	111	106	7
Se	3.04	88	88	98	102	103	102	95	95	97	97	98	97	101	112	94	102	97	5
Tl	3.06	92	92	90	93	95	96	89	90	98	99	98	101	99	107	94	105	95	4
Sb	1.845	100	98	92	91	98	97	95	100	101	95	94	96	98	111	91	98	96	3

¹ Average percent recovery (%R) is based upon n = 16 initial calibration verification percent recoveries.

Table 18 Target Analyte Metal Percent Recovery of Laboratory Control Standard¹

Beginning of Day	Date	8/20	8/21	8/22	8/25	8/26	8/28	Avg.	%			
LOQ	Certified value							%R	RSD			
Element	(mg/kg)	(mg/kg)	%R	%R	%R	%R	%R	%R	%R			
Ag	0.023	0.212	105	112	113	102	94	114	107	7		
Mn	0.0033	0.372	126	127	125	104	97	105	114	12		
Zn	0.012	0.936	105	105	103	102	111	96	104	5		
Cr	0.108	0.293	121	120	123	108	105	106	114	8		
Ni	0.014	0.423	127	127	122	112	101	107	116	9		
Al	0.069	0.966	115	115	108	112	119	113	114	3		
Be	0.009	0.205	99	98	100	101	96	101	99	2		
Cu	0.013	0.540	108	110	110	108	95	110	107	6		
V	0.010	1.494	90	91	94	108	103	109	99	9		
Ba	0.004	0.306	114	117	116	108	96	107	110	7		
Co	0.027	1.251	102	103	102	110	112	109	106	4		
Fe	0.012	2.049	95	97	101	103	104	106	101	4		
Cd	0.008	0.636	89	88	90	103	98	101	95	7		
Pb	0.115	1.728	85	84	88	104	100	102	94	10		
End of Day											Cumulative	%
											Avg (n=12)	RSD
Ag	0.023	0.212	94	101	99	87	89	99	95	6	101	9
Mn	0.0033	0.372	115	118	109	93	101	98	106	10	110	11
Zn	0.012	0.936	111	114	99	104	121	103	109	7	106	6
Cr	0.108	0.293	114	116	113	100	110	106	110	5	112	7
Ni	0.014	0.423	123	123	118	93	107	110	112	10	114	10
Al	0.069	0.966	104	102	96	111	125	112	108	9	111	7
Be	0.009	0.205	88	89	90	91	96	93	91	3	95	5
Cu	0.013	0.540	90	95	95	91	94	97	93	3	100	8
V	0.010	1.494	79	84	84	97	106	100	92	12	95	11
Ba	0.004	0.306	94	95	99	89	93	93	94	3	102	10
Co	0.027	1.251	94	100	93	106	120	109	104	10	105	7
Fe	0.012	2.049	91	94	91	99	108	100	97	7	99	6
Cd	0.008	0.636	92	94	89	109	110	110	101	10	98	9
Pb	0.115	1.728	86	92	88	104	110	108	98	11	96	10

¹ERA WastWatR #9967

TABLE 19. Percent Recovery (%R) of a Laboratory Control Standard Soil Based Upon Certified Reference and Theoretical made-to Values

Element	certified value ¹			made-to value ³		
	(mg/kg)	Ave ² %R	% RSD	(mg/kg)	Ave ² %R	% RSD
K	3250	138	14	4224	106	14
Ca	3710	114	7	4325	98	7
Mg	2490	124	7	3213	96	7
Na	167	169	14	182	155	14
Ag	89	105	8	108	87	8
Mn	298	132	11	348	113	11
Zn	110	117	7	133	97	7
Cr	71.6	122	5	82.2	107	5
Ni	68.5	102	7	85.3	82	7
Al	6070	202	15	7040	174	15
Be	122	99	7	161	75	7
Cu	173	97	8	204	83	8
V	105	144	6	127	119	6
Ba	161	104	10	197	85	10
Co	94.2	115	5	127	86	5
Fe	12500	247	5	13600	227	5
As	65.4	121	6	87.1	91	6
Cd	85.4	98	7	104	81	7
Pb	70.4	105	6	87.1	85	6
Se	152	104	4	193	82	4
Tl	71.8	125	11	123	73	11
Sb	38.1	358	3	186	73	3

¹Certified concentrations provided by ERA for PriorityPollutnT/CLP Soil #228

²n = 9 over seven analysis days

³Theoretical made-to concentration for PriorityPollutnT/CLP Soil #228

Table 20 Target Analyte Metals Percent Recovery of Interferents A & AB Solutions

Date	8/20		8/21		8/22		8/25		8/26		8/28		Sol A		Sol AB			
	Sol A ¹	Sol AB ²	Sol A	Sol AB	Sol A	Sol AB	Sol A	Sol AB	Sol A	Sol AB	Sol A	Sol AB	Sol A	Sol AB	Avg ³	%	Avg ³	%
Element	values (mg/kg)	values (mg/kg)	%R	%R	%R	%R	%R	%R	%R	%R	%R	%R	%R	%R	%	RSD	%	RSD
Ca	500.6	500.6	104	104	102	102	99	94	94	97	98	101	94	95	98	4	99	4
Mg	500.8	500.8	106	106	107	108	99	95	100	103	106	109	102	99	103	3	103	5
Ag		1.000		96		101		93		98		94		106			98	5
Mn		0.504	2	102	2	105	2	91	2	100	2	94	2	100	2	10	98	5
Zn		1.000		99		99		88		95		101		90			95	6
Cr		0.504		98		99		90		94		92		97			95	4
Ni		1.000		97		98		86		82		88		96			91	7
Al	500.3	500.3	104	104	106	104	98	93	107	107	103	103	106	105	104	3	103	5
Be		0.500		100		99		89		100		93		100			97	5
Cu		0.500		97		100		92		102		90		105			98	6
V		0.500		97		100		90		97		94		100			96	4
Ba		0.500	1	95	2	100	1	87	2	101	1	93	2	106	1	12	97	7
Co		0.504		93		96		87		96		94		94			93	3
Fe	200.5	200.5	91	90	93	92	91	86	89	92	89	92	94	93	91	2	91	3
Cd		1.000		92		93	1	84	1	96		91	1	94	1	8	92	5
Pb		1.000	16	104	18	111	15	99	19	112	17	108	19	106	17	9	107	5

¹Solution (Sol A) contains only Ca, Mg, Al, and Fe but is run for all elements ² solution (Sol AB) contains both the interfering elements and the analyte elements.

²Solution (Sol AB) contains both the interfering elements and the analyte elements.

³ Average %R for n = 6, one per sample day of study

TABLE 21. Comparison of Field vs. Commercial Laboratory Results for Pb and Cd

Sample ID	Field (mg/kg)		Commercial Laboratory (mg/kg)		Pb	Cd
	Lead	Cadmium	Lead	Cadmium	RPD ¹	RPD ¹
S1-B11-(5-6)	19	0.48	15.4	<0.94	21	
S2-B1-(16-18)	194	0.35	222	<0.89	-13	
S2-B2-(20-22)	16	<0.33	29	<0.95	-58	
S2-B6-(2-4)	46	<0.33	168	<1.05	-114	
S2-B10-(10-12)	8	<0.33	11.6	<0.95	-37	
S2-B12-(12-14)	11	<0.33	19.4	<0.84	-55	
S2-B15A-(10-12)	14	<0.33	20	<1.04	-35	
S3-B1-(13-15)	65	6	75.6	6.49	-15	-8
S3-B6-(10-12)	19	0.62	28.7	<1.07	-41	
S3-B14-(6-8)	5	<0.33	2.94	1.74	52	
S3-B23-(13-15)	9	0.36	9.96	<0.84	-10	

¹(Tufts Field Lab-Fixed Base Lab)/((Tufts Field Lab + Fixed Base Lab)/2)

Table 22. HAFB Site Sample Target Analyte Metals Measurement Precision

Sample I.D.	S2-B1-(16-18)						S2-B2-(20-22)						S2-B4-(19-21)					
Date	8/20						8/20						8/21					
Element	LOQ	(1)	(2)	(3)	Average	%	(1)	(2)	(3)	Average	%	(1)	(2)	(3)	Average	%		
	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	RSD	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	RSD	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	RSD		
K	38.0	1625	1766	872	1421	34	1264	1154	1387	1268	9	1507	3069	1409	1995	47		
Ca	0.36	947	1152	504	810	38	723	724	707	718	1	875	1080	946	967	11		
Mg	5.00	3073	3107	1787	2480	28	2243	2012	2405	2220	9	2715	3338	2722	2925	12		
Na	11.0	84.4	102	55.9	75.4	29	78.6	63.7	69.3	70.5	11	102	152	91	114.9	29		
Ag	1.00	2.74	3.25	1.64	2.38	32	2.24	1.96	2.43	2.21	11	3.13	3.85	2.55	3.17	20		
Mn	0.14	131	132	83	108	24	92.5	76.5	99.3	89.4	13	117	144	104	121	17		
Zn	0.52	21.0	21.5	12.3	17.0	28	20.3	19.8	21.3	20.5	4	21.8	34.4	18.4	24.9	34		
Cr	0.47	19.2	16.7	10.8	14.6	28	12.9	11.4	13.2	12.5	8	13.3	19.6	14.5	15.8	21		
Ni	0.60	12.2	13.2	8.1	10.4	24	8.94	9.08	9.62	9.21	4	9.70	17.24	9.28	12.1	37		
Al	3.00	7453	7901	4577	6205	27	6149	5429	6963	6180	12	6674	14259	6085	9006	51		
Be	0.04	0.300	0.317	0.209	0.257	21	0.250	0.212	0.276	0.246	13	0.282	0.621	0.250	0.384	53		
Cu	0.56	8.35	6.92	4.28	6.09	32	5.66	5.37	6.62	5.88	11	6.32	10.02	4.23	6.86	43		
V	0.41	17.0	16.6	9.7	13.5	28	12.2	10.6	13.8	12.2	13	14.2	22.0	12.4	16.2	31		
Ba	0.16	25.2	25.3	13.7	20.0	31	23.3	21.2	27.5	24.0	14	18.7	53.0	17.9	29.9	67		
Co	1.16	4.22	4.57	2.66	3.57	27	3.80	3.43	3.70	3.64	5	3.92	6.21	3.66	4.60	31		
Fe	0.52	11360	10867	6627	8983	27	6729	6167	7342	6746	9	8904	12000	7633	9512	24		
As	15.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND			
Cd	0.33	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND			
Pb	5.00	194	185	113	153	27	16.1	15.6	17.0	16.2	4	19.0	61.7	18.1	33.0	76		
Se	10.8	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND			
Tl	19.4	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND			
Sb	11.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND			

Table 22 continued. HAFB Site Sample Target Analyte Metals Measurement Precision

Sample I.D.	S2-B15-(14-16)						S1-B17A-(5.75-8)						S1-B1A-(2-4)				
Date	8/22						8/22						8/25				
Element	LOQ (mg/kg)	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD	
K	38.0	1134	1212	1208	1185	4	569	624	742	645	14	2259	2602	2073	2311	12	
Ca	0.36	751	845	887	828	8	385	507	551	481	18	1089	1297	1214	1200	9	
Mg	5.00	2973	2567	2376	2639	12	1086	1233	1377	1232	12	2658	3537	2661	2952	17	
Na	11.0	155	174	184	171	9	56.7	60.8	61.4	59.6	4	151	235	145	177	28	
Ag	1.00	2.47	2.43	2.69	2.53	5	1.85	1.96	2.14	1.98	7	3.57	3.87	3.68	3.71	4	
Mn	0.14	120	94.5	99.7	105	13	55.5	62.7	75.9	64.7	16	142	142	125	136	7	
Zn	0.52	38.1	37.4	41.3	38.9	5	30.9	29.3	34.5	31.6	9	34.4	32.8	31.4	32.9	5	
Cr	0.47	29.6	26.6	27.9	28.0	5	7.72	8.99	8.96	8.55	8	17.2	19.3	16.5	17.65	8	
Ni	0.60	12.8	12.0	11.7	12.2	5	10.35	4.14	4.69	6.39	54	7.43	7.98	6.06	7.16	14	
Al	3.00	6506	6234	6449	6396	2	5735	5896	6573	6068	7	8164	9268	8033	8488	8	
Be	0.04	0.243	0.259	0.272	0.26	6	0.280	0.232	0.259	0.26	9	0.299	0.281	0.278	0.286	4	
Cu	0.56	13.1	13.6	14.0	13.5	3	6.05	6.05	5.81	5.97	2	11.8	14.5	19.9	15.4	27	
V	0.41	13.7	11.8	14.0	13.1	9	11.0	12.4	12.5	12.0	7	21.1	23.6	21.1	21.9	6	
Ba	0.16	19.4	22.5	21.9	21.3	8	17.9	19.6	20.1	19.2	6	37.2	47.1	36.1	40.1	15	
Co	1.16	4.04	3.58	4.05	3.89	7	1.70	1.73	2.13	1.86	13	4.30	4.19	3.57	4.02	10	
Fe	0.52	10252	8496	8386	9045	12	6281	6612	6949	6614	5	13214	12400	12411	12675	4	
As	15.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND		
Cd	0.33	6.29	6.80	6.72	6.60	4	ND	ND	ND	ND		0.440	0.475	0.434	0.450	5	
Pb	5.00	361	344	357	354	2	37.4	33.7	40.2	37	9	24.4	20.9	15.1	20	23	
Se	10.8	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND		
Tl	19.4	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND		
Sb	11.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND		

Table 22 continued. HAFB Site Sample Target Analyte Metals Measurement Precision

Sample I.D.	S3-B02-(17-19)						S1-B11-(2-4)					S3-B16-(6-8)				
Date	8/25						8/26					8/28				
Element	LOQ (mg/kg)	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD	(1) (mg/kg)	(2) (mg/kg)	(3) (mg/kg)	Average (mg/kg)	% RSD
K	38.0	1624	1457	888	1323	29	1983	1925	2025	1978	3	1033	1114	1066	1071	4
Ca	0.36	1304	1100	830	1078	22	1379	1173	1436	1329	10	621	829	697	716	15
Mg	5.00	2848	2495	1949	2431	19	3122	3394	3305	3274	4	2173	2014	1953	2047	6
Na	11.0	115	101	107	108	7	188	170	210	189	10	73.7	89.1	76.8	79.8	10
Ag	1.00	2.62	2.14	1.44	2.07	29	3.63	3.12	2.68	3.14	15	2.04	1.84	2.08	1.99	6
Mn	0.14	137	103	79	106	27	166	162	166	165	1	83.1	97.6	79.7	86.8	11
Zn	0.52	25.4	23.4	96.6	48.5	86	24.9	25.0	29.5	26.4	10	21.6	54.8	13.3	29.9	74
Cr	0.47	16.6	13.3	10.1	13.3	24	19.4	19.0	18.4	18.9	3	11.4	10.2	10.9	10.8	5
Ni	0.60	13.6	11.7	8.84	11.4	21	7.92	8.51	9.03	8.49	7	9.35	9.05	10.24	9.55	6
Al	3.00	6778	5932	4616	5775	19	10175	10000	11167	10447	6	5695	5842	5394	5644	4
Be	0.04	0.255	0.211	0.162	0.209	22	0.367	0.351	0.406	0.375	8	0.168	0.197	0.250	0.205	20
Cu	0.56	7.68	6.01	4.41	6.03	27	9.9	9.9	10.2	10.01	2	6.33	6.41	7.25	6.66	8
V	0.41	15.6	13.7	9.76	13.0	23	23.7	23.9	24.8	24.1	2	11.6	11.0	10.4	11.0	5
Ba	0.16	23.3	23.8	13.6	20.3	28	38.1	40.9	42.2	40.4	5	15.9	16.4	18.4	16.9	8
Co	1.16	6.60	5.56	4.24	5.47	22	5.25	5.14	5.53	5.30	4	3.42	3.06	3.66	3.38	9
Fe	0.52	8297	7106	6045	7149	16	13276	13276	14165	13572	4	6289	6051	5597	5979	6
As	15.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND	
Cd	0.33	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND	
Pb	5.00	ND	ND	ND	ND		11.4	9.9	11.1	ND		ND	ND	ND	ND	
Se	10.8	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND	
Tl	19.4	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND	
Sb	11.0	ND	ND	ND	ND		ND	ND	ND	ND		ND	ND	ND	ND	

Table 27. IFD and EnviroQuant Sample Data Comparison

S2-B01-(20-22)					S2-B02-(20-22)				
PTMS031.D	RL	IFD	ENVQ	RPD	PTMS046.D	RL	IFD	ENVQ	RPD
1,1,1-Trichloroethane	20	<46.	57	NA	1,1,1-Trichloroethane	20	300	300	0%
cis-1,2-Dichloroethene	20	120	130	8%	1,1-Dichloroethane	20	41.	23	55%
Ethylbenzene	20	830	840	1%	1,1-Dichloroethene	20	30.	28	7%
m/p-Xylene	40	3400	3400	0%	cis-1,2-Dichloroethene	20	560	570	2%
o-Xylene	50	750	750	0%	Ethylbenzene	20	990	1000	1%
Tetrachloroethene	20	110	120	9%	m/p-Xylene	40	7400	7500	1%
Toluene	50	22000	21000	5%	o-Xylene	50	2300	2300	0%
					Tetrachloroethene	20	120	120	0%
					Toluene	50	37000	36000	3%
S2-B14-(08-10)					S2-B12-(09-11)				
PTMS032.D	RL	IFD	ENVQ	RPD	PTMS047.D	RL	IFD	ENVQ	RPD
m/p-Xylene	40	54.	54	0%	m/p-Xylene	40	51.	53	4%
Toluene	50	43.	51	17%	Toluene	50	73.	68	7%
S2-B13-(18-20)					S2-B01-(12-14)				
PTMS036.D	RL	IFD	ENVQ	RPD	PTMS052.D	RL	IFD	ENVQ	RPD
1,1,1-Trichloroethane	20	210	440	71%	1,1,1-Trichloroethane	20	36.	38	7%
1,1-Dichloroethane	20	15.	35	77%	Ethylbenzene	20	53.	54	2%
1,1-Dichloroethene	20	51.	63	20%	m/p-Xylene	40	140	144	3%
cis-1,2-Dichloroethene	20	15000	31000	70%	o-Xylene	50	54.	54	1%
Ethylbenzene	20	230	460	67%	Tetrachloroethene	20	170	164	4%
m/p-Xylene	40	640	1300	68%	Toluene	50	390	370	5%
o-Xylene	50	190	370	64%					
Tetrachloroethene	20	25.	52	68%	S2-B02-(13-15)				
Toluene	50	5500	11000	67%	PTMS064.D	RL	IFD	ENVQ	RPD
trans-1,2-Dichloroethene	20	45.	92	69%	1,1,1-Trichloroethane	20	20.	33	50%
					Ethylbenzene	20	550	1059	63%
					m/p-Xylene	40	2100	3962	61%
					o-Xylene	50	980	1900	64%
					Tetrachloroethene	20	540	1025	62%
					Toluene	50	9100	16744	59%
S2-B14-(18-20)					S2-B13-(14-16)				
PTMS038.D	RL	IFD	ENVQ	RPD	PTMS065.D	RL	IFD	ENVQ	RPD
1,1,1-Trichloroethane	20	24.	22	9%	1,1,1-Trichloroethane	20	31.	47	40%
cis-1,2-Dichloroethene	20	110	140	24%	cis-1,2-Dichloroethene	20	380	440	15%
Ethylbenzene	20	28.	31	10%	Ethylbenzene	20	160	178	10%
m/p-Xylene	40	86.	96	11%	m/p-Xylene	40	210	240	14%
o-Xylene	50	67.	61	8%	o-Xylene	50	95.	109	13%
Toluene	50	390	460	16%	Tetrachloroethene	20	42.	52	22%
					Toluene	50	1600	1789	11%
S2-B03-(10-12)					S2-B04-(13-15)				
PTMS042.D	RL	IFD	ENVQ	RPD	PTMS066.D	RL	IFD	ENVQ	RPD
Ethylbenzene	20	98.	100	2%	Ethylbenzene	20	390	888	78%
o-Xylene	50	170	180	6%	m/p-Xylene	40	870	1957	77%
					o-Xylene	50	970	2167	76%
					Tetrachloroethene	20	41.	93	79%
					Toluene	50	1100	2301	71%
S2-B04-(19-21)									
PTMS044.D	RL	IFD	ENVQ	RPD					
Ethylbenzene	20	410	550	29%					
m/p-Xylene	40	640	850	28%					
o-Xylene	50	530	700	28%					

RL = Reporting Limit
 IFD = Ion Fingerprint Detection Software, Sample Results (ppb)
 ENVQ = EnviroQuant Software, Sample Results (ppb)
 RPD = Relative Percent Difference

Table 27 continued. IFD and EnviroQuant Sample Data Comparison

Sample ID	RL	IFD	ENVQ	RPD
S2-B02-(20-22) FD1				
PTMS067.D				
Ethylbenzene	20	26.	18	36%
m/p-Xylene	40	92.	64	35%
o-Xylene	50	81.	30	92%
Toluene	50	120	110	9%
S2-B02-(20-22) FD2				
PTMS069.D				
Toluene	50	58.	60	3%
S2-B15A-(14-16)				
PTMS070.D				
1,1,1-Trichloroethane	20	110	160	37%
cis-1,2-Dichloroethene	20	40.	60	39%
Ethylbenzene	20	50.	70	34%
m/p-Xylene	40	140	210	40%
o-Xylene	50	280	400	35%
Toluene	50	1500	2100	33%
S2-B03-(13-15)				
PTMS071.D				
Ethylbenzene	20	1700	2500	38%
m/p-Xylene	40	17000	27000	45%
o-Xylene	50	4300	6500	41%
Toluene	50	24000	35000	37%
S2-B03-(16-18)				
PTMS072.D				
Ethylbenzene	20	640	610	5%
m/p-Xylene	40	15000	14000	7%
o-Xylene	50	2500	2300	8%
Toluene	50	9600	8700	10%
S1-B04-(6.2-08)				
PTMS095.D				
1,1,1-Trichloroethane	20	500	580	15%
1,1-Dichloroethane	20	130	150	14%
1,2-Dichloroethane	20	95.	74	26%
cis-1,2-Dichloroethene	20	40000	44000	10%
Tetrachloroethene	20	330	360	9%
trans-1,2-Dichloroethene	20	1400	1500	7%
Trichloroethene	20	10000	10000	0%
S1-B04-(9.5-12)				
PTMS098.D				
cis-1,2-Dichloroethene	20	150	150	0%
Toluene	50	110	110	0%
S3-B01-(17-19)				
PTMS100.D				
trans-1,2-Dichloroethene	20	23.	0	200%
S2-B02-(16-18)				
PTMS102.D				
1,1,1-Trichloroethane	20	15000	19000	24%
1,1-Dichloroethane	20	3200	3700	14%
1,1-Dichloroethene	20	870	0	200%
1,2-Dichloroethane	20	290	0	200%
Benzene	20	24.	29	17%
Chlorobenzene	20	32.	38	18%
cis-1,2-Dichloroethene	20	1600	1900	17%
Ethylbenzene	20	1900	2400	23%
m/p-Xylene	40	12000	14000	15%
o-Xylene	50	3500	4200	18%
Tetrachloroethene	20	920	1100	18%
Toluene	50	36000	120000	108%
Trichloroethene	20	67.	79	17%
S3-B05-(10-12)				
PTMS104.D				
cis-1,2-Dichloroethene	20	110	130	17%
Ethylbenzene	20	35.	40	14%
m/p-Xylene	40	86.	89	4%
o-Xylene	50	39.	0	200%
Toluene	50	140	150	7%
S3-B04-(12.5-14)				
PTMS106.D				
Ethylbenzene	20	670	680	1%
m/p-Xylene	40	3600	3600	0%
o-Xylene	50	1800	1800	0%
Toluene	50	200	190	5%
S2-B04-(16-18)				
PTMS107.D				
Ethylbenzene	20	190	260	31%
m/p-Xylene	40	340	440	26%
o-Xylene	50	490	530	8%
Toluene	50	250	550	75%
S2-B03-(18-20)				
PTMS109.D				
Ethylbenzene	20	340	390	14%
m/p-Xylene	40	2100	2500	17%
o-Xylene	50	530	590	11%
Toluene	50	930	1100	17%

RL = Reporting Limit
 IFD = Ion Fingerprint Detection Software,
 Sample Results (ppb)
 ENVQ = EnviroQuant Software, Sample Results (ppb)
 RPD = Relative Percent Difference

Table 27 continued. IFD and EnviroQuant Sample Data Comparison

S3-B01-(10-12)					S1-B04-(02-04)				
PTMS139.D	RL	IFD	ENVQ	RPD	PTMS160.D	RL	IFD	ENVQ	RPD
Ethylbenzene	20	730	720	1%	1,1,1-Trichloroethane	20	540	550	2%
m/p-Xylene	40	9100	9000	1%	1,1-Dichloroethane	20	150	150	0%
o-Xylene	20	1300	1200	8%	1,2-Dichloroethane	20	120	100	18%
Toluene	20	390	370	5%	cis-1,2-Dichloroethene	20	160000	150000	6%
S3-B02-(12-14)					Ethylbenzene	20	38.	41	6%
PTMS140.D					m/p-Xylene	40	170	180	6%
Ethylbenzene	20	22.	21	4%	o-Xylene	20	73.	74	2%
m/p-Xylene	40	130	130	0%	Tetrachloroethene	20	110	120	9%
o-Xylene	20	40.	29	33%	Toluene	20	1500	1500	0%
S3-B04-(10-12)					trans-1,2-Dichloroethene	20	1500	1500	0%
PTMS141.D					Trichloroethene	20	4600	4400	4%
Ethylbenzene	20	280	280	0%	S1-B10A-(4.5-06)				
m/p-Xylene	40	110	120	9%	PTMS161D.D				
o-Xylene	20	1000	1000	0%	cis-1,2-Dichloroethene	20	300	300	0%
Toluene	20	60.	61	2%	Trichloroethene	20	61.	61	0%
S3-B08-(06-08)					S3-B08-(10-12)				
PTMS143.D					PTMS177.D				
cis-1,2-Dichloroethene	20	830	830	0%	cis-1,2-Dichloroethene	20	250	320	25%
Ethylbenzene	20	460	460	0%	Ethylbenzene	20	400	500	22%
m/p-Xylene	40	860	870	1%	m/p-Xylene	40	1600	2100	27%
o-Xylene	20	430	420	2%	o-Xylene	20	350	430	21%
Toluene	20	1400	1400	0%	Toluene	20	2400	2900	19%
trans-1,2-Dichloroethene	20	32.	0	200%	trans-1,2-Dichloroethene	20	19.	26	29%
S3-B06-(10-12)					S3-B06-(13-15)				
PTMS144.D					PTMS178.D				
Ethylbenzene	20	22.	20	8%	1,1,1-Trichloroethane	20	80.	21	118%
m/p-Xylene	40	28.	26	9%	1,1-Dichloroethene	20	24.	0	200%
Toluene	20	33.	27	20%	Ethylbenzene	20	50.	36	33%
S3-B07-(10-12)					m/p-Xylene	40	150	150	0%
PTMS150.D					o-Xylene	20	94.	38	85%
cis-1,2-Dichloroethene	20	24.	28	14%	Styrene	20	48.	0	200%
S3-B05-(14-16)					Tetrachloroethene	20	61.	62	2%
PTMS156.D					Toluene	20	22.	8	99%
Ethylbenzene	20	4300	4800	11%	trans-1,2-Dichloroethene	20	160	30	136%
m/p-Xylene	40	31000	33000	6%	S3-B23-(13-15)				
o-Xylene	20	12000	12000	0%	PTMS179.D				
Toluene	20	15000	16000	6%	Tetrachloroethene	20	23.	0	200%

RL = Reporting Limit IFD = Ion Fingerprint Detection Software, Sample Results (ppb) ENVQ = EnviroQuant Software, Sample Results (ppb) RPD = Relative Percent Difference

Table 27 continued. IFD and EnviroQuant Sample Data Comparison

S1-B17A-(02-04)
PTMS187.D

	RL	IFD	ENVQ	RPD
1,1,1-Trichloroethane	20	210	210	0%
cis-1,2-Dichloroethene	20	4800	4900	2%
Tetrachloroethene	20	66.	81	21%
trans-1,2-Dichloroethene	20	180	200	11%
Trichloroethene	20	82.	88	7%

S1-B17A-(5.75-08)
PTMS188.D

1,1,1-Trichloroethane	20	18.	24	26%
cis-1,2-Dichloroethene	20	810	800	1%
Trichloroethene	20	29.	29	0%

S1-B1A-(09-11)
PTMS193.D

Ethylbenzene	20	61.	61	0%
m/p-Xylene	40	280	290	4%
o-Xylene	20	69.	69	0%

S1-B1A-(09-11)
PTMS194.D

1,1,1-Trichloroethane	20	41.	6	152%
1,1-Dichloroethene	20	20.	0	200%
Ethylbenzene	20	39.	37	7%
m/p-Xylene	40	150	190	24%
o-Xylene	20	81.	69	16%

S1-B1A-(09-11)
PTMS195.D

1,1,1-Trichloroethane	20	33.	3	163%
Ethylbenzene	20	41.	39	4%
m/p-Xylene	40	160	200	22%
o-Xylene	20	85.	57	39%

S1-B13-(06-08)
PTMS196.D

Ethylbenzene	20	1700	1780	5%
m/p-Xylene	40	1600	1600	0%
o-Xylene	20	370	350	6%

S1-B16A-(06-08)
PTMS207.D

cis-1,2-Dichloroethene	20	42.	47	12%
Ethylbenzene	20	40.	40	1%
m/p-Xylene	40	44.	46	4%
Toluene	20	140	140	0%

S1-B16A-(09-11)
PTMS208.D

cis-1,2-Dichloroethene	20	120	130	8%
Ethylbenzene	20	230	230	0%
m/p-Xylene	40	330	340	3%
o-Xylene	20	110	120	9%
Toluene	20	230	250	8%

RL = Reporting Limit IFD = Ion Fingerprint Detection Software, Sample Results (ppb) ENVQ = EnviroQuant Software, Sample Results (ppb) RPD = Relative Percent Difference

TABLE 28. VOC 5-point Calibration Linear Regression Analysis and Average RF

Compound	Tufts purge and trap TD			Tekmar purge and trap		
	(Slope ± Std.Dev) x 10 ³	(Intercept ± Error) x 10 ²	RFcal ± %RSD	(Slope ± Std.Dev) x 10 ³	(Intercept ± Error) x 10 ²	RFcal ± %RSD
1,1,1-Trichloroethane	16.6 ± 0.1	1.23 ± 3.70	1.13 ± 4	7.39 ± 0.05	-1.81 ± 1.60	0.72 ± 3
1,1-Dichloroethane	17.2 ± 0.1	6.27 ± 4.96	1.19 ± 4	9.54 ± 0.17	-7.91 ± 6.09	0.89 ± 8
1,1-Dichloroethene	3.21 ± 0.04	0.301 ± 1.539	0.22 ± 4	0.699 ± 0.008	-0.133 ± 0.291	0.07 ± 5
1,2-Dichloroethane	6.62 ± 0.09	2.96 ± 3.17	0.46 ± 6	4.83 ± 0.04	-3.69 ± 1.34	0.45 ± 6
Benzene	19.8 ± 0.1	21.2 ± 4.6	1.51 ± 12	8.95 ± 0.10	0.567 ± 3.496	2.18 ± 4
Carbon Tetrachloride	11.1 ± 0.3	10.2 ± 11.1	0.79 ± 7	4.21 ± 0.02	-0.472 ± 0.626	0.95 ± 2
Chlorobenzene	28.7 ± 0.2	10.0 ± 8.7	2.00 ± 5	23.8 ± 0.2	1.17 ± 7.61	0.92 ± 10
Chloroform	22.3 ± 0.2	-2.35 ± 7.41	1.50 ± 4	14.7 ± 0.1	-8.19 ± 4.61	0.41 ± 4
cis-1,2-Dichloroethene	10.7 ± 0.2	2.63 ± 5.59	0.74 ± 5	5.62 ± 0.07	-1.81 ± 2.52	2.39 ± 4
Ethylbenzene	31.5 ± 2.1	144 ± 76	2.89 ± 14	34.1 ± 0.3	8.06 ± 9.78	1.39 ± 5
m/p-Xylene	31.2 ± 0.5	76.9 ± 35.1	2.33 ± 7	26.0 ± 0.2	-2.57 ± 14.13	0.54 ± 5
o-Xylene	36.9 ± 0.3	25.3 ± 9.1	2.64 ± 6	32.8 ± 0.3	1.95 ± 10.70	3.50 ± 5
Styrene	29.1 ± 0.4	8.14 ± 15.25	2.03 ± 6	33.1 ± 0.4	-11.0 ± 13.5	2.60 ± 4
Tetrachloroethene	6.25 ± 0.06	0.920 ± 2.133	0.43 ± 5	2.58 ± 0.01	0.549 ± 0.387	3.30 ± 5
Toluene	29.3 ± 0.3	21.2 ± 9.2	2.13 ± 8	16.1 ± 0.1	-3.48 ± 4.87	3.20 ± 5
trans-1,2-Dichloroethene	3.08 ± 0.03	1.29 ± 1.19	0.21 ± 4	0.620 ± 0.005	0.195 ± 0.185	0.26 ± 3
Trichloroethene	7.58 ± 0.09	1.27 ± 3.20	0.53 ± 7	3.14 ± 0.02	0.356 ± 0.590	1.58 ± 4
Summary:						
	≤ 30%		17			17
	≤ 40%		0			0
	> 40%		0			0

TABLE 28 continued. VOC 5-point Calibration Linear Regression Analysis and Average RF

Compound	Tufts purge and trap TD			Tekmar purge and trap		
	(Slope ± Std.Dev) x 10 ³	(Intercept ± Error) x 10 ²	RFcal ± %RSD	(Slope ± Std.Dev) x 10 ³	(Intercept ± Error) x 10 ²	RFcal ± %RSD
1,1,1-Trichloroethane	11.5 ± 0.2	-6.72 ± 5.75	0.72 ± 6	8.05 ± 0.07	-0.705 ± 2.652	0.80 ± 3
1,1-Dichloroethane	15.9 ± 0.4	-7.74 ± 10.22	1.00 ± 6	9.97 ± 0.11	2.46 ± 4.31	1.02 ± 7
1,1-Dichloroethene	0.509 ± 0.009	-0.0319 ± 0.2620	0.03 ± 5	0.564 ± 0.005	0.463 ± 0.187	0.06 ± 8
1,2-Dichloroethane	6.38 ± 0.16	-4.87 ± 3.85	0.39 ± 9	5.37 ± 0.06	0.674 ± 2.103	0.54 ± 3
Benzene	12.3 ± 0.3	-4.11 ± 8.11	0.81 ± 7	7.81 ± 0.07	8.40 ± 2.78	2.35 ± 1
Carbon Tetrachloride	6.71 ± 0.17	-3.60 ± 4.10	0.43 ± 5	4.06 ± 0.02	0.388 ± 0.699	1.19 ± 1
Chlorobenzene	34.4 ± 1.5	-51.4 ± 36.7	1.95 ± 10	25.1 ± 0.2	10.9 ± 5.7	0.91 ± 19
Chloroform	35.3 ± 1.0	-26.4 ± 23.6	2.17 ± 6	17.2 ± 0.1	-1.78 ± 5.31	0.41 ± 4
cis-1,2-Dichloroethene	8.06 ± 0.24	-5.87 ± 5.89	0.49 ± 8	6.06 ± 0.03	2.55 ± 1.16	2.67 ± 8
Ethylbenzene	49.0 ± 2.2	-57.6 ± 57.3	2.94 ± 9	36.1 ± 0.2	16.9 ± 9.1	1.73 ± 5
m/p-Xylene	36.9 ± 1.7	-105 ± 82	2.14 ± 10	26.9 ± 0.1	12.8 ± 10.8	0.64 ± 5
o-Xylene	54.3 ± 2.4	-76.7 ± 59.6	3.16 ± 11	35.6 ± 0.1	11.5 ± 5.1	3.84 ± 8
Styrene	44.4 ± 2.0	-69.9 ± 50.1	2.52 ± 13	33.5 ± 0.2	15.7 ± 8.2	2.81 ± 7
Tetrachloroethene	2.84 ± 0.10	-3.20 ± 2.49	0.17 ± 7	2.27 ± 0.01	0.146 ± 0.378	3.75 ± 7
Toluene	23.9 ± 0.9	-17.7 ± 21.3	1.53 ± 10	14.3 ± 0.1	5.55 ± 2.64	3.54 ± 7
trans-1,2-Dichloroethene	0.487 ± 0.020	-0.0723 ± 0.5334	0.03 ± 9	0.610 ± 0.003	0.390 ± 0.130	0.23 ± 3
Trichloroethene	5.62 ± 0.28	-5.43 ± 6.77	0.34 ± 8	2.70 ± 0.01	0.640 ± 0.398	1.52 ± 9
Summary:						
	≤ 30%		17			17
	≤ 40%		0			0
	> 40%		0			0

TABLE 29. VOC Tufts purge and trap TD GC/MS MDL Study, 6.7 ppb Measured

Repetitions Compounds	n = 1	2	3	4	5	6	7	Conc. Average ppb	%RSD	STDEV	MDL ppb	Tekmar P&T MDL ppb
	1,1,1-Trichloroethane	2.1	1.8	1.9	1.9	1.8	1.8	1.8	1.9	5	0.10	
1,1-Dichloroethane	1.9	1.8	1.9	1.9	1.9	1.9	1.9	1.9	3	0.06	0.2	6
1,1-Dichloroethene	3.1	2.2	1.5	2.9	3.0	3.0	3.0	2.7	22	0.6	2	3
1,2-Dichloroethane	1.8	1.7	1.7	1.7	2.0	1.8	1.9	1.8	7	0.1	0.4	13
Benzene	3.0	2.7	2.7	2.6	2.5	2.6	2.8	2.7	7	0.2	0.6	3
Carbon Tetrachloride	2.6	2.3	2.4	2.5	2.2	2.3	2.2	2.4	6	0.2	0.5	4
Chlorobenzene	1.5	1.7	1.5	1.5	1.5	1.3	1.5	1.5	7	0.1	0.3	8
Chloroform	2.2	1.9	2.0	2.0	2.1	2.1	2.0	2.	4	0.08	0.3	8
cis-1,2-Dichloroethene	1.8	1.8	1.9	1.9	1.8	1.9	1.9	1.9	2	0.03	0.1	5
Ethylbenzene	1.7	1.8	1.7	1.7	1.6	1.5	1.7	1.7	5	0.08	0.3	6
m/p-Xylene	3.6	3.8	3.6	3.5	3.5	3.2	3.5	3.5	5	0.2	0.6	16
Methylene Chloride	9.3	9.2	9.7	9.9	10.0	10.4	10.3	9.8	5	0.5	1	23
o-Xylene	1.5	1.7	1.6	1.5	1.5	1.4	1.5	1.5	6	0.08	0.3	3
Styrene	1.3	1.3	1.3	1.2	1.3	1.1	1.2	1.2	5	0.06	0.2	6
Tetrachloroethene	2.7	3.0	2.7	2.5	2.6	1.7	2.7	2.6	16	0.4	1	6
Toluene	1.9	2.0	1.9	2.0	1.9	1.8	2.0	1.9	4	0.08	0.2	10
trans-1,2-Dichloroethene	3.5	3.8	4.3	4.3	4.3	4.3	4.2	4.1	8	0.3	1	6
Trichloroethene	1.8	1.9	1.9	1.9	1.8	1.8	1.9	1.9	3	0.06	0.2	18

Notes

- MDL was calculated based on seven repetitions of a 6.7ppb standard
- MDL was calculated using 3.14 times the Std. Deviation
- For Tekmar MDL, See Table 13

Table 30. Comparison of Tekmar and Tufts Purge and Trap GC/MS

Sample ID: S2-B1-(12-14)				Sample ID: S3-B5-(10-12)			
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
Ethylbenzene	10	23	20	Chloroform	2	4	15
m/p-xylene	9	46	33	cis-1,2-dichloroethene	4	3	6
o-xylene	21	32	11	m/p-xylene	2	5	21
Tetrachloroethene	17	39	20	o-xylene	4	4	2
Toluene	21	57	23	Toluene	17	4	12
Sample ID: S3-B8-(6-8)				Sample ID: S2-B11-(16-18)			
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
Chloroform	ND	6		Toluene	1	5	34
cis-1,2-dichloroethene	12	26	18	Sample ID: S2-B12-(18-20)			
Ethylbenzene	3	4	8	<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
m/p-xylene	3	14	32	m/p-xylene	ND	4	
o-xylene	10	12	6	Styrene	ND	4	
Toluene	3	22	38	Toluene	1	13	43
Sample ID: S3-B2-(12-14)				Sample ID: S2-B15A-(10-12)			
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
m/p-xylene	4	5	10	Toluene	ND	33	
Toluene	1	7	37	Sample ID: S2-B3-(18-20)			
Sample ID: S3-B3-(10-12)				<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	Chloroform	2	5	22
m/p-xylene	1	4	29	Ethylbenzene	39	30	7
Sample ID: S2-B12-(9-11)				m/p-xylene	38	25	10
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	o-xylene	115	66	14
Chloroform	2	6	24	Toluene	7	12	15
m/p-xylene	ND	5		Sample ID: S2-B4-(10-12)			
Sample ID: S2-B13-(8-10)				<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	Toluene	2	5	22
Toluene	1	5	33	Trichloroethene	ND	4	
Sample ID: S2-B7-(8-10)				Sample ID: S2-B9-(6-8)			
<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>	<u>Compounds</u>	<u>PT</u>	<u>TD</u>	<u>RPD</u>
Toluene	2	4	18	m/p-xylene	7	10	9

Table 30 continued. Comparison of Tekmar and Tufts Purge and Trap GC/MS

Sample ID: S2-B1-(20-22)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>RPD</u>
Ethylbenzene	6	5	5	5 (14)	9	8	17	11 (39)	18
m/p-xylene	68	59	60	62 (8)	66	108	111	95 (26)	10
o-xylene	166	143	145	151 (8)	119	146	159	141 (14)	2
Styrene	ND	ND	ND		5	4	3	4 (32)	
Tetrachloroethene	45	34	35	38 (16)	64	85	112	87 (28)	20
Toluene	64	49	49	54 (16)	65	88	93	82 (18)	10

Sample ID: S2-B5-(8-10)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>RPD</u>
Toluene	ND	ND	ND		4	18	23	15 (65)	

Sample ID: S2-B14-(8-10)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>RPD</u>
Toluene	ND	ND	ND		5	7	4	5 (27)	

Sample ID: S2-B2-(10-12)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>RPD</u>
o-xylene	2	3	3	3 (22)	8	5	4	6 (35)	20
Styrene	ND	ND	ND		10	6	5	7 (31)	
Toluene	3	3	2	3 (22)	10	8	8	9 (9)	27

Sample ID: S3-B4-(12.5-14)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>RPD</u>
Chloroform	1	2	2	2 (35)	2	7	5	5 (55)	25
Ethylbenzene	3	2	1	2 (50)	4	9	7	7 (33)	27
m/p-xylene	5	3	3	4 (35)	13	20	10	14 (33)	29
o-xylene	15	10	6	10 (44)	13	19	14	15 (22)	10
Toluene	4	2	2	3 (46)	12	22	14	16 (33)	35

Table 30 continued. Comparison of Tekmar and Tufts Purge and Trap GC/MS

Sample ID: S2-B10-(6-8)

<u>Compounds</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>PT</u>	<u>RPD</u>
Toluene	7	5	4	6 (25)	ND	

Sample ID: S3-B4-(10-12)

<u>Compounds</u>	<u>TD1</u>	<u>TD2</u>	<u>TD3</u>	<u>Ave (RSD)</u>	<u>PT</u>	<u>RPD</u>
Ethylbenzene	19	11	17	16 (28)	7	20
m/p-xylene	14	20	15	17 (20)	18	5
o-xylene	23	21	21	22 (6)	38	21
Toluene	13	12	16	14 (14)	ND	

Sample ID: S3-B3-(6-8)

<u>Compounds</u>	<u>PT1</u>	<u>PT2</u>	<u>PT3</u>	<u>Ave (RSD)</u>	<u>TD</u>	<u>RPD</u>
Toluene	2	1	2	2 (35)	4	20

Table 31. ICP/OES Instrument Detection Limit Study for Metals

Analyte	Wavelength (nm)		Repetitions							Std. Dev.	IDL x3.143
	Backgrd.		1	2	3	4	5	6	7		
K	766.490	766.422	1.926	2.001	2.096	2.045	1.951	2.147	2.158	0.0919	0.2889
Ca	317.933	317.902	0.0295	0.0282	0.0306	0.0303	0.0308	0.0301	0.0297	0.0009	0.0027
Mg	279.079	279.109	0.0883	0.1094	0.1192	0.1072	0.0911	0.090	0.0917	0.0122	0.0385
Na	589.592	589.535	0.4510	0.4773	0.3972	0.4081	0.4194	0.4356	0.4303	0.0270	0.0848
Ag	328.068	328.033	0.0397	0.0396	0.0406	0.0389	0.0454	0.0419	0.0381	0.0024	0.0077
Mn	257.610	257.589	0.0037	0.0041	0.0042	0.0047	0.0044	0.0044	0.0047	0.0004	0.0011
Zn	206.200	206.220	0.0120	0.0121	0.0154	0.0129	0.0122	0.0134	0.0142	0.0013	0.0040
Cr	267.716	267.690	0.0215	0.0189	0.020	0.0206	0.0218	0.0222	0.0201	0.0011	0.0036
Ni	231.604	231.583	0.0459	0.0452	0.0437	0.0463	0.0456	0.0484	0.0471	0.0015	0.0047
Al	308.215	308.248	0.1569	0.1473	0.1469	0.1577	0.1374	0.1476	0.1425	0.0073	0.0229
Be	313.042	313.011	0.0008	0.0008	0.0008	0.0008	0.0008	0.0008	0.0008	0.0000	0.0000
Cu	324.754	324.789	0.0168	0.018	0.0148	0.0168	0.0153	0.015	0.0180	0.0014	0.0043
V	292.401	292.429	0.0228	0.0218	0.0234	0.0220	0.0213	0.0208	0.0207	0.0010	0.0032
Ba	455.403	455.355	0.0025	0.0035	0.0026	0.0023	0.0031	0.0029	0.0027	0.0004	0.0013
Co	228.616	228.592	0.0179	0.025	0.0227	0.0207	0.0252	0.0206	0.0189	0.0028	0.0089
Fe	259.940	259.915	0.0159	0.0152	0.0185	0.0167	0.0173	0.0183	0.0159	0.0013	0.0040
As	193.695	193.712	0.1632	0.1446	0.1557	0.0954	0.2071	0.1382	0.1923	0.0367	0.1155
Cd	214.438	214.423	0.0057	0.0061	0.0069	0.0054	0.0072	0.0049	0.0063	0.0008	0.0026
Pb	220.353	220.335	0.1081	0.1078	0.1159	0.0993	0.1319	0.1032	0.1267	0.0122	0.0382
Se	196.026	196.038	0.1404	0.1676	0.1574	0.1366	0.1050	0.1785	0.1781	0.0266	0.0835
Tl	190.801	190.789	0.1048	0.0842	0.1338	0.0533	0.1858	0.0911	0.1682	0.0476	0.1496
Sb	206.833	206.853	0.0857	0.1219	0.0742	0.1252	0.0652	0.0787	0.0568	0.0268	0.0841

Note: EPA 40 CFR Ch. 1 (7-1-92) Pt. 136, App. B

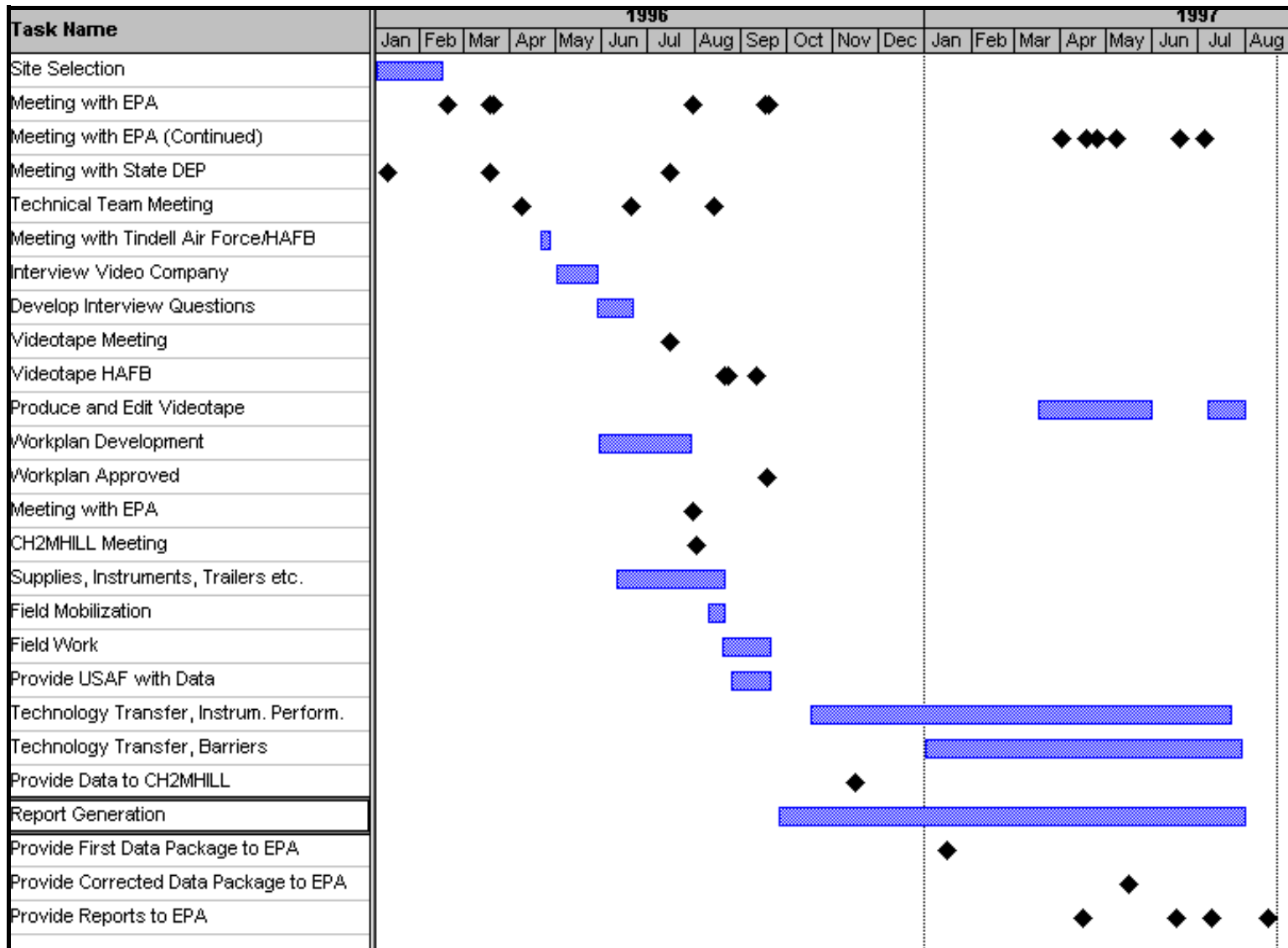
Table 32. Summary of Detection Limits for Mobile ICP/OES

Element	IDL	MDL IDL x 43.28	LOQ MDL x 3	LOQ Blank LOQ / 43.28	10 DAF (mg/kg)
K	0.2889	12.50	38	0.87	NA
Ca	0.0027	0.12	0.36	0.01	NA
Mg	0.0385	1.67	5.00	0.12	NA
Na	0.0848	3.67	11.0	0.25	NA
Ag	0.0077	0.33	1.00	0.02	17
Mn	0.0011	0.05	0.14	0.003	NA
Zn	0.0040	0.17	0.52	0.01	6000
Cr	0.0036	0.16	0.47	0.01	19
Ni	0.0047	0.20	0.60	0.01	65
Al	0.0229	0.99	3.00	0.07	NA
Be*	0.0003	0.01	0.04	0.001	32
Cu	0.0043	0.19	0.56	0.01	NA
V	0.0032	0.14	0.41	0.01	3000
Ba	0.0013	0.05	0.16	0.004	800
Co	0.0089	0.39	1.16	0.03	NA
Fe	0.0040	0.17	0.52	0.01	NA
As	0.1160	5.02	15.0	0.35	15
Cd	0.0026	0.11	0.33	0.008	4
Pb	0.0382	1.65	5.00	0.12	NA
Se	0.0835	3.61	10.8	0.25	3
Tl	0.1496	6.47	19.4	0.45	0.4
Sb	0.0841	3.64	11.0	0.25	3

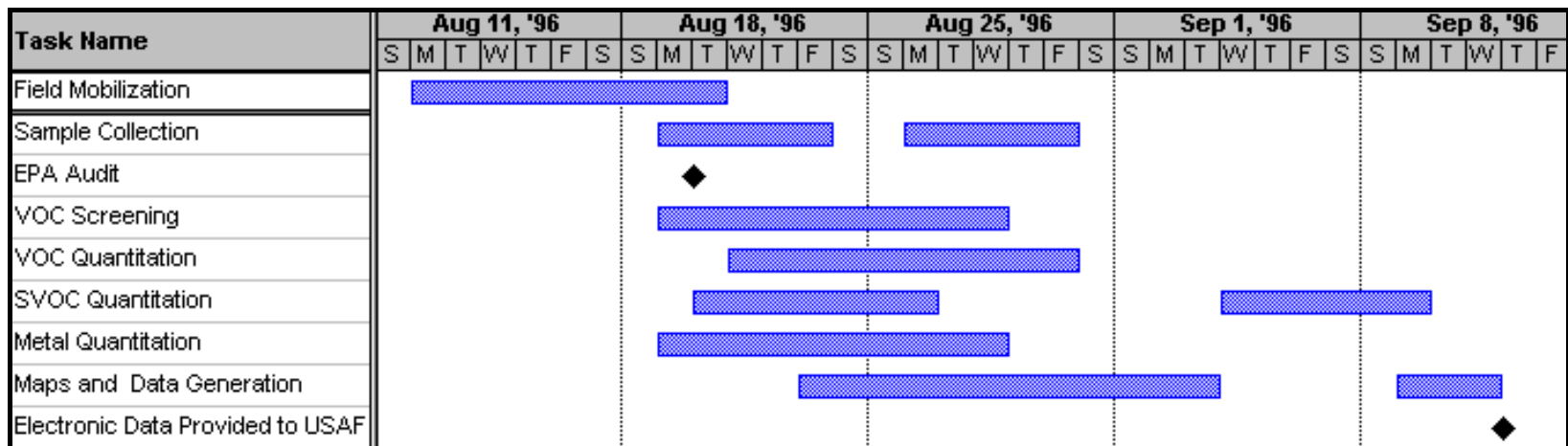
* Used beryllium theoretical value because experimental sensitivity was lower

Appendix II

**Illustrated on the following pages are the
Environmental Technology Initiative and HAFB Field Investigation Timelines.**



ETI Project Timeline



Field Investigation Timeline

Appendix III

The field method Standard Operating Procedures are provided in this section

STANDARD OPERATING PROCEDURE

HANSCOM AFB, MASSACHUSETTS

IRP JUN 1996, Contract No. F41624-94-D-8053, Delivery Order 0014

FIELD GC/MS METHOD FOR ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SOIL AND WATER

- SCOPE:** This procedure is for the analysis of volatile organic compounds (VOCs) in soil and water by purge and trap (P&T) concentration followed by gas chromatography/mass spectrometry (GC/MS).
- SUMMARY:** Both water and soil samples are added to a sparge vessel and purged with helium using a Tekmar 3000 concentration device. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept onto a sorbent column where the volatile organic compounds are trapped. After purging is completed the sorbent trap is flash heated and the volatile compounds are transferred via a heated line onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the volatile compounds which are then detected with a mass spectrometer.
- REQUIRES:** This procedure requires that the operator be familiar with the set-up and operation of both P&T apparatus and GC/MS. This SOP covers procedures for sample and standard preparation, calibrations, quantitation, identification, and quality control. The procedure does not cover data operating systems or reporting. A P&T concentration device, HP GCD GC/MS (a HP5890GC/5972MS can be used as well) and acquisition software are required for the procedure.

1.0 EQUIPMENT AND RUN CONDITIONS

1.1 Purge & Trap Device. A Tekmar LSC 3000 or equivalent is recommended for the Hanscom project. The Purge and Trap (P&T) is a concentration device for volatiles in both soil and water. A sparger vessel, a removable glass tube, is used to hold either the water or soil samples. A polymer trap is used to trap the volatiles as they are purged from the sample. After the volatiles are purged from the sample, the trap is flash heated and the volatiles are transferred to the GC for separation, identification, and quantitation.

1.2 Purge & Trap Run Conditions. Run conditions for the Hanscom project are listed below.

- Polymer trap: Supelco (# 3, Tenex, charcoal, and silica gel) or equivalent.
- Purge rate: 30 mL/minute \pm 5 mL/minute.
- Purge: 5 minutes at a temperature of 40°C.
- Desorb: 2 minutes at 225°C.
- Bake: 6 minutes at 230°C.
- Mount temperature: off.
- Line and valve temperatures: 180°C.
- Bake gas delay: on for 2 minutes.

1.3 HP G1800A GCD System Run Conditions. The HP-GCD system consist of a gas chromatograph, an electron ionization detector (EID), and a data system. The gas chromatograph provides compound separation while the detector generates traditional retention time and abundance information. The detector is capable of scanning from 1 to 415 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The MS requires no external cooling or an environmentally controlled room for operation. The following run conditions are recommended for the Hanscom project, however they may be altered by the field chemist based on actual site conditions.

- Carrier gas: Helium at 99.999% purity.
- Flow of 2.0 mL/minute.
- Injector temperature: 225°C.
- Initial temperature: 50°C.
- Initial time: 4 minutes.
- Rate: 8°C/minute.
- Final temperature: 150°C.
- Final time: 0 minutes.
- Run Time: 16.5 minutes.
- Detector temperature: 280°C.
- Mass range: 45 to 260 amu.
- Solvent delay: 3.0 minutes.
- Split flow: 40 mL/minute.
- Split ratio: 20:1

1.4 Column. A number of columns are available for volatile analysis, however, for the Hanscom project a DB-624, 60 meter, 0.25mm ID microbore column, 0.25 μ m film thickness (or equivalent) will be used.

2.0 CONVENTIONS.

2.1 Standards. Standard conventions are established to ensure consistent procedures from project to project and from operator to operator. This includes standard preparation, documentation, calculations, and tracking.

2.1.1 Stock Standards. Stock chemical standards will be purchased from Supelco, Inc., Chem Service, Inc., or an equivalent supplier. All standard information will be logged in a bound logbook with the pages sequentially numbered. This information will include, at a minimum:

- Vendor name supplying standards.
- Name and concentration of the standard (this can be done by attaching the label from the standard to the logbook).
- Lot number of the standard.
- Expiration date of the standard, if listed.

2.1.2 Working Standards. Working chemical standards may either be purchased through an approved vendor or prepared from stock standards. Working standards will be made by diluting neat or prepared stock standards. For standards made from neat solutions, the compound density or weight will be used in calculating the appropriate amount of compound and solvent to be combined. All working standards will be labeled with an identification code (see below), compound or mix name, and concentration. The associated logbook will contain the following information at a minimum:

- The Stock solution used to make the working standard, including either the information required for logging the stock solution or a reference to where that information can be found.
- Both a written description and calculation of how the working standard was prepared.
- The solvent(s) and associated lot number(s) used in preparing the standards.
- The final concentration of the working standard.
- The GC/MS code associated with the standard.
- Standard and stock solutions will be kept refrigerated at less than 10°C when not in use.
- The surrogate standard is made separately from the working standards.

2.2 GC/MS Coding System. The GC/MS coding system is used to trace all standards back to the vendor. All standard are required to have the appropriate code assigned when they are prepared and must be labeled accordingly. The GC/MS code will follow the format:

- FGCXXXXXXWWYZPPPP Where:
- XXXXXX is the month, day, and year that the standard was made, i.e., 041696 = April 16, 1996.
- WW is the page in the logbook where the standard can be found, i.e., 01 to 99.
- Y is where the standard fell chronologically on the page, i.e., A through Z.
- Z is the logbook number where the standard can be found, i.e., 1 through 9.
- PPPP is the project identifier, i.e., USAF = United States Air Force, a short character identification of the project from 1 to 4 characters in length.

2.3 Logbook Entries. While the style and specific requirements for logbook entries will vary between operators and projects, certain information is required for all projects. At a minimum this will include:

- A table of contents listing what and where specific information is located.
- A listing of P&T and GC/MS run conditions and set points.
- A chemical standards preparation section containing the required standards information.
- A run log section containing at a minimum: sample identification, run number or computer file identification, sample amount (weight or volume), spiked surrogate amount and % recovery, standard code for each standard used, amount of standard(s) used, an example calculation for any calculations performed, dilution factor, and a remarks column with any pertinent information (e.g., unusual sample color or odor, unusable blanks, failed standards, coelutions, reruns, etc.).
- An equipment maintenance section containing both routine and equipment malfunction maintenance (e.g., column replacement, electronic parts replacement, GC/MS repair, replacement, or cleaning, etc.)

3.0 TUNING

3.1 HP-GCD Tune. The HP-GCD uses an auto-tune macro to tune the mass spectrometer. Three masses of the calibration compound (PFTBA) are selected: 69, 219, and 414. The procedure is automatically implemented by the GCD control software, once every 24 hours. For all 3 masses, starting with 69 amu, the MS system adjusts the mass, peak width and energy in that order until it achieves the desired values. The resolution is set to unity with a *ca.* 10% valley definition; this is done by measuring the actual peak width and adjusting the peak width parameter accordingly. The GC/MS system will be tuned at the start of each day prior to analyzing samples. The tune is considered valid for each 24 hour period. The following conditions must be met before the HP-GCD auto-tune will pass verification:

- Base peak must be mass 69 and fall between 68.80 and 69.20 amu.
- Position of mass 219 must be between 218.80 and 219.20 amu.
- Position of mass 414 must be between 413.80 and 414.20 amu.
- Position of isotope mass 70 must be between 69.80 and 70.20 amu.

- Position of isotope mass 220 must be between 219.80 and 220.20 amu.
- Position of isotope mass 415 must be between 414.80 and 415.20 amu.
- Ratio of mass 70 to mass 69 must be between 0.5 and 1.6%.
- Ratio of mass 220 to mass 219 must be between 3.2 and 5.4%.
- Ratio of mass 415 to mass 414 must be between 6.8 and 11.2%.
- Ratio of mass 219 to 69 should be > 15%.
- Ratio of mass 414 to 69 should be > 0.2%.
- Mass 69 precursor <= 3%.
- Mass 219 precursor <= 6%.
- Mass 414 precursor <= 12%.
- Ratio of mass 18 to 69 should be <20%.
- Ratio of mass 28 to 69 should be <10%.

4.0 CALIBRATION.

4.1 Multilevel Calibration. In the initial multilevel calibration, a minimum of five levels (concentrations) of standards are analyzed. The standard concentrations are selected to cover the concentration range of contaminants expected. This includes a low concentration standard at or near the method detection limit (MDL). In addition, the upper level standard needs to be within the working range of both the detector and the column. The expected calibration range is from 20 to 800 ppb.

4.2 Daily Calibration. After mobilizing the GC/MS unit to the field, the analyst must prepare calibration standards at a minimum of five concentrations levels for each compound by carefully adding amounts of one or more secondary dilution standards to reagent water for purging. One of the calibration standards should be at a concentration near, but above, the MDL. The other concentrations should correspond to the expected range of concentrations found in the "real-world" sample or the establishment of the GC/MS dynamic range.

4.3 Internal Standard and Surrogate Calibration Procedure. Prepare a solution that contains at least Toluene-d8 (internal standard), and another one that contains 4-Bromofluorobenzene (surrogate) and 1,4-Difluorobenzene (surrogate). using the procedures described in Sections 2.0 and 2.1. The concentrations of all three compounds should be 100 ppb or 100-ng/ μ L. Toluene-d8 is selected as the internal standard because of its retention characteristics relative to VOCs on USEPA's Target Compound List (TCL). It does not coelute with any of the compounds of interest; the closest eluting compound, Toluene, is baseline separated.

4.4 Calibration Curve. Add 5 mL of analyte free water to a luer-lock syringe. With separate syringes add the internal standard solution, surrogate solution, and the target volatile mix to the luer-lock syringe. Connect the luer-lock syringe to the Tekmar 3000 sparger apparatus and load the sample for purging. The internal standard and surrogate concentrations should be 100 ppb (inject 5 μ L of 100 ng/ μ L internal standard solution and 5 μ L of 100 ng/ μ L surrogate solution into 5.0 mL reagent water). Add 1 μ L of total volatile mix at 100 ng/ μ L to the 5 mL of reagent water (containing the internal standard and surrogate solution) for a concentration of 20 ppb.

Repeat the process for each additional concentration level by increasing the amount of volatile mix added to the 5 mL of reagent water, i.e., 5 µL of volatile solution would be equal to 100 ppb, etc.). The internal standard amount and surrogate amounts remain constant during the calibration process. Note: The surrogate solution is normally varied in concentration as is the volatile mix, however for this project it will be kept constant to allow for alternative choices for the internal standard.

4.5 Response Factors. Repeat analysis one time for each concentration. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using Equation 1:

$$RF = (A_x)(C_{is}) / (A_{is})(C_x) \quad [1]$$

where, A_x = area of the standard target analyte ion current signal, C_{is} = concentration of the internal standard, A_{is} = area of the internal standard ion current signal, C_x = concentration of the standard target analyte.

If the RF value is constant (i.e., < 30% relative standard deviation, with no more than one third of the samples with > 30% RSD for quantitative GC/MS) over the dynamic range, the RF can be assumed to be invariant and the average from the RF value(s) can be used for quantitation. If the average RF values for any analytes fall outside of the 40% RSD window, the instrument must be checked for mass drift and the point or points skewing the curve must be performed again.

4.6 Continuing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard. If all of the analyte RF's from the continuing standard have respective %D's within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the continuing calibration criteria are not achieved, then a new calibration curve with a minimum of five concentration levels will be generated.

4.6 Closing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard (400 ppb). If all of the analyte RF's from the continuing standard have respective %D's within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the closing calibration is still not in control, a new calibration curve must be generated and all samples analyzed during the previous calibration period must either be re-analyzed or flagged to indicate that the system was not within the QC requirements.

5.0 METHOD DETECTION LIMITS

5.1 MDL Determination. The MDLs are determined for each compound in both water and soil prior to the analysis of samples. This is accomplished by analyzing seven replicate low concentration standards near the expected MDL. From these analysis a standard deviation is calculated and multiplied by 3.14 to establish the MDL. This correlates to a student's t of 99% for (n-1).

5.1.1 Water MDL. Water MDLs will be performed using 5 mL of analyte free reagent water spiked with the target compounds at 20 ppb.

5.1.2 Soil MDL. Soil MDLs will be performed using a minimum of 5 grams of blank soil (ERA volatiles blank soil, catalog # 054) spiked with the target compounds at 20 ppb.

6.0 COMPOUND IDENTIFICATION

6.1 Total Ion Current Mode. Compounds will be identified using the total ion current mode (TIC). TIC's for the primary ion and at least two secondary ions for each compound of interest must be identified. The following criteria must be met to make a qualitative identification.

6.1.1 Characteristic Ions. The characteristic ions of each compound of interest must maximize within one scan of each other.

6.1.2 Retention Time. The retention time of the suspected analyte must fall within ± 30 seconds of the retention time of the actual target analyte as determined by the calibration data.

6.1.3 Relative Peak Heights/Areas. The relative peak heights/areas of the three characteristic ions in the TIC's must fall within $\pm 40\%$ of the relative intensities of these ions according to the mass spectra obtained during calibration for targeted analytes or from a reference mass spectrum for non-targeted analytes.

6.1.4 Structural Isomers. Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mixture is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

<u>Compounds</u>	<u>Ions</u>
Vinyl Chloride	62,64,61
1,1-Dichloroethene	96,61,98
Methylene Chloride	84,49,51,86
trans-1,2-Dichloroethene	96,61,98
1,1-Dichloroethane	63,65,83
cis-1,2-Dichloroethene	96,61,98
Chloroform	83,85,47
1,1,1-Trichloroethane	97,99,117
Carbon Tetrachloride	117,119,121
Benzene	78,52,71
1,2-Dichloroethane	62,64,98
Trichloroethene	130,95,97,132
†Toluene-d8	98,100,70
Toluene	92,91,65
Tetrachloroethene	164,129,131,166
Chlorobenzene	112,114,77
Ethylbenzene	106,91
m/p-Xylene	106,91
o-Xylene	106,91
Styrene	104,103,78,51,77
‡1,4-Difluorobenzene	114,63,88
‡4-Bromofluorobenzene	95,174,176
† Internal Standard,	
‡ Surrogate Compounds.	

7.0 CALCULATIONS

7.1 Quantitation. When a compound has been identified the quantitation of that compound should be based on the integrated abundance from the TIC of the primary ion. If the sample produces an interference for the primary ion then a secondary characteristic ion will be used to quantitate.

7.1.1 Water Samples. Calculate the concentration of the compound using the internal standard calculation procedure described in Section 4.5 and Equation 2:

$$\text{Concentration } \mu\text{g/L} = (A_x)(C_{is})/(A_{is})(RF)(V_o) \quad [2]$$

where the parameters used in equation [2] are described in Section 4 and V_o = volume of water purged (mL), taking into consideration any dilutions made.

7.1.2 Soil Samples. Calculate the concentration of the compound using the internal standard calculation procedure described in Section 4.5 and Equation 3:

$$\text{Concentration } \mu\text{g/kg} = (A_x)(C_{is})/(A_{is})(RF)(W_s)(D) \quad [3]$$

where the parameters used in equation [3] are described in Section 4, W_s = weight of sample extracted or purged (g), and D = % dry weight of sample/100, or 1 for a wet weight basis.

8.0 BLANKS

8.1 Volatile Blanks. There are two types of blanks associated with purge and trap analysis; low level method blanks and cleaning blanks.

8.1.1 Low Level Method Blanks. For routine analysis, a low level method blank must be analyzed before samples are analyzed. A low level method blank consist of 5 mL (or at the volume that samples are to be analyzed, i.e., 25 mL, 50 mL, etc.) of analyte free water that has a surrogate and internal standard added. Method blanks are acceptable if no target compounds are present above the reporting limit. Samples should not be analyzed until an acceptable method blank is run demonstrating that the instrument is free of interferences.

8.1.2 Cleaning Blanks. A cleaning blank is 5 mL of reagent water only. Blanks will be analyzed after any high level sample to ensure that carryover is not occurring. A high level sample is defined as having a concentration 5 times higher than the highest calibration point. Cleaning blanks will be analyzed until the analysis of further samples will not be affected by carryover.

9.0 SAMPLE PREPARATION

9.1 Sample Preparation. Sample analysis and preparation techniques have been adapted from protocols outlined in SW-846 3rd ed. USEPA Purge and Trap method 8260A (USEPA 1986).

9.1.1 Water Samples. Rinse a 5 mL (or 25 mL, depending on sample aliquot) syringe with one volume of sample. Draw the sample into the syringe, invert and remove all air adjusting the final volume to 5.0 mL. Add the appropriate amount of surrogate and internal standard directly to the sample. Deliver the sample to the sparger and start the purge process.

9.1.2 Soil Samples. Weigh 5 g +/- 0.5 g (or appropriate sample amount) into the sparging vessel. Add surrogate and internal standard to 5.0 mL reagent water (or to an equal amount of water that correlates to the soil amount) and add into the sparging vessel and start the purge cycle.

9.1.3 Total Solids. All sample results will be reported on a dry weight basis. Place approximately 10 grams of wet soil into a pre-weighed tin and dry in an oven (or use a heat lamp) until repetitive weighings show no further drying is occurring. Subtract the weight of the tin from the wet weight and from the dry weight. Divide the adjusted dry weight by the adjusted wet weight and multiply by 100 to get the percent total solids.

10 QUALITY CONTROL

10.1 Quality Control. The following procedures will be implemented by the field chemist to insure standardization of the operating procedures:

10.1.1 Review. The field chemist will review each chromatogram before analyzing the next sample. The review will include the calculation of surrogate recoveries, comparison of surrogate and target compound retention times to calibration standards, and the evaluation of carryover potential.

10.1.2 Water Surrogate Recoveries. 4-Bromofluorobenzene and 1,4-Difluorobenzene will be the primary surrogates for the Hanscom project. Surrogate recoveries for water samples must be greater than 50 percent and less than 150 percent. Water samples with surrogate recoveries of less than 50 percent must be reanalyzed to confirm matrix interferences. Water samples with recoveries greater than 150 percent will be noted in the log book. If the surrogate recovery is less than 50 percent for the water sample then the sample will be reanalyzed. If the surrogate fails the second analysis the results will be reported and the recovery noted in the logbook.

10.1.3 Soil Surrogate Recoveries. 4-Bromofluorobenzene and 1,4-Difluorobenzene will be the primary surrogates for the Hanscom project. Surrogate recoveries for soil samples must be greater than 30 percent and less than 200 percent. Soil samples with surrogate recoveries of less than 30 percent must be reanalyzed to confirm matrix interferences. Soil samples with recoveries greater than 200 percent will be noted in the log book. If the surrogate recovery is less than 30 percent for the soil sample then the sample will be reanalyzed. If the surrogate fails the second analysis the results will be reported and the recovery noted in the logbook.

10.1.4 Duplicates and Triplicates. Duplicates (or triplicates) will be analyzed for 5% of the analytical samples to calculate precision. When duplicates are analyzed a 30% RPD criteria will be used as the upper control limit. When triplicates are analyzed a 30% RSD will be used as the upper control limit. All results will be calculated for review in the logbook and in a quality control summary if requested.

11 REPORTING

11.0 Reporting. Data from all sample analyses and relevant calibration and blank analyses will be documented in the project GCMS run logbook. A quality control summary may be generated at the completion of the project and will include some or all of the following as required: initial calibrations, continuing calibrations, surrogate recoveries, method blanks, dilutions, reanalyzes, observations of the field chemist, problems and fixes, unknown peaks, raw data, etc..

REFERENCES:

U.S. Environmental Protection Agency, 1986, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 8260A; SW-846"; Office of Solid Waste and Emergency Response, Washington, D.C.

STANDARD OPERATING PROCEDURE ADDENDUM

1.0 TUNING OF THE BRUKER TDGC/MS

1.1 Bruker MS Tune - For the automatic tuning of the instrument, eight masses of the calibration compound (FC-77, a mixture of fluorinated hydrocarbons) are selected: 69, 100, 119, 169, 219, 269, 331, and 397 as well as argon ($m/z = 70$) and moisture ($m/z = 18$) in the carrier gas (air). The procedure is automatically implemented upon manual selection of the automatic calibration feature of the instrument. For all 10 masses, starting with 18 amu, the MS system adjusts the mass, peak width and energy in that order until it achieves the optimized values. The resolution is set to unit with a ca. 10% valley definition; this is done by measuring the actual peak width and adjusting the peak width parameter accordingly.

2.0 TDGC/MS FIELD SCREENING METHOD FOR VOLATILES

2.1 Thermal desorption gas chromatograph/mass spectrometer (Bruker Instruments, Billerica, MA) will be used to initially screen the 4-ft. collection liners from the geoprobe. The GC will be held isothermal at 50°C, the Bruker thermal desorption probe will be held isothermal at 45°C, and the mass spectrometer assigned to target compounds known to be present (from past data) at the site.

2.1.1 At the start of each day on site, the Bruker TDGC/MS is tuned according to the criteria listed above. 620-ng of target analytes are then injected into the center of a geoprobe sleeve filled with blank soil. After 2 minutes, the thermal desorption probe is held over a hole cut in the sleeve directly above the location of the injection, and responses are noted for each target analyte. This is to test the sensitivity of the method at approximately the 10-ppb level (based on the sample size of 62-g, the approximate weight of indigenous soil contained in a 2-in slice of the geoprobe sleeve). A one point calibration is performed using the above procedure at approximately the 40-ppb level in order to provide rough quantitative data.

2.1.2 Each 4-ft geoprobe plastic sleeve received on site is marked to show 1-ft sections along the length of the tube. A hole is cut using a clean knife at the approximate center of the 1-ft section. The Bruker's thermal desorption probe is then held directly over the hole and responses are noted for each target analyte. If there is no response after a period of 1-min, the section is considered blank. This procedure is then repeated for each 1-ft section of the sleeve, and responses noted.

2.1.3 Data generated using this screening method is used to determine which, if any, 2-ft section of the geoprobe boring is to be sampled for further laboratory analysis. This data is also used to quickly determine whether more 'step-out' boring samples are needed at a particular boring location.

2.1.4 The following analytes are to be targeted for on-site screening analysis:

Vinyl Chloride
Dichloroethene
Dichloroethane
Trichloroethene
Trichloroethane
Tetrachloroethene
Chloroform
Benzene
Toluene
Ethylbenzene
Xylene

STANDARD OPERATING PROCEDURE

HANSCOM AFB, MASSACHUSETTS

IRP JUN 1996, Contract No. F41624-94-D-8053, Delivery Order 0014

FIELD TDGC/MS METHOD FOR ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SOIL AND WATER

SCOPE: This procedure is for the analysis of volatile organic compounds (VOCs) in soil and water by purge and trap (P&T) concentration followed by Thermal Desorption Gas Chromatography/Mass Spectrometry (TDGC/MS).

SUMMARY: Both water and soil samples are added to a sparge vessel and purged with high purity nitrogen (or equivalent). The purgeables are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile organic compounds are trapped. After purging is completed the tube is placed into an thermal desorption injection port attached to the GC oven. The sorbent tube is heated and flushed with carrier gas. The VOC's are thermally desorbed from the sorbent and swept onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the volatile compounds which are then detected with a mass spectrometer.

REQUIRES: This procedure requires that the operator be familiar with the set-up and operation of both P&T apparatus and TDGC/MS. This SOP covers procedures for sample and standard preparation, calibrations, quantitation, identification, and quality control. The procedure does not cover data operating systems or reporting. A P&T concentration device, HP GCD TDGC/MS (or equivalent) and acquisition software are required for the procedure.

1.0 EQUIPMENT AND RUN CONDITIONS

1.1 Purge & Trap Device. The purge and trap device consists of three separate parts, the purge gas, the purge vessel, and the trap.

1.1.1 The sample purge vessel is designed to hold 20-mL, 40-mL, or 100-mL equivalent samples of water and soil. The purge vessel used for this method is available commercially by Wheaton Scientific, Millville, NJ. If samples of higher concentration are to be analyzed, the 20-mL impinger can be used. The purge gas must be introduced not more than 5-mm from the base of the water column. High purity nitrogen gas is attached at the top of the glass tube which is used to introduce purge gas through the water and soil samples.

1.1.2 The trap used in these methods, consists of a 100-mm long, 8-mm OD glass tube (SKC, Eighty Four, PA). The tubes can be filled with different sorbents for optimum trapping of VOCs. A 100-mg Tenax/50 mg Charcoal combination is recommended for the Hanscom project.

1.2 Purge & Trap Run Conditions. Run conditions for the Hanscom project are listed below.

- Polymer tubes: SKC, Supelco or equivalent (Tenax to charcoal 2:1).
- Purge rate: 30 mL/minute \pm 5 mL/minute.
- Purge: 5 minutes at a temperature of ambient temperature.
- Predesorb in the Thermal Desorber for 3 minutes at 235 °C.
- Desorb: 2 minutes at 235°C.
- Close valve and open Thermal Desorber, bake at 235°C.
- Bake: overnight at 180°C in a separate oven with nitrogen at low flow.

1.3 HP G1800A GCD System Run Conditions. The HP-GCD system consist of a gas chromatograph, an electron ionization detector (EID), and a data system. The gas chromatograph provides compound separation while the detector generates traditional retention time and abundance information. The detector is capable of scanning from 1 to 415 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The MS requires no external cooling or an environmentally controlled room for operation. The following run conditions are recommended for the Hanscom project, however they may be altered by the field chemist based on actual site conditions.

- Carrier gas: Helium at 99.999% purity.
- Flow of 2.0 mL/minute.
- Injector temperature: 225°C.
- Thermal Desorber: 235°C.
- Initial temperature: 0°C.
- Initial time: 1 minute.
- Rate: 50°C/minute.
- Final temperature: 50°C.
- Final time: 2 minutes.
- Rate A: 14°C/minute.
- Final temperature A: 180°C.

- Final time: 2.7 minutes.
- Run Time: 16.0 minutes.
- Detector temperature: 280°C.
- Mass range: 45 to 260 amu.
- Solvent delay: 1.0 minutes.
- Split flow: 40 mL/minute.
- Split ratio: 20:1

1.4 Column. A number of columns are available for volatile analysis, however, for the Hanscom project a DB-624, 60 meter, 0.25mm ID microbore column, 0.25µm film thickness (or equivalent) will be used.

2.0 CONVENTIONS.

2.1 Standards. Standard conventions are established to ensure consistent procedures from project to project and from operator to operator. This includes standard preparation, documentation, calculations, and tracking.

2.1.1 Stock Standards. Stock chemical standards will be purchased from Supelco, Inc., Chem Service, Inc., or an equivalent supplier. All standard information will be logged in a bound logbook with the pages sequentially numbered. This information will include, at a minimum:

- Vendor name supplying standards.
- Name and concentration of the standard (this can be done by attaching the label from the standard to the logbook).
- Lot number of the standard.
- Expiration date of the standard, if listed.

2.1.2 Working Standards. Working chemical standards may either be purchased through an approved vendor or prepared from stock standards. Working standards will be made by diluting neat or prepared stock standards. For standards made from neat solutions, the compound density or weight will be used in calculating the appropriate amount of compound and solvent to be combined. All working standards will be labeled with an identification code (see below), compound or mix name, and concentration. The associated logbook will contain the following information at a minimum:

- The Stock solution used to make the working standard, including either the information required for logging the stock solution or a reference to where that information can be found.
- Both a written description and calculation of how the working standard was prepared.
- The solvent(s) and associated lot number(s) used in preparing the standards.
- The final concentration of the working standard.
- The GC/MS code associated with the standard.

- Standard and stock solutions will be kept refrigerated at less than 10°C when not in use.
- The surrogate standard is made separately from the working standards.

2.2 GC/MS Coding System. The GC/MS coding system is used to trace all standards back to the vendor. All standard are required to have the appropriate code assigned when they are prepared and must be labeled accordingly. The GC/MS code will follow the format:

- FGCXXXXXXXXWWYZPPPP Where:
- XXXXXX is the month, day, and year that the standard was made, i.e., 041696 = April 16,1996.
- WW is the page in the logbook where the standard can be found, i.e., 01 to 99.
- Y is where the standard fell chronologically on the page, i.e., A through Z.
- Z is the logbook number where the standard can be found, i.e., 1 through 9.
- PPPP is the project identifier, i.e., USAF = United States Air Force, a short character identification of the project from 1 to 4 characters in length.

2.3 Logbook Entries. While the style and specific requirements for logbook entries will vary between operators and projects, certain information is required for all projects. At a minimum this will include:

- A table of contents listing what and where specific information is located.
- A listing of P&T and GC/MS run conditions and set points.
- A chemical standards preparation section containing the required standards information.
- A run log section containing at a minimum: sample identification, run number or computer file identification, sample amount (weight or volume), spiked surrogate amount and % recovery, standard code for each standard used, amount of standard(s) used, an example calculation for any calculations performed, dilution factor, and a remarks column with any pertinent information (e.g., unusual sample color or odor, unusable blanks, failed standards, coelutions, reruns, etc.).

3.0 TUNING

3.1 HP-GCD Tune. The HP-GCD uses an auto-tune macro to tune the mass spectrometer. Three masses of the calibration compound (PFTBA) are selected: 69, 219, and 502. The procedure is automatically implemented by the GCD control software, once every 24 hours. For all 3 masses, starting with 69 amu, the MS system adjusts the mass, peak width and energy in that order until it achieves the desired values. The resolution is set to unity with a *ca.* 10% valley definition; this is done by measuring the actual peak width and adjusting the peak width parameter accordingly. The GC/MS system will be tuned at the start of each day prior to analyzing samples. The tune is considered valid for each 24 hour period. The following conditions must be met before the HP-GCD auto-tune will pass verification:

- Base peak must be mass 69 and fall between 68.80 and 69.20 amu.

- Position of mass 219 must be between 218.80 and 219.20 amu.
- Position of mass 414 must be between 413.80 and 414.20 amu.
- Position of isotope mass 70 must be between 69.80 and 70.20 amu.
- Position of isotope mass 220 must be between 219.80 and 220.20 amu.
- Position of isotope mass 415 must be between 414.80 and 415.20 amu.
- Ratio of mass 70 to mass 69 must be between 0.5 and 1.6%.
- Ratio of mass 220 to mass 219 must be between 3.2 and 5.4%.
- Ratio of mass 415 to mass 414 must be between 6.8 and 11.2%.
- Ratio of mass 219 to 69 should be > 15%.
- Ratio of mass 414 to 69 should be > 0.2%.
- Mass 69 precursor <= 3%.
- Mass 219 precursor <= 6%.
- Mass 414 precursor <= 12%.
- Ratio of mass 18 to 69 should be <20%.
- Ratio of mass 28 to 69 should be <10%.

4.0 CALIBRATION.

4.1 Multilevel Calibration. In the initial multilevel calibration, a minimum of five levels (concentrations) of standards are analyzed. The standard concentrations are selected to cover the concentration range of contaminants expected. This includes a low concentration standard at or near the method detection limit (MDL). In addition, the upper level standard needs to be within the working range of both the detector and the column. The expected calibration range is from 20 to 800 ppb.

4.2 Daily Calibration. After mobilizing the GC/MS unit to the field, the analyst must prepare calibration standards at a minimum of five concentrations levels for each compound by carefully adding amounts of one or more secondary dilution standards to reagent water for purging. One of the calibration standards should be at a concentration near, but above, the MDL. The other concentrations should correspond to the expected range of concentrations found in the "real-world" sample or the establishment of the GC/MS dynamic range.

4.3 Internal Standard and Surrogate Calibration Procedure Prepare a solution that contains at least Toluene-d8 (internal standard), and another one that contains 4-Bromofluorobenzene (surrogate) and 1,4-Difluorobenzene (surrogate). using the procedures described in Sections 2.0 and 2.1. The concentration of all three compounds should be 66.7 ppb or 100-ng/ μ L. Toluene-d8 is selected as the internal standard because of its retention characteristics relative to VOCs on USEPA's Target Compound List (TCL). It does not coelute with any of the compounds of interest; the closest eluting compound, toluene, is baseline separated.

4.4 Calibration Curve. Add 15 mL of analyte free water to the 20 mL impinger. With separate syringes add the internal standard solution, surrogate solution, and the target volatile mix to the impinger. Connect the top of the impinger, attach the Tenex/charcoal sorption tube and start the nitrogen purge flow. The internal standard and surrogate concentrations should be

66.7 ppb (inject 10 μL of 100 ng/ μL internal standard solution and 10 μL of 100 ng/ μL surrogate solution into 15.0 mL reagent water). Add 2 μL of total volatile mix at 100 ng/ μL to the 15 mL of reagent water (containing the internal standard and surrogate solution) for a concentration of 13.3 ppb. Repeat the process for each additional concentration level by increasing the amount of volatile mix added to the 5 mL of reagent water, i.e., 5 μL of volatile solution would be equal to 33.3 ppb, etc.). The internal standard amount and surrogate amounts remain constant during the calibration process. Note: The surrogate solution is normally varied in concentration as is the volatile mix, however for this project it will be kept constant to allow for alternative choices for the internal standard.

4.5 Response Factors. Repeat analysis one time for each concentration. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using Equation 1:

$$\text{RF} = (A_x)(C_{is}) / (A_{is})(C_x) \quad [1]$$

where A_x = area of the standard target analyte ion current signal, C_{is} = concentration of the internal standard, A_{is} = area of the internal standard ion current signal, and C_x = concentration of the standard target analyte.

If the RF value is constant (i.e., < 30% relative standard deviation, with no more than one third of the samples with > 30% RSD for quantitative GC/MS) over the dynamic range, the RF can be assumed to be invariant and the average from the RF value(s) can be used for quantitation. If the average RF values for any analytes fall outside of the 40% RSD window, the instrument must be checked for mass drift and the point or points skewing the curve must be performed again.

4.6 Continuing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard. If all of the analyte RFs from the continuing standard have respective %Ds within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the continuing calibration criteria are not achieved, then a new calibration curve with a minimum of five concentration levels will be generated.

4.7 Closing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard. If all of the analyte RFs from the continuing standard have respective %Ds within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the closing calibration is still not in control, a new calibration curve must be generated and all samples analyzed during the previous calibration period must either be re-analyzed or flagged to indicate that the system was not within the QC requirements..

5.0 METHOD DETECTION LIMITS

5.1 MDL Determination. The MDLs are determined for each compound in both water and soil prior to the analysis of samples. This is accomplished by analyzing seven replicate low concentration standards near the expected MDL. From these analysis a standard deviation is calculated and multiplied by 3.14 to establish the MDL. This correlates to a student's t of 99% for $(n-1)$.

5.1.1 Water MDL. Water MDLs will be performed using 15 mL of analyte free reagent water spiked with the target compounds at 6.7 ppb.

5.1.2 Soil MDL. Soil MDLs will be performed using a minimum of 15 grams of blank soil (ERA volatiles blank soil, catalog # 054) spiked with the target compounds at 6.7 ppb.

6.0 COMPOUND IDENTIFICATION

6.1 Total Ion Current Mode. Compounds will be identified using the total ion current mode (TIC). TICs for the primary ion and at least two secondary ions for each compound of interest must be identified. The following criteria must be met to make a qualitative identification.

6.1.1 Characteristic Ions. The characteristic ions of each compound of interest must maximize within one scan of each other.

6.1.2 Retention Time. The retention time of the suspected analyte must fall within ± 30 seconds of the retention time of the actual target analyte as determined by the calibration data.

6.1.3 Relative Peak Heights/Areas. The relative peak heights/areas of the three characteristic ions in the TICs must fall within $\pm 40\%$ of the relative intensities of these ions according to the mass spectra obtained during calibration for targeted analytes or from a reference mass spectrum for non-targeted analytes.

6.1.4 Structural Isomers. Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mixture is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

<u>Compounds</u>	<u>Ions</u>
Vinyl Chloride	62,64,61
1,1-Dichloroethene	96,61,98
Methylene Chloride	84,49,51,86
trans-1,2-Dichloroethene	96,61,98
1,1-Dichloroethane	63,65,83
cis-1,2-Dichloroethene	96,61,98
Chloroform	83,85,47
1,1,1-Trichloroethane	97,99,117
Carbon Tetrachloride	117,119,121
Benzene	78,52,71
1,2-Dichloroethane	62,64,98
Trichloroethene	130,95,97,132
†Toluene-d8	98,100,70
Toluene	92,91,65
Tetrachloroethene	164,129,131,166
Chlorobenzene	112,114,77
Ethylbenzene	106,91
m/p-Xylene	106,91
o-Xylene	106,91
Styrene	104,103,78,51,77
‡1,4-Difluorobenzene	114,63,88
‡4-Bromofluorobenzene	95,174,176
† Internal Standard,	
‡ Surrogate Compounds.	

7.0 CALCULATIONS

7.1 Quantitation. When a compound has been identified the quantitation of that compound should be based on the integrated abundance from the TIC of the primary ion. If the sample produces an interference for the primary ion then a secondary characteristic ion will be used to quantitate.

7.1.1 Water Samples. Calculate the concentration of the compound using the internal standard calculation procedure described in Section 4.5 and Equation 2:

$$\text{Concentration } \mu\text{g/L} = (A_X)(C_{IS})/(A_{IS})(RF)(V_o) \quad [2]$$

where the parameters used in equation [2] are described in Section 4, and V_o = volume of water purged (mL), taking into consideration any dilutions made.

7.1.2 Soil Samples. Calculate the concentration of the compound using the internal standard calculation procedure described in Section 4.5 and Equation 3:

$$\text{Concentration } \mu\text{g/kg} = (A_X)(C_{IS})/(A_{IS})(RF)(W_S)(D) \quad [3]$$

where the parameters used in equation [3] are described in Section 4, W_S = weight of sample extracted or purged (g), and D = % dry weight of sample/100, or 1 for a wet weight basis.

8.0 BLANKS

8.1 Volatile Blanks. There are two types of blanks associated with purge and trap analysis; low level method blanks and cleaning blanks.

8.1.1 Low Level Method Blanks. For routine analysis, a low level method blank must be analyzed before samples are analyzed. A low level method blank consist of 15 mL (or at the volume that samples are to be analyzed, i.e., 25 mL, 50 mL, etc.) of analyte free water that has a surrogate and internal standard added. Method blanks are acceptable if no target compounds are present above the reporting limit. Samples should not be analyzed until an acceptable method blank is run demonstrating that the instrument is free of interferences

8.1.2 Cleaning Blanks. A cleaning blank is 15 mL of reagent water only. Blanks will be analyzed after any high level sample to ensure that carryover is not occurring. A high level sample is defined as having a concentration 5 times higher than the highest calibration point. Cleaning blanks will be analyzed until the analysis of further samples will not be affected by carryover.

9.0 SAMPLE PREPARATION

9.1 Sample Preparation. Sample analysis and preparation techniques have been adapted from protocols outlined in SW-846 3rd ed. USEPA Purge and Trap method 8260A (USEPA 1986).

9.1.1 Water Samples. Rinse a 25 mL syringe with one volume of sample. Draw the sample into the syringe, invert and remove all air adjusting the final volume to 15.0 mL. Add the appropriate amount of surrogate and internal standard directly to the sample. Deliver the sample to the impinger, connect the Tenex/charcoal sorption tube, start the nitrogen purge gas and start the purge process. After the purge cycle is complete, remove the sorbent trap and transfer to the thermal desorber for analysis.

9.1.2 Soil Samples. Weigh 15 g +/- 0.5 g (or appropriate sample amount) into the impinger. Add surrogate and internal standard to 10.0 mL reagent water, deliver the solution to the impinger, connect the Tenex/charcoal sorption tube, start the nitrogen purge gas and start the purge process. After the purge cycle is complete, remove the sorbent trap and transfer to the thermal desorber for analysis.

9.1.3 Total Solids. All sample results will be reported on a dry weight basis. Place approximately 10 grams of wet soil into a pre-weighed tin and dry in an oven (or use a heat lamp) until repetitive weightings show no further drying is occurring. Subtract the weight of the tin

from the wet weight and from the dry weight. Divide the adjusted dry weight by the adjusted wet weight and multiply by 100 to get the percent total solids.

10 QUALITY CONTROL

10.1 Quality Control. The following procedures will be implemented by the field chemist to insure standardization of the operating procedures:

10.1.1 Review. The field chemist will review each chromatogram before analyzing the next sample. The review will include the calculation of surrogate recoveries, comparison of surrogate and target compound retention times to calibration standards, and the evaluation of carryover potential.

10.1.2 Water Surrogate Recoveries. 4-Bromofluorobenzene and 1,4-Difluorobenzene will be the primary surrogates for the Hanscom project. Surrogate recoveries for water samples must be greater than 50 percent and less than 150 percent. Water samples with surrogate recoveries of less than 50 percent must be reanalyzed to confirm matrix interferences. Water samples with recoveries greater than 150 percent will be noted in the log book. If the surrogate recovery is less than 50 percent for the water sample then the sample will be reanalyzed. If the surrogate fails the second analysis the results will be reported and the recovery noted in the logbook.

10.1.3 Soil Surrogate Recoveries. 4-Bromofluorobenzene and 1,4-Difluorobenzene will be the primary surrogates for the Hanscom project. Surrogate recoveries for soil samples must be greater than 30 percent and less than 200 percent. Soil samples with surrogate recoveries of less than 30 percent must be reanalyzed to confirm matrix interferences. Soil samples with recoveries greater than 200 percent will be noted in the log book. If the surrogate recovery is less than 30 percent for the soil sample then the sample will be reanalyzed. If the surrogate fails the second analysis the results will be reported and the recovery noted in the logbook.

10.1.4 Duplicates and Triplicates. Duplicates (or triplicates) will be analyzed after every twenty samples to calculate precision. When duplicates are analyzed a 30% RPD criteria will be used as the upper control limit. When triplicates are analyzed a 30% RSD will be used as the upper control limit. All results will be calculated for review in the logbook and in a quality control summary if requested.

11 REPORTING

11.0 Reporting. Data from all sample analyses and relevant calibration and blank analyses will be documented in the project GCMS run logbook. A quality control summary may be generated at the completion of the project and will include some or all of the following as required: initial calibrations, continuing calibrations, surrogate recoveries, method blanks, dilution's, reanalyzes, observations of the field chemist, problems and fixes, unknown peaks, raw data, etc..

REFERENCES:

U.S. Environmental Protection Agency, 1986, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 8260A; SW-846"; Office of Solid Waste and Emergency Response, Washington, D.C.

STANDARD OPERATING PROCEDURE**HANSCOM AFB, MASSACHUSETTS**

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FIELD TDGC/MS METHOD FOR ANALYSIS OF PAHs AND PCBs IN SOIL**SCOPE:**

The method is capable of providing either semi-quantitative or quantitative analysis for the presence of polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyl's (PCBs) in organic extracts prepared from non-aqueous solid materials. The method uses a solvent extraction followed by thermal desorption gas chromatography/mass spectrometry (TDGC/MS). Concentrations reported for PCBs will be reported as Total PCB levels. The PAH analyzed by this method are as follows:

Polycyclic Aromatic Hydrocarbon	CAS Number ^a
Naphthalene	91-20-3
Acenaphthylene	208-96-8
Acenaphthene	83-32-9
Fluorene	86-73-7
¹ Phenanthrene	85-01-8
¹ Anthracene	120-12-7
² Fluoranthene	206-44-0
² Pyrene	129-00-0
³ Chrysene	218-01-9
³ Benzo(a)anthracene	56-55-3
Benzo(a)pyrene	50-32-8
⁴ Benzo(b)fluoranthene	205-99-2
⁴ Benzo(k)fluoranthene	207-08-9
⁵ Dibenz(a,h)anthracene	53-70-3
⁵ Indeno(1,2,3-cd)pyrene	193-39-5

^aCAS Number = Chemical Abstracts Service registry number

Note: Compounds with same superscript number coelute.

SUMMARY: The soil sample is micro-extracted using methylene chloride. An aliquot of the extract and a known amount of the internal standard are injected onto a thermal desorption tube. The solvent is allowed to volatilize and the remaining compounds are then thermally desorbed and analyzed by the GC/MS system.

REQUIRES: This procedure requires that the operator be familiar with the set-up and operation of the GC/MS system. This SOP covers procedures for sample and standard preparation, calibrations, quantitation, identification, and quality control. The procedure does not cover data operating systems or reporting. A TDGC/MS (Tufts University model or equivalent) and acquisition software are required for the procedure.

1.0 EQUIPMENT AND RUN CONDITIONS

1.1 Thermal Desorber Run Conditions. The thermal desorber is attached directly to the TDGC/MS injection port. The desorption tube is inserted into the desorber at the start of analysis. The sample is flash heated off from the sorbent tube onto the capillary column for separation and identification.

- Desorber temperature: 280°C.
- Desorb time: 1.5 minutes

1.2 TDGC/MS (HP5972) System Run Conditions. The TDGC/MS system consist of a gas chromatograph, a mass spectrometer , and a data system. The gas chromatograph provides compound separation while the mass spectrometer generates ion abundance information. The following run conditions are recommended for the Hanscom project, however they may be altered by the field chemist based on actual site conditions.

1.3 TDGC Conditions. The analytes are thermally desorbed off of the thermal desorption tube by heating the thermal desorption port isothermally at 280°C. The sample is introduced at the head of the capillary column by volatilization at 280°C, the carrier gas flow rate through the thermal desorption port, and the temperature gradient between the thermal desorption port and the initial column temperature. The column temperature is held at 150°C for 60 seconds followed by linear temperature programmed heating to 320°C at 50°C/minute. Finally, the temperature program is held isothermal, heating period of 330 seconds at 320°C. The total analysis time (including oven cooling time) is approximately 15 minutes.

1.4 MS Conditions. MS data is collected as a total ion current chromatogram (TIC). The mass range is 125 and 500 amu (Tufts TDGC/MS) with the MS scan time of 1-2 sec depending upon the target analytes at a specific site.

- Carrier gas: Helium at 99.999% purity.
- Flow of 1.0 mL/minute.
- Injector temperature: 280°C.
- Initial temperature: 150°C.
- Rate: 50°C/minute.
- Final temperature: 320°C.
- Final time: 5.5 minutes.
- Run Time: 10 minutes.
- Interface temperature: 280°C.
- Mass range: 120 to 500 amu.
- Solvent delay: 1.5 minutes.

1.5 Column. A number of columns are available for semi-volatile analysis, however, for the Hanscom project a DB-5MS, 20 meter, 0.25mm ID microbore column, 0.25µm film thickness (or equivalent) will be used.

2.0 CONVENTIONS.

2.1 Standards. Standard conventions are established to ensure consistent procedures from project to project and from operator to operator. This includes standard preparation, documentation, calculations, and tracking.

2.1.1 Stock Standards. Stock chemical standards will be purchased from Supelco, Inc., Chem Service, Inc., or an equivalent supplier. All standard information will be logged in a bound logbook with the pages sequentially numbered. This information will include, at a minimum:

- Vendor name supplying standards.
- Name and concentration of the standard (this can be done by attaching the label from the standard to the logbook).
- Lot number of the standard.
- Expiration date of the standard, if listed.

2.1.2 Working Standards. Working chemical standards may either be purchased through an approved vendor or prepared from stock standards. Working standards will be made by diluting neat or prepared stock standards. For standards made from neat solutions, the compound density or weight will be used in calculating the appropriate amount of compound and solvent to be combined. All working standards will be labeled with an identification code (see below), compound or mix name, and concentration. The associated logbook will contain the following information at a minimum:

- The Stock solution used to make the working standard, including either the information required for logging the stock solution or a reference to where that information can be found.
- Both a written description and calculation of how the working standard was prepared.

- The solvent(s) and associated lot number(s) used in preparing the standards.
- The final concentration of the working standard.
- The GC/MS code associated with the standard.
- Standard and stock solutions will be kept refrigerated at less than 10°C when not in use.
- The surrogate standard is made separately from the working standards.

2.2 GC/MS Coding System. The GC/MS coding system is used to trace all standards back to the vendor. All standard are required to have the appropriate code assigned when they are prepared and must be labeled accordingly. The GC/MS code will follow the format:

- FGCXXXXXXXXWWYZPPPP Where:
 XXXXXX is the month, day, and year that the standard was made,
 i.e., 041696 = April 16,1996.
- WW is the page in the logbook where the standard can be found, i.e., 01 to 99.
- Y is where the standard fell chronologically on the page, i.e., A through Z.
- Z is the logbook number where the standard can be found, i.e., 1 through 9.
- PPPP is the project identifier, i.e., USAF = United States Air Force, a short character identification of the project from 1 to 4 characters in length.

2.3 Logbook Entries. While the style and specific requirements for logbook entries will vary between operators and projects, certain information is required for all projects. At a minimum this will include:

- A table of contents listing what and where specific information is located.
- A listing of GC/MS run conditions and set points.
- A chemical standards preparation section containing the required standards information.
- A run log section containing at a minimum: sample identification, run number or computer file identification, sample amount (weight or volume), standard code for each standard used, amount of standard(s) used, an example calculation for any calculations performed, dilution factor, and a remarks column with any pertinent information (e.g., unusual sample color or odor, unusable blanks, failed standards, coelutions, reruns, etc.).
- An equipment maintenance section containing both routine and equipment malfunction maintenance (e.g., column replacement, electronic parts replacement, GC/MS repair, replacement, or cleaning, etc.)

3.0 TUNING

3.1 TDGC/MS Tune. The HP-5890 uses a standard autotune macro to tune the mass spectrometer. Three masses of the calibration compound (PFTBA) are selected: 69, 219, and 502. . The procedure is automatically implemented upon manual selection of the automatic tune feature of the instrument. For all 3 masses, starting with 69 amu, the MS system adjusts the mass, peak width and energy in that order until it achieves the desired values. The resolution is

set to unity with a *ca.* 10% valley definition; this is done by measuring the actual peak width and adjusting the peak width parameter accordingly. The GC/MS system will be tuned at the start of each day prior to analyzing samples. The tune is considered valid for each 24 hour period.

4.0 CALIBRATION.

4.1 Multilevel Calibration. In the initial multilevel calibration, a minimum of five levels (concentrations) of standards are analyzed. The standard concentrations are selected to cover the concentration range of contaminants expected (approximately 0.1 to 100 ppm). This includes a low concentration standard at or near the method detection limit (MDL). In addition, the upper level standard has to be within the working range of both the detector and the column.

4.2 Daily Calibration. After mobilizing the TDGC/MS unit to the field, the analyst must prepare calibration standards at a minimum of five concentration levels for each compound. The concentrations should correspond to the expected range of concentrations found in the "real-world" sample or the establishment of the GC/MS dynamic range.

4.3 Internal Standard Calibration Procedure. Prepare a solution that will contain at least one internal standard (Pyrene-d10 has been selected for the Hanscom project) using the procedures described in Sections 2.0 and 2.1. The internal standard concentration should be 50 ppb. Pyrene-d10 is selected as the internal standard because of its retention characteristics relative to PAHs and PCBs.

4.4 Calibration Curve. Inject calibration standards containing the internal standard onto the desorption tube and analyze using TDGC/MS. The internal standard concentration should be 50 ppb. Analyze a calibration sample at a concentration near but above the MDL. Repeat analysis one time for each of the five concentration levels to cover the linear range.

4.5 Response Factors. Repeat analysis one time for each concentration. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using Equation 1:

$$RF = (A_X)(C_{IS}) / (A_{IS})(C_X) \quad [1]$$

where, A_X = area of the standard target analyte ion current signal, C_{IS} = concentration of the internal standard, A_{IS} = area of the internal standard ion current signal, and C_X = concentration of the standard target analyte

If the RF value is constant (i.e., < 30% relative standard deviation, with no more than one third of the samples with > 30% RSD for quantitative GC/MS) over the dynamic range, the RF can be assumed to be invariant and the average from the RF value(s) can be used for quantitation. If the average RF values for any analytes fall outside of the 40% RSD window, the instrument must be checked for mass drift and the point or points skewing the curve must be performed again.

4.6 Continuing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard. If all of the analyte RF's from the continuing

standard have respective %D's within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the continuing calibration criteria are not achieved then a new calibration curve with a minimum of five concentration levels will be generated.

4.7 Closing Calibration. The working calibration curve or RF must be verified each working day with a mid-level calibration standard. If all of the analyte RF's from the continuing standard have respective %D's within the 30% and 40% requirements then analysis of samples may begin. If the response for 1/3 of the compounds varies from the initial calibration curve response factor by more than 40% then the test must be repeated using a fresh calibration standard at mid-level concentration. This is repeated until third failure, if the closing calibration is still not in control, then a new calibration curve must be generated and all samples analyzed during the previous calibration period must either be re-analyzed or flagged to indicate that the system was not within the QC requirements.

5.0 METHOD DETECTION LIMITS

5.1 MDL Determination. The MDLs are determined for each compound in soil prior to the analysis of samples. This is accomplished by analyzing seven replicate low concentration standards near the expected MDL. From these analysis a standard deviation is calculated and multiplied by 3.14 to establish the MDL. This correlates to a student's *t* of 99% for (n-1).

5.1.1 Soil MDL. Soil MDLs will be performed using a minimum of 2 grams of blank soil spiked with the target compounds at 1 to 10 ppm.

6.0 COMPOUND IDENTIFICATION

6.1 Total Ion Current Mode. Compounds will be identified using the total ion current mode (TIC). TIC's for the primary ion and at least two secondary ions for each compound of interest must be identified. The following criteria must be met to make a qualitative identification.

6.1.1 Characteristic Ions. The characteristic ions of each compound of interest must maximize within one scan of each other.

6.1.2 Retention Time. The retention time of the suspected analyte must fall within ± 30 seconds of the retention time of the actual target analyte as determined by the calibration data.

6.1.3 Relative Peak Heights/Areas. The relative peak heights/areas of the three characteristic ions in the TIC's must fall within $\pm 40\%$ of the relative intensities of these ions according to the mass spectra obtained during calibration for targeted analytes or from a reference mass spectrum for non-targeted analytes.

6.1.4 Structural Isomers. Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mixture is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

<u>Polycyclic Aromatic Hydrocarbons</u>	<u>Ions</u>
Naphthalene	128, 129, 127
Acenaphthylene	152, 151, 153
Acenaphthene	154, 153, 152, 151
Fluorene	166, 165, 167, 163
¹ Phenanthrene	178, 176, 179, 177
¹ Anthracene	178, 176, 179, 177
² Fluoranthene	202, 203, 200, 201
² Pyrene	202, 203, 200, 201
³ Chrysene	228, 226, 229, 227
³ Benzo(a)anthracene	228, 226, 229, 227
Benzo(a)pyrene	252, 250, 253, 251
⁴ Benzo(b)fluoranthene	252, 250, 253, 251
⁴ Benzo(k)fluoranthene	252, 250, 253, 251
⁵ Dibenz(a,h)anthracene	278, 279
⁵ Indeno(1,2,3-cd)pyrene	276, 277, 275, 274
Benzo(g,h,i)perylene	276, 277, 274
[†] Pyrene-d10	212, 211, 210
[‡] Octachloronaphthalene	402, 404, 406
[†] Internal Standard,	
[‡] Surrogate	

Note: Compounds with the same superscript number coelute.

7.0 CALCULATIONS

7.1 Quantitation. When a compound has been identified the quantitation of that compound should be based on the integrated abundance from the TIC of the primary ion. If the sample produces an interference for the primary ion then use a secondary characteristic ion to quantitate.

7.1.1 Soil Samples. The quantitation of an identified compound is based on the integrated area of the analyte's primary ion (100% relative abundance) extracted from the total ion current chromatogram.

Calculate the concentration of the analytes in soil/sediment sample by using the following equation:

$$\text{Concentration (ng/g)} = K(A_x)(C_{is})/(A_{is})(RF)(W_s)(D)$$

where K = dilution factor, W_s = weight of the sample (g), and D = (100-% moisture in the sample)/100 if appropriate.

8.0 BLANKS

8.1 PAHs and PCBs Blanks. Prior to analysis of any calibration or actual site samples, a method blank analysis must be performed. A method blank is performed where the extraction solvent, apparatus, and thermal desorption tube are checked for background contamination. A blank extraction procedure is performed and an aliquot of the resulting extract solvent is placed on the thermal desorption tube and allowed to volatilize. The residue is then thermally desorbed off of the tube by heating the thermal desorption port at 280°C, and analysis is performed as discussed below. The solvent residue is thus analyzed using the method GC temperature program with MS detection for the presence/absence of background contaminants.

8.2 Corrective Action. If background contamination is found, a stepwise process of elimination will be performed from the thermal desorption tube back to the initial extraction solvent to isolate and eliminate the source of contamination. Prior to any analyses (calibration or site samples) the system must be found to be free of any outside sources of contamination. If the internal standard (e.g., pyrene-d₁₀) is suspected to be contaminated, this procedure must be repeated with the addition of the internal standard for thermal desorption.

8.3 Interferences. Contamination by carry-over can occur whenever low-level samples are analyzed after high-level samples. This can be avoided by baking out the thermal desorption port and the capillary column at 280° and 240° respectively, for a few minutes after high-level samples are analyzed. The thermal desorption tube should be cleaned with analyte-free solvent after each analysis. The instrument will be monitored after every sample analysis, by targeting specific hydrocarbon ions, to ensure background currents reach normal levels. The use of high purity (HPLC grade) reagents will help to minimize interference problems.

9.0 SAMPLE PREPARATION

9.1 Sample Preparation The soil sample is thoroughly homogenized in the sample container and a sub-sample (2.0g±0.2g) is weighed and placed in a 8-mL sample vial with a Teflon lined screw cap. To this vial is added the surrogate compound as required. The surrogate will be added at a concentration in the mid-range of the calibration (50-100 ppm final concentration). Approximately 2g of anhydrous sodium sulfate are added and the sample is thoroughly mixed with a Teflon coated spatula to form a free-flowing mixture. 2.0-mL methylene chloride is then added to the sample. This solution is then hand shaken for 2 minutes, allowed to settle briefly (< 30 seconds or use a centrifuge). The extract is then transferred into a 2-mL glass sample vial using a disposable glass Pasteur pipette. The extract may be used directly for TDGC/MS analysis. Batch lots of these apparatus must be analyzed through the method blank and found to be free of interfering contaminants.

9.2 Sample Introduction. An aliquot of the extract (50 µL or method detection limit study volume) is injected onto a thermal desorption tube along with 2.5 µL of 20 ng/µL internal standard solution previously prepared. Internal standard concentration will vary dependent upon individual instrument sensitivity and levels of monitored analytes expected to be found at the sampling

location, however, 50 ng is generally a sufficient quantity. After the solvent has vaporized, the residues of the sample extract and the internal standard are then thermally desorbed into the capillary column of the TDGC at a temperature of 280°C.

10 QUALITY CONTROL

10.1 Quality Control. The following procedures will be implemented by the field chemist to insure standardization of the operating procedures:

10.1.1 Review. The field chemist will review each chromatogram before analyzing the next sample. The review will include the comparison of target compound retention times to calibration standards, and the evaluation of carryover potential.

10.1.2 Soil Surrogate Recoveries. Octochloronaphthalene will be the surrogate for the Hanscom project. Surrogate recoveries for soil samples must be greater than 30 percent and less than 200 percent. Soil samples with surrogate recoveries of less than 30 percent must be reanalyzed to confirm matrix interferences. Soil samples with recoveries greater than 200 percent will be noted in the log book. If the surrogate recovery is less than 30 percent for the soil sample then the sample will be reanalyzed. If the surrogate fails the second analysis the results will be reported and the recovery noted in the logbook.

10.1.3 Duplicates. Duplicate analysis will be performed at a rate of every twenty samples following the first five samples analyzed. When possible, the duplicate will be chosen which was found to contain target analyte. In such cases, it is preferable (time permitting) to perform triplicate analysis and establish measurement precision criteria. Duplicate analysis measurements must result in < 60% RPD for samples with concentrations higher than 5x PQL. The measurements must result in < 100% RPD for the samples with concentrations in the range from 1x to 5x PQL. Triplicate analysis measurements must result in < 60% RSD for samples with concentrations higher than 5x PQL. The measurements must result in < 100% RSD for the samples with concentrations in the range from 1x to 5x PQL.. The RPD is measured as:

$$\text{RPD} = (\delta/y) \times 100$$

where,

δ = the absolute value of the difference between the duplicate measurements.

y = the mean of the two measurements.

If duplicate analysis criteria fails, the system will be checked for mass alignment, sensitivity, and re-tuned prior to re-homogenization and re-analysis of the sample.

10.1.4 Internal Standard. The internal standard(s) will be monitored for each analysis. The area count for the internal standard must fall within -50% to +150% of the average internal standard area count established for the initial calibration curve. If the internal standard area count falls outside this range, the system will be checked for mass alignment, sensitivity, and re-tuned if necessary prior to re-analysis. If the internal standard area count falls outside of the criteria upon re-analysis, a new three point calibration curve will be established with new internal standard area count limits.

11 REPORTING

11.0 Reporting. Data from all sample analyses and relevant calibration and blank analyses will be documented in the project GCMS run logbook. A quality control summary may be generated at the completion of the project and will include some or all of the following as required: initial calibrations, continuing calibrations, surrogate recoveries, method blanks, dilutions, reanalyzes, observations of the field chemist, problems and fixes, unknown peaks, raw data, etc..

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TUFTS UNIVERSITY STANDARD OPERATING PROCEDURE

TUFTS UNIVERSITY SITE OPERATION PLAN FOR FIELD TRANSPORTABLE ICP-AES METHOD FOR ANALYSIS OF PRIORITY INORGANIC POLLUTANTS IN SOIL AND WATER MATRICES

1.0 SCOPE AND APPLICATION

1.1 A quantitative method based upon the contract laboratory program using a specially modified ICP-AES built to withstand transport to a field environment. The instrument in all other respects performs exactly like its laboratory counterpart.

2.0 SUMMARY OF METHOD

2.1 Soil, sediment and water samples are digested using microwave acid digestion followed by filtration and dilution. The filtrate is then nebulized into the ICP-AES via an autosampler for analysis.

3.0 INTERFERENCES

3.1 Background contamination of trace metals is negligible when utilizing microwave acid digestion vessels due to the chemically inert nature of the Teflon™ material. Post digestion cleanup of vessels requires only a ASTM Type I rinse until all visual particles have been removed.

3.2 Contaminants can be minimized during filtration by using a Teflon™ column assembly utilizing positive air pressure to push the filtrate through a disposable 30-60 micron Teflon™ membrane supported upon a porous polypropylene frit. Filtrates are collected in HDPE containers.

3.3 Contaminants during ICP-AES can be introduced by memory effect in which metals adhere to the walls of the pumping system, nebulization chamber and nebulization device. This can be detected by instrument scans of blanks and minimized with properly set rinse out times between sample runs.

3.4 Interelement effects due to matrix constituents will be corrected by interelement and background check standards obtained from an independent source if necessary. The PS-1000M (Leeman Laboratories) modified at Tufts University has a high resolution monochromator which enables unambiguous detection of most analytes at specific wavelengths. Background correction points are typically set on-site after test samples have been scanned against the standard correction library.

4.0 APPARATUS AND MATERIALS

4.1 CEM MDS-2000 (CEM Corp. Matthews, NC)

4.1.1 This microwave system allows solid and liquid samples to be digested in PFA lined Teflon™ bombs with working pressures up to 200psi and a rupture disc protection at 220 psi. There is one control vessel per twelve which has both pressure and temperature sensor capabilities. The oven operates at 110-120V 20A 60Hz with 630 Watts of microwave power at a frequency of 2450Mhz. The instrument has a built-in LCD panel for programming both temperature and pressure controlled methods. After a digestion the PFA liners are cleaned with ASTM Type I water.

4.1.2 Sample Digestion Tools and Equipment

- Microwave unit
- (24) advanced composite vessels with (2) racks
- (6) extra PFA liners
- PFA rupture discs
- Analytical balance $250g \pm 0.1mg$
- (2) sensor head vessels
- 3-prong outlet
- Exhaust port
- (6) PFA coated thermowell sleeves
- (2) Fiber-optic temperature probes
- 1L Teflon™ Bottle
- An adjustable volume bottle-top dispenser (Teflon™ lined)
- Weighing paper
- (4) Spatulas
- (2) Concentrated acid bottle-top dispensers (For waters analysis only)

4.1.3 Filtration Apparatus

- Zitex porous Teflon™ membrane 30-60 micron (sheets)
- Air cylinder / regulator
- PFA columns
- Gas manifold
- Hole boring tool
- 60ml HDPE sample containers (1 per sample)
- 1000ml Teflon™ Bottle with Type I water
- An adjustable volume bottle-top dispenser (Teflon™ lined)

4.2 The PS1000 M Inductively Coupled Plasma/Atomic Emission Spectroscopy Echelle Sequential Spectrometer (Leeman Labs, Lowell MA) was modified at Tufts University.

4.2.1 This instrument uses the hildebrand nebulizer to introduce samples into the plasma. It features an autosampler which will be used in conjunction with macros within the instrument software to allow for continuous utilization.

4.2.2 Lab Bench Requirements

Spectrometer	Power Supply
Width: 60"	Width: 20"
Depth: 35"	Depth: 35"
Height: 40"	Height: 14"
Weight: 375lb. (170kg)	Weight: 200lb (91kg)

The instrument will not be located near ventilation ducts from heat or air conditioning in order to ensure temperature stability.

4.2.3 Environmental Conditions

Temperature range: 15-30°C (60-86°F)

Temperature variation: Should not exceed 2°C (3.6°F) per hour and 10°C (18°F) during the work day. Instrument modifications have be done to increase temperature drift tolerances to ± 20 °F.

Relative humidity: 20-80% non-condensing

4.2.4 Electrical Power

One 190-230 V, 30 Amp, 50/60Hz grounded single phase line connected to circuit breaker with Hubbell Twist-Lock (Part # 2620A) receptacle.

Two 115 V, 15 Amp, 60 Hz or 220 V, 10 Amp, 50Hz double-plug outlets for Spectrometer.

Three regular three prong outlets.

4.2.5 ICP Exhaust

10-12" stainless steel or corrosion resistant exhaust 42-45" above lab bench surface.

4.2.6 Argon Gas Supply

One Liquid Ar Dewar in use and one more as a backup. (purified grade)

4.2.7 Tools and Equipment

- (2) Hildebrand Grid Nebulizers in reserve
- (2) torches in reserve at all times
- Autosampler
- Certified Standards and Check Solutions are Plasma-Pure (Leeman Labs, Lowell, MA)
- Independent standards (multi-element)
- Polymeric fine tip transfer pipets

- Teflon™ bottles for standards
- Amber HDPE bottles for Ag containing solutions
- Carboy filled with Type I water
- HDPE test tubes with caps
- 16mm test tube racks
- Teflon™ wash bottles
- (2) packages sample and waste tubing
- Consumables kit for routine maintenance
- Tool kit designed for instrument
- 1L volumetric flask
- 1L Teflon™ bottle for reagent blank
- 1L HDPE bottle for waste
- Digital thermometer

5.0 REAGENTS

5.1 Hydrochloric and nitric acid for digestion and standard preparation are of trace metals grade quality. Water is ASTM Type I 18 MegaOhm.

5.2 Preparation Standards will be purchased from the same commercial source with the exception of the Laboratory Control Standard.

5.2.1 Certified Standard single-element and multi-element solutions of all metals traceable to NIST.

5.2.2 Compatible multi-element standard solutions will be prepared as necessary from their stock solutions by gravimetric dilution on an analytical balance to the nearest 0.1mg. The sample acid concentration will be used as the concentration of the diluent.

5.2.3 An initial calibration verification standard will be run to check if the calibration is acceptable.

5.2.4 An independent multi-element standard from a different source than used in the calibration standards will be run for QA/QC purposes and diluted to the appropriate level when necessary.

5.2.5 A CLP interelement and background correction standard will be used.

5.2.6 Spike standards will be used if necessary.

5.2.7 A Laboratory control soil sample will be run, PriorityPoluntnT™ (ERA, Averada, CO).

5.2.8 A laboratory control water sample will be run, WasteWatR™ (ERA, Averada, CO).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Most samples will be analyzed upon receipt and when appropriate will be preserved by a suitable technique.

7.0 ICP-AES OPERATION AND MAINTENANCE

7.1 The instrument will be allowed at least 1.5 hours for warm-up of torch and sample introduction apparatus. During the initial warm-up period the source mirror will be peaked on the Mn 257.610 nm line. This operation determines nebulizer performance and assures optimal alignment of the plasma and monochromator. Once a good MnY profile is obtained the operator will peak all analytical wavelengths. The following parameters made need to be adjusted by the operator:

Power	Pump Rate
Nebulization pressure	Integration time
Coolant Flow	Sample uptake time
Auxiliary Flow	Rinse out time
Elemental wavelength	Background correction point(s)

7.1.1 Daily Maintenance may require

- a) Changing pump tubings...typically every 3-4 day
- b) Emptying of waste container
- c) Sample introduction disassembly...
 - 1) to replace O-rings...typically twice per month
 - 2) check nebulizer spray visually and audibly
 - 3) cleaning of torch....only when needed
 - 4) cleaning of spray chamber...only when needed

7.2 Maintain an Instrument Maintenance Log Book recording any changes such as:

- 7.2.1 Changing sample introduction components
- 7.2.2 Changing Argon supplies
- 7.2.3 Any preventive maintenance performed
- 7.2.4 Any significant changes to the instruments environment
 - a) HVAC difficulties
 - b) new equipment coming on line
 - c) power interruptions
 - d) temperature variance

7.3 Weekly Maintenance

- 7.3.1 Change pump tubing and O-rings

- 7.3.2 Clean and lubricate the autosampler
- 7.3.3 Check RF contact strip for corrosion and looseness
- 7.3.4 Check water level in water recirculator
- 7.3.5 Check air filters on power supply and spectrometer, clean as necessary

7.4 Monthly

- 7.4.1 Change O-rings
- 7.4.2 Clean filters on power supply and spectrometer
- 7.4.3 Drain water recirculator and refill with **FRESH DI WATER**

7.5 Semiannual

- 7.5.1 Clean water recirculator
 - a) Drain completely
 - b) Wipe reservoir clean
 - c) Refill with **FRESH DI WATER**
- 7.5.2 Acid rinse the cooling water lines
 - a) Disconnect lines at the recirculator
 - 1) Drain water from instrument
 - b) Fill water lines with 5% HCL 5% HNO₃
 - 1) Acid solution should **never** enter the recirculator
 - 2) Wait **5 minutes**
 - c) Flush lines with at least 20 liters of DI water
 - d) Reconnect the recirculator
 - 1) Fill with **FRESH DI WATER**
 - 2) Use of algaecides is not recommended
- 7.5.3 Replace inline Argon filter

8.0 PROCEDURE

8.1 Soil Sample Preparation

8.1.1 Samples will be logged into a Sample Preparation Logbook.

8.1.2 1.00 ±0.001 grams of soil will be weighed by analytical balance on weigh paper and transferred to a PFA liner.

8.1.3 Two microwave acid digestion matrices are available depending on the elements of interest. The 50% nitric digestion is based upon EPA method 3051 and recovers all target analyte metals except Ag and Sb. Complete recovery of these elements can be accomplished using 50% 3:2 HNO₃:HCl. The digestion parameters are shown in the following table:

Matrix I.D.*	1	2
Acid	50% HNO ₃	50% 3:2 HNO ₃ :HCl
volume (ml)	20	20

sample weight (g)	1.00		1.00	
# of vessels	12		12	
stages	(1)	(2)	(1)	(2)
power %	100	100	100	100
temperature (°C)	130	175	120	165
pressure (psi)	120	190	120	200
time allotted (min)	20.0	20.0	10.0	20.0
ramp time (min)	----	----	----	----
hold (min)	5.0	5.0	5.0	5.0

8.1.4 Once samples have cooled the 20ml digestate is transferred by Teflon™ funnel to a filtration column and 10-20 psi of air pressure applied once the gas assembly has been sealed. The filtrate is dilute to 25% total acid volume with 20ml Type I water from a dispenser and the tared weight is recorded in the sample preparation logbook.

8.1.5 Filtration columns are disassembled and thoroughly rinsed with Type I water and then a new membrane is loaded.

8.2 Water Sample Preparation

8.2.1 Samples will be logged into Sample Preparation Logbook.

8.2.2 15.00 ±0.001 grams of water will be directly weighed into a PFA liner.

8.2.3 Two microwave digestion matrices are available as indicated in *Section 8.1.3*. However, the only change shall be that the acid will be volumetrically delivered via a bottle-top dispenser in concentrated form. Matrix 1 will be 5ml concentrated nitric acid and Matrix 2 shall be 3ml concentrated nitric and 2ml hydrochloric acid. The same time parameters will be employed, but the *time allotted* can be increased if the correct temperature profile is not obtained.

8.2.4 When the samples have cooled the analyst will determine if any suspended particulates are present. If not, then sample will be transferred directly to sample bottles with the tared weight recorded into the sample preparation logbook. If so, then they shall be filtered through Teflon™ columns. The air pressure will be reduced to 2-5 psi.

8.3 When the Operator determines that the instrument has adequately warmed up and is stable the following actions are taken:

8.3.1 Peak in all metals and set integration time. Integration time will be dependent upon matrix interferences and sensitivity requirements.

8.3.2 Scan a peaking standard as well as a method blank to select best background correction point(s). Follow this by scanning the test sample matrix, adjust accordingly.

8.3.3 Run calibration standards manually.

8.3.4 The calibration curve for each metal is graphically displayed and the operator determines if the calibration meets QC/QA criteria. The operator must accept each calibration curve before proceeding to sample analysis. If an element fails QC/QA requirements then the element is scanned and reaped. After running the standards for this metal if it still does not meet QC/QA requirements then instrument parameters need to be adjusted by the operator. As a last resort new standards should be prepared.

8.3.5 If the calibration curves pass QC/QA criteria then samples can be analyzed by transferring samples to HDPE test tubes and programming a autosampler rack file and engaging the autosampler macro. Additionally all solutions run are entered into a sample sequence logbook. All actions taken by the instrument are physically recorded on a dot matrix printer and assigned a unique sequence number.

9.0 QUALITY CONTROL/QUALITY ASSURANCE

9.1 QC/QA will follow, for the most part, CLP SOW ILM01.0 which is summarized in Appendix 12.1

10.0 INSTRUMENT DETECTION LIMITS

10.1 Note that method detection limits will be sample dependent and vary according to changes in the sample matrix. The Instrument Detection Limits and Limit of Quantitation values in the table below are typical for the PS-1000M instrument.

Analyte	PS 1000 IDL (ug/kg) ¹	Matrix 1 LOQ (mg/kg) ²	Matrix 2 LOQ (mg/kg) ²
Aluminum	37	5	5
Antimony	46	6	6
Arsenic	51	7	7
Barium	1.1	0.2	0.2
Beryllium	0.20	0.02	0.02
Cadmium	2	0.22	0.22
Calcium	8.0	1.1	1.1
Chromium	8.0	1.0	1.0
Cobalt	10	1.3	1.3
Copper	4.0	0.55	0.55
Iron	3.0	0.4	0.4
Lead	40	5.0	5.0
Magnesium	57	7.5	7.5
Manganese	1.0	0.07	0.07
Nickel	10	1.25	1.25
Potassium	550	73	73
Selenium	123	16	16
Silver	5.1	0.7	0.7
Sodium	45	6	6
Thallium	78	10.5	10.5
Vanadium	3.0	0.4	0.4
Zinc	7.4	1	1

1 IDL determined using EPA 40 CFR Ch. I Pt. 136, App B.

2 Limit of Quantitation values based upon 1.00g soil sample and calculated by multiplying (IDL) x (sample dilution factor) x (3).

11.0 DATA REPORTING

11.1 Field data will be reported in a format which maximizes transfer to site mapping technologies. The final report will include full QA/QC data in publication format.

12.0 APPENDIX

12.1 QC/QA Requirements for ICP-AES:

Requirement	Superfund Hazardous Waste Analysis CLP SOW ILM01.0
Initial Calibration Frequency	1 standard and a blank Daily
Calibration Verification Frequency Criteria	Mid-range standard Beginning, end, and every 10 samples or every 2 hrs 80-120% recovery
Other Standards Frequency Criteria	Standard at 3x CRDL or IDL Beginning and end of each run or 2 every 8 hrs EPA QC limits
Interference Check Sample Frequency Criteria	EPA Solution A and AB Beginning of each day 80-120% recovery
Calibration Blanks Frequency Criteria	diluent matrix Beginning, end, and 10% of samples or every 2 hrs All analytes \leq CRDL
Preparation Blank Frequency Criteria	run through sample treatment process 1 per SDG* or digestion batch All analytes \leq CRDL
QC Check Sample / LCS Frequency Criteria	SRM or site sample 1 per SDG or digestion batch for each matrix 80-120% recovery
Triplicate Samples Frequency Criteria	10% or 1 per SDG per matrix per level (predigestion) $< 20\%$ RPD for values $\geq 5x$ CRDL

* Sample Delivery Group

Corrective Actions

Initial Calibration Verification (ICV): Terminate analysis, correct problem, recalibrate, reverify standards concentrations. Examine the RSD's of the reps. If they are excessively high it might indicate a sample introduction problem. Recalibrate. If RSD's of reps still high adjust rinse and uptake time.

Continuing Calibration Verification (CCV): Same as ICV, all samples to last compliant CCV must be reanalyzed upon correcting problem.

Other Standards: Verify linearity near CRDL or IDL whichever is greater. Acceptance criteria have not been determined by EPA yet. Just report value (All analyte wavelengths except for Al, Ba, Ca, Fe, Mg, Na, K).

Interference Check Sample (ICS): Terminate analysis , correct problem, recalibrate. Two solutions A (interferents) and AB (analytes and interferents). Run A followed by AB.

Calibration Blank: Terminate analysis, correct problem, recalibrate, and reanalyze last 10 samples after last compliant CB.

Preparation Blank: Lowest concentration of analyte must be 10x the concentration of the PB. If sample falls between CRDL and 10x then those samples must be redigested and reanalyzed for the analytes.

QC/ LCS (Laboratory Control Sample): Terminate analysis, correct problem. Samples associated with that LCS redigested and reanalyzed.

Matrix Spike Samples: Indicate by flagging those samples in the data report. Except where sample concentration exceeds spike concentration by four.

Duplicate Samples: If the data falls outside the control limits it must be flagged when reported.

TUFTS UNIVERSITY STANDARD OPERATING PROCEDURE

TUFTS UNIVERSITY SITE OPERATION PLAN FOR FIELD TRANSPORTABLE EDXRF METHOD FOR ANALYSIS OF PRIORITY INORGANIC POLLUTANTS IN SOIL

1.0 SCOPE AND APPLICATION

1.1 A quantitative and semi-quantitative method for analyzing most metals on the priority pollutant target analyte list using a bench top instrument platform.

2.0 SUMMARY OF METHOD

2.1 Soil samples are directly transferred into a 32mm XRF cell and covered with prolene film. The sample is irradiated with x-rays resulting in fluorescent emission which is detected by a semi-conducting crystal. Energy resolution is used to separate the signal for each element and produce an intensity response. Software processes the count rates for each element via fundamentals parameters.

3.0 INTERFERENCES

3.1 When element energy peaks are not resolved the software uses predetermined ratios to calculate the relative contribution due to each element. Interferences are minimized by subjecting a representative soil sample to a multistage excitation procedure which optimizes the resolution between each elemental peak and provides the maximum intensity response.

4.0 APPARATUS AND MATERIALS

4.1 QuanX XRF (TN Spectrace, Sunnyvale, CA)

4.1.1 The Spectrace QuanX is a compact energy dispersive x-ray fluorescence (EDXRF) system. The analyzer uses an x-ray tube excitation source and a solid state detector to provide spectroscopic analysis of elements ranging from sodium to uranium in atomic number. Instrument control and data analysis are performed by a personal computer that is connected to the system. Fundamental parameters (Section 8.2) are determined and used by the computer to calculate element concentrations. No further calculations (e.g. sample moisture or sample size) are necessary. The system requires regular A/C line voltage and current. The use of a

vacuum pump (Alcatel Pascal Model 2010, Kurt J. Lesker Co., PA) is necessary for elements less than eighteen in atomic number.

4.1.2 Sample Preparation

- 32mm double open-ended (Chemplex, Tuckahoe, NY)
- Microporous Film (Chemplex)
- Prolene x-ray film 4.0 microns (Chemplex)
- (4) Spatulas

5.0 REAGENTS

5.1 National Institute for Standards and Testing (NIST) soil SRMs 2704, 2709, 2710.

5.2 Water is ASTM Type I 18 MegaOhm.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples can be directly analyzed or sealed in appropriate jars and stored in a refrigerator for an extended period of time.

7.0 EDXRF OPERATION AND MAINTENANCE

7.1 The instrument will be left on at all times. The solid state detector is thermoelectrically cooled and must remain on to achieve the best results.

7.1.1 Daily Maintenance entails running a test spectrum of a copper disc referred to as an energy calibration to assign the proper position of the K_{α} and K_{β} for copper which are 8.047 and 8.904 keV respectively.

PROCEDURE

8.1 Soil Sample Preparation

8.1.1 Samples will be logged into a Sample Preparation Logbook.

8.1.2 One side of the XRF cell is covered with the microporous film, flipped over and soil is placed into the cell to a height of approximately $\frac{3}{4}$ of the cup volume. The prolene film is then stretched over the opening with a collar and the cell is shaken and place prolene film side down on a clean surface.

8.2 Instrument

8.2.1 NIST SRMs 2704, 2709, 2710 are loaded into the instrument and analyzed for a total of 200 lifetime seconds for each step in the analysis. When the instrument is operated for 200 lifetime seconds the data is essentially quantitative whereas 120 seconds can be employed for a semi-quantitative screening of soil samples. The XRF analysis parameters with their corresponding elements are provided below.

	Step 1	Step 2	Step 3	Step 4
Elements	Al,Si,S	K,Ca,Ti,V,Cr,Mn	Fe,Ni,Cu,Zn,Sr,Zr,Pb	Cd,Ba,Ag
Tube Voltage (kV)	6	12	30	50
Tube Current (mA)	0.24	0.34	0.26	0.40
Filter	none	Al	Pd thick	Cu thick
Range (kV)	1-10	1-10	1-20	1-40
Atmosphere	vacuum	vacuum	air	air

8.2.2 Fundamental parameters is a complicated mathematical algorithm which characterizes the emission response of individual elements from a homogenous, infinitely thick sample surface. The software calculates pure element count rates (signal response) for each element in the certified reference standard. Soil sample concentrations are derived by comparing the pure element count rates of the sample versus the standard for each element.

10.0 INSTRUMENT DETECTION LIMITS

10.1 Since EDXRF is a surface technique the concentration value reflects the total amount of an element in the sample. The SRM certified values are produced by total dissolution of the soil matrix.

Analyte	Lowest Limit of Detection** (mg/kg)
Aluminum	500
Barium	105
Cadmium	2
Calcium	100
Chromium	21
Cobalt	15
Copper	10
Iron	15
Lead	8
Manganese	22
Nickel	15
Potassium	135
Silicon*	200
Silver	5
Strontium*	8
Sulfur*	90
Titanium*	24
Vanadium	20
Zinc	6

*not listed in target inorganic list **200 seconds livetime

11.0 DATA REPORTING

11.1 Concentration data is a direct readout from the instrument, with no further calculations needed. Data will be reported for those elements which can be analyzed by XRF and appear in the ICP-OES target list of analytes. The methodology will be optimized to provide maximum detection of both Cd and Pb. Those elements which are insensitive to excitation or whose energy lines are outside the energy range of the detector include Sb, As, Be, Mg, Na, and Tl.

12.0 APPENDIX

12.1 There are no established QA/QC procedures or criteria for this method. A soil standard obtained from Environmental Research Associates (Arvada, CO) will be run every 20 samples to track instrument performance.

Appendix IV

This section illustrates the volume estimate calculation for Sites 1, 2, and 3.

Volume Estimate Calculations

Illustrated below are two hypothetical volume calculations used as examples to explain the volume estimate calculations made for Sites 1, 2, and 3. Figure 1 shows the same seven borings, with one (Case A) and two (Case B) contaminated borings, respectively. The same method is used to estimate soil contaminant volumes.

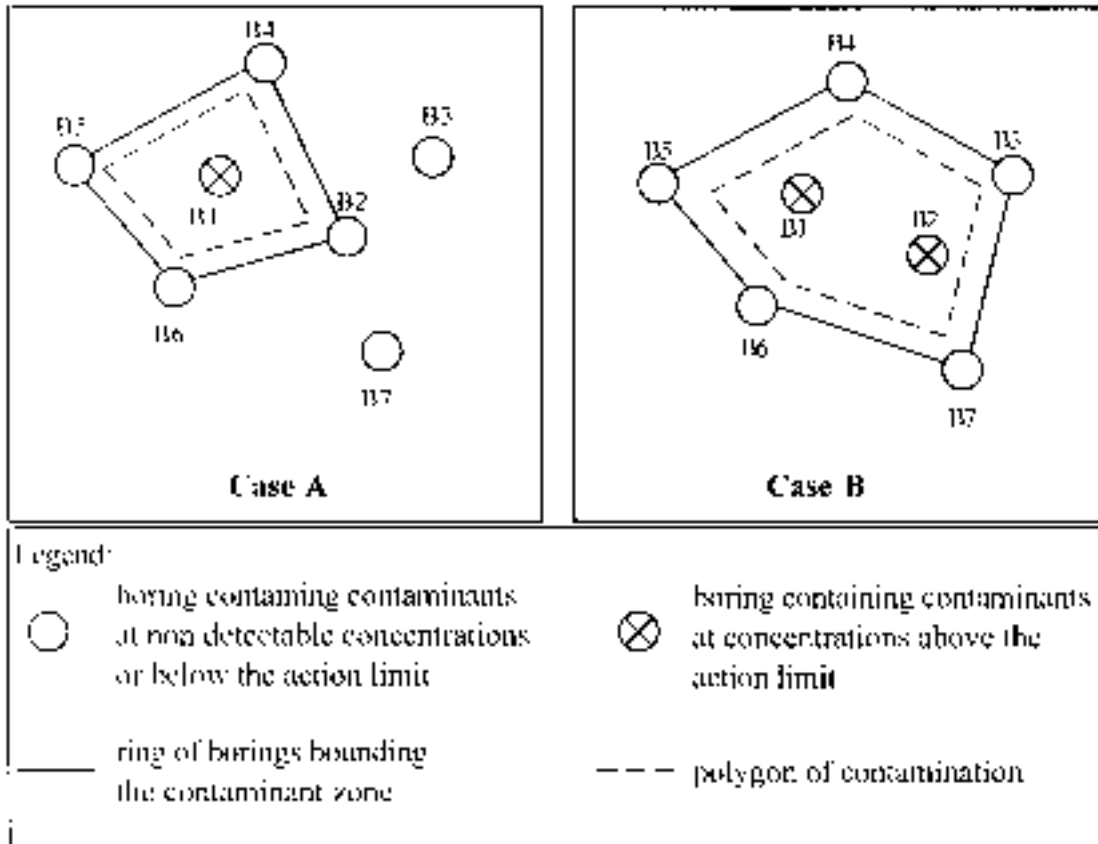


Figure 1. The Two Cases Considered for Volume Estimation

For example, in Case A the following data is obtained for boring B1 (the contaminated Boring):

Screening Depth	Value	Quantitative Depth	Value
0-5 ft	ND	2-4 ft	ND
6-12 ft	Hit	6-8 ft	3-10DAF
12-15ft	ND	10-12 ft	10-10DAF
		13-15 ft	ND

Note that screening data was produced every 1-ft during the HAFB investigation. The data indicates contamination between 6 and 12-ft, with quantitative data confirming this at the intervals shown in the table. This describes the contamination profile in the vertical direction for boring B1.

The horizontal contamination profile is calculated based on a linear interpolation between B1 and its nearest neighbor borings (2, 4, 5, and 6) where analyses indicate concentrations at or below the action level (designated as ND).

For example, the horizontal profile is shown in Figure 2 for the B1-B2 pair. Boring B2 is on the right and boring B1 on the left, with the distance between them 40-ft. The 6-ft to 8-ft interval is estimated separately from the 10-ft to 12-ft interval, with contaminant distances between them linearly estimated as well. Consistent with this calculation is the depth estimate from 5-ft to 6-ft and 12-ft to 13-ft. The process is repeated for each contaminant/ND boring pair (B1/B2, B1/B4, B1/B5, B1/B6). A 3-dimensional polygon depicting site contamination is obtained, which results in the total volume estimate for the site.

A more complicated situation occurs when an adjacent boring is also contaminated, see Case B. The first step in calculating the total volume estimate is to determine which boring pairs bound the site. For example, B1 is bound by B4, B5, and B6, while B2 is bound by B4, B3, B7, and B6.

Based on the concentration data produced at each boring, the calculation process introduced in Case A can now be used to calculate the total site contamination volume for Case B. Note that this process is used irrespective of whether the same contamination profile exists at each boring with respect to depth. For example B1 may be contaminated between 6-ft and 12-ft while B2 may be contaminated between 6-ft and 15-ft.

At Hanscom Air Force Base, Site 3 was calculated as described in Case A, with four different contaminated borings used to estimate the total site volume (66,000-ft³). Site 1 contained two adjacent borings whose contaminant profile was consistent with depth. Case B was used to calculate the total volume estimate (28,000-ft³). Site 2 contained seven contaminated borings that were interconnected at various depths, a more complicated example of Case B. Total estimated soil contamination was calculated to be 243,000-ft³. Table 1 lists the contaminated and ND borings for each site, see Figures 7 to 9 in the body of the report for locations. Also tabulated are the calculated horizontal areas of contamination as a function of depth for each site.

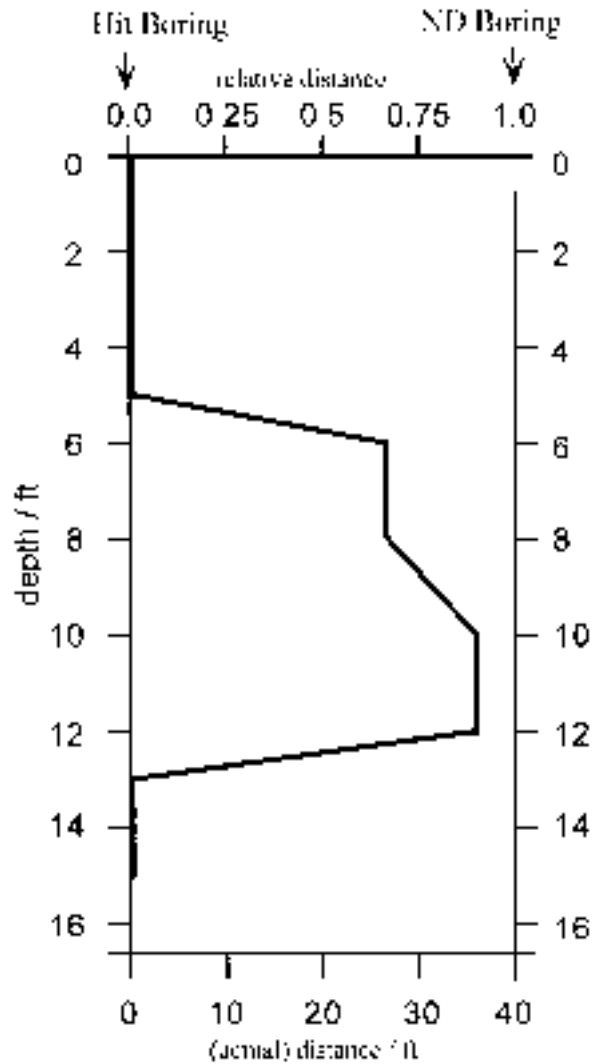


Figure 2. Calculated Vertical Profile of the Extent of Contamination

Appendix V

The following pages include all data generated as part of the HAFB field investigation.

The following data was reported without data flags to reduce paper volume. The data was flagged to identify compound measured in blank above MDL, B; compound estimate above highest point in calibration curve, E; and compound measured between MDL and RL, J. A full data package can be obtained upon request by contacting Albert Robbat (Tufts University) or Nora Conlon (EPA Region I).

Although sample preparation and analyses were performed in separate sections of the mobile laboratory, site samples became contaminated with methylene chloride during the semivolatile sample preparation procedure. Methylene chloride, analyzed as part of the original HAFB target compound list, was taken out of the data set.

Organics Analysis by Field Gas Chromatography/Mass Spectrometry

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B01-A (02-04)	S1 B01-A (02-04) FD#1	S1 B01-A (02-04) FD#2	S1 B01-A (02-04) FD#3	S1 B01-A (06-08)	S1 B01-A (09-11) R1	S1 B01-A (09-11) R2
Volatile Organics				N/A			
1,1,1-Trichloroethane	<8.	<9.	<9.		<9.	<9.	41
1,1-Dichloroethane	<6.	<6.	<6.		<7.	<7.	<7.
1,1-Dichloroethene	<3.	<3.	<3.		<3.	<3.	20
1,2-Dichloroethane	<14.	<14.	<14.		<14.	<15.	<15.
Benzene	<3.	4	5		4	<3.	<3.
Carbon Tetrachloride	<4.	<4.	<4.		<4.	<4.	<4.
Chlorobenzene	<8.	<9.	<9.		<9.	<9.	<9.
Chloroform	<8.	<9.	<9.		<9.	<9.	<9.
cis-1,2-Dichloroethene	<5.	<5.	<5.		<6.	<6.	<6.
Ethylbenzene	<6.	<6.	<6.		<7.	61	39
m/p-Xylene	<17.	<17.	<17.		<18.	280	150
Methylene Chloride	67	66	64		65	55	41
o-Xylene	5	<3.	<3.		<3.	69	81
Styrene	<6.	<6.	<6.		<7.	<7.	7
Tetrachloroethene	<6.	<6.	<6.		<7.	<7.	<7.
Toluene	<10	<11.	<11.		<11.	<11.	<11.
trans-1,2-Dichloroethene	<6.	<6.	<6.		<7.	<7.	12
Trichloroethene	<19.	<19.	<19.		<20.	<20.	<20.
Vinyl Chloride	<34	<35.	<35.		<37.	<37.	<37.
Semi-Volatile Organics							N/A
Acenaphthene	<42	<85	<85	<85	<44	<45	
Acenaphthylene	<63	<130	<130	<130	<67	<67	
Anthracene/Phenanthrene	<136	<280	<280	<280	<144	<145	
Benzo(a)pyrene	<52	<110	<110	<110	<56	<56	
Benzo(g,h,i)perylene	<94	<190	<190	<190	<100	<100	
Benzo[a]anthracene/Chrysene	<167	<340	<340	<340	<178	<179	
Benzo[b]/(k)fluoranthene	<157	<320	<320	<320	<167	<167	
Dibenz(a,h)anthracene	<84	<170	<170	<170	<89	<89	
Fluoranthene	<52	<110	<110	<110	<56	<56	
Fluorene	<84	<170	<170	<170	<89	<89	
Indeno(1,2,3-c,d)pyrene	<94	<190	<190	<190	<100	<100	
Naphthalene	<199	<410	<410	<410	<211	<212	
Pyrene	<73	<150	<150	<150	<78	<78	
Cl-1	<10	<21	<21	<21	<11	<11	
Cl-2	<31	<64	<64	<64	<33	<33	
Cl-3	<31	<64	<64	<64	<33	<33	
Cl-4	<21	<43	<43	<43	<22	<22	
Cl-5	<21	<43	<43	<43	<22	<22	
Cl-6	<21	<43	<43	<43	<22	<22	
Cl-7	<31	<64	<64	<64	<33	<33	
Cl-8	<31	<64	<64	<64	<33	<33	
Cl-10	<52	<110	<110	<110	<56	<56	
Total PCB	ND	ND	ND	ND	ND	ND	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B01-A (09-11) R3	S1 B01-A (12-14)	S1 B02 (02-04)	S1 B02 (06-08)	S1 B03 (02-04)	S1 B03 (06-08)	S1 B04 (02-04)	S1 B04 (6.2-08)	S1 B04 (9.5-12)
Volatile Organics									
1,1,1-Trichloroethane	33	<9.	<8.	<9.	<9.	<9.	540	500	<10.
1,1-Dichloroethane	<7.	<7.	<6.	<6.	<6.	<6.	150	130	<7.
1,1-Dichloroethene	15	<3.	<3.	<3.	<3.	<3.	<4.	<4.	<4.
1,2-Dichloroethane	<15.	<14.	<14.	<14.	<14.	<14.	120	95	<16.
Benzene	<3.	<3.	<3.	7	<3.	4	17	16	<4.
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<4.	<5.	<6.	<5.
Chlorobenzene	<9.	<9.	<8.	<9.	<9.	<9.	<10.	<11.	<10.
Chloroform	<9.	<9.	<8.	<9.	<9.	<9.	<10.	<11.	<10.
cis-1,2-Dichloroethene	6	<5.	<5.	<5.	<5.	<5.	160000	40000	150
Ethylbenzene	41	<7.	<6.	<6.	<6.	<6.	38	<8.	<7.
m/p-Xylene	160	<17.	<17.	<17.	<17.	<17.	170	<22.	<20.
Methylene Chloride	54	69	64	88	65	71	250	1700	930
o-Xylene	85	<3.	<3.	<3.	<3.	7	73	<4.	4
Styrene	9	<7.	<6.	<6.	<6.	<6.	<7.	<8.	<7.
Tetrachloroethene	<7.	<7.	<6.	<6.	<6.	<6.	110	330	<7.
Toluene	<11.	<11.	<10.	<11.	<11.	<11.	1500	<14.	110
trans-1,2-Dichloroethene	12	<7.	<6.	<6.	<6.	<6.	1500	1400	<7.
Trichloroethene	<20.	<20.	<19.	<19.	<19.	<19.	4600	10000	<22.
Vinyl Chloride	<37.	<36.	<35.	<35.	<35.	<36.	<40.	<46.	<40.
Semi-Volatile Organics									
	N/A								
Acenaphthene		<87	<84	<87	<86	<43	<96	1300	<98
Acenaphthylene		<130	<130	<130	<130	<65	<140	710	<150
Anthracene/Phenanthrene		<280	<270	<280	<280	<141	<310	5100	<320
Benzo(a)pyrene		<110	<100	<110	<110	<54	<120	<140	<120
Benzo(g,h,i)perylene		<200	<190	<190	<190	<97	<220	<250	<220
Benzo[a]anthracene/Chrysene		<350	<340	<340	<340	<173	<380	2000	<390
Benzo[b]/(k)fluoranthene		<330	<310	<320	<320	<162	<360	<420	<370
Dibenz(a,h)anthracene		<170	<170	<170	<170	<86	<190	<56	<200
Fluoranthene		<110	<100	<110	<110	<54	<120	840	<120
Fluorene		<170	<170	<170	<170	<86	<190	1900	<200
Indeno(1,2,3-c,d)pyrene		<200	<190	<190	<190	<97	<220	<84	<220
Naphthalene		<410	<400	<410	<410	<205	<460	<84	<470
Pyrene		<150	<150	<150	<150	<76	<170	1200	<170
Cl-1		<22	<21	<22	<22	<11	<24	3800	<25
Cl-2		<65	<63	<65	<65	<32	<72	<220	<74
Cl-3		<65	<63	<65	<65	<32	<72	<250	<74
Cl-4		<43	<42	<43	<43	<22	<48	<530	<49
Cl-5		<43	<42	<43	<43	<22	<48	<360	<49
Cl-6		<43	<42	<43	<43	<22	<48	<200	<49
Cl-7		<65	<63	<65	<65	<32	<72	<28	<74
Cl-8		<65	<63	<65	<65	<32	<72	<84	<74
Cl-10		<110	<100	<110	<110	<54	<120	8200	<120
Total PCB		ND	ND	ND	ND	ND	ND	12000	ND

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B05 (13-15)	S1 B06 (12-13.5)	S1 B07 (13.5-14.5)	S1 B08 (06-08)	S1 B09 (09-11)	S1 B09 (09-11) FD #1	S1 B09 (09-11) FD #2
Volatile Organics							
1,1,1-Trichloroethane	<9.	<9.	<9.	<8.	<8.	<9.	<9.
1,1-Dichloroethane	<7.	<6.	<7.	<6.	<6.	<6.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<3.
1,2-Dichloroethane	<14.	<14.	<15.	<13.	<14.	<14.	<14.
Benzene	<3.	<3.	<3.	<3.	<3.	4	8
Carbon Tetrachloride	<4.	<4.	<5.	<4.	<4.	<4.	<4.
Chlorobenzene	<9.	<9.	<9.	<8.	<8.	<9.	<9.
Chloroform	<9.	<9.	<9.	<8.	<8.	<9.	<9.
cis-1,2-Dichloroethene	<5.	<5.	<6.	<5.	<5.	<5.	<5.
Ethylbenzene	<7.	<6.	<7.	<6.	<6.	<6.	<6.
m/p-Xylene	<17.	<17.	<18.	<17.	<17.	<17.	<17.
Methylene Chloride	61	69	74	65	74	74	66
o-Xylene	9	<3.	<3.	<3.	<3.	<3.	<3.
Styrene	<7.	<6.	<7.	<6.	<6.	<6.	<6.
Tetrachloroethene	<7.	<6.	<7.	<6.	<6.	<6.	<6.
Toluene	<11.	<11.	<11.	<10.	<11.	<11.	<11.
trans-1,2-Dichloroethene	<7.	<6.	<7.	<6.	<6.	<6.	<6.
Trichloroethene	<20.	<19.	<20.	<19.	<19.	<19.	<19.
Vinyl Chloride	<36.	<35.	<37.	<34.	<35.	<35.	<35.
Semi-Volatile Organics							
Acenaphthene	<87	<86	<90	<83	<85	<85	N/A
Acenaphthylene	<130	<130	<140	<120	<130	<130	
Anthracene/Phenanthrene	<280	<280	<290	<270	<280	<280	
Benzo(a)pyrene	<110	<110	<110	<100	<110	<110	
Benzo(g,h,i)perylene	<200	<190	<200	<190	<190	<190	
Benzo[a]anthracene/Chrysene	<350	<340	<360	<330	<340	<340	
Benzo[b]/(k)fluoranthene	<330	<320	<340	<310	<320	<320	
Dibenz(a,h)anthracene	<170	<170	<180	<170	<170	<170	
Fluoranthene	<110	<110	<110	<100	<110	<110	
Fluorene	<170	<170	<180	<170	<170	<170	
Indeno(1,2,3-c,d)pyrene	<200	<190	<200	<190	<190	<190	
Naphthalene	<410	<410	<430	<390	<400	<400	
Pyrene	<150	<150	<160	<140	<150	<150	
Cl-1	<22	<21	<23	<21	<21	<21	
Cl-2	<66	<64	<68	<62	<63	<63	
Cl-3	<66	<64	<68	<62	<63	<63	
Cl-4	<44	<43	<45	<41	<42	<42	
Cl-5	<44	<43	<45	<41	<42	<42	
Cl-6	<44	<43	<45	<41	<42	<42	
Cl-7	<66	<64	<68	<62	<63	<63	
Cl-8	<66	<64	<68	<62	<63	<63	
Cl-10	<110	<110	<110	<100	<110	<110	
Total PCB	ND	ND	ND	ND	ND	ND	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B09 (09-11) FD #3	S1 B10 (06-08)	S1 B10-A (02-04)	S1 B10-A (4.5-06)	S1 B11 (02-04)	S1 B11 (05-06)	S1 B11 (08-9.5)	S1 B12-A (02-04)	S1 B12-A (05-6.5)
Volatile Organics									
1,1,1-Trichloroethane	<9.	<9.	<8.	<8.	<9.	<9.	<10.	<9.	<9.
1,1-Dichloroethane	<6.	<7.	<6.	<6.	<6.	<6.	<7.	<6.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<4.	<3.	<3.
1,2-Dichloroethane	<14.	<14.	<13.	<14.	<14.	<14.	<16.	<14.	<14.
Benzene	7	<3.	<3.	<3.	<3.	<3.	<4.	<3.	<3.
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<4.	<5.	<4.	<4.
Chlorobenzene	<9.	<9.	<8.	<8.	<9.	<9.	<10.	<9.	<9.
Chloroform	<9.	<9.	<8.	<8.	<9.	<9.	<10.	<9.	<9.
cis-1,2-Dichloroethene	<5.	<6.	<5.	300	<5.	<5.	<6.	<5.	<5.
Ethylbenzene	<6.	<7.	<6.	<6.	<6.	<6.	<7.	<6.	<6.
m/p-Xylene	<17.	<18.	<16.	<17.	<17.	<17.	<20.	<17.	<17.
Methylene Chloride	65	300	74	69	83	360	430	75	63
o-Xylene	<3.	11	<3.	<3.	<3.	8	<4.	<3.	<3.
Styrene	<6.	<7.	<6.	<6.	<6.	<6.	<7.	<6.	<6.
Tetrachloroethene	<6.	<7.	<6.	<6.	<6.	<6.	<7.	<6.	<6.
Toluene	<11.	<11.	<10.	<11.	<11.	<11.	<12.	<11.	<11.
trans-1,2-Dichloroethene	<6.	<7.	<6.	10	<6.	<6.	<7.	<6.	<6.
Trichloroethene	<19.	<20.	<19.	61	<19.	<19.	<22.	<19.	<19.
Vinyl Chloride	<35.	<37.	<34.	<35.	<35.	<35.	<41.	<35.	<35.
Semi-Volatile Organics									
	N/A								
Acenaphthene		<88	<82	<84	<86	<86	<99	<85	<85
Acenaphthylene		<130	<120	<130	<130	<130	<150	<130	<130
Anthracene/Phenanthrene		<290	<270	<270	<280	<280	<320	<280	<280
Benzo(a)pyrene		<110	<100	<110	<110	<110	<120	<110	<110
Benzo(g,h,i)perylene		<200	<190	<190	<190	<190	<220	<190	<190
Benzo[a]anthracene/Chrysene		<350	<330	<340	<340	<340	<390	<340	<340
Benzo[b]/(k)fluoranthene		<330	<310	<320	<320	<320	<370	<320	<320
Dibenz(a,h)anthracene		<180	<160	<170	<170	<170	<200	<170	<170
Fluoranthene		<110	<100	<110	<110	<110	<120	<110	<110
Fluorene		<180	<160	<170	<170	<170	<200	<170	<170
Indeno(1,2,3-c,d)pyrene		<200	<190	<190	<190	<190	<220	<190	<190
Naphthalene		<420	<390	<400	<410	<410	<470	<410	<410
Pyrene		<160	<140	<150	<150	<150	<170	<150	<150
Cl-1		<22	<21	<21	<21	<21	<25	<21	<21
Cl-2		<66	<62	<63	<64	<64	<74	<64	<64
Cl-3		<66	<62	<63	<64	<64	<74	<64	<64
Cl-4		<44	<41	<42	<43	<43	<49	<43	<43
Cl-5		<44	<41	<42	<43	<43	<49	<43	<43
Cl-6		<44	<41	<42	<43	<43	<49	<43	<43
Cl-7		<66	<62	<63	<64	<64	<74	<64	<64
Cl-8		<66	<62	<63	<64	<64	<74	<64	<64
Cl-10		<110	<100	<110	<110	<110	<120	<110	<110
Total PCB		ND	ND	ND	ND	ND	ND	ND	ND

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B12-D (06-08)	S1 B12-E (5-7)	S1 B13 (02-04)	S1 B13 (06-08)	S1 B13-A (06-08)	S1 B14 (02-04)	S1 B14 (04-06)	S1 B15 (02-04)	S1 B15 (06-08)	S1 B16-A (06-08)
Volatile Organics										
1,1,1-Trichloroethane	<9.	<8.	<9.	<44.	<8.	<9.	<9.	<9.	<9.	<11.
1,1-Dichloroethane	<6.	<6.	<7.	<33.	<6.	<7.	<6.	<7.	<7.	<8.
1,1-Dichloroethene	<3.	<3.	<3.	<16.	<3.	<3.	<3.	<3.	<3.	<4.
1,2-Dichloroethane	<14.	<14.	<14.	<71.	<14.	<14.	<14.	<14.	<15.	<18.
Benzene	<3.	7	4	<16.	<3.	<3.	<3.	<3.	4	<4.
Carbon Tetrachloride	<4.	<4.	<4.	<22.	<4.	<4.	<4.	<4.	<5.	<6.
Chlorobenzene	<9.	<8.	<9.	<44.	<8.	<9.	<9.	<9.	<9.	<11.
Chloroform	<9.	<8.	<9.	<44.	<8.	<9.	<9.	<9.	<9.	<11.
cis-1,2-Dichloroethene	<5.	<5.	<5.	<27.	<5.	<5.	<5.	<5.	<6.	42
Ethylbenzene	<6.	<6.	<7.	1700	<6.	<7.	<6.	<7.	<7.	40
m/p-Xylene	<17.	<17.	<18.	1600	<17.	<17.	<17.	<17.	<18.	44
Methylene Chloride	67	79	57	<120	68	67	70	66	72	130
o-Xylene	<3.	<3.	13	370	<3.	<3.	<3.	<3.	<3.	16
Styrene	<6.	<6.	<7.	<33.	<6.	<7.	<6.	<7.	<7.	<8.
Tetrachloroethene	<6.	<6.	<7.	<33.	<6.	<7.	<6.	<7.	<7.	<8.
Toluene	<11.	<11.	<11.	<54.	<11.	<11.	<11.	<11.	<11.	140
trans-1,2-Dichloroethene	<6.	<6.	<7.	<33.	<6.	<7.	<6.	<7.	<7.	<8.
Trichloroethene	<19.	<19.	<20.	<98.	<19.	<20.	<19.	<20.	<21.	<25.
Vinyl Chloride	<35.	<35.	<36.	<180	<35.	<36.	<35.	<36.	<38.	<46.
Semi-Volatile Organics										
Acenaphthene	<86	<85	<88	<87	N/A	<87	<86	<87	<91	<110
Acenaphthylene	<130	<130	<130	<130		<130	<130	<130	<140	<170
Anthracene/Phenanthrene	<280	<270	<290	<280		<280	<280	<280	<300	<360
Benzo(a)pyrene	<110	<110	<110	<110		<100	<110	<110	<110	<140
Benzo(g,h,i)perylene	<190	<190	<200	<200		<200	<190	<200	<210	<250
Benzo[a]anthracene/Chrysene	<340	<340	<350	<350		<350	<350	<350	<370	<440
Benzo[b]/(k)fluoranthene	<320	<320	<330	<330		<330	<320	<330	<340	<420
Dibenz(a,h)anthracene	<170	<170	<180	<170		<170	<170	<170	<180	<220
Fluoranthene	<110	<110	<110	<110		<110	<110	<110	<110	<140
Fluorene	<170	<170	<180	<170		<170	<170	<170	<180	<220
Indeno(1,2,3-c,d)pyrene	<190	<190	<200	<200		<200	<190	<200	<210	<250
Naphthalene	<410	<400	<420	<410		<410	<410	<410	<430	<530
Pyrene	<150	<150	<150	<150		<150	<150	<150	<160	<190
Cl-1	<21	<21	<22	<22		<22	<22	<22	<23	<28
Cl-2	<64	<63	<66	<65		<65	<65	<65	<68	<83
Cl-3	<64	<63	<66	<65		<65	<65	<65	<68	<83
Cl-4	<43	<42	<44	<44		<43	<43	<44	<46	<55
Cl-5	<43	<42	<44	<44		<43	<43	<44	<46	<55
Cl-6	<43	<42	<44	<44		<43	<43	<44	<46	<55
Cl-7	<64	<63	<66	<65		<65	<65	<65	<68	<83
Cl-8	<64	<63	<66	<65		<65	<65	<65	<68	<83
Cl-10	<110	<110	<110	<110		<110	<110	<110	<110	<140
Total PCB	ND	ND	ND	ND		ND	ND	ND	ND	ND

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B16-A (09-11)	S1 B17-1 (02-04)	S1 B17-1 (02-04) FD #1	S1 B17-1 (02-04) FD #2	S1 B17-2 (10-12)	S1 B17-A (02-04)	S1 B17-A (10-12)	S1 B17-A (5.75-08)
Volatile Organics								
1,1,1-Trichloroethane	<13.	<9.	<8.	<8.	<9.	210	<9.	18
1,1-Dichloroethane	<10.	<6.	<6.	<6.	<7.	<7.	<6.	<7.
1,1-Dichloroethene	<5.	<3.	<3.	<3.	<3.	<4.	<3.	<3.
1,2-Dichloroethane	<21.	<14.	<14.	<14.	<14.	<15.	<14.	<14.
Benzene	10	10	8	<3.	6	12	7	<3.
Carbon Tetrachloride	9	<4.	<4.	<4.	<4.	<5.	<4.	<4.
Chlorobenzene	<13.	<9.	<8.	<8.	<9.	<10.	<9.	<9.
Chloroform	<13.	<9.	<8.	<8.	<9.	<10.	<9.	<9.
cis-1,2-Dichloroethene	120	<5.	<5.	<5.	<5.	4800	9	810
Ethylbenzene	230	<6.	<6.	<6.	<7.	<7.	<6.	<7.
m/p-Xylene	330	<17.	<17.	<17.	<17.	<19.	<17.	<17.
Methylene Chloride	190	78	76	78	75	130	69	71
o-Xylene	110	<3.	<3.	<3.	<3.	6	<3.	<3.
Styrene	<10.	<6.	<6.	<6.	<7.	<7.	<6.	<7.
Tetrachloroethene	<10.	<6.	<6.	<6.	<7.	66	<6.	<7.
Toluene	230	<11.	<11.	<11.	<11.	<12.	<11.	<11.
trans-1,2-Dichloroethene	<10.	<6.	<6.	<6.	<7.	180	<6.	11
Trichloroethene	<30.	<19.	<19.	<19.	<20.	82	<19.	29
Vinyl Chloride	<54.	<35.	<35.	<35.	<36.	<39.	<36.	<36.
Semi-Volatile Organics								
Acenaphthene	<130	N/A	<85	N/A	<87	<200	<43	<220
Acenaphthylene	<200		<130		<130	<290	<65	<330
Anthracene/Phenanthrene	<430		<270		<280	<630	<140	<700
Benzo(a)pyrene	<160		<110		<110	<240	<54	290
Benzo(g,h,i)perylene	<300		<190		<200	<440	<97	<490
Benzo[a]anthracene/Chrysene	<530		<340		<350	900	<172	<870
Benzo[b]/(k)fluoranthene	<490		<320		<330	<730	<161	<810
Dibenz(a,h)anthracene	<260		<170		<170	<390	<86	<430
Fluoranthene	<170		<110		<110	<240	<54	<270
Fluorene	<260		<170		<170	<390	<86	<430
Indeno(1,2,3-c,d)pyrene	<300		<190		<200	<440	<97	<490
Naphthalene	<630		<400		<410	<930	<204	<1000
Pyrene	<230		<150		<150	<340	<75	<380
Cl-1	<33		<21		<22	<50	<11	<54
Cl-2	<99		<63		<65	<150	<32	<160
Cl-3	<99		<63		<65	<150	<32	<160
Cl-4	<66		<42		<44	<100	<22	<110
Cl-5	<66		<42		<44	<100	<22	<110
Cl-6	<66		<42		<44	<100	<22	<110
Cl-7	<99		<63		<65	<150	<32	<160
Cl-8	<99		<63		<65	<150	<32	<160
Cl-10	<170		<110		<110	<240	<54	<270
Total PCB	ND		ND		ND	ND	ND	ND

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S1 B18 (05-07)	S1 B18 (05-07)	S1 B18 (10-12)	S2 B01 (12-14)	S2 B01 (20-22)	S2 B02 (13-15)	S2 B02 (13-15)-R	S2 B02 (16-18)	S2 B02 (20-22)
Volatile Organics		N/A							
1,1,1-Trichloroethane	<9.		<9.	36	<46.	20	<9.	15000	300
1,1-Dichloroethane	<7.		<6.	<6.	<34.	<7.	<7.	3200	41
1,1-Dichloroethene	<3.		<3.	<3.	<17.	<3.	<3.	870	30
1,2-Dichloroethane	<15.		<14.	<14.	<75.	<14.	<15.	290	<15.
Benzene	<3.		<3.	<3.	<17.	<3.	<3.	24	<3.
Carbon Tetrachloride	<4.		<4.	<4.	<23.	<4.	<4.	<9	<5.
Chlorobenzene	<9.		<9.	<9.	<46.	<9.	<9.	32	<9.
Chloroform	<9.		<9.	<9.	<46.	<9.	<9.	<18	<9.
cis-1,2-Dichloroethene	<6.		<5.	10	120	13	<6.	1600	560
Ethylbenzene	<7.		<6.	53	830	550	58	1900	990
m/p-Xylene	<18.		<17.	140	3400	2100	<18.	12000	7400
Methylene Chloride	120		68	700	3700	810	280	370	520
o-Xylene	4		<3.	54	750	980	290	3500	2300
Styrene	<7.		<6.	<6.	<34.	<7.	<7.	<13	<7.
Tetrachloroethene	<7.		<6.	170	110	540	42	920	120
Toluene	<11.		<11.	390	22000	9100	<11.	36000	37000
trans-1,2-Dichloroethene	<7.		<6.	<6.	<34.	<7.	<7.	19	<7.
Trichloroethene	<20.		<19.	<19.	<100	<20.	<20.	67	<21.
Vinyl Chloride	<37.		<35.	<35.	<190	<36.	<37.	<73	<38.
Semi-Volatile Organics				N/A		N/A	N/A	N/A	N/A
Acenaphthene	<450	<90	<85		<93				
Acenaphthylene	<670	<130	<130		190				
Anthracene/Phenanthrene	<1500	<290	<280		<300				
Benzo(a)pyrene	<560	<110	<110		<120				
Benzo(g,h,i)perylene	<1000	<200	<190		<210				
Benzo[a]anthracene/Chrysene	<1800	<360	<340		<370				
Benzo[b]/(k)fluoranthene	<1700	<340	<320		<350				
Dibenz(a,h)anthracene	<900	<180	<170		<190				
Fluoranthene	<560	<110	<110		<120				
Fluorene	<900	<180	<170		<190				
Indeno(1,2,3-c,d)pyrene	<1000	<200	<190		<210				
Naphthalene	<2100	<430	<400		<440				
Pyrene	<800	<160	<150		<160				
Cl-1	<110	<22	<21		<23				
Cl-2	<340	<67	<64		<70				
Cl-3	<340	<67	<64		<70				
Cl-4	<230	<45	<43		<47				
Cl-5	<230	<45	<43		<47				
Cl-6	<230	<45	<43		<47				
Cl-7	<340	<67	<64		<70				
Cl-8	<340	<67	<64		<70				
Cl-10	<560	<110	<110		<120				
Total PCB	ND	ND	ND		ND				

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B02 (20-22) FD1	S2 B02 (20-22) FD2	S2 B02-1A (9.5-11)	S2 B03 (10-12)	S2 B03 (13-15)	S2 B03 (16-18)	S2 B03 (18-20)	S2 B04 (10-12)	S2 B04 (13-15)
Volatile Organics									
1,1,1-Trichloroethane	<10.	<10.	<9.	<9.	<9.	<9.	<9.	<9.	<9.
1,1-Dichloroethane	<7.	<7.	<7.	<6.	<6.	<7.	<7.	<7.	<6.
1,1-Dichloroethene	<4.	<4.	<3.	<3.	<3.	<3.	<4.	<3.	<3.
1,2-Dichloroethane	<15.	<15.	<15.	<14.	<14.	<14.	<15.	<14.	<14.
Benzene	<4.	<4.	4	<3.	<3.	4	<4.	<3.	<3.
Carbon Tetrachloride	<5.	<5.	<5.	<4.	<4.	<4.	<5.	<4.	<4.
Chlorobenzene	<10.	<10.	<9.	<9.	<9.	<9.	<9.	<9.	<9.
Chloroform	<10.	<10.	<9.	<9.	<9.	<9.	<9.	<9.	<9.
cis-1,2-Dichloroethene	7	<6.	<6.	<5.	<5.	<6.	<6.	<5.	<5.
Ethylbenzene	26	<7.	<7.	98	1700	640	340	<7.	390
m/p-Xylene	92	<19.	<18.	<17.	17000	15000	2100	<18.	870
Methylene Chloride	740	480	91	640	630	470	430	630	400
o-Xylene	81	6	<3.	170	4300	2500	530	8	970
Styrene	<7.	<7.	<7.	<6.	<6.	<7.	<7.	<7.	<6.
Tetrachloroethene	<7.	<7.	<7.	<6.	<6.	16	<7.	<7.	41
Toluene	120	58	<11.	<11.	24000	9600	930	<11.	1100
trans-1,2-Dichloroethene	<7.	<7.	<7.	<6.	<6.	<7.	<7.	<7.	<6.
Trichloroethene	<21.	<21.	<21.	<19.	<19.	<20.	<21.	<20.	<19.
Vinyl Chloride	<39.	<39.	<38.	<35.	<35.	<36.	<39.	<36.	<35.
Semi-Volatile Organics									
Acenaphthene	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	370
Acenaphthylene									1200
Anthracene/Phenanthrene									<280
Benzo(a)pyrene									970
Benzo(g,h,i)perylene									<190
Benzo[a]anthracene/Chrysene									<340
Benzo[b]/(k)fluoranthene									770
Dibenz(a,h)anthracene									<170
Fluoranthene									1100
Fluorene									<170
Indeno(1,2,3-c,d)pyrene									<190
Naphthalene									5400
Pyrene									690
Cl-1									<21
Cl-2									<64
Cl-3									<64
Cl-4									<43
Cl-5									<43
Cl-6									1300
Cl-7									<64
Cl-8									7500
Cl-10									<110
Total PCB									8800

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B04 (16-18)	S2 B04 (19-21)	S2 B05 (02-04)	S2 B05 (06-08)	S2 B05 (08-10)	S2 B05 (10-12)	S2 B06 (02-04)	S2 B06 (06-08)	S2 B06 (10-12)	S2 B07 (02-04)
Volatile Organics										
1,1,1-Trichloroethane	<9.	<9.	<8.	<9.	<9.	<9.	<9.	<9.	<9.	<9.
1,1-Dichloroethane	<7.	<7.	<6.	<7.	<7.	<7.	<6.	<6.	<7.	<6.
1,1-Dichloroethene	<3.	<4.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.
1,2-Dichloroethane	<15.	<15.	<13.	<15.	<14.	<15.	<14.	<14.	<15.	<14.
Benzene	<3.	<4.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	4
Carbon Tetrachloride	<4.	<5.	<4.	<4.	<4.	<5.	<4.	<4.	<5.	<4.
Chlorobenzene	<9.	<9.	<8.	<9.	<9.	<9.	<9.	<9.	<9.	<9.
Chloroform	<9.	<9.	<8.	<9.	16	<9.	<9.	<9.	<9.	<9.
cis-1,2-Dichloroethene	<6.	<6.	<5.	<6.	<6.	10	<5.	<5.	<6.	<5.
Ethylbenzene	190	410	<6.	<7.	<7.	<7.	<6.	<6.	<7.	<6.
m/p-Xylene	340	640	<17.	<18.	<18.	<19.	<17.	<17.	<18.	<17.
Methylene Chloride	37	42	86	540	410	650	440	400	380	72
o-Xylene	490	530	<3.	<3.	5	<3.	<3.	<3.	<3.	<3.
Styrene	<7.	<7.	<6.	<7.	<7.	<7.	<6.	<6.	<7.	<6.
Tetrachloroethene	<7.	<7.	<6.	<7.	<7.	<7.	<6.	<6.	<7.	<6.
Toluene	250	20	<10.	<11.	<11.	<12.	<11.	<11.	<11.	<11.
trans-1,2-Dichloroethene	<7.	<7.	<6.	<7.	<7.	<7.	<6.	<6.	<7.	<6.
Trichloroethene	<20.	<21.	<19.	<20.	<20.	<21.	<19.	<19.	<20.	<19.
Vinyl Chloride	<37.	<39.	<34.	<37.	<37.	<38.	<35.	<35.	<38.	<35.
Semi-Volatile Organics										
Acenaphthene	N/A	250	N/A	N/A	<89	N/A	N/A	N/A	N/A	<85
Acenaphthylene		1300			<130					<130
Anthracene/Phenanthrene		<310			<290					<280
Benzo(a)pyrene		<120			<110					<110
Benzo(g,h,i)perylene		<210			<200					<190
Benzo[a]anthracene/Chrysene		<380			<350					<340
Benzo[b]/(k)fluoranthene		<350			<330					<320
Dibenz(a,h)anthracene		<190			<180					<170
Fluoranthene		340			<110					<110
Fluorene		<190			<180					<170
Indeno(1,2,3-c,d)pyrene		<210			<200					<190
Naphthalene		17000			<420					<400
Pyrene		360			<160					<150
Cl-1		<24			<22					<21
Cl-2		<71			<67					<64
Cl-3		<71			<67					<64
Cl-4		<47			<44					<43
Cl-5		<47			<44					<43
Cl-6		<47			<44					<43
Cl-7		<71			<67					<64
Cl-8		<71			<67					<64
Cl-10		<120			<110					<110
Total PCB		ND			ND					ND

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B07 (05-07)	S2 B07 (08-10)	S2 B08 (08-10)	S2 B08 (12-14)	S2 B08 (15-17)	S2 B08 (18-20)	S2 B09 (06-08)	S2 B09 (10-12)	S2 B10 (06-08)	S2 B10 (10-12)
Volatile Organics										
1,1,1-Trichloroethane	<9.	<9.	<9.	<8.	<9.	20	<9.	<9.	<9.	<9.
1,1-Dichloroethane	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<6.	<6.	<7.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.
1,2-Dichloroethane	<14.	<14.	<14.	<14.	<15.	<15.	<14.	<14.	<14.	<14.
Benzene	<3.	4	8	<3.	<3.	<3.	<3.	<3.	4	4
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<5.	<4.	<4.	<4.	<4.
Chlorobenzene	<9.	<9.	<9.	<8.	<9.	<9.	<9.	<9.	<9.	<9.
Chloroform	<9.	<9.	<9.	<8.	<9.	26	<9.	<9.	<9.	<9.
cis-1,2-Dichloroethene	<5.	<5.	<5.	<5.	<6.	<6.	<5.	<5.	<5.	<5.
Ethylbenzene	<6.	<6.	<6.	<6.	<7.	15	<6.	<6.	<6.	<7.
m/p-Xylene	<17.	<17.	<17.	<17.	<18.	34	<17.	<17.	<17.	<17.
Methylene Chloride	60	790	72	350	380	400	820	760	820	340
o-Xylene	<3.	<3.	<3.	<3.	<3.	44	<3.	<3.	<3.	<3.
Styrene	<6.	<6.	<6.	<6.	<7.	18	<6.	<6.	<6.	<7.
Tetrachloroethene	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<6.	<6.	<7.
Toluene	<11.	<11.	<11.	<10.	<11.	48	<11.	<11.	<11.	<11.
trans-1,2-Dichloroethene	<6.	<6.	<6.	<6.	<7.	22	<6.	<6.	<6.	<7.
Trichloroethene	<19.	<19.	<19.	<19.	<20.	<21.	<19.	<19.	<19.	<20.
Vinyl Chloride	<35.	<35.	<36.	<34.	<37.	<38.	<35.	<35.	<35.	<36.
Semi-Volatile Organics										
Acenaphthene	<85	N/A	N/A	<84	N/A	N/A	N/A	N/A	N/A	N/A
Acenaphthylene	<130			<130						
Anthracene/Phenanthrene	<280			<270						
Benzo(a)pyrene	<110			<100						
Benzo(g,h,i)perylene	<190			<190						
Benzo[a]anthracene/Chrysene	<340			<340						
Benzo[b]/(k)fluoranthene	<320			<310						
Dibenz(a,h)anthracene	<170			<170						
Fluoranthene	<110			<100						
Fluorene	<170			<170						
Indeno(1,2,3-c,d)pyrene	<190			<190						
Naphthalene	<400			<400						
Pyrene	<150			<150						
Cl-1	<21			<21						
Cl-2	<64			<63						
Cl-3	<64			<63						
Cl-4	<43			<42						
Cl-5	<43			<42						
Cl-6	<43			<42						
Cl-7	<64			<63						
Cl-8	<64			<63						
Cl-10	<110			<100						
Total PCB	ND			ND						

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B11 (08-10)	S2 B11 (12-14)	S2 B11 (14-16)	S2 B11 (16-18)	S2 B12 (09-11)	S2 B12 (12-14)	S2 B12 (18-20)	S2 B12 (18-20) FD #1
Volatile Organics								
1,1,1-Trichloroethane	<9.	<9.	<9.	<10.	<9.	<9.	<9.	<10.
1,1-Dichloroethane	<6.	<6.	<6.	<7.	<6.	<7.	<7.	<7.
1,1-Dichloroethene	<3.	<3.	<3.	<4.	<3.	<3.	<3.	<4.
1,2-Dichloroethane	<14.	<14.	<14.	<16.	<14.	<14.	<15.	<15.
Benzene	<3.	<3.	<3.	<4.	9	<3.	<3.	9
Carbon Tetrachloride	<4.	<4.	<4.	<5.	<4.	<4.	<5.	<5.
Chlorobenzene	<9.	<9.	<9.	<10.	<9.	<9.	<9.	<10.
Chloroform	<9.	<9.	<9.	<10.	14	<9.	<9.	<10.
cis-1,2-Dichloroethene	<5.	<5.	<5.	<6.	<5.	<5.	<6.	<6.
Ethylbenzene	<6.	<6.	<6.	<7.	19	<7.	<7.	<7.
m/p-Xylene	<17.	<17.	<17.	<19.	51	<17.	<18.	<19.
Methylene Chloride	730	300	270	630	500	340	630	76
o-Xylene	<3.	<3.	<3.	12	30	<3.	<3.	<4.
Styrene	<6.	<6.	<6.	<7.	<6.	<7.	<7.	<7.
Tetrachloroethene	<6.	<6.	<6.	<7.	<6.	<7.	<7.	<7.
Toluene	<11.	<11.	<11.	<12.	73	<11.	<11.	<12.
trans-1,2-Dichloroethene	<6.	<6.	<6.	<7.	<6.	<7.	<7.	<7.
Trichloroethene	<19.	<19.	<19.	<22.	<19.	<20.	<20.	<21.
Vinyl Chloride	<35.	<35.	<35.	<40.	<35.	<36.	<38.	<39.
Semi-Volatile Organics								
Acenaphthene	N/A	N/A	N/A	N/A	N/A	<87	<91	N/A
Acenaphthylene						<130	<140	
Anthracene/Phenanthrene						<280	<300	
Benzo(a)pyrene						<110	<110	
Benzo(g,h,i)perylene						<200	<200	
Benzo[a]anthracene/Chrysene						<350	<360	
Benzo[b]/(k)fluoranthene						<330	<340	
Dibenz(a,h)anthracene						<170	<180	
Fluoranthene						<110	<110	
Fluorene						<170	<180	
Indeno(1,2,3-c,d)pyrene						<200	<200	
Naphthalene						<410	<430	
Pyrene						<150	<160	
Cl-1						<22	<23	
Cl-2						<66	<68	
Cl-3						<66	<68	
Cl-4						<44	<45	
Cl-5						<44	<45	
Cl-6						<44	<45	
Cl-7						<66	<68	
Cl-8						<66	<68	
Cl-10						<110	<110	
Total PCB						ND	ND	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B12 (18-20) FD #2	S2 B13 (08-10)	S2 B13 (14-16)	S2 B13 (14-16)-R	S2 B13 (18-20)	S2 B14 (08-10)	S2 B14 (12-14)	S2 B14 (18-20)	S2 B15 (10-12)
Volatile Organics									
1,1,1-Trichloroethane	<10.	<9.	31	<94	210	<9.	<9.	24	<9.
1,1-Dichloroethane	<7.	<6.	<6.	<70	15	<7.	<6.	<7.	<7.
1,1-Dichloroethene	<4.	<3.	<3.	<35	51	<3.	<3.	<4.	<3.
1,2-Dichloroethane	<15.	<14.	<14.	<150	<15.	<14.	<14.	<15.	<14.
Benzene	5	<3.	<3.	<35	9	<3.	<3.	<4.	<3.
Carbon Tetrachloride	<5.	<4.	<4.	<47	<4.	<4.	<4.	<5.	<4.
Chlorobenzene	<10.	<9.	<9.	<94	<9.	<9.	<9.	<9.	<9.
Chloroform	<10.	<9.	<9.	<94	<9.	<9.	<9.	<9.	<9.
cis-1,2-Dichloroethene	<6.	30	380	<59	15000	<5.	<5.	110	<5.
Ethylbenzene	<7.	<6.	160	<59	230	14	<6.	28	<7.
m/p-Xylene	<19.	<17.	210	2200	640	54	<17.	86	<17.
Methylene Chloride	74	490	640	990	160	410	390	200	400
o-Xylene	<4.	<3.	95	2700	190	27	10	67	<3.
Styrene	<7.	<6.	<6.	<70	<7.	<7.	<6.	<7.	<7.
Tetrachloroethene	<7.	<6.	42	<70	25	<7.	<6.	<7.	<7.
Toluene	<12.	<11.	1600	<120	5500	43	<11.	390	<11.
trans-1,2-Dichloroethene	<7.	<6.	<6.	<70	45	<7.	<6.	<7.	<7.
Trichloroethene	<21.	<19.	<19.	<210	<20.	<20.	<19.	<21.	<20.
Vinyl Chloride	<39.	<35.	<35.	<390	<37.	<36.	<35.	<39.	<36.
Semi-Volatile Organics									
Acenaphthene	N/A	N/A	<85	N/A	N/A	N/A	N/A	<94	N/A
Acenaphthylene			1200					<140	
Anthracene/Phenanthrene			<280					<310	
Benzo(a)pyrene			830					<120	
Benzo(g,h,i)perylene			<190					<210	
Benzo[a]anthracene/Chrysene			290					<380	
Benzo[b]/(k)fluoranthene			620					<350	
Dibenz(a,h)anthracene			260					<190	
Fluoranthene			<110					<120	
Fluorene			<170					<190	
Indeno(1,2,3-c,d)pyrene			240					<210	
Naphthalene			650					<450	
Pyrene			410					<170	
Cl-1			<21					<24	
Cl-2			<64					<71	
Cl-3			56					<71	
Cl-4			<43					<47	
Cl-5			<43					<47	
Cl-6			1200					<47	
Cl-7			<64					<71	
Cl-8			5600					<71	
Cl-10			<110					<120	
Total PCB			6856					ND	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S2 B15 (14-16)	S2 B15 (14-16) R1	S2 B15-A (10-12)	S2 B15-A (14-16)-R	S2 B15-A (18-20)-R	S2 B27-A (09-11)	S3 B01 (10-12)	S3 B01 (13-15)	S3 B01 (17-19)
Volatile Organics									
1,1,1-Trichloroethane	99	140	<9.	<9.	<9.	<9.	<9.	<86.	<9.
1,1-Dichloroethane	68	<33.	<6.	<6.	<7.	<7.	<7.	<64.	<7.
1,1-Dichloroethene	100	<17.	<3.	<3.	<4.	<4.	<3.	<32.	<3.
1,2-Dichloroethane	30	<72.	<14.	<14.	<15.	<15.	<15.	<140	<14.
Benzene	330	140	<3.	<3.	<4.	5	<3.	<32.	4
Carbon Tetrachloride	<9.	<22.	<4.	<4.	<5.	<5.	<4.	<43.	<4.
Chlorobenzene	36	<44.	<9.	<9.	<9.	<9.	<9.	<86.	<9.
Chloroform	<18.	53	<9.	<9.	<9.	<9.	<9.	<86.	<9.
cis-1,2-Dichloroethene	2200	430	<5.	<5.	<6.	<6.	<6.	<54.	11
Ethylbenzene	500	1800	<6.	54	63	<7.	730	13000	<7.
m/p-Xylene	1300	1400	<17.	150	300	<19.	9100	320000	<18.
Methylene Chloride	450000	34000	410	240	120	84	180	880	430
o-Xylene	1000	1400	4	410	430	<4.	1300	83000	8
Styrene	<13.	<33.	<6.	<6.	<7.	<7.	<7.	<64.	<7.
Tetrachloroethene	250	260	<6.	<6.	<7.	<7.	<7.	<64.	<7.
Toluene	8300	4300	<11.	120	60	<12.	390	280000	<11.
trans-1,2-Dichloroethene	1500	460	<6.	<6.	<7.	<7.	<7.	<64.	23
Trichloroethene	2100000	810000	<19.	<19.	<21.	<21.	<20.	<190	<20.
Vinyl Chloride	<73.	<180	<35.	<35.	<39.	<39.	<37.	<350	<37.
Semi-Volatile Organics									
Acenaphthene	380	N/A	N/A	N/A	N/A	N/A	N/A	<86	N/A
Acenaphthylene	1100							<130	
Anthracene/Phenanthrene	<580							<280	
Benzo(a)pyrene	850							<110	
Benzo(g,h,i)perylene	780							<190	
Benzo[a]anthracene/Chrysene	<710							<340	
Benzo[b]/(k)fluoranthene	870							<320	
Dibenz(a,h)anthracene	590							<170	
Fluoranthene	960							<110	
Fluorene	<350							<170	
Indeno(1,2,3-c,d)pyrene	550							<190	
Naphthalene	2600							<410	
Pyrene	800							<150	
Cl-1	<44							<21	
Cl-2	<133							<64	
Cl-3	<133							<64	
Cl-4	<88							820	
Cl-5	<88							1200	
Cl-6	<88							57	
Cl-7	<133							<64	
Cl-8	<133							690	
Cl-10	<221							<110	
Total PCB	ND							2767	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S3 B02 (10-12)	S3 B02 (12-14)	S3 B02 (12-14) FD1	S3 B02 (12-14) FD2	S3 B02 (17-19)	S3 B03 (02-04)	S3 B03 (06-08)	S3 B03 (10-12)	S3 B03 (14-16)
Volatile Organics									
1,1,1-Trichloroethane	<9.	<9.	<8.	<8.	<9.	<9.	<8.	<9.	<9.
1,1-Dichloroethane	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<7.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.
1,2-Dichloroethane	<14.	<14.	<14.	<14.	<15.	<14.	<14.	<14.	<14.
Benzene	4	5	4	<3.	<3.	<3.	<3.	<3.	<3.
Carbon Tetrachloride	<4.	<4.	7	7	<5.	<4.	<4.	<4.	<4.
Chlorobenzene	<9.	<9.	<8.	<8.	<9.	<9.	<8.	<9.	<9.
Chloroform	<9.	<9.	<8.	<8.	<9.	<9.	<8.	<9.	<9.
cis-1,2-Dichloroethene	10	<5.	5	<5.	<6.	<5.	<5.	26	<5.
Ethylbenzene	<6.	22	<6.	<6.	67	<7.	<6.	38	14
m/p-Xylene	<17.	130	<17.	<17.	720	<18.	<17.	<17.	99
Methylene Chloride	820	91	78	79	100	830	670	860	160
o-Xylene	<3.	40	17	4	310	<3.	<3.	<3.	5600
Styrene	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<7.	<6.
Tetrachloroethene	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<7.	<6.
Toluene	<11.	<11.	<11.	<11.	150	<11.	<11.	<11.	160
trans-1,2-Dichloroethene	<6.	<6.	<6.	<6.	<7.	<7.	<6.	<7.	<6.
Trichloroethene	<19.	<19.	<19.	<19.	<20.	<20.	<19.	<20.	<19.
Vinyl Chloride	<35.	<35.	<35.	<35.	<37.	<36.	<35.	<36.	<35.
Semi-Volatile Organics									
Acenaphthene	N/A	N/A	N/A	N/A	N/A	<88	N/A	<87	N/A
Acenaphthylene						<130		<130	
Anthracene/Phenanthrene						<290		<280	
Benzo(a)pyrene						<110		<110	
Benzo(g,h,i)perylene						<200		<200	
Benzo[a]anthracene/Chrysene						<350		<350	
Benzo[b]/(k)fluoranthene						<330		<330	
Dibenz(a,h)anthracene						<180		<170	
Fluoranthene						<110		<110	
Fluorene						<180		<170	
Indeno(1,2,3-c,d)pyrene						<200		<200	
Naphthalene						<420		<410	
Pyrene						<150		<150	
Cl-1						<22		<22	
Cl-2						<66		<65	
Cl-3						<66		<65	
Cl-4						<44		<44	
Cl-5						<44		160	
Cl-6						<44		200	
Cl-7						<66		<65	
Cl-8						<66		<65	
Cl-10						<110		<110	
Total PCB						ND		360	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S3 B04 (10-12)	S3 B04 (12.5-14)	S3 B05 (10-12)	S3 B05 (14-16)	S3 B05 (17-19)	S3 B06 (10-12)	S3 B06 (13-15)	S3 B06 (16-18)	S3 B07 (10-12)
Volatile Organics									
1,1,1-Trichloroethane	<9.	<9.	<9.	<9.	<9.	<9.	80	<10.	<9.
1,1-Dichloroethane	<7.	<7.	<7.	<6.	<7.	<7.	<7.	<7.	<7.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	24	<4.	<3.
1,2-Dichloroethane	<14.	<15.	<14.	<14.	<14.	<14.	<14.	<16.	<14.
Benzene	6	<3.	4	<3.	<3.	5	6	<4.	5
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<4.	9	<5.	<4.
Chlorobenzene	<9.	<9.	<9.	<9.	<9.	<9.	<9.	<10.	<9.
Chloroform	<9.	<9.	<9.	<9.	<9.	<9.	19	<10.	<9.
cis-1,2-Dichloroethene	<5.	<6.	110	<5.	6	16	9	<6.	24
Ethylbenzene	280	670	35	4300	<7.	22	50	<7.	8
m/p-Xylene	110	3600	86	31000	<17.	28	150	<19.	<18.
Methylene Chloride	130	410	860	33	390	73	100	65	87
o-Xylene	1000	1800	39	12000	<3.	18	94	<4.	17
Styrene	<7.	<7.	<7.	<6.	<7.	<7.	48	<7.	<7.
Tetrachloroethene	<7.	<7.	<7.	<6.	<7.	<7.	61	<7.	<7.
Toluene	60	200	140	15000	<11.	33	22	<12.	<11.
trans-1,2-Dichloroethene	<7.	<7.	<7.	<6.	<7.	<7.	160	<7.	<7.
Trichloroethene	<20.	<20.	<20.	<19.	<20.	<20.	<20.	<22.	<20.
Vinyl Chloride	<36.	<37.	<36.	<35.	<36.	<37.	<36.	<40.	<37.
Semi-Volatile Organics									
Acenaphthene	N/A	N/A	N/A	N/A	<87	220	N/A	<97	N/A
Acenaphthylene					<130	<130		<150	
Anthracene/Phenanthrene					<280	900		<320	
Benzo(a)pyrene					<110	<110		<120	
Benzo(g,h,i)perylene					<200	<200		<220	
Benzo[a]anthracene/Chrysene					<350	<360		<390	
Benzo[b]/(k)fluoranthene					<330	<330		<360	
Dibenz(a,h)anthracene					<170	<180		<190	
Fluoranthene					<110	<110		<120	
Fluorene					<170	340		<190	
Indeno(1,2,3-c,d)pyrene					<200	<200		<220	
Naphthalene					<410	<420		<460	
Pyrene					<150	<160		<170	
Cl-1					<22	<22		<24	
Cl-2					<65	<67		<73	
Cl-3					<65	<67		<73	
Cl-4					<44	220		<48	
Cl-5					<44	1200		<48	
Cl-6					<44	370		<48	
Cl-7					<65	<67		<73	
Cl-8					<65	560		<73	
Cl-10					<110	<110		<120	
Total PCB					ND	2350		ND	

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S3 B08 (06-08)	S3 B08 (10-12)	S3 B08 (12-14)	S3 B09 (06-08)	S3 B09 (10-12)	S3 B10 (06-08)	S3 B10 (10-12)	S3 B11 (06-08)	S3 B11 (10-12)	S3 B12 (05-07)
Volatile Organics										
1,1,1-Trichloroethane	<9.	<9.	<9.	<8.	<9.	<8.	<9.	<9.	<9.	<8.
1,1-Dichloroethane	<7.	<7.	<6.	<6.	<7.	<6.	<7.	<7.	<7.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<4.	<3.	<3.	<3.
1,2-Dichloroethane	<15.	<14.	<14.	<13.	<14.	<13.	<15.	<15.	<14.	<14.
Benzene	12	15	3	<3.	<3.	<3.	<4.	<3.	<3.	<3.
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<4.	<5.	<5.	<4.	<4.
Chlorobenzene	<9.	<9.	<9.	<8.	<9.	<8.	<9.	<9.	<9.	<8.
Chloroform	<9.	<9.	<9.	<8.	<9.	<8.	<9.	<9.	<9.	<8.
cis-1,2-Dichloroethene	830	250	<5.	<5.	<6.	<5.	<6.	<6.	<6.	<5.
Ethylbenzene	460	400	<6.	<6.	<7.	<6.	<7.	<7.	<7.	<6.
m/p-Xylene	860	1600	<17.	<16.	<18.	<17.	<19.	<18.	<18.	<17.
Methylene Chloride	120	78	55	60	54	66	65	71	63	360
o-Xylene	430	350	<3.	<3.	<3.	<3.	<4.	<3.	<3.	<3.
Styrene	<7.	<7.	<6.	<6.	<7.	<6.	<7.	<7.	<7.	<6.
Tetrachloroethene	<7.	<7.	<6.	<6.	<7.	<6.	<7.	<7.	<7.	<6.
Toluene	1400	2400	<11.	<10.	<11.	<10.	<12.	<11.	<11.	<10.
trans-1,2-Dichloroethene	32	19	<6.	<6.	<7.	<6.	<7.	<7.	<7.	<6.
Trichloroethene	<20.	<20.	<19.	<19.	<20.	<19.	<21.	<21.	<20.	<19.
Vinyl Chloride	<37.	<37.	<36.	<34.	<37.	<34.	<39.	<38.	<37.	<34.
Semi-Volatile Organics										
Acenaphthene	<89	N/A	<87	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Acenaphthylene	<130		<130							
Anthracene/Phenanthrene	<290		<280							
Benzo(a)pyrene	<110		<110							
Benzo(g,h,i)perylene	<200		<190							
Benzo[a]anthracene/Chrysene	<360		<350							
Benzo[b]/(k)fluoranthene	<330		<320							
Dibenz(a,h)anthracene	<180		<170							
Fluoranthene	<110		<110							
Fluorene	<180		<170							
Indeno(1,2,3-c,d)pyrene	<200		<190							
Naphthalene	<420		<410							
Pyrene	<160		<150							
Cl-1	<22		<22							
Cl-2	<67		<65							
Cl-3	<67		<65							
Cl-4	<45		<43							
Cl-5	<45		<43							
Cl-6	<45		<43							
Cl-7	<67		<65							
Cl-8	<67		<65							
Cl-10	<110		<110							
Total PCB	ND		ND							

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S3 B12 (09-11)	S3 B12 (09-11) FD1	S3 B12 (09-11) FD2	S3 B13 (10-12)	S3 B14 (06-08)	S3 B15 (06-08)	S3 B16 (06-08)	S3 B16-A (09-11)	S3 B17 (06-08)
Volatile Organics									
1,1,1-Trichloroethane	<9.	<9.	<9.	<9.	<8.	<8.	<8.	<8.	<8.
1,1-Dichloroethane	<7.	<7.	<7.	<7.	<6.	<6.	<6.	<6.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.
1,2-Dichloroethane	<15.	<15.	<15.	<14.	<14.	<14.	<13.	<14.	<13.
Benzene	<3.	<3.	5	<3.	<3.	5	6	3	<3.
Carbon Tetrachloride	<5.	<5.	<5.	<4.	<4.	<4.	<4.	<4.	<4.
Chlorobenzene	<9.	<9.	<9.	<9.	<8.	<8.	<8.	<8.	<8.
Chloroform	<9.	<9.	<9.	<9.	<8.	<8.	<8.	<8.	<8.
cis-1,2-Dichloroethene	<6.	<6.	<6.	<5.	<5.	<5.	<5.	<5.	<5.
Ethylbenzene	<7.	<7.	<7.	<7.	<6.	<6.	<6.	<6.	<6.
m/p-Xylene	<18.	<18.	<18.	<17.	<17.	<17.	<16.	<17.	<17.
Methylene Chloride	110	100	100	67	66	68	66	65	60
o-Xylene	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.	<3.
Styrene	<7.	<7.	<7.	<7.	<6.	<6.	<6.	<6.	<6.
Tetrachloroethene	<7.	<7.	<7.	<7.	<6.	<6.	<6.	<6.	<6.
Toluene	<11.	<11.	<11.	<11.	<11.	<10.	<10.	<10.	<10.
trans-1,2-Dichloroethene	<7.	<7.	<7.	<7.	<6.	<6.	<6.	<6.	<6.
Trichloroethene	<21.	<21.	<21.	<20.	<19.	<19.	<18.	<19.	<19.
Vinyl Chloride	<38.	<38.	<38.	<36.	<35.	<34.	<34.	<35.	<34.
Semi-Volatile Organics									
Acenaphthene	<92	<89		N/A	N/A	N/A	N/A	N/A	N/A
Acenaphthylene	<140	<130							
Anthracene/Phenanthrene	<300	<290							
Benzo(a)pyrene	<110	<110							
Benzo(g,h,i)perylene	<210	<200							
Benzo[a]anthracene/Chrysene	<370	<360							
Benzo[b]/(k)fluoranthene	<340	<340							
Dibenz(a,h)anthracene	<180	<180							
Fluoranthene	<110	<110							
Fluorene	<180	<180							
Indeno(1,2,3-c,d)pyrene	<210	<200							
Naphthalene	<440	<420							
Pyrene	<160	<160							
Cl-1	<23	<22							
Cl-2	<69	<67							
Cl-3	<69	<67							
Cl-4	<46	<45							
Cl-5	<46	<45							
Cl-6	<46	<45							
Cl-7	<69	<67							
Cl-8	<69	<67							
Cl-10	<110	<110							
Total PCB	ND	ND							

All measurements in ppb / dry weight

Organic Compound Analysis for the Hanscom Air Force Base ETI Project

Sample ID	S3 B18 (06-08)	S3 B19 (09-11)	S3 B20 (10-12)	S3 B21 (13.5-15)	S3 B22 (10-12)	S3 B23 (08-10)	S3 B23 (13-15)	S3 B23 (16-18)	S3 B24 (9.5-11)
Volatile Organics									
1,1,1-Trichloroethane	<8.	<8.	<8.	<8.	<8.	<8.	<8.	<9.	<8.
1,1-Dichloroethane	<6.	<6.	<6.	<6.	<6.	<6.	<6.	<7.	<6.
1,1-Dichloroethene	<3.	<3.	<3.	<3.	<3.	<3.	15	<3.	<3.
1,2-Dichloroethane	<13.	<13.	<13.	<14.	<14.	<14.	<14.	<15.	<14.
Benzene	<3.	<3.	5	<3.	7	<3.	4	<3.	6
Carbon Tetrachloride	<4.	<4.	<4.	<4.	<4.	<4.	6	<5.	<4.
Chlorobenzene	<8.	<8.	<8.	<8.	<8.	<8.	<8.	<9.	<8.
Chloroform	<8.	<8.	<8.	<8.	<8.	<8.	<8.	<9.	<8.
cis-1,2-Dichloroethene	<5.	<5.	<5.	<5.	<5.	<5.	<5.	<6.	<5.
Ethylbenzene	<6.	<6.	<6.	<6.	<6.	<6.	7	<7.	<6.
m/p-Xylene	<16.	<16.	<17.	<17.	<17.	<17.	<17.	<18.	<17.
Methylene Chloride	64	63	59	70	63	63	81	68	73
o-Xylene	<3.	<3.	<3.	<3.	<3.	<3.	17	<3.	<3.
Styrene	<6.	<6.	<6.	<6.	<6.	<6.	<6.	<7.	<6.
Tetrachloroethene	<6.	<6.	<6.	<6.	<6.	<6.	23	<7.	<6.
Toluene	<10.	<10.	<10.	<11.	<10.	<11.	<10.	<11.	<11.
trans-1,2-Dichloroethene	<6.	<6.	<6.	<6.	<6.	<6.	<6.	<7.	<6.
Trichloroethene	<19.	<18.	<19.	<19.	<19.	<19.	<19.	<21.	<19.
Vinyl Chloride	<34.	<34.	<34.	<35.	<34.	<35.	<34.	<38.	<35.
Semi-Volatile Organics									
Acenaphthene	N/A	N/A	N/A	N/A	N/A	<85	N/A	N/A	N/A
Acenaphthylene						<130			
Anthracene/Phenanthrene						<280			
Benzo(a)pyrene						<110			
Benzo(g,h,i)perylene						<190			
Benzo[a]anthracene/Chrysene						<340			
Benzo[b]/(k)fluoranthene						<320			
Dibenz(a,h)anthracene						<170			
Fluoranthene						<110			
Fluorene						<170			
Indeno(1,2,3-c,d)pyrene						<190			
Naphthalene						<400			
Pyrene						<150			
Cl-1						<21			
Cl-2						<64			
Cl-3						<64			
Cl-4						<42			
Cl-5						<42			
Cl-6						<42			
Cl-7						<64			
Cl-8						<64			
Cl-10						<110			
Total PCB						ND			

All measurements in ppb / dry weight

Metals Analysis by Field Inductively Coupled Plasma/Optical Emission Spectroscopy

	Field ICAP		Field ICAP Dry		
	% Solids	Wet Value		Value	
		Pb	Cd	Pb	Cd
	mg/kg	mg/kg	mg/kg	mg/kg	
S1-B10-(6-8)	90.4	6.3	nd	7.0	nd
S1-B11-(2-4)	93.4	11	0.40	11	0.43
S1-B11-(5-6)	93.4	18	0.45	19	0.48
S1-B11-(8-9.5)	81.1	8.9	0.43	11	0.53
S1-B12A-(2-4)	93.7	7.3	0.53	7.8	0.57
S1-B12A-(5-6.5)	93.7	10	0.36	11	0.38
S1-B13-(2-4)	91.0	7.5	nd	8.2	nd
S1-B17A-(10-12)	93.1	7.2	nd	7.8	nd
S1-B17A-(2-4)	84.2	28	0.33	34	0.39
S1-B17A-(5.75-8)	92.3	34	0.41	37	0.44
S1-B1A-(12-14)	92.3	nd	nd	nd	nd
S1-B1A-(2-4)	95.5	21	nd	21	nd
S1-B1A-(2-4) FD	94.4	22	0.39	23	0.41
S1-B1A-(6-8)	90.0	nd	nd	nd	nd
S1-B1A-(9-11)	89.6	7.3	nd	8.2	nd
S1-B2-(2-4)	95.5	12	0.42	12	0.44
S1-B2-(6-8)	91.6	8.1	0.40	8.8	0.44
S1-B3-(2-4)	92.9	19	0.42	20	0.45
S1-B3-(6-8)	92.5	9.5	0.66	10	0.71
S1-B4-(2-4)	83.5	14	nd	17	nd
S1-B4-(6.2-8)	71.6	16	nd	22	nd
S1-B4-(9.5-12)	81.6	13	nd	16	nd

	% Solids	Field ICAP Wet Value		Field ICAP Dry Value	
		Pb	Cd	Pb	Cd
		mg/kg	mg/kg	mg/kg	mg/kg
S2-B1-(12-14)	93.9	243	nd	259	nd
S2-B1-(16-18)	93.4	181	0.33	194	0.35
S2-B1-(20-22)	86.8	12	0.38	14	0.44
S2-B1-(9-11)	90.5	19	nd	21	nd
S2-B10-(10-12)	92.1	7.2	nd	7.8	nd
S2-B10-(6-8)	94.4	7.2	nd	7.6	nd
S2-B11-(12-14)	93.5	8.1	0.36	8.7	0.38
S2-B11-(14-16)	92.6	5.0	nd	5.4	nd
S2-B11-(16-18)	82.2	nd	nd	nd	nd
S2-B11-(8-10)	92.7	8.7	nd	9.4	nd
S2-B12-(12-14)	91.6	10	0.37	11	0.40
S2-B12-(18-20)	88.1	6.4	nd	7.3	nd
S2-B12-(18-20)FD	84.2	5.4	nd	6.4	nd
S2-B12-(9-11)	93.6	14	nd	15	nd
S2-B13-(14-16)	94.0	25	nd	26	nd
S2-B13-(18-20)	89.4	7.6	nd	8.5	nd
S2-B13-(8-10)	93.4	13	nd	14	nd
S2-B14-(12-14)	93.0	11	0.35	12	0.38
S2-B14-(18-20)	84.8	6.5	nd	7.6	nd
S2-B14-(8-10)	92.1	13	0.36	14	0.39
S2-B15-(10-12)	91.9	12	nd	13	nd
S2-B15-(14-16)	90.5	327	5.7	362	6.29
S2-B15A-(10-12)	92.9	13	nd	14	nd
S2-B15A-(14-16)	93.6	13	nd	14	nd
S2-B15A-(18-20)	84.4	nd	nd	nd	nd
S2-B2-(10-12)	90.0	107	nd	119	nd
S2-B2-(13-15)	91.0	94	nd	103	nd
S2-B2-(16-18)	90.5	60	0.55	67	0.60
S2-B2-(20-22)	85.7	14	nd	16	nd
S2-B2-(20-22) FD	83.9	18	0.36	21	0.43
S2-B3-(10-12)	92.0	38	nd	42	nd
S2-B3-(13-15)	94.4	106	nd	112	nd
S2-B3-(16-18)		70	0.38		
S2-B3-(18-20)	85.0	9.4	0.93	11	1.09
S2-B4-(10-12)	90.9	18	nd	20	nd
S2-B4-(13-15)	93.5	204	0.49	218	0.52
S2-B4-(16-18)	88.9	58	nd	65	nd
S2-B4-(19-21)	85.0	16	nd	19	nd

	Field ICAP		Field ICAP Dry		
	% Solids	Wet Value		Value	
		Pb	Cd	Pb	Cd
	mg/kg	mg/kg	mg/kg	mg/kg	
S2-B5-(10-12)	86.3	5.6	nd	6.5	nd
S2-B5-(2-4)	96.5	13	nd	13	nd
S2-B5-(6-8)	88.9	nd	nd	nd	nd
S2-B5-(8-10)	90.2	9.6	nd	11	nd
S2-B6-(10-12)	87.9	6.5	nd	7.4	nd
S2-B6-(2-4)	92.5	42	0.33	46	0.36
S2-B6-(6-8)	94.2	nd	nd	nd	nd
S2-B7-(2-4)	94.0	5.2	nd	5.5	nd
S2-B7-(5-7)	94.1	5.9	nd	6.3	nd
S2-B7-(8-10)	93.9	8.6	nd	9.2	nd
S2-B8-(12-14)	95.5	7.2	nd	7.5	nd
S2-B8-(15-17)	88.8	nd	nd	nd	nd
S2-B8-(18-20)	86.9	6.9	nd	7.9	nd
S2-B8-(8-10)	92.9	21	0.37	23	0.40
S2-B9-(10-12)	92.5	5.5	nd	5.9	nd
S2-B9-(6-8)	94.2	7.7	nd	8.1	nd

	% Solids	Field ICAP Wet Value		Field ICAP Dry Value	
		Pb	Cd	Pb	Cd
		mg/kg	mg/kg	mg/kg	mg/kg
S3-B1-(10-12)	89.5	34	11	38	12
S3-B1-(13-15)	93.2	61	5.2	65	5.6
S3-B1-(17-19)	90.4	52	1.1	57	1.2
S3-B10-(10-12)	85.6	5.8	nd	6.8	nd
S3-B10-(6-8)	96.9	5.1	nd	5.2	nd
S3-B11-(10-12)	90.1	6.5	nd	7.2	nd
S3-B11-(6-8)	85.9	6.3	nd	7.4	nd
S3-B12-(5-7)	96.2	7.4	0.49	7.6	0.51
S3-B12-(9-11)	87.1	nd	0.37	nd	0.42
S3-B12-(9-11) FD	89.5	5.3	0.44	6.0	0.49
S3-B13-(10-12)	91.7	5.9	nd	6.4	nd
S3-B14-(6-8)	94.8	5.0	nd	5.3	nd
S3-B15-(6-8)	96.1	5.4	nd	5.7	nd
S3-B16-(6-8)	97.7	nd	nd	nd	nd
S3-B16A-(9-11)	95.3	6.2	0.41	6.5	0.43
S3-B17-(6-8)	96.7	nd	nd	nd	nd
S3-B18-(6-8)	97.0	5.6	nd	5.7	nd
S3-B19-(9-11)	97.8	nd	nd	nd	nd
S3-B2-(10-12)	92.8	20	1.7	21	1.8
S3-B2-(12-14)	94.2	12	0.85	13	0.90
S3-B2-(12-14) FD	95.1	13	0.71	14	0.75
S3-B2-(17-19)	88.1	5.9	0.35	6.7	0.40
S3-B20-(10-12)	96.4	7.6	nd	7.9	nd
S3-B21-(13.5-15)	94.4	7.2	nd	7.6	nd
S3-B22-(10-12)	96.0	nd	nd	nd	nd
S3-B23-(13-15)	96.1	8.6	0.35	8.9	0.36
S3-B23-(16-18)	87.3	nd	nd	nd	nd
S3-B23-(8-10)	94.3	11	0.84	11	0.89
S3-B24-(9.5-11)	94.4	7.0	0.59	7.4	0.62
S3-B3-(10-12)	91.8	34	10	37	10.9
S3-B3-(2-4)	90.6	8.3	nd	9.1	nd
S3-B3-(6-8)	95.3	8.0	0.56	8.4	0.59
S3-B4-(10-12)	92.2	27	21	29	23
S3-B4-(12.5-14)	89.0	20	14	22	16
S3-B5-(10-12)	91.7	39	33	43	36
S3-B5-(14-16)	93.3	38	25	41	26
S3-B5-(17-19)	91.8	5.5	0.91	6.0	0.99
S3-B6-(10-12)	90.0	17	0.56	19.3	0.62

	Field ICAP Wet Value		Field ICAP Dry Value		
	% Solids	Pb mg/kg	Cd mg/kg	Pb mg/kg	Cd mg/kg
S3-B6-(16-18)	82.5	5.4	nd	6.5	nd
S3-B7-(10-12)	90.4	28	0.43	31	0.48
S3-B8-(10-12)	89.7	13	nd	15	nd
S3-B8-(12-14)	92.4	7.0	nd	7.6	nd
S3-B8-(6-8)	89.6	24	0.40	27	0.45
S3-B9-(10-12)	89.9	nd	nd	nd	nd
S3-B9-(6-8)	97.1	5.5	nd	5.7	nd

Summary of Lead and Cadmium Values by Energy Dispersive X-ray Fluorescence

Tufts University Laboratory

#	Sample ID	Pb* (mg/kg)	Cd* (mg/kg)
1	S1-B11-(5-6)	29.7	ND
2	S1-B17-2(10-12)	22.7	ND
3	S1-B18-(5-7)	51.8	ND
4	S2-B1-(9-11)	29.5	ND
5	S2-B1-(12-14)	226	ND
6	S2-B1-(16-18)	121	ND
7	S2-B2-(20-22)	23.7	ND
8	S2-B3-(10-12)	66.5	2.4
9	S2-B3-(18-20)	19.2	4.5
10	S2-B4-(10-12)	38.9	ND
11	S2-B4-(16-18)	82.3	ND
12	S2-B5-(2-4)	31.1	ND
13	S2-B6-(2-4)	53.0	ND
14	S2-B7-(8-10)	23.1	ND
15	S2-B8-(8-10)	45.3	ND
16	S2-B10-(10-12)	14.4	ND
17	S2-B11-(16-18)	16.7	3.1
18	S2-B12-(12-14)	19.5	3.7
19	S2-B13-(14-16)	28.1	ND
20	S2-B15-(14-16)	418	14.9
21	S2-B15A-(10-12)	24.3	ND

#	Sample ID	Pb* (mg/kg)	Cd* (mg/kg)
22	S3-B1-(10-12)	46.8	43.5
23	S3-B1-(13-15)	63.4	12.5
24	S3-B1-(17-19)	51.1	2.4
25	S3-B2-(10-12)	25.1	ND
26	S3-B2-(12-14)	24.0	ND
27	S3-B2-(12-14) FD	26.6	2.9
28	S3-B2-(17-19)	18.1	ND
29	S3-B3-(6-8)	24.1	ND
30	S3-B3-(10-12)	44.8	30.1
31	S3-B4-(10-12)	33.7	53.8
32	S3-B4-(12.5-14)	27.1	40.0
33	S3-B5-(10-12)	63.1	137
34	S3-B5-(14-16)	50.2	70.4
35	S3-B5-(17-19)	17.3	ND
36	S3-B6-(10-12)	28.6	ND
37	S3-B6-(16-18)	23.6	3.4
38	S3-B7-(10-12)	40.9	2.4
39	S3-B8-(10-12)	23.6	ND
40	S3-B8-(12-14)	14.4	ND
41	S3-B8-(6-8)	45.3	ND
42	S3-B9-(6-8)	20.4	5.5
43	S3-B9-(10-12)	21.3	7.5
44	S3-B10-(6-8)	19.6	ND
45	S3-B10-(10-12)	17.5	5.0
46	S3-B11-(6-8)	19.1	3.6
47	S3-B11-(10-12)	21.0	ND
48	S3-B12-(5-7)	21.8	ND
49	S3-B12-(9-11)	24.0	ND
50	S3-B12-(9-11)FD	18.1	ND
51	S3-B13-(10-12)	19.1	ND
52	S3-B14-(6-8)	21.8	ND
53	S3-B15-(6-8)	17.0	ND
54	S3-B16-(6-8)	17.2	2.1
55	S3-B16A-(9-11)	16.8	ND
56	S3-B17-(6-8)	15.8	ND
57	S3-B18-(6-8)	15.2	ND
58	S3-B19-(9-11)	20.2	ND
59	S3-B20-(10-12)	24.2	ND
60	S3-B21-(13.5-15)	16.6	ND
61	S3-B22-(10-12)	16.0	ND
62	S3-B23-(8-10)	14.5	ND
63	S3-B23-(13-15)	18.6	ND
64	S3-B23-(16-18)	11.7	2.4
65	S3-B24-(9.5-11)	17.2	4.0
Detection Limit		7.8	1.8

*Livetime : 200 sec (n = 1)