EVALUATION REPORT
With Appendix: Certification Statement

for

HAPSITE®
Portable Gas Chromatograph Mass Spectrometer

Manufactured by:

INFICON® Inc.

Two Technology Place, East Syracuse, New York 13057

Certificate Number 04-01-042

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CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY
DEPARTMENT OF TOXIC SUBSTANCES CONTROL

CALIFORNIA ENVIRONMENTAL
TECHNOLOGY CERTIFICATION PROGRAM

Cal/EPA
California Environmental Protection Agency

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ACRONYMS AND ABBREVIATIONS

APCL	Applied Physics & Chemistry Laboratory
BFB	bromofluorobenzene
BPFB	bromopentafluorobenzene
ºC	degrees centigrade
Cal/EPA	California Environmental Protection Agency
CCC	continuing calibration compound
CST	civilian support team
D	difference
DTSC	California Department of Toxic Substances Control
DHS	California Department of Health Services
DOE	United States Department of Energy
EHL	Environmental Health Laboratory
ELAP	Environmental Laboratory Accreditation Program
ETV	Environmental Technology Verification
ºF	degree Fahrenheit
FPA	Field Portable Analytical, Inc
FN	false negative
FP	false positive
GC	gas chromatography
GC/MS	gas chromatography mass spectrometer
GPS	Global position system
HML	Hazardous Materials Laboratory
MDL	method detection limit
MAFB	McClellan Air Force Base
mg/kg	milligram per kilogram
min	minute
mL	milliliter
mm	millimeter
MS	mass spectrum
MS	matrix spike
MSD	matrix spike duplicate
MTBE	methyl tert-butyl ether
NEG	non-evaporable getter
NIST	National Institute of Standards and Technology
ppmv	part per million volume
ppbv	part per billion volume
PCE	perchloroethene
PE	proficiency evaluation
PQL	practical quantitation limit
PT	performance testing
QA/QC	quality assurance/ quality control
R	correlation coefficient
RSD	relative standard deviation
RPD	Relative Percent Difference
sec	second
SIM	selective ion monitoring
SOP	standard operating procedure
SRS	Savannah River Site
TCE	trichloroethene
TRIS	1,3,5-trifluoromethylbenzene

USACE	US Army Corp of Engineering
U.S. EPA	United States Environmental Protection Agency
VOA	volatile organic analysis
VOC	volatile organic compound
µg/L	microgram per liter
µg/kg	microgram per kilogram

FOREWORD

Effective January 1, 1994, Section 25200.1.5 of the California Health and Safety Code authorizes the State of California to certify the performance of environmental technologies that offer an environmental benefit. The term "technology" in this certification program designates a system, which consists of equipment, material, operating knowledge and skills, and a quality control/quality assurance program on the part of both the technology vendor and the user. As part of this program, the State Department of Toxic Substances Control has evaluated the efficacy, efficiency, reliability, and safety of measurement technologies to protect public health and the environment. The primary goals are to evaluate: (1) the performance of field technologies to support the dynamic field activities for expediting the hazardous waste site process for public health protection, and (2) the alternative test methods to support the function of Environmental Laboratory Accreditation Program in the Department of Health Services to meet the California regulatory requirements. The Field-Portable Gas Chromatograph Mass Spectrometer is one of the innovations for on-site measurement of organic pollutants in environmental media. This report was prepared to show the performance of a portable gas chromatograph - mass spectrometer system HAPSITE® manufactured by INFICON®, Inc. for on-site measurement of volatile organic compounds in air water, soil and gas samples. The evaluation is based on a detailed review of information and data packages submitted by the technology proponent, field data generated by independent parties, and new data collected under the oversight of the DTSC whose findings the evaluation team considered reliable.

INTRODUCTION

In complying with the 1990 Clean Air Act amendments, the HAPSITE was originally designed to meet the requirements for source emission testing. Afterwards, the instrument was developed for on-site determination of volatile organic compounds in air, water and soil. In 1997 the U.S. EPA conducted a field demonstration of Field Portable GC/MS for the measurement of chlorinated volatile organic compounds in water. The INFICON HAPSITE was one of the technologies participating in this study conducted at the DOE Savannah River Site (SRS) in South Carolina and at McClellan Air Force Base (MAFB) in California. The reports obtained from these groundwater studies revealed a highly linear correlation between the HAPSITE data and the laboratory data[A4]. The HAPSITE had been used by the US Army Corp of Engineering (USACE) to monitor TCE contamination in groundwater[A5], followed by continuing investigations of the extent of groundwater contamination at Monterey Peninsula Airport. INFICON has been certified under I.S. EN ISO 9001 by National Standards Authority of Ireland. This certification remains valid to December 14, 2005[A11, A29]. To extend the scope of applications, INFICON is applying for the Cal/EPA Certification to verify the analytical capabilities of HAPSITE as a field-based analytical method as well as a laboratory instrument in determining VOCs in water, soil and soil gas[A3].

As is the case with many technologies, INFICON over time improves its products. An improved model of the HAPSITE system was released in the fall of 2001. Due to the short period of commercialization of the new HAPSITE model, the performance data for this evaluation were based on data generated by the original HAPSITE model. However, both the original and the improved HAPSITE® model are operated by the same principle. Field Portable Analytical, Inc (FPA), an independent contractor, has used the HAPSITE for many projects throughout California and many states in the United States. The data packages used for this evaluation were mostly generated by the FPA. The purpose of this evaluation is to verify the efficacy, reliability, and safety of the HAPSITE analytical system for the environmental applications in the field as well as in the laboratory.

SUMMARY OF SCIENTIFIC PRINCIPLE

This technology is based on the principle of quadrupole GC/MS for compound identification and quantitation. The sample components are separated by a GC column and passed into the mass spectrometer (MS) through a membrane interface. The interface between the GC and the MS is a 70% dimethyl silicone/30% polycarbonate membrane that provides the permeability for volatile organic compounds to the MS, but excludes inorganic constituents, such as nitrogen carrier gas, from the MS. As each compound emerges from the GC column, it passes through the selected membrane into the MS where the sample is fragmented by high-energy electron impact ionization. The mass fragments are then detected through quadruple filter. Compound identifications may be achieved by matching ion spectra in the National Institute of Standards and Technology (NIST) library.

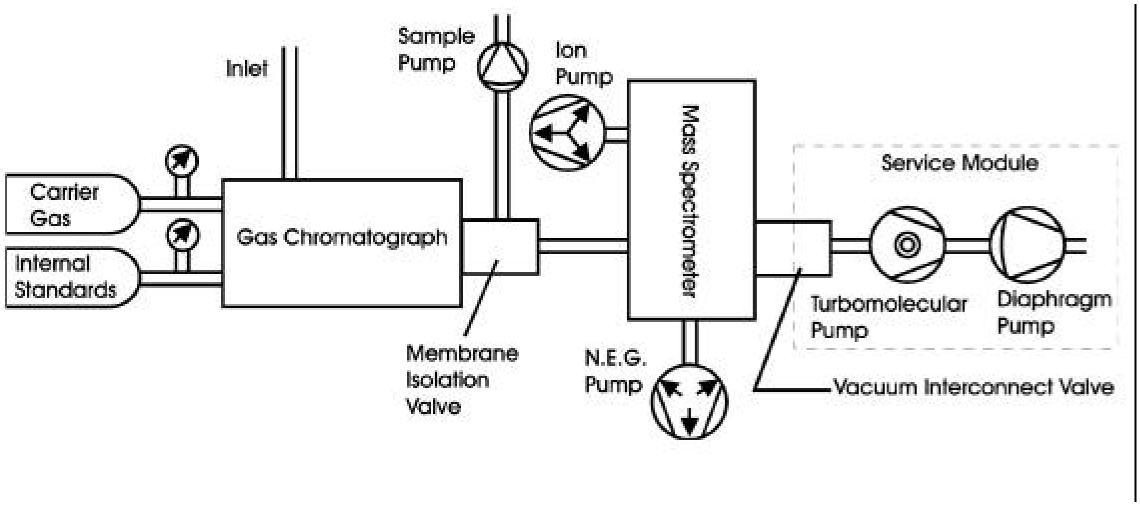
The HAPSITE is designed to analyze volatile organic compounds in air, so samples must be introduced in a gas phase. In conjunction with a headspace equilibrium sampling accessory the instrument has the capability to analyze water and soil samples. The headspace analysis involves heating water or VOC contaminated soil (in water) in a closed sample container to a known temperature. Heat forces the volatile compounds to partition between the water and the space above the sample. After allowing sufficient time for equilibrium, the headspace containing VOCs from the sample is introduced to the HAPSITE as a gas sample. The HAPSITE is capable of measuring volatile organic compounds with molecular weight typically 45 to 300 amu, boiling point approximately from -50ºC to +180ºC. The internal standard gas is used as mass calibrator for compound identification and quantitation[A6].

BRIEF DESCRIPTION OF THE TECHNOLOGY

1. System Overview

The HAPSITE System has four components: the HAPSITE instrument, a Service Module, a Hand Control Unit and Headspace Sampling System. Figure 1 is a diagram of the major subsystems of the instrument[A32].

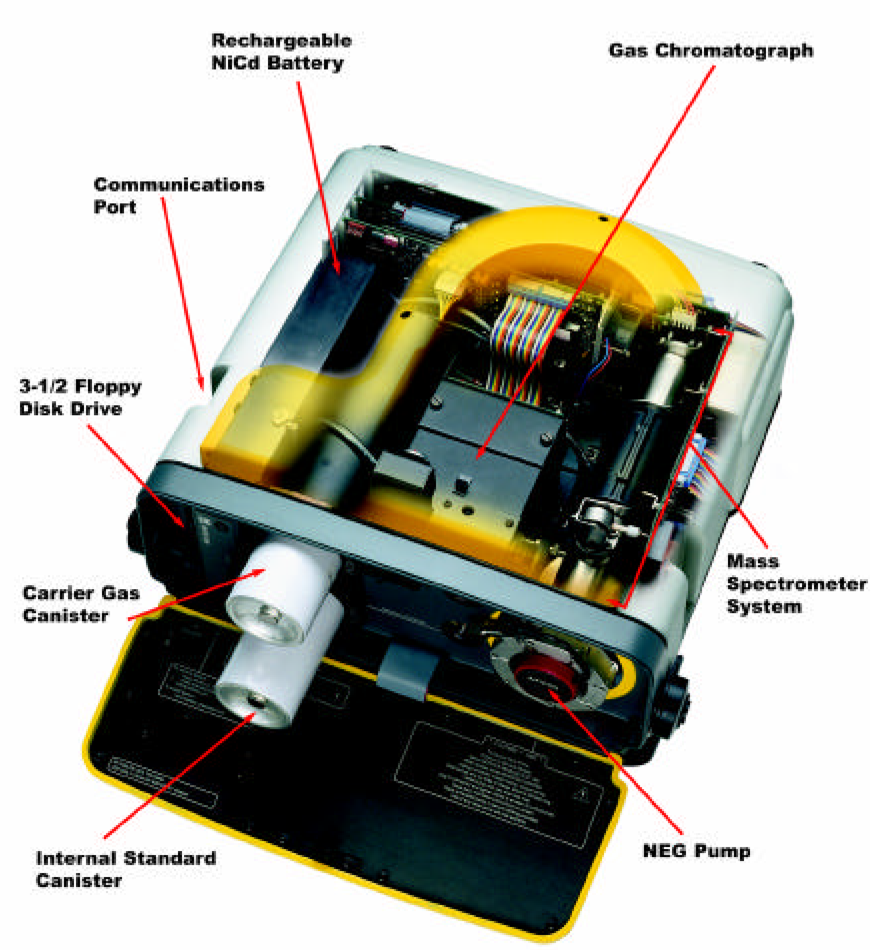
Figure 1 Major HAPSITE Components



HAPSITE

The HAPSITE is the major operating unit. It consists of seven parts: (1) gas chromatography, (2) mass spectrometer, (3) cylinders of carrier gas and internal standards gas, (4) high vacuum pumps, (5) the control electronic and software systems, (6) battery and charger, and (7) a key board and display. A system overview and major components of the HAPSITE system is presented in Figure 2[A32].

Figure 2 HAPSITE Portable GC/MS System



Service Module

The Service Module contains the turbo-molecular high vacuum pump and its auxiliary diaphragm vacuum pump, the mechanism for operating the interconnecting valve, a battery-charger and a power supply. The Service Module is required when changing the NEG pump or the ionizer.

Hand Control Unit/Inlet Line

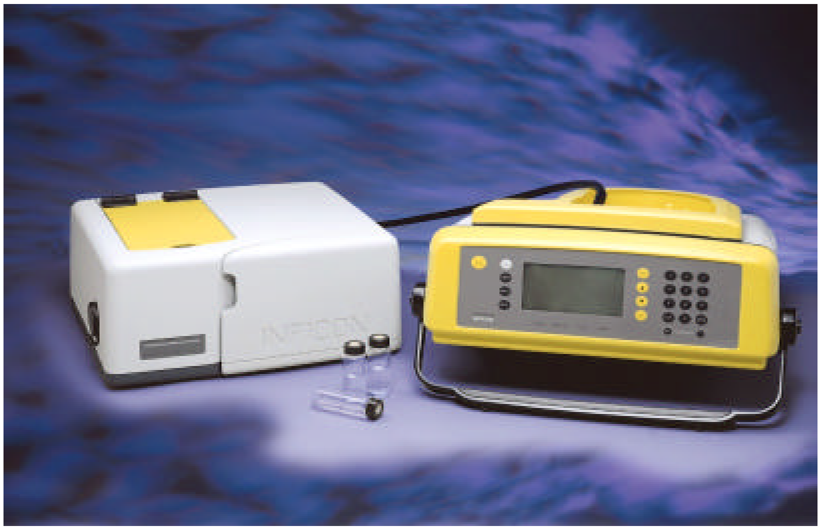
This unit feature, referred to as a “probe”, is a LCD display with a keypad which allows the operator to control the instrument. The hand control unit is a sampling point to the Analytical Module (Figure 3[A32]). This inlet line is connected to the HAPSITE and provides an inert heated conduit for conduction of a sample flow into the HAPSITE. The inlet line is temperature controlled, and usually maintained at 85° to 95°F.

Figure 3 HAPSITE Field – Portable Mode

Headspace Sampling System

The headspace sampling system is an accessory connected to the HAPSITE portable GC/MS which allows testing for volatile compounds in various solid and liquid matrices (Figure 4[A32]).

Figure 4 HAPSITE Field-Portable Headspace Sampling System



The oven accommodates four standard 40-ml vials. A sampling needle can be easily inserted into the vial for withdrawal of the headspace for the analysis. The sampling system operates on a rechargeable battery or from a 24-volt converter when external power is available.

The HAPSITE dimensions are 18? length, 17? width, and 7? height. The instrument can be operated in three modes:

Field-Portable Mode

The HAPSITE can be operated from a battery and utilizes a proprietary chemical getter pumping system to maintain the vacuum in the MS system. The pumping system provides a vacuum for 120 hours of actual sample analysis time. HAPSITE contains all the required accessories, such as low-pressure canisters for carrier gas (nitrogen) and internal standards, for the analysis of VOC in the field. The system is completely self-contained and portable, weighing only 35 pounds.

Transportable Mode

The HAPSITE GC/MS is mounted on service module that contains a turbo molecular pump and a roughing pump in a van. A DC converter is also available for non-portable operation. The system weighs 75 pounds in the transportable mode.

Stationary Mode

The HAPSITE GC/MS can be set up in a laboratory for operation as laboratory equipment using the carrier gas from a high-pressure cylinder. INFICON HASITE® is designed for outdoor environments as well as for laboratory analysis.

The HAPSITE uses Windows-based software which allows customized data analysis and methods development for compound identification and quantitation. The MS can be operated in a full scan mode, selective ion mode (SIM) or a direct injection to the MS mode (MS only). However, in most cases the unit is operated in full scan mode for compound identifications. With the built in sampling and analytical programs, the instrument automatically acquires and analyzes samples accordingly. Within minutes of sampling, results are displayed on the front panel. The instrument automatically saves analytical results plus chromatographic and spectral data on its hard drive along with time, date and GPS coordinates. More information can be found in the following website: http://www.HAPSITE.com

2. Operating Procedures

The detailed startup procedures to prepare the HAPSITE for the field operation are described in the User Guide, section 2[A7]. A non-evaporable getter (NEG) pump is required for the portable mode to maintain the vacuum in the MS system. If the instrument is being used in a battery-operated mode, a minimum of two fully-charged batteries must be on hand, because each battery supplies 2.5 to 3.5 hours of operation. In order to meet the U.S. EPA requirements for method 8260B, field operation involves three preparation steps outlined below.

2.1 Mass Spectrometer Tuning and Mass Calibration

The mass spectrometer must be tuned to the following criteria as specified in U.S. EPA Method 8260B for 4-Bromofluorobenzene (Table 1)

Table 1 4-Bromofluorobenzene Mass Intensity Criteria

Mass Fragment	Relative Abundance
50	15-40%
75	30-60%
95	100%, Base Peak
96	5-9%
173	<2% of mass 174
174	50-100%
175	5-9%
176	95-101% of mass 174
177	5-9%

The MS tune must be verified daily by a mass calibration check to meet the manufacturer's specification. The tune check is performed by introducing 1, 3, 5-trifluoromethylbenzenzene (TRIS) and bromopentafluorobenzene (BPFB) into the mass spectrometer via membrane interface. The acceptance criteria for BPFB mass relative abundance are listed in Table 2.

Table 2 Criteria of BPFB Mass Calibration Check

Mass Fragment	Relative Abundance
50	0.5-2.5%
55	2-8%
69	8-18%
93	15-25%
117	100% Base Peak
167	50-70%
213	10-30%
246	15-40%
263	5-15%
282	5-15%

2.2 Data Acquisition

2.2.1 Instrument Calibration

The initial instrument calibration contains a minimum of 5 concentration levels. The lower level must be no more than 5 times the reporting limit. The highest level should encompass the linear range of the instrument. The general acceptance criteria for initial calibration are 30% relative standard deviation (RSD) for all compounds. A solution containing targets of interests from an independent source should be analyzed to confirm the calibration. The acceptance criteria are 30%D compared to the initial calibration[A15, A16, A17]. After the instrument tuning and before sample analysis, the continuing calibration compounds (CCCs) must be analyzed. The CCCs are 1, 1-Dichloroethene, Chloroform, 1, 2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride. The general acceptance criteria for continuing calibration are ±25% difference for the CCCs. All other compounds must be within ±50% recovery from the initial calibration.

2.2.2 Sample Preparation and Analysis

The operating procedures for the analysis of water, soil, and soil gas are described in the Standard Operating Procedures[A15, A16, A17]. With slightly variations, the typical sample preparations for water, soil, and air analysis are as follows:

(1) Water

Twenty milliliters of water is placed in a 40-ml VOA vial sealed with PTFE coated septum screw cap. A solution containing internal standards and surrogates is added to the sample prior to equilibrium. The vials are then placed in a heated chamber, maintained at 60°C for 20 minutes prior to head space analysis.

(2) Soil

Ten grams of soil is weighed and placed into a 40-ml VOA screw cap vial. The soil is weighed on a wet basis. Twenty ml of water is withdrawn from the syringe and added to the VOA vial. The vial is then sealed with a PTFE coated septum and the sample is sonicated for 5 minutes. Prior to sonication, internal standards and surrogates are added to the sample. The sample vials are then placed in a heated chamber, maintained at 60°C for 20 minutes prior to head space analysis.

(3) Air, Soil Gas and Other Gas Analysis

The gas sample is collected in a 1.0 liter Tedlar bag. The Tedlar bag is attached to the GC/MS sampling probe using a short piece of Teflon tubing. When the start button on the sampling probe is pressed, the internal pump pulls the sample through the sample loop for a predetermined amount of time, usually 30 to 60 seconds. In the meanwhile, internal standards are drawn into the instrument from the onboard internal standard cylinder at a 1:10 ratio to the sample. After the sampling is completed, the valve is automatically switched to the inject position which sweeps the sample and internal standards onto the pre-column. After 100 seconds any heavier compounds, such as diesel, are back-flushed out of the system. To ensure that representative samples are analyzed, the sample line and loop are completely flushed with sample for 30 to 60 seconds before sample injection onto the GC column. The GC column is a 30m long Supelco SPB 1 (100% methyl silicone phase, 0.32 mm i.d. coated with 1.0 µm film).

As an alternative to the Tedlar bag method of sampling, air and other gas samples may also be drawn directly from the source, through the sampling probe and into the HAPSITE without using a Tedlar bag as a collection device. This is a major advantage of the HAPSITE in that direct sampling will better preserve the integrity of the sample and allow for faster turn around time.

For the analysis of liquid or solid samples a 40-mL VOA vial is capped and placed in one of the cells in the headspace accessory then heated to 60°C for 20 min. This allows the VOCs in the sample to equilibrate with the headspace above the sample in the VOA vial. At the end of heating period, the pump draws the headspace from VOA vial for 10 seconds (sec) into the HAPSITE for analysis. The content of the sample loop is then injected onto a GC column.

3. Operating Conditions

The instrument is designed for use outdoors in ambient temperature from 32 to 113ºF (0 to 45ºC). If HAPSITE is being used in a hazardous location, it should always be removed to the decontamination site before the front panel is opened. The HAPSITE can be used over the full range of relative humidity (from 0 to 100%) and in the mist or light rain[A8]. With the adequate supplies of batteries to provide power source and NEG pump the HAPSITE can be operated in most environmental conditions in the field.

PERFORMANCE CLAIMS

INFICON claims the HAPSITE Portable GC/MS has the analytical capability to meet the following performance criteria:

(1) The method practical quantitation limits will meet criteria as presented in Tables 3, 4, and 5 for air (including soil gas and other gas phase), water, and soil analyses.
(2) With a 5-point calibration, daily calibrations and associated quality control procedures, the HAPSITE has the capability to generate data comparable to laboratory data.
(3) The HAPSITE identifies and quantifies volatile organic compounds in air or gases at the site of investigation.
(4) With the associated headspace accessory, the HAPSITE can analyze liquid and solid samples comparable to the U.S. EPA SW-846 Method 8260B.
(5) The instrument can be used as laboratory instrument.

Table 3 HAPSITE Practical Quantitation Limit in Water

Compound	CAS Number	PQL mg/L	Quan Mass
Benzene	71-43-2	5	78
Bromobenzene	108-86-1	10	77
Bromochloromethane	74-97-5	15	49
Bromodichloromethane	75-27-4	5	83
Bromoform	75-25-2	15	173
Bromomethane	74-83-9	5	94
Carbon Tetrachloride	56-23-5	5	117
Chlorobenzene	108-90-7	5	112
Chloroethane	75-00-3	10	64
Chloroform	67-66-3	5	83
Chloromethane	74-87-3	5	50
Dibromochloromethane	124-48-1	5	129
1,2-Dibromomethane	106-93-4	5	107
Dibromomethane	95-50-1	5	174
Dichlorofluoromethane	75-71-8	10	85
1,1-Dichloroethane	75-35-3	5	63
1,2-Dichloroethane	107-06-2	5	62
1,1-Dichloroethene	75-35-4	5	61
cis-1,2-Dichloroethane	156-60-5	5	61
trans-1,2-Dichloroethene	156-60-5	5	61
1,2-Dichloropropane	78-87-5	10	63
2,2-Dichloropropane	594-20-7	10	77
1,1-Dichloropropane	563-58-6	10	75
Cis-1,3-Dichlorpropene	10061-01-5	10	75
Trans-1,3-Dichloropropene	10061-02-6	10	75
Ethyl Benzene	100-41-4	5	91
Isopropyl benzene	98-82-8	10	105
Methylene chloride	75-09-2	5	49
Styrene	100-42-5	5	104
1,1,1,2-Tetrachloroethane	630-20-6	20	131
1,1,2,2-tetrachloroethane	79-34-5	20	83
Tetrachloroethene	127-18-4	5	166
Toluene	108-88-3	5	91
1,1,1-Trichloroethane	71-55-6	5	97
1,1,2-Trichloroethane	79-00-5	5	97
Trichloroethene	79-01-6	5	95
1,2,3-Trichloropropane	96-18-4	15	75
Vinyl Chloride	75-01-4	5	62
o-Xylene	95-47-6	5	91
m-Xylene	108-38-3	5	91
p-Xylene	106-42-3	5	91

Table 4 HAPSITE Practical Quantitation Limit in Soil

Compound	CAS Number	PQL mg/kg	Quan Mass
Benzene	71-43-2	10	78
Bromodichloromethane	75-27-4	10	83
Bromoform	75-25-2	30	173
Bromomethane	74-83-9	10	94
Carbon Tetrachloride	56-23-5	10	117
Chlorobenzene	108-90-7	10	112
Chloroethane	75-00-3	20	64
Chloroform	67-66-3	10	83
Chloromethane	74-87-3	10	50
Dibromochloromethane	124-48-1	10	129
1,2-Dibromomethane	106-93-4	10	107
Dibromomethane	95-50-1	10	174
Dichlorodifluoromethane	75-71-8	20	85
1,1-Dichloroethane	75-35-3	10	63
1,2-Dichloroethane	107-06-2	10	62
1,1-Dichloroethene	75-35-4	10	61
Cis-1,2-Dichloroethane	156-60-5	10	61
Trans-1,2-Dichloroethene	156-60-5	10	61
1,2-Dichloropropane	78-87-5	20	63
Ethyl benzene	100-41-4	10	91
Methylene chloride	75-09-2	10	49
Styrene	100-42-5	10	104
1,1,1,2-Tetrachloroethane	630-20-6	40	131
1,1,2,2-tetrachloroethane	79-34-5	40	83
Tetrachloroethene	127-18-4	10	166
Toluene	108-88-3	10	91
1,1,1-Trichloroethane	71-55-6	10	97
1,1,2-Trichloroethane	79-00-5	10	97
Trichloroethene	79-01-6	10	130
Trichlorofluoromethane	75-69-4	10	101
Vinyl chloride	75-01-4	10	62
o-Xylene	95-47-6	10	91
m-Xylene	108-38-3	10	91
p-Xylene	106-42-3	10	91
Acetone	67-64-1	20	58
2-Butanone	78-93-3	20	57
2-Hexanone	591-78-6	20	58
Methyl *t*-Butyl Ether	1634	20	73
1,2-Dichlorobenzene	95-50-1	20	146
1,3-Dichlorobenzene	541-73-1	20	146
1,4-Dichlorobenzene	106-46-7	20	146

Table 5 HAPSITE Practical Quantitation Limit in Soil Gas

Compound	CAS Number	PQL ppmv	Quan Mass
Benzene	71-43-2	0.2	78
Carbon tetrachloride	56-23-5	0.2	117
Chlorobenzene	108-90-7	0.2	112
Chloroform	67-66-3	0.2	83
1,2-Dibromomethane	106-93-4	0.2	107
Dichlorodifluoromethane (Fr12)	75-71-8	0.2	85
1,1-Dichloroethane	75-35-3	0.2	63
1,2-Dichloroethane	107-06-2	0.2	62
1,1-Dichloroethene	75-35-4	0.2	61
Cis-1,2-Dichloroethene	156-60-5	0.2	61
Trans-1,2-Dichloroethene	156-60-5	0.2	61
Ethyl benzene	100-41-4	0.2	91
Methylene chloride	75-09-2	0.2	49
Styrene	100-42-5	0.2	104
1,1,2,2-tetrachloroethane	79-34-5	0.2	83
Tetrachloroethene	127-18-4	0.2	166
Toluene	108-88-3	0.2	91
1,1,1-Trichloroethane	71-55-6	0.2	97
1,1,2-Trichloroethane	79-00-5	0.2	97
Trichloroethene	79-01-6	0.2	130
Trichlorofluoromethane	75-69-4	0.2	101
Vinyl chloride	75-01-4	0.2	62
o-Xylene	95-47-6	0.2	91
m-Xylene	108-38-3	0.2	91
p-Xylene	106-42-3	0.2	91
Acetone	67-64-1	0.2	58
Freon 113	765-13-1	0.2	135
n-Hexane	110-54-3	0.2	57
1,3,5-Trimethylbenzene	108-67-8	0.2	105
1,2,4- Trimethylbenzene	95-63-6	0.5	105
1,3-Butadiene	106-99-0	0.5	54
Cyclohexane	110-82-7	0.5	84
2,2,4-Trimethyulpentane	540-84-1	0.5	57
Methyl *t*-Butyl Ether	1634-04-4	0.2	73

TECHNICAL EVALUATION: GENERAL CONSIDERATIONS

An effective analytical method must meet criteria for method sensitivity, selectivity, precision and accuracy. For a field instrument, there are several additional factors to be evaluated: the portability and ruggedness of an instrument under the environmental conditions, the complexity of operating procedures, the utility requirements, the waste generation and the safety issues. The

HAPSITE field measurement system was evaluated on the following aspects:

Method Sensitivity: Sensitivity is the ability to detect the substance of interest at a specified concentration level and within the dynamic range of measurement. Method sensitivity is usually expressed in detection limits, quantitation limits and the frequency of false positive and false negative results relative to a specified target concentration.

Precision and Accuracy: Precision is the agreement among replicate measures of a sample under prescribed analytical conditions. Accuracy is the closeness of measurement to a true value. A good analytical method will have a high degree of reproducibility and good recovery data from various sample matrices under the described analytical conditions, such as consistent recoveries from spiked real-world samples.

Operating Conditions: Experimental and environmental conditions under which the product claims have been validated.

Data Comparability: The capability of a method to generate data comparable or highly correlated to a reference method. Depending on the data available and the sampling method, the data comparability can be evaluated by the relative percent of difference (RPD) of pair data or the correlation coefficient of two data sets. For the inter-method comparison or the results obtained from collocated samples instead of homogenous split sample, the difference of a factor of two can be used as criteria for method comparison.

Quality Control and Quality Assurance: Documentations of the quality control (QC) measurements and sample handling to ensure the integrity of the data and the data of known quality commensurate with its intended use.

Quality Assurance in Manufacture: The ruggedness of an instrument under various environmental conditions and the ability to produce consistent results with good precision and accuracy.

TECHNICAL EVALUATION: SPECIFIC TO THIS PRODUCT

1. Performance Specification

The INFICON HAPSITE was designed to analyze gas samples in the parts per billion (ppbv) to parts per million (ppmv) range. The instrument operates on pre-programmed sets of instructions known as methods. The instrument performance specifications are presented in Table 6.

Table 6 HAPSITE Performance Specification

Mass range	1-300 AMU
Scan rate	1000 amu/sec at 10 points per amu
Ionization mode	70 eV electron impact
Vacuum system	15 l/sec NEG pump; 0.2 l/sec sputter-ion pump
Operating temperature	0°C to 45°C (32 to 113°F)
Internal power consumption	30 watts average, 24 volts DC
Carrier gas	Nitrogen
Temperature range	Inlet & GC 15°C above ambient to 80°C, isothermal
Max. sample moisture content	8% by weight
SIM channels	10 sets / 20 ions per set
External communications	Parallel port

2. Performance Data

2.1 Sensitivity

2.1.1 Method Detection Limit and Practical Quantitation Limit

INFICON determined the method detection limit by performing seven replicate analyses of the low standard in sample matrices. The method detection limit (MDL) is defined as standard deviation of these replicates multiplied by the student's t at the 99% confidence level of 3.14. INFICON defines the practical quantitation limit (PQL) as the lower bound of the calibration range and represents a peak-to-peak signal to noise ratio of 10:1. The PQLs for analytes in water, soil, and air or soil gas are presented in Table 3, 4, and 5, respectively. The HAPSITE can be operated in full scan mode or selective ion monitoring (SIM). Based on the information provided by the FPA, the sensitivity increases 2 to 10 times by using SIM.

To study the MDL, FPA spiked VOC standards in various environmental media and analyzed the sample according to the procedures described in section 2.3. Results for the MDL studies are presented as follows:

(1) Water

The MDL for water analysis were demonstrated in two studies. In one study, the spiked sample contained 12 commonly found chlorinated and aromatic hydrocarbons (Appendix A). The spiked concentration was 5 µg/L. The average recoveries for each compounds ranged from 3.3 to 5.5 µg/L, with %RSD from 3.3 to 10.3%. In another study, the spiked sample contains 36 VOC standards. The average recoveries of these standards were between 3 to 5 µg/L with a %RDS from 8 to 35%, except bromoform, which was detected at 2.1 µ/L with an RDS at 50%.

(2) Soil

Since soil samples were prepared by sonication in water and analyzed by headspace, the calibration curve established for water analysis is also applicable for soil analysis. No MDL data was provided for the soil analysis.

(3) Air and Soil Gas

For the air and soil gas analysis, the VOC standards were introduced into a 1-liter Tedlar bag at 0.2 ppmv for each compound. A gas standard at a specified concentration was prepared by injecting 1 µl of the dilute standard into a Teflon gas-sampling bag filled with 5 liters of nitrogen. Three sets of MDL studies were submitted for evaluation in which two different instruments (Speedy and TAZ) were used to demonstrate the consistent responses of different instruments (Appendix A). The average recoveries for each compound ranged from 0.15 to 0.2 ppmv with all the RSD less than 20% (n=7). A total of 38 VOCs were studied. A MDL study recently carried out by the IT Corporation, an independent consultant company, indicated that on the basis of 3 standards, 0.2 ppmv is the MDL for the most of the VOCs included in the study (Appendix A).

2.1.2 Precision and Accuracy

(1) Precision

The method precision is assessed by the relative percent difference (RPD) for duplicate analyses or the percent relative standard deviation (%RSD) for multiple analyses. Under the normal environmental conditions, the RSD of HAPSITE analysis is expected to be =20% over the working range of the instrument. By adjusting the sample amount, the working range of the instrument can be adjusted from the lower limit upward to allow measurement within the linear dynamic range of the instrument. For the chemicals listed in Tables 3, 4, and 5, the instrument dynamic range is over 10^4.

(2) Accuracy

The instrument accuracy is assessed by the recoveries of PE samples and spiked matrices. In conjunction with a headspace system, the recoveries for water analysis at the 95% confidence are expected to be at ±25% of the spiked values or better over the calibration range level[A4].

2.2 Environmental Sample Analysis and Validation Studies

2.2.1 Water Analysis

Previously, INFICON HAPSITE had participated in two U.S. EPA technology evaluation programs. These included the 1997 Environmental Technology Verification (ETV) program and the 1999 Innovation in Site Characterization for real-time VOC analysis using a Field Portable GC/MS. The data obtained from these two programs were accepted by this program for the evaluation of water analysis. To ensure HAPSITE maintained the performance standard over time, FPA acquired a new set of groundwater data, under the oversight of the DTSC, for this technology evaluation. The results of these demonstrations are summarized in the following:

(1) U.S. EPA Environmental Technology Demonstration for Groundwater Analysis

This field demonstration was held in September of 1997 at two DOE sites: the Savannah River Site (SRS) in South Carolina and the McClellan Air Force Base (MAFB) in California[4A]. The primary objectives of this demonstration were to test and verify the performance of field-portable technologies for characterization and monitoring of chlorinated volatile organic compounds in groundwater. Comprehensive studies had been designed in this program to evaluate the qualitative and quantitative factors of a technology for water analysis. The details of this demonstration plan are available in the U.S. EPA ETV website (http://www.epa.gov/etv) for "Well-Head Monitoring-VOCs"[B7], or directly at www.epa.gov/etv/pdfs/vrvs/01_vr_inf.pdf. The results of HAPSITE analysis in this technology demonstration are summarized in the following:

- *Analysis of PE Sample*

A comprehensive QC study was conducted by the U.S. EPA ETV program. One hundred and sixty six proficiency evaluation (PE) samples with known VOC concentrations and compositions were supplemented to each set of site samples for the analysis. The HAPSITE completed 165 of 166 PE samples. These PE samples were designed to evaluate the method precision and accuracy at different concentration levels and the rates of false positive (FP) and false negative (FN) results. Different PE samples were incorporated to the SRS and the MAFB sites (Table 7). In this study, ten replicate PE samples at 10µg/L (typical regulatory limit) were provided for the analysis to determine the FP and FN at this target concentration. No FP was found in HAPSITE analysis. As shown in Table 7, some FN results were found on each site, bromoform having relatively lower response in which 9 of 10 samples were not identified at this target concentration.

Table 7 Results of HAPSITE Analysis on Very Low-Level Water PE Sample

SRS PE (10 µg/L)		**MAFB PE (10 µg/L)**	
Compound	**False Negative**	**Compound**	**False Negative**
1,1-Dichloroethene	0 of 10	*trans*-1,2-Dichloroethene	0 of 10
Dichloromethane	0 of 10	1,2-Dichloroethane	0 of 10
1,1-dichloroethane	0 of 10	1,1,1-Trichloroethane	0 of 10
Chloroform	0 of 10	Benzene	0 of 10
Carbon Tetrachloride	1 of 10	Bromodichloromethane	0 of 10
1,2-dichloropropane	0 of 10	*cis*-1,1-Dichloropropene	0 of 10
Trichloroethene	0 of 10	*Trans*-1,3-Dichloropropene	0 of 10
1,1,2-Trichloroethane	4 of 10	Toluene	0 of 10
Dichlorobromomethane	2 of 10	Ethyl benzene	0 of 10
Tetrachloroethene	4 of 10	Bromoform	9 of 10
Chlorobenzene	1 of 10	1,1,2,2-tetrachlorothane	3 of 10
2-chloroethyl vinyl ether	No calibration		
Trichlorofluoromethane	No calibration		
1,2-Dichlorobenzene	No calibration		

Four replicate PE samples at low, mid, and high levels were analyzed to assess the method's precision and accuracy at various concentration levels. Six target analytes, which are regulated under the Safe Drinking Water Act, spiked to the low level PE samples were also analyzed. The same numbers of PE samples were distributed to the laboratory for analysis to compare the performance with the reference method, U.S. EPA SW-846 Method 8260B[5B]. The precision and accuracy of HAPSITE analysis on six target compounds are shown in Table 8.

The RSD of HAPSITE analysis is expected to be =20% and the recovery to be within the range of 75 to 125 percent as specified by the INFICON. Based on these performance criteria, the precision and recovery data outside the specified range are flagged in "+". In general, the RSDs of six target compounds at all concentrations met the criteria of =20% (Table 8), with the exceptions of 1,1,2-Trichloroethane at the high concentration levels and 1, 2-Dichloropropane and PCE at low levels but all within 30%.

Table 8 Precision of HAPSITE Analysis for PE Samples at SRS and MAFB

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

Data presented was obtained from Cal/EPA Environmental Technology Verification Report, Table 5-4. The composition and concentrations of PE sample were given in Appendix B (Table 3-5 and Table 3-6). The RSD greater than the INFICON specified level, 20%, are flagged in"+".

Table 9 Recoveries of PE Target Compounds at SRS and MAFB

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

Data presented was obtained from Cal/EPA Environmental Technology Verification Report Table 5-6. The composition and concentrations of PE sample were given in Appendix B (Table 3-5 and Table 3-6)

The HAPSITE accuracy was determined by comparing the average value of four HAPSITE replicate analyses with the known concentrations in the PE sample. All the recovery data are within the acceptable range of 75 to 125 percent (Table 9). These results demonstrated the precision and accuracy of the HAPSITE analysis are comparable to that of the U.S. EPA Method

8260B.

- *Groundwater Analysis*

To evaluate the data comparability, a total of 33 ground water samples collected from 10 monitoring wells were analyzed at each demonstration site. Replicate site samples with concentrations ranging from very low to very high levels across each site were collected for the analysis. Replicate sample sets were composed of either 3 or 4 samples from each well. Average laboratory results from each replicate set were used as the reference values for the comparison with the HAPSITE data. A comparison of laboratory and the HAPSITE analyses are presented in Table 10 (SRS) and Table 11 (MAFB).

Table 10 Results of HAPSITE and Laboratory Analysis for SRS Groundwater

Sample Description	Compound	Replicate	Lab. Avg. (µg/L)	Lab RSD (%)	HAPSITE Avg. (µg/L)	HAPSITE RSD (%)	Recovery (%)
Very low 1	TCE	3	9.0	11	8.8	43^{+}	98
	PCE		3.5	14	2.5	89	71
Very low 2	TCE	3	2.4	34	1.9	32	79
Low 1	TCE	3	11	5	13	11	118
	PCE		27	6	28	19	104
Low 2	TCE	4	27	7	27	12	100
	PCE		22	9	19	17	86
	Chloroform		1.3	0	NR	NR	NA
	Carbon tetrachloride		1.0	15	NR	NR	NA
Mid 1	TCE	4	150	9	126	7	84
	PCE		87	12	68	12	78
Mid 2	TCE	3	35	7	29	19	83
	PCE		240	4	186	15	78
	1,1-Dichloroethene		12	8	7.3	19	61^{+}
High 1	TCE	3	747	1	726	8	97
	PCE		33	2	37	11	112
High 2	TCE	4	1875	12	1703	8	91
	PCE		520	8	454	23^{+}	87
	1,1-Dichloroethene		32	8	26	42^{+}	81
Very high 1	TCE	3	1367	8	1460	6	107
	PCE		800	6	898	12	112
Very high 2	TCE	3	4933	6	4783	3	97
	PCE		3668	6	3197	12	87
Range				0 – 34		3 – 89	61- 118
Median				8		12	87
95th Percentile				15		43	

NR = not reported NA= not applicable

Data presented is obtained from U.S. EPA Environmental Technology Verification Report [A4]. The RSD greater than 20% and the recovery outside the range of 75 to 125% are flagged in "+". The percent recovery is calculated from the average value of HAPSITE analyses relative to the average of the Laboratory analyses[1].

Table 11 Results of HAPSITE and Laboratory Analysis for MAFB Groundwater

Sample Description	Compound	Replicate	Lab. Avg. (µg/L)	Lab RSD (%)	HAPSITE Avg. (µg/L)	HAPSITE RSD (%)	Recovery (%)
Very low 1	TCE	3	4.6	5	12.8	93	278
	1,1-dichloroethene		7.7	9	6.3	7	82
Very low 2	TCE	3	13	0	15	15	115
	PCE		2.0	6	NR	NR	NA
	Chloroform		9.0	1	9.0	12	100
	1,1-Dichloroethene		3.8	3	2.9	20	76
	Carbon tetrachloride		137	4	140	22^{+}	102
Low 1	1,1-Dichloroethene	4	2.5	7	NR	NR	NA
	1,1-Dichloroethane		15	0	13	15	87
	Carbon tetrachloride		7.5	2	6.0	30	80
	Chloroform		4.8	2	4.5	4	94
	TCE		16	4	17	27^{+}	106
Low 2	Freon 11	3	20	6	NR	NR	NA
	1,1-Dichloroethene		1.5	12	NR	NR	NA
	1,1-Dichloroethane		5.1	4	4.0	23^{+}	78
	cis-1,2-Dichloroethene		1.5	4	1.9	34	127
	Carbon tetrachloride		1.4	4	NR	NR	NA
	TCE		22	5	25.1	11	114
Mid 1	1,1-Dichloroethene	4	180	12	189	23^{+}	105
	1,1-Dichloroethane		3.0	9	NR	NR	NA
	cis-1,2-Dichloroethene		3.3	13	4.0	12	121
	1,1,1-Trichloroethane		6.8	12	5.3	34^{+}	78
	TCE		114	11	111	12	97
	PCE		1.2	14	NR	NR	NA
Mid 2	*cis*-1,2-Dichloroethene	3	15	4	24	8	160^{+}
	Chloroform		3.5	5	3.1	16	86
	TCE		280	4	264	3	94
High 1	*cis*-1,2-Dichloroethene	3	38	4	56	11	147^{+}
	Chloroform		6.9	21	6.4	5	93
	TCE		238	2	240	11	101
High 2	*tans*-1,2-Dichloroetnene	4	7.7	4	7.1	10	92
	cis-1,2-Dichloroethene		66	5	97	12	147^{+}
	Chloroform		42	5	45	5	107
	1,2-Dibromochloropropane		6.1	6	4.7	19	77
	TCE		380	5	398	9	105
Very high 1	1,1-Dichloroethene	3	690	3	1032	33^{+}	150^{+}
	cis-1,2-Dichloroethene		237	7	426	9	180^{+}
	Chloroform		397	5	418	8	105
	Benzene		283	5	265	12	94
	TCE		10,667	5	11,714	13	110
	Carbon tetrachloride		350	5	565	44	161^{+}
Very high 2	*cis*-1,2-Dichloroethene	3	207	10	299	16	144^{+}
	Chloroform		63	6	67	3	103
	Carbon tetrachloride		51	5	60	23^{+}	117
	TCE		6167	8	6821	15	111
Range				0 – 21		3 - 93	76 – 180
Median				5		13	105
95th Percentile				13		36	

NR = not reported NA= not applicable

Data presented is from in U.S. EPA Environmental Technology Verification Report. The RSD greater than 20% and the recoveries outside the range of 75 to 125% are flagged in"+". The percent recovery is calculated from the average value of the HAPSITE analyses relative to the average of the laboratory analyses. Concentrations less than HAPSITE PQL (5 µg/L) were not included for the evaluation.

- *Method Comparison of Ground Water Analysis*

For the method comparison, only those compounds having concentrations greater than the PQL (mostly 5 µg/L) were evaluated. The method precision and recoveries of two analytical systems are assessed by the range, the median, and the 95th percentile distribution of RSDs of replicate analyses by each method.

At the SRS, the major contaminates in groundwater were TCE and PCE. Depending on the analyte and concentration, the HAPSITE values were 61 to 118% of the laboratory values. At the MAFB site, the contaminants were TCE, PCE, 1,1-dichloroethene, 1,2-dichloroethene, Chloroform, Carbon Tetrachloride, 1,1-dichloroethane, 1,1,1-trichloroethane, and 1,2-dibromochlorpropane (Table 11). The values obtained from HAPSITE analysis were 78 to 180 % of the laboratory values. The RSDs of these groundwater analyses were mostly =20% as specified by the INFICON, with few exceptions flagged in "+".

Due to the wide concentration range in these samples, the data were divided into two groups at the cutoff value of 100 µg/L. The calculated correlation coefficient (r^2) between the HAPSITE data and the U.S. EPA Method 8260B data were all greater than 0.97 for both low and high concentration samples collected from both sites (Table 12).

Table 12 Correlation Coefficient of HAPSITE and Laboratory Water Data

Site	**Correlation Coefficient (r^2)**	
	Low Concentration =100 µg/L	High Concentration >100 µg/L
SRS	0.983	0.996
MAFB	0.978	1.000

(2) On-Site VOC Analysis at the Monterey Peninsula Airport[A5]

In this project, the HAPSITE GC/MS was loaded in a van was used as an "on-site laboratory". The purpose was installing a monitoring well network to detect TCE contamination at the Monterey Peninsula Airport (MPA). Ground water samples were collected from open borings using bailers and transferred to 40-mL VOA vials. Within minutes after sample collection, the VOA vials were handed to the HAPSITE operator, who poured the VOA vial's contents into a 50 mL gas tight syringe and introduced exactly 20 mL of water into a headspace vial and sealed the vial. Because the sample was analyzed immediately, no preservative was added to the sample. Fourteen groundwater samples collected from borehole locations were quantitatively analyzed for VOCs by HAPSITE GC/MS. A set of QC samples which included blanks, duplicates, and MS/MSD were concurrently tested with all the site samples.

The method accuracy is assessed by the recoveries of surrogate compounds and matrix spike added to the sample. The recoveries of surrogate compounds were ranged from 78 to 127%

(Table 13) and matrix spikes from 86 to 94% (Table 14). These recovery data fall within the acceptance criteria of 75 to 125%, with the exception of one duplicate sample at 127%. The method precision, as determined by the RPDs of duplicate analyses (6 to 17%) and MS/MSD (0 or 1%), were all within the specified range of =25% (Table 14). These QC data established the data quality of HAPSITE analysis to guide plume delineation and monitoring well placement.

Table 13 Percent Recoveries of Surrogate

Boring	DBF[1]	Toluene–D8	BFB[2]	Run Date
MPA-B7-GW	109	92.5	120	6/18/99
MPA-B7-GW Duplicate	114	96.6	127	6/18/99
MPA-B7A-GW	117	91.8	103	6/18/99
MPA-B8-GW	91.3	101	93.2	6/8/99
MPA-B9-GW	111	91.8	83.0	6/14/99
MPA-B10-GW	78.7	104	105	6/8/99
MPA-B10-GW Duplicate	82.2	95.3	81.3	6/8/99
MPA-B11-GW	86.0	93.5	80.9	6/10/99
MPA-B11-GW Duplicate	87.0	99.4	89.7	6/10/99
MPA-B12-GW	94.1	99.0	84.6	6/9/99
MPA-B12-GW Duplicate	85.2	94.1	97.7	6/9/99
MPA-B13-GW	120	95.5	112	6/15/99
MPA-B15-GW	111	97.7	112	6/16/99
MPA-B18-GW	116	94.2	118	6/16/99

[1] DBF = dibromofluoromethane
[2] BFB = bromofluorobenzene

Table 14 Results of Duplicate Analysis and Matrix Spike

Duplicate Analysis

Boring (ground water)	Cis-1,2-Dichloroethene (µg/L)	RPD (%)	Trichloroethene (µg/L)	RPD (%)
MPA-B7-GW	100	10	160	6
MPA-B7-GW Dup	110		170	
MPA-B10-GW	<5	-	25	17
MPA-B10-GW Dup	<5		21	
MPA-B11-GW	<5	-	490	10
MPA-B11-GW Dup	6.8		540	
MPA-B12-GW	<5	-	52	6
MPA-B12-GW Dup	<5		55	

Matrix Spike Recovery and Precision

Matrix Spike	Trichloroethene (µg/L)	RPD (%)	Benzene (µg/L)	RPD (%)	Toluene (µg/L)	RPD (%)
MPA-B12-MS	162 (88%)	2	116 (93%)	1	108 (86%)	0
MPA-B12-MS Dup	164 (90%)		117 (94%)		108 (86%)	

- *Data Comparability*

The USACE used the VOC results on a real-time basis to determine the installation of a monitoring well (MW). After monitoring wells were installed and fully developed in selected borings, ground water samples were collected from the wells for VOC analysis using SW-846 Method 8260B. The laboratory applied the same acceptance criteria as the field method for the QC program. The paired data obtained for the boring ground water and the monitoring well are presented in Table 15.

Table 15 Comparison of Boring and Monitoring Well Data

Boring/Monitoring Well Location	***cis*-1,2-Dichloroethene (μg/L)**		**Trichloroethene (μg/L)**	
	Boring (HAPSITE)	**Well (Laboratory)**	**Boring (HAPSITE)**	**Well Laboratory**
B7/MW11	55	18	330	350
B10/MW10	<5	0.8 (estimate)	25	48
B12/MW9	<5	1	52	58
B13/MW14	<5	1.8	17	110
B17/MW16	7.1	6.9	170	220
B18/MW12	32	110	890	1300

Since these paired data were obtained from the monitoring well and the paired boring, instead of split samples, there are some variations in results (Table 15). These variations can be contributed by the physical disturbance in drilling activities, spatial and temporal variation in sampling as well as sampling techniques in obtaining boring ground water and well water. However, the results obtained from both the field and laboratory analyses were consistent in identifying two major contaminants, TCE and cis-1,2-Dichloroethene and 75% of the data were within a factor of two for decision making.

(3) Alameda Point, Operable Unit 5 [A18, A19]

To ensure that INFICON maintains the previous performance as claimed over time, HML in coordination with the US Department of the Navy carried out a study to verify the performance of HAPSITE. The testing site was the Naval Air Station (NAS) in Alameda CA, referred to as Site 25 or Operable Unit 5 in the test plan[A28]. The U.S. EPA, the DTSC and the State Water Resources Board in California were the responsible parties to oversee this site remediation project. The Navy contracted IT Corporation (now Shaw Environmental and Infrastructure) to conduct the environmental sampling and analysis for this investigation. Eleven groundwater samples of the existing monitoring wells were collected and split for VOC analysis by the HAPSITE in the field and the GC/MS in a fixed laboratory.

- *Test Plan for Method Comparison*

The analytical data obtained from previous investigations indicated that petroleum-related constituents, BTEX, low MW PAHs and possible MTBE are the most commonly found chemicals in groundwater. IT conducted groundwater sampling on May 31, 2001. The sample at each monitoring well was collected using a 2' Grundfos submersible pump which was placed in the well. The well was purged of three well volumes prior to sample collection. Immediately following collection of the Navy sample, the split sample was collected for on-site analysis by the HAPSITE. A set of QC samples prepared by the HML was included in this batch of groundwater samples for the analysis by both the laboratory and the HAPSITE. These QC samples, including one trip blank (VOC-free water), one PE sample, one groundwater sample (P181-MW 46, used to prepare the MS/MSD) and MS/MSD, were analyzed by HML to verify the concentration using U.S. EPA Method 8260B. The same set of QC samples, double blind to the participants, was analyzed by the APCL and the FPA. The procedures for the preparation of QC samples were given in Appendix C. The HAPSITE analysis was performed on-site under HML oversight. Because of high level of carbonates, these groundwater samples were not acid preserved but kept in a cooler for transport and stored until analysis. The *Applied Physics & Chemistry Laboratory* (APCL) in Chino CA was the confirmatory laboratory. The APCL is NELAC certified and also a Navy approved laboratory for VOC analysis.

- *Results of QC Analysis*

To evaluate the method precision, four replicate PE samples were analyzed by the HAPSITE and the average of four analyses was used for the method comparison(Table 16).

Table 16 Results of HAPSITE Analysis on PE Samples[1]

Compounds	Certified Value (µg/L)	Performance Acceptance Limits (µg/L)	PE1	PE2	PE3 & dupl.	PE4	Mean (µg/L)	%RSD n=5
Benzene	50	40.8 – 59.6	27	23	34, 31	23	27.6	18
Bromodichloromethane	27.3	21.9 – 32.9	20	17	21,23	19	20	11
Bromoform	25.6	19.2 – 32.0	18	14	20, 21	20	18.6	15
Carbon tetrachloride	40.7	30.6 – 52.1	16	15	19, 19	16	17.2	13
Chlorobenzene	54.7	43.7 – 64.5	37	37	46, 45	31	39.2	16
Chlorodibromomethane	22.6	17.4 – 27.4	16	13	20, 20	16	17.2	19
Chloroform	72.1	56.9 – 85.2	36	32	45, 44	32	37.8	17
1,2- Dichlorobenzene	11.4	8.92 – 13.8	NR	NR	NR	NR		
1,3- Dichlorobenzene	32.2	25.6 – 37.4	NR	NR	NR	NR		
1,4- Dichlorobenzene	17.4	13.7 – 21.0	NR	NR	NR	NR		
1,2- Dichloroethane	44.7	35.7 – 54.8	37	28	38, 37	34	34.8	12
Ethylbenzene	58.1	44.9 – 69.8	31	30	41, 40	23	33	23
Methylene chloride*	58.6	44.3 – 73.3	350	350	380,340	310	346	7
4-Methyl-2-pentanone	80.9	49.8 - 107	46	54	52, 46	58	51.2	10
1,1,2,2-Tetrachloroethane	89.o	64.2 - 112	110	100	110, 100	100	104	5
Tetrachloroethene	27.1	20.7 – 32.1	9.2	9.2	16, 15	8.1	11.5	32
Toluene	53	42.8 – 61.4	26	22	31, 31	18	26	22
1,1,1-Trichloroethane	35.6	27.1 – 43.0	15	12	20, 19	11	15.4	26
Trichloroethene	36.6	27.7 – 43.5	14	14	20, 19	12	15.8	22
***m*-Xylene**	28.4	19.8 – 35.4	NR	NR	NR	NR	NA	NA
***p*-Xylene**	25.8	18.0 – 32.1	NR	NR	NR	NR	NA	NA
Xylene, total	54.2	37.8 – 67.5	28	27	36, 36	22	29.8	20

This PE sample was prepared according to Environmental Resource Associates Lot No. 608, Catalog No.710.
*Contamination from the sample preparation methylene chloride data were excluded from the evaluation.

The certified value and the performance acceptance limit of each compound given by the ERA are included in the table for comparison. As indicated in Table 3, three compounds, 1,2-, 1,3-, and 1,4-Dichlorobenzene, were excluded from INFICON's claim and were not calibrated for the analysis[2A]. For these analyses, the HAPSITE and two laboratories found high levels of methylene chloride were in all the PE samples, but not in the travel blank. Apparently the contamination was introduced during the preparation of PE samples. For this reason, the result of methylene chloride is excluded from data evaluation. The sum of *o-, m-, and p*-Xylene are reported as total xylene for all the analyses. These results showed that the average HAPSITE recoveries of each compound were mostly below the performance acceptance limits. The %RSD of five analyses was 5 to 32 %, mostly less than 20%, except ethylbenzene, tetrachloroethene, toluene, and trichloroethene.

Table 17 compares QC results obtained from HML, APCL and HAPSITE. The percent recovery of each compound was based on the certified values. The percent recoveries of MS and MSD were based on the spiked concentration at 40 µg/L of each compound. A low level groundwater sample, P181 MW-46, was transported in a cooler to HML at the same day to prepare MS/MSD. There are summarized in the following sections:

Table 17 Comparison of PE Results by HML, APCL, and FPA

		HML		**APCL**		HAPSITE**	
Compounds	**Certified Value**	**Conc. (µg/L)**	**Rec. (%)**	**Conc. (µg/L)**	**Rec. (%)**	**Conc. (µg/L)**	**Rec. (%)**
Benzene	50	48	96	40	80	27.6	55.2
Bromodichloromethane	27.3	28	103	21	77	20	73.3
Bromoform	25.6	21	82	20	78	18.6	72.7
Carbon tetrachloride	40.7	28	69	25	61	17.2	42.3
Chlorobenzene	54.7	53	97	45	82	39.2	71.7
Chlorodibromomethane	22.6	21	93	20	88	17.2	76.1
Chloroform	72.1	73	101	58	80	37.8	52.4
1,2-Dichlorobenzene	11.4	11	96	9	79	NA	NA
1,3-Dichlorobenzene	32.2	31	96	26	81	NA	NA
1,4-Dichlorobenzene	17.4	17	98	10	57	NA	NA
1,2-Dichloroethane	44.7	46	103	34	76	34.8	77.9
Ethylbenzene	58.1	54	93	40	69	33	56.8
Methylene chloride*	58.6	580*	990*	541*	923*	346*	590*
4-Methyl-2-pentanone	80.9	76	94	ND	ND	51.2	63.4
1,1,2,2-Tetrachloroethane	89.o	100	112	59	66	104	117
Tetrachloroethene	27.1	19	70	20	74	11.5	42.4
Toluene	53	49	92	39	74	26	48.3
1,1,1-Trichloroethane	35.6	28	79	24	67	15.4	43.2
Trichloroethene	36.6	31	85	29	79	15.8	43.3
***m*-Xylene**	28.4	NR	NR	NR	NR	NR	NR
***p*-Xylene**	25.8	NR	NR	NR	NR	NR	NR
Xylene, total	54.2	50	92	37	68	29.8	55

* The results of methylene chloride are excluded from comparison due to background contamination

** The value is the average of four replicate analyses.

Results of HML Analysis

As indicated in Table 17, HML found the average recovery of compounds in the PE sample was 92%, ranging from 69 to 112%. The results of PE analyses were within the performance acceptance limits of ±20%, with the exception of carbon tetrachloride (69%), tetrachloroethene (70%) and 1,1,1-Trichloroethane (79%). The background sample (P181 MW-46) that was used to prepare MS/MSD was found to contain benzene, toluene, and MTBE and other volatile organic compounds which include ethylbenzene, styrene, xylene isomers and very high level of naphthalene. This finding was consistent with the previous studies by the Navy. As shown in Table 18, after subtraction of the background level, the recoveries of six spiked compounds ranged from 74 to 98% and the %RSD of 0 to 9.2%. Results of HML, APCL, and HAPSITE analyses on the MS/MSD are presented in Table 18.

Results of APCL Analysis

The average recovery of compounds in the PE sample was 74%, ranging from 57 to 80% (Table 17) in which ten compounds were out of the performance acceptable limits and one compound, 4-methyl-2-pententanone, was not identified. The APCL did not identify any background contaminants in the groundwater (P181 MW-46) except low levels of ethylbenzene (2.3 µg/L) and MTBE (19 µg/L). As indicated in Table 18, three out of six spiked compounds in the matrix spike were not detected (1,1-dichloroethene and MTBE were not detected and toluene at 11 µg/L was below the background level found in P181 MW-46). The recoveries of other three compounds, chlorobenzene, trichloroethene and benzene, were 56, 68 and 18% respectively. The recovery of benzene was based on the background level of 67 µg/L determined by HML. The results of APCL analyses for the background sample and the MS/MSD samples were not acceptable, both in quality and in quantity.

Results of HAPSITE Analysis

The average recovery of HAPSITE analyses was 62%, ranging from 42 to 117%. As shown in Table 17, the recoveries of HAPSITE analyses were apparently lower than those of the laboratory analysis. All the compounds were identified but only five compounds were within the performance acceptable limits. The HAPSITE identified all the contaminants in the groundwater sample, including high level of naphthalene and other compounds as indicated in HML analysis, but extremely high level of benzene was found in the groundwater sample. After reviewing the sample analysis sequence, the high level of benzene appears to be the carryover from a previous sample which had benzene concentration at the ppm level (results of APCL analysis 1,620 µg/L; HAPSITE 1800 µg/L). For this reason, the recoveries of benzene and toluene for HAPSITE analysis were calculated on the basis of the background concentrations found by the HML analysis. As indicated in Table 18, the recoveries of HAPSITE analysis on the matrix spike ranged from 55 to 77% with RSDs =10%. The criteria specified in U.S. EPA Method 8260B are RSD of =30% and the acceptable recovery range of 70 to 130%. The results obtained from these analyses suggested that the HAPSITE data were in general 20% to 30% less than that of the laboratory data. To avoid the cross contamination problem, the analysis of a method blank must be followed after a highly contaminated sample.

Table 18 Comparison of MS and MSD Recoveries by HML, APCL and HAPSITE

	Spiked Compound	Benzene	Chloro-benzene	Toluene	Trichloro-ethene	1,1-Dichloro ethene	MTBE
	Concentration	**(µg/l)**	**(µg/l)**	**(µg/l)**	**(µg/l)**	**(µg/l)**	**(µg/l)**
HML	P181 MW-46	67	ND	30	ND	ND	16
	MS	98	33	60	31	29	57
	MSD	96	33	60	31	30	52
	%RPD	2.1	0.0	0.0	0.0	3.4	9.2
	Average	97		60	31	30	55
	%Recovery*	75	83	75	78	74	98
APCL	P181-MW-46	ND	ND	ND	ND	ND	19
	MS	83	22	11[a]	28	ND[a]	ND[a]
	MSD	64[a]	23	11[a]	26	ND[a]	ND[a]
	%RPD	12.9	2.2	0.0	3.7	NA	NA
	Average	74	23	11	27	NA	NA
	%Recovery	18[b]	56	NA	68	NA	NA
HAPSITE	P181 MW-46	110	ND	53[b]	ND	ND	21
	MS	90	26	57	22	21	47
	MSD	89	26	57	22	19	46
	%RPD	5.7	0.0	0.0	0.0	10	2.2
	Average	90	26	57	22	20	47
	%Recovery	58[b]	65	68	55	59	77

*The recovery was based on the calculated concentration of 40 µg/L of each compound.

[a] Value is below the spiked or the background concentration found in P181 MW-46.

[b] The recovery was based on the background concentration determined by the HML.

- *Results of Environmental Analysis*

Eleven well samples were split for the analysis between the HAPSITE and the APCL laboratory. Inconsistent analytical results were received from the HAPSITE and the APCL laboratory. Based of the unsuccessful performance of APCL on the associated QC samples, the groundwater data generated by the APCL can not be validated as reference data to evaluate the HAPSITE performance. For this reason, the HAPSITE performance in this study can only compare to the HML QC data as discussed in the previous section.

2.2.2 Soil Analysis

(1) Laboratory Proficiency Testing

As part of the requirements by the National Environmental Laboratory Accreditation Program (NELAP), INFICON participated in two inter-laboratory proficiency studies for the analysis of VOCs in soil (LPTP 01-S2 and LPTP 01-S3) during the year of 2001. The Laboratory Proficiency Testing was sponsored by the R. T. Corporation (RTC) in Laramie WY. In these studies, the HAPSITE data were compared to that of U.S. EPA Method 8260B. The FPA used HAPSITE to analyze the following two PE samples:

Study LPTP 01-S2 (Commencement date: May 7, 2001), the sample (0011-605-VOAs) contained 20 VOCs with concentrations from "non-detect to 100 µg/kg"

Study LPTP 01-S3 (Commencement date: August 6, 2001), the sample contained 23 VOCs at the low and high concentrations; the low level sample (0109-609-VOAs) with concentrations from 1 to 210 µg/kg, and the high level sample (0109-608-VOAs) from 10 to 6900 µg/kg.

The RTC evaluated the performance according to a calculated Z-score for every analyte that is assigned a "Detect" status. The equations for calculating a Z-score and the acceptance limits are as follows:

Z-score = (reported value – study mean)/ study standard deviations
A z-score between -3 and +3 is considered "Acceptable".

Based on these evaluation criteria, the reported HAPSITE values were 100% within the acceptable limits. Copies of RTC reports on the HAPSITE analysis are included in Appendix D. for reference. It is noticeable that HAPSITE reported values were in general less than the average mean values for the low level soil. For soil concentrations below 100 µg/kg, the HAPSITE values were approximately 50% of the mean value, for soil concentrations less than 200 µg/kg approximately 30% of the mean value. These results suggest that the HAPSITE analysis is more favorable for the high level soil than the low level soil.

(2) Environmental Sample Analysis

In coordination with the Hazardous Materials Laboratory, a study for soil analysis was conducted on February 28, 2002. The DTSC, Site Mitigation Unit collected eighteen soil samples from a Hi-Tech Iron Works in Los Angeles area for VOC analysis. These samples were split for analysis between the FPA and the HML. The laboratory used U.S. EPA Method 5035A in conjunction with Method 8260B for the analysis.

- *Sample Preparation and Analysis*

The level of VOC contamination in these samples was unclear from previous investigations. To ensure the analysis would cover both the high and low level samples, each sample was prepared in a 40-mL VOA vial submitted to HML in three preparations: "unpreserved soil", "methanol-preserved soil", and "soil in 20% sodium bisulfate solution. Two unused vials containing water were shipped with samples as trip blank. An additional set of soil samples with no preservative added was taken for the determination of moisture or other uses as necessary. All the VOA vials containing 20 mL water, were shipped by FPA to the field for sampling. Split samples were prepared in the field and shipped in a 4°C cooler with a chain of custody form, by overnight express to the HML in Berkeley and to FPA in Cameron Park, CA for the analysis.

Analysis of Low Level Soil

For the laboratory analysis, 5 g of soil was collected in a 40-mL VOA vial containing

5 mL of 20% sodium bisulfate solution and was subjected to analysis using purge and trap/GCMS method in a closed system.

For the field analysis, the addition of a preservative is usually unnecessary. To evaluate the applications of HAPSITE for laboratory use, these samples were run in a laboratory setting. FPA analyzed both the preserved and unpreserved samples. The co-located samples were used for the analysis of low level samples. FPA analyzed these samples according to INFICON SOP for soil analysis described in section 2.3.

Analysis of High Level Soil

Five grams of soil was added directly into a 40-ml VOA vial containing 5 mL of methanol and sent to the HML for analysis using U.S. EPA SW-846 Method 8260B. Following the laboratory analysis, one mL of each methanol extract was split and given to the FPA for HAPSITE analysis.

- *Results of Analysis*

As a routine practice, HML first analyzed the methanol extract for the high level sample. None of target compounds listed in U.S. EPA Method 8260B were detected in these samples or in the method blank (water). The HML quantitation limits for these compounds were 0.5 mg/kg. The recoveries of all the surrogates (1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene) were within the specified QC limits. Two pairs of matrix spike and matrix spike duplicate were analyzed. The RPDs of five surrogates ranged from 0 to 1.97%. Since none of the target compounds were detected by the laboratory, FPA was not required to analyze the split methanol extracts of these samples.

For the low level analysis, samples were preserved in sodium bisulfate solution for Purge and Trap analysis in the laboratory. FPA analyzed both the preserved and unpreserved samples. The recoveries of surrogates, Toluene-d8 and 4-bromofluorobenzene, for every sample were ranged from 84 to 109% (within the acceptable range of 70 to 130%). However, as shown in Table 19, no significant levels of VOC were detected by either the laboratory or the field method. Trace levels of acetone (in every sample), 2-butanone (in 7 samples) and tetrachloroethene (in one sample) were found in the laboratory analysis. However, concentrations of these compounds were mostly below the PQL of the HAPSITE (20 µg/kg for acetone, 20 µg/kg for butanone, and 10 µg/kg for tetrachloroethene). The HAPSITE detected acetone in three preserved samples but the values were not in consistent with the laboratory data. Methylene chloride and toluene with concentrations varying from 5 to 15 µg/kg were found in the water blank and in every soil sample of the HAPSITE analysis. Apparently the water used by the FPA for analysis was contaminated with these two compounds. No other VOCs were identified either by the HAPSITE or the laboratory analysis.

Table 19 Results of VOC Analysis in Soil Samples

	Compound Identified					
Collector's #	**Acetone (µg/kg)**		**2-Butanone (µg/kg)**		**Tetrachloroethene (µg/kg)**	
	Method 8260[1]	**HAPSITE**[2] **pres./unpres.**	**Method 8260**[1]	**HAPSITE**[2] **pres. /unpres.**	**Method 8260**[1]	**HAPSITE**[2] **pres./unpres.**
HA-1-05	13	ND/ND	D	ND/ND	6.1	ND/7.8
HA-1-5	24*	ND/ND	7.5*	ND/ND	D	4.5/ND
HA-1-5A(D)	20	ND/ND	5.9	ND/ND	D	ND/ND
HA-2-05	46	ND/ND	ND	ND/ND	ND/ND	ND/ND
HA-2-5	18	NA/NA	ND	NA/NA	ND/ND	NA/ND
HA-3-05	15	58/ND	ND	ND/ND	ND/ND	ND/ND
HA-3-5	12	ND/ND	ND	ND/ND	ND/ND	ND/ND
HA-4-05	15	ND/ND	ND	ND/ND	ND/ND	ND/ND
HA-4-5	13	ND/ND	D	ND/ND	ND/ND	ND/ND
HA-5-05	19	ND/ND	16	ND/ND	ND/ND	ND/ND
HA-5-5	31	390/ND	7.5	ND/ND	ND/ND	ND/ND
HA-6-05	33	ND/ND	11	ND/ND	ND/ND	ND/ND
HA-6-05 (D)	17	ND/ND	5.7	ND/ND	ND/ND	ND/ND
HA-6-5	21	ND/ND	5.2	ND/ND	ND/ND	ND/ND
HA/7/05	18	119/ND	ND	ND/ND	ND/ND	ND/ND
HA-7-5	16	ND/ND	D	ND/ND	ND/ND	ND/ND
HA-8-05	31	ND/ND	20	ND/ND	ND/ND	NDND
HA-8-5	NA	ND/ND	NA	ND/ND	NA/NA	ND/NA

ND = not detected; NA = not analyzed; D = detected below quantitation limit.
*estimate value due to possible matrix interference
[1] The Quantitation Limit of U.S. EPA Method 8260:
Acetone = 10 µg/kg 2-Butanone = 5 µg/kg Tetrachloroethene = 5µg/kg
[2] The Quantitation Limit of HAPSITE analysis:
Acetone = 20 µg/kg 2-Butanone = 20 µg/kg Tetrachloroethene = 10µg/kg

2.2.3 Air and Soil Gas Analysis

Since the principle of the HAPSITE operation is based on detecting the chemical equilibrium concentration in vapor phase, the air and soil gas analyses have been the most widely used environmental applications. A recent study on Air QC samples performed under the oversight of DTSC staff and two existing soil gas data sets submitted by INFICON were available for this evaluation.

(1) Air QC Study

In coordination with CA DHS Environmental Health Laboratory (EHL) a test plan was developed to verify the HAPSITE air analysis. The analyses were performed in the Hazardous Materials Laboratory by the FPA on March 12, 2002. The purpose of this study was to compare the performance of HAPSITE analysis with a laboratory GC/MS method on split sample analysis.

- *Sample Preparation and Analysis*

A methanol standard solution containing the following eight target compounds was prepared for the study.

Methyl tert-butyl Ether (MTBE)
Chloroform
1,1,1-trichloroethane
Benzene
Carbon Tetrachloride
Toluene
Chlorobenzene
m-xylene

Procedures for Sample Preparation

The standards and samples were prepared in a 6-L Restek Silicocanisters™ and pressurized to approximately 7 liters. Gas standards were prepared by injecting a known volume of a liquid mixture into a heated injector port and purged into the canister with humidified air (approximately 50% RH). Seven liters of diluted air were determined by flow rate and time rather than using a pressure transducer to measure the internal canister pressure. To avoid the variation in gas standards, the same set of gas standards were split between the EHL and FPA to create the calibration curves for each analyte. Gas standards, in canisters, were prepared at 0.25, 0.5, 1, 2.5, 5.0, and 10 ppmv. Quality control samples were prepared at concentrations 1 and 5 ppmv. Two canisters were prepared at 6.4 and 9.6 ppmv, but were not reported by EHLB because these samples exceeded the upper quantation range of the laboratory GC/MS. To avoid introducing additional uncertainty, dilution of samples above 5 ppmv was not performed. A Finnigan 4500 GC/MS equipped with a J&W 60 meter DB-VRX column was used for the laboratory analysis. The U.S. EPA Method TO-15, *Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specifically-prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry,* was followed for the laboratory analysis.

For the sample splitting, the over pressurized volume of 1 L was transferred into a Tedlar bag and submitted to FPA for analysis within 10 minutes of their preparation. The remaining air sample (6 liters) retained in a canister was used for the laboratory analysis. Two QC samples at 1 and 5 ppmv were generated from a liquid standard prepared independently by another analyst to verify the accuracy of the standard concentrations. These two QC samples were held for 2 hours to check the degradation of the analytes. All samples submitted to FPA were analyzed within 2 hours. A set of samples consisting of a blank (air), six known samples, one duplicate, and two QC samples (at the low and high concentrations) were prepared by the EHL for this study. A total of 10 samples were generated and split between the EHL and FPA for analysis

- *Results of QC Study*

In both GC/MS and HAPSITE analyses, all the data were acquired in a full scan mode. The quantification was based on the primary ion of each analyte as shown in Table 20. The results of

QC samples (1ppm and 5ppm) indicated that all compounds prepared under the operation conditions were stable for the time required for the FPA to complete the analysis. Two high concentration samples (sample 5 and 6) exceeding the dynamic range of the laboratory instrument was analyzed by the HAPSITE only. Consequently, the comparison of two data sets was based on the data of 8 samples. The results of laboratory and HAPSITE analysis are summarized in Table 20. The correlation coefficients of data obtained from two analytical systems are all greater than 0.99 for each analyte (Table 21). One duplicate analysis, Sample 1 for the HAPSITE and Sample 3 for the laboratory, was performed by each method. The RPDs of duplicate analysis of each analyte ranged from 0 to 12% for the laboratory analysis and 0 to 28% for the HAPSITE analysis. All the recoveries are within ± 25% of the spiked concentrations, with the exception of MTBE spiked at 0.43 ppmv marked in "gray". These results meet the acceptance criteria of RPD =30% and the recoveries of MD/MSD between 70 to 130% of the spiked concentrations (Table 22). For the analytes and the concentrations tested in this study, the results showed good correlation of the HAPSITE data obtained from Tedlar bags and GC/MS laboratory data obtained from Silicocanisters. This study also demonstrated the stability of analytes in Tedlar bags within two hours of holding period. These results demonstrate that vapor analysis using HAPSITE and U.S. EPA Method TO 15 give comparable results within the experimental parameters of this study. In addition, the HAPSITE has demonstrated the advantage over the GC or GC/MS in analyzing high concentration samples.

Table 20 Summary of Laboratory and HAPSITE Analysis

Sample No		Chemical (Quan Ion)	MTBE (73)	Chloroform (83)	1,1,1-TCE (97)	CCl_4 (117)	Benzene (78)	CL-Benzene (112)	*m*-Xylene (91)	Toluene 91	
		Concentration (ppmv)									
		Spiked	Found							Spiked	Found
Sample 1	EHL	0.43	0.48	0.46	0.45	0.43	0.46	0.42	0.44	0.50	0.56
	HAPSITE		0.23	0.46	0.44	0.34	0.42	0.38	0.32		0.42
Sample 1D	HAPSITE		0.24	0.45	0.35	0.45	0.43	0.37	0.32		0.41
Sample 2	EHL	0.64	0.66	0.64	0.65	0.64	0.66	0.65	0.67	0.74	0.86
	HAPSITE		0.55	0.65	0.60	0.61	0.57	0.57	0.49		0.63
Sample 3	EHL	0.96	1.0	0.99	1.0	0.93	0.99	0.98	1.0	1.1	1.2
Sample 3D	EHL	0.96	1.1	1.0	1.1	0.99	0.99	1.1	1.1	1.1	1.3
	HAPSITE		0.89	0.97	0.98	0.99	0.93	0.91	0.86		1.0
Sample 4	EHL	4.3	4.7	4.6	4.6	4.5	4.7	4.5	4.4	5.0	5.0
	HAPSITE		4.8	4.6	4.6	5	4.6	5	5		5.5
Sample5	EHL	6.4	NR	NR	NR	NR	NR	NR	NR	7.4	NR
	HAPSITE		7.1	6.4	6.5	6.7	6.4	7.2	7.3		8.0
Sample6	EHL	9.6	NR	NR	NR	NR	NR	NR	NR	11	NR
	HAPSITE		11	9.5	9.4	9.8	9.7	11	11		12
QCL	EHL	1	1	0.92	0.91	0.9	0.96	0.96	0.92	1.0	1.0
	HAPSITE		1.1	1.0	0.97	0.98	0.95	0.95	0.86		0.99
QCH	EHL	5	4.8	4.6	4.7	4.6	4.7	4.7	4.6	5.0	4.9
	HAPSITE		5.8	4.6	4.9	5.2	4.9	5	5.2		5.2

Table 21 Comparison of Laboratory and HAPSITE Data on Split Sample Analysis

	Correlation Coefficient of Two Data Set[1]							
.	**MTBE**	**Chloroform**	**1,1,1-TCE**	**CL_4**	**Benzene**	**Cl-Benzene**	***m*-Xylene**	**Toluene**
r square	0.9928	0.9998	0.9991	0.9990	0.9992	0.9990	0.9992	0.9993
Relative Percent Difference of Duplicate Analysis[2]								
EHL	9.5	1	9.5	6.3	0	12	0	8
HAPSITE	4.3	2.2	23	28	2.4	2.7	0	2.4

[1] Correlation coefficients were calculated from the results of split sample analysis of 4 unknown and 2 QC samples.

[2] RPD was calculated from the duplicate analysis of HAPSITE (sample 1) and Method TO-15 (sample 3).

Table 22 Percent Recovery of Spiked Analyte from HAPSITE and Laboratory Analyses

Sample No	**Analysis**	**Analyte (Quan Ion)**	**MTBE (73)**	**Chloroform (83)**	**1,1,1-TCE (97)**	**CCl_4 (117)**	**Benzene (78)**	**CL-Benzene (112)**	***m*-Xylene (91)**	**Toluene (91)**	
		Spiked Conc. (ppmv)	Recovery (%)							Spiked (ppmv)	Recovery (%)
Sample 1	EHL	0.43	112	107	105	100	107	97.7	102	0.50	112
	HAPSITE		53.5$^+$	107	102	79	97.7	88.4	74.7		84
Sample 1D	HAPSITE		56$^+$	105	81	105	100	86	74.4		82
Sample 2	EHL	0.64	103	100	10-2	100	103	102	105	0.74	116
	HAPSITE		86	102	94	95	89	89	76.6		85
Sample 3	EHL	0.96	105	103	104	97	103	102	105	1.1	109
Sample 3D	EHL	0.96	110	107	113	103	103	111	113	1.1	120
	HAPSITE		92.7	101	102	103	96.9	94.8	89.4		90.1
Sample 4	EHL	4.3	109	107	106	105	109	105	101	4.96	101
	HAPSITE		112	107	107	116	107	116	116		111
Sample5	EHL	6.4	NR	NR	NR	NR	NR	NR	NR	7.4	NR
	HAPSITE		111	100	102	105	100	113	114		108
Sample6	EHL	9.6	NR	NR	NR	NR	NR	NR	NR	11	NR
	HAPSITE		114	99	98	102	101	114	114		108
QCL	EHL	1	100	92	91	90	96	96	92	1.0	104
	HAPSITE		110	100	97	98	95	95	86		99
QCH	EHL	5	96	91.6	94.6	92	94.4	94.8	91.4	5.0	99
	HAPSITE		116	92	98	104	98	100	104		104

D = duplicate

$^+$The recovery is out of ±25% of the spiked level.

(2) Radian Tracy Army Depot Project

In 1998, the FPA was contracted by PEG, a private engineering firm, to investigate BTEX contamination in Santa Monica, California. The role of FPA was to provide on-site analysis to delineate a soil gas plume to make decisions for on site cleanup activities. In order to select a qualified contractor for the project, PEG sent a blind audit sample to the FPA and other two laboratories for the analysis. Both laboratories used U.S. EPA Method TO-14 for the analysis. The results of inter-laboratory study on this pre-audit sample are presented in Table 23. No sample information was given to the participants in this proficiency testing.

Table 23 Inter-laboratory Comparison of Gas Analysis

Target Analyte	HAPSITE[1]	MSD[2] (FPA)	Lab 1[3]	Lab 2[3]	Mean	Std. Dev
Benzene	**Concentration (ppmv)**					
B-10-10	0.04	0.03	0.04	0.05	0.04	0.008
B-10-20	0.02	0.04	0.09	0.03	0.045	0.031
B-10-40	ND	ND	0.02	<0.04	NA	NA
Toluene						
B-10-10	0.02	0.02	0.05	0.07	0.04	0.025
B-10-20	0.06	0.09	0.22	0.10	0.12	0.07
B-10-40	0.01	ND	0.05	<0.04	NA	NA
Ethyl Benzene						
B10-10	0.41	0.37	0.32	0.41	0.38	0.043
B10-20	0.49	0.45	0.21	0.40	0.39	0.13
B10-40	ND	ND	0.02	<0.04	NA	NA
Xylenes						
B10-10	1.6	1.5	1.5	1.9	1.63	0.19
B10-20	2.0	1.8	0.29	1.8	1.47	0.79
B10-40	0.01	ND	0.06	0.05	NA	NA
TCE						
B19-20	0.1	0.07	0.05	0.19	0.10	0.062
B19-40	ND	ND	0.02	0.06	NA	NA
B-19-60	0.04	NA	NA	NA	NA	NA
B19-60	0.14	0.06	0.11	0.21	0.13	0.063

NA= Not Applicable; ND= Not Detected

[1] HAPSITE MDL is 0.2 ppmv. Only values above MDL were used for method comparison (marked in Grey).

[2] The MSD at the FPA was a HP 5973.

[3] Lab 1 and Lab 2 used U.S. EPA Method TO-14; with MDL from 0.002 ppmv to 0.004 ppmv.

For method comparison, only those values above the HAPSITE MDL are used for evaluation (Table 23). Although the composition and concentrations of this proficiency sample were not released, the HAPSITE values are consistent with the laboratory data.

- *Soil Gas Data*

Soil gas samples from the Army facility in Tracy, CA were collected to investigate the TCE and PCE contamination. Forty-seven soil gas samples, representing 10% of the total collected samples analyzed by the HAPSITE, were split for confirmation by a fixed laboratory. A summary of these analyses submitted by the FPA is presented in Table 24[A20].

Table 24 Comparison of Soil Gas Data between HAPSITE and Laboratory Analysis

Sample ID	TCE (ppmv)		RPD[1] (%)	PCE (ppmv)		RPD[1] (%)
	HAPSITE	TO-14		HAPSITE	TO-14	
69GS001	0.96	0.91	5	<0.2	0.017	NA
23GS001	<0.2	<0.001	NA	<0.2	<0.001	NA
46GS002	<0.2*	0.28	NA	<0.2	0.18	NA
52GS002	<0.2	0.11	NA	<0.2	0.11	NA
53GS003	<0.2	0.034	NA	<0.2	0.038	NA
56GS002	<0.20	0.083	NA	<0.2	0.11	NA
89GS002	0.56	0.57	2	<0.2	0.078	NA
89GS003	15	8.2	83	2.1	1.4	50
90GS001	0.23	0.27	15	<0.2	0.09	NA
92GS001	0.76	0.82	7	0.51	0.60	16
93GS001	0.42	0.48	13	0.42	0.52	23
96GS001	1.0	1.1	10	<0.2	0.19	NA
96GS002	8.5	4.6	85	1.3	0.94	32
71GS001	<0.2	0.095	NA	<0.2	0.081	Na
74GS001	<0.2	0.026	NA	<0.2	0.025	NA
75GS001	<0.2	0.014	NA	<0.2	0.015	NA
75GS002	<0.2	0.025	NA	<0.2	0.023	NA
75GS003	<0.2	0.037	NA	<0.2	0.045	NA
91GS004	<0.2	0.04	NA	0.34	0.37	8
69GS004	<0.2	0.13	NA	1.2	1.1	9
62GS004	<0.2	0.013	NA	<0.2	0.068	NA
62GS001	<0.2	0.16	NA	<0.2	0.077	NA
66GS001	9.0	8.3	8	<0.2	0.069	NA
67GS001	5.3	5.2	2	<0.2	0.075	NA
308GS002	0.61	0.53	15	1.4	1.0	33
310GS001	0.89	0.76	17	1.2	1.0	18
310GS002	0.85	0.71	20	1.1	0.95	15
311GS002	2.6	2.0	30	1.8	1.4	25
312GS001	12	4.5	166^{+}	0.32	0.21	44
312GS002	3.8	2.9	31	0.37	0.39	5
318GS001	0.27	0.33	18	0.26	0.33	24
318GS002	0.5	0.54	8	0.44	0.41	7
318GS003	<0.2	0.21	NA	0.29	0.30	3
319GS001	3.8	4.0	5	1.8	1.8	0
319GS002	2.2	2.2	0	1.1	1.1	0
317GS003	2.1	2.2	5	4.5	4.8	6
315GS002	1.3	1.4	7	4.5	4.5	0
215GS001	7.4	6.9	7	2.4	1.9	23
215GS003	0.47	0.51	8	<0.2*	0.27	NA
216GS003	0.63	0.62	2	4.6	4.6	0
217GS001	14	8.2	71	22	12	83
216GS001	16	7.2	122^{+}	49	24	104^{+}
216GS002	3.2	3.2	0	19	17	11
181GS003	<0.2	<0.001	NA	<0.2	<0.001	NA
185GS001	<0.2	<0.001	NA	<0.2	<0.001	NA
188GS002	<0.2	<0.001	NA	<0.2	<0.001	NA
191GS003	<0.2	<0.001	NA	<0.2	<0.001	NA

NA = Not Applicable *False negative result

[1] RPD = (HAPSITE data - laboratory data)/laboratory × 100 The RPD greater than 100% are marked in "+"

The laboratory analyses were performed by Performance Analytical Inc. in Simi Valley, California. These soil gas samples were collected and pressurized in canisters. FPA prepared the split sample by filling a Tedlar bag from the pressurized canister. The bag was analyzed by the FPA and the canister was sent to the laboratory for analysis using U.S. EPA Method TO-14. However, neither the laboratory analysis nor the HAPSITE analysis was accompanied with the associated QC data. However, neither the laboratory analysis nor the HAPSITE analysis was accompanied with the associated QC data. Using laboratory data as a reference, the HAPSITE data were assessed by the numbers of false positive (FP) and false negative (FN) results, the RPDs of each paired samples, and the correlation coefficient of linear regression analysis between two sets of data sets.

- *The Number of the False Positive and False Negative Results*

In this study, a FN result is defined as a sample that was determined greater than 0.2 ppmv by the reference method but not detected by the HAPSITE. A FP result is a sample that was determined less than 0.2 ppmv by the reference method and was detected by the HAPSITE. Out of 47 samples, one FN for TCE and one FN for PCE were incorrectly identified by the HAPSITE (Table 24). No false positive results were found in this data set. The concentrations of these FN were 0.28 and 0.27 ppmv which were closed to the MDL of HAPSITE, 0.2 ppmv. The uncertainty of data at this marginal level is expected.

- *The Relative Percent Difference*

On the basis of the laboratory data the RPD between the HAPSITE and the laboratory data was calculated. For the inter-method comparison the RPD greater than 100% (the absolute value of the HAPSITE data less than one half or more than double of the laboratory value) is considered apparent difference between two values. These results showed out of 47 samples, three RPDs (two for TCE and one for PCE) were over 100%. Based on this criteria, 95% of the HAPSITE data were consistent with the data generated by the U.S. EPA Method TO-14.

- *Linear Regression Analysis*

On the bases of positively identified samples, the correlation coefficient of the HAPSITE data and the TO-14 data was assessed by a linear regression analysis. A summary of statistical output with linear fit plots of TCE and PCE are presented in Figures 5 and 6. The correlation coefficients (r^2) for TCE and PCE are 0.85 and 0.93, respectively. However, due to the significant deviations of two high values of TCE and PCE in the data set, in which the HAPSITE values are approximately two times of the laboratory data (Table 24), the linear regression analysis indicated nearly 50% positive bias for the HAPSITE analysis. Since it has been well demonstrated in many cases that the HAPSITE system has the capability to analyze more highly concentrated vapor samples than the laboratory GC or GC/MS system, it is unclear whether the low values of the laboratory analysis are due to the instrument over saturation or the inaccuracy of the HAPSITE analyses. The high correlation coefficients of the TCE and PCE data suggested that the HAPSITE analyses on soil gas data are well correlated to the U.S. EPA Method TO-14.

TCE Regression Statistics	
Multiple R	0.91947
R Square	0.84543
Adjusted R Square	0.83948
Standard Error	1.96916
Observations	28

PCE Regression Statistics	
Multiple R	0.96462
R Square	0.93050
Adjusted R Square	0.92734
Standard Error	2.92854
Observations	24

Figure 5 Correlation of HAPSITE TCE Analysis and Method TO14

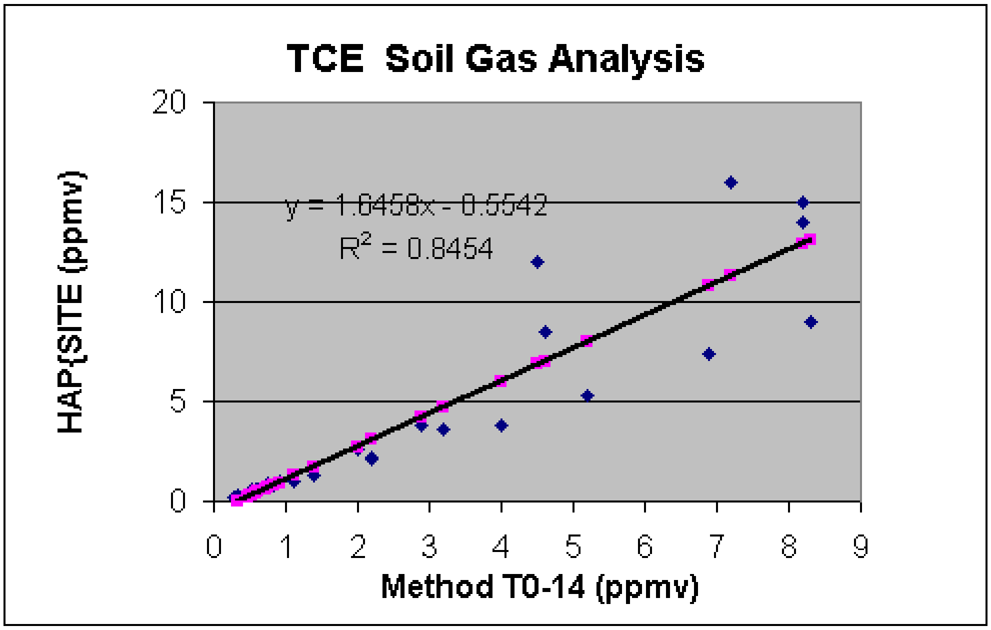
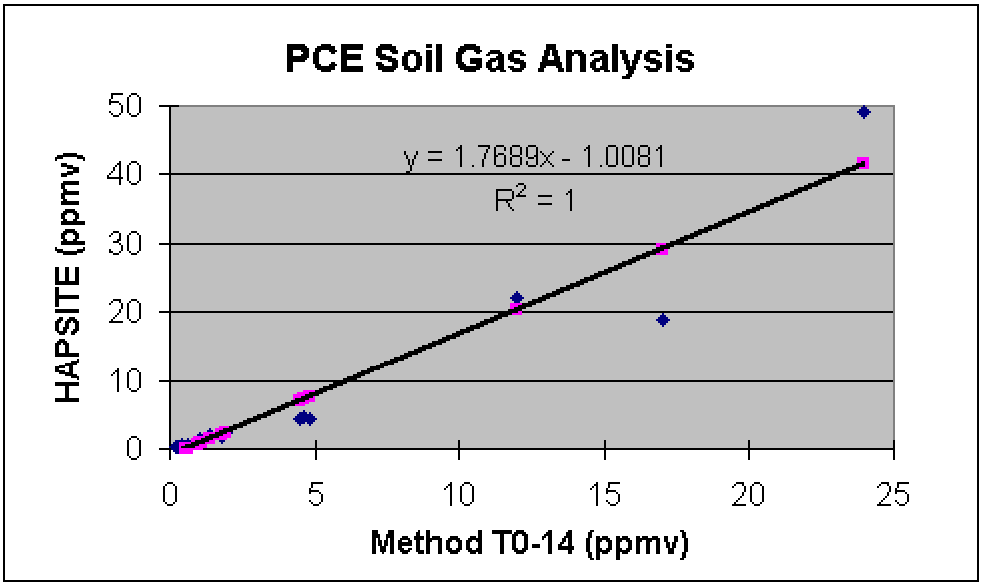


Figure 6 Correlation of HAPSITE Analysis and Method TO-14



(3) Data From an Independent Third Party

IT OHM Corporation (now, part of the Shaw Environmental and Infrastructures) contracted by the US Department of the Navy, had used HAPSITE to monitor VOC contaminations in San Diego area (North Island) for the last several years. The soil gas samples on a 10% basis had been split for analysis by the laboratory for confirmation. The results of split sample analyses on soil gases, from January to April of 2002, are presented in Table 25. The QC data accompanied with this set of laboratory analysis were submitted by IT for the evaluation. Five major compounds, 1,1,1-trichloroethene, 1,1-dichloroethane, 1,1-dichloroethene, *cis*-1,2-dichloroethene and trichloroethene, were identified in four samples. In this project, the intended use of the data was not to achieve low detection limit but to identify high level of contaminants. For this reason, high calibration standards were used to minimize the number and magnitude of the sample dilutions. Thus, the reporting limit of 1 ppmv is for the project-specific determination, and not a limitation of HAPSITE analysis. On the basis of the laboratory data, the RPD of the paired samples was calculated and presented in Table 25. The RPDs of these analytes ranged from 1.4% to 47% which were less than a factor of two. For the inter-method comparison, this variation is within the acceptable range.

Table 25 IT Soil Gas Split Sample Analysis by HAPSITE and U.S. EPA Method TO-14

Sample Location **Sample Date**	**OU-19/20-VP03C** 4/4/02			**OU19/20PW07** 3/6/02			**OU19/20-VP03B** 3/6/02			**OU19/20-VP03B** 0108/02		
Concentration	**HAPS** (ppmv)	**LAB** (ppmv)	**RPD** (%)	**HAPS** (ppmv)	**LAB** (ppmv)	**RPD** (%)	**HAPS** (ppmv)	**LAB** (ppmv)	**RPD** (%)	**HAPS** (ppmv)	**LAB** (ppmv)	**RPD** (%)
1,1,1-Trichloroethane	28.2	22	28	2.09	2	4.5	23.5	16	47	NA	NA	NA
1,1-Dichloroethane	NA	NA	NA	NA	NA	NA	NA	NA	NA	2.9	4	27
1,1-Dichloroethene	NA	NA	NA	NA	NA	NA	1.2	1.1	9.1	2.3	2.6	12
***cis*-1,2-Dichloroethene**	75.8	63	20	5.48	5.4	1.5	27.2	20	36	57.1	49	16
Trichloroethene	65.2	51	28	142	130	9.2	70	50	40	155	130	19

3. Quality Assurance and Quality Control (QA/AC)

The QA/QC procedures for the analysis of water, soil and soil gas must be documented to ensure the data quality. The same QC procedures used in the confirmatory laboratory are applied to the field analysis.

3.1 Instrument Calibration and QC samples

The GC/MS is tuned with bromofluorobenzene (BFB) as specified in U.S. EPA Method 8260B. Alternatively, the HAPSITE manufacturer uses a mass calibration procedure that will yield standard spectra to meet the project need as specified in Table 2. The continuing calibration is analyzed at the beginning of each analytical shift. The acceptance criteria for Quality control samples are specified in Table 26.

Table 26 Instrument Calibration and QC Samples

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Instrument tuning of BFB	Every 12 hours	Ion abundance criteria as specified in Table 1	Reanalyze BFB and adjust parameters to meet tune criteria
5-point calibration	At the beginning of project or as required to meet specified criteria	%RSD of water = 30%; soil =20%;and soil gas =25%	Rerun levels which do not meet criteria
Continuing calibration	Beginning of each day	Difference of the expected concentration for the CCC compounds of water: ± 25%;soil: ± 20% , and soil gas: ± 30%	Recalibrate the instrument using new standards
End calibration	End of each analytical shift	±30% D of the initial calibration	Flag data if end check is out
Method blank	After beginning of day CCC	Concentration of all calibrated compounds < PQL	Re-run blanks until criteria are met.
Duplicates	10% of the samples	RPD = 30%	Analyzed a third aliquot and flag reported data
MS/MSD (soil only)	5% of the sample	30% recovery of spike compounds	Rerun new spike samples and flag reported data
Surrogates	Each standard solution, method blank, sample and matrix spike,	70% to 130% recovery	Re-analyzed the sample or flag reported data, unless due to matrix effects.

For the quantitative analysis a calibration curve with a minimum of 5 concentrations must be prepared for each target analyte. Duplicates are analyzed to evaluate the precision of the analysis. Due to matrix effects and technical problems in collecting homogeneous samples, specifically for soil samples, the results of duplicate analysis can vary to some extent. Surrogates are spiked into all the samples to evaluate the effectiveness of the analytical process. Method blanks with same sample matrix are analyzed concurrently through the entire analytical process to assess the possibility of extraneous contamination[A5, A15, A16, A17].

3.2 Operator's Training

The chemist who operates the instrument must have knowledge to design a QC procedure that is sufficient to ensure that the resulting data will be of known quality meeting the project-specific objectives. In general, it requires three days of training to introduce technical personnel familiar with GC/MS operation. The training for field operation requires an additional day[A4]. Information obtained from users' survey suggests that operators must continue using the instrument to maintain the skill. A background in chemistry and computers would be helpful for the operator training[B12].

4. Applications, Advantages and Disadvantages

4.1 Applications

The HAPSITE GC/MS is designed for both indoor and outdoor use. The HAPSITE, small and light weight, contains all the necessary accessories for field analysis. The major applications of the HAPSITE analytical system can be classified into three areas:

(1) Emergency Response

The instrument can be operated by a single operator while being carried. The technology has been effectively used by HAZMAT and National Guard Civilian Support Teams (CST) to detect and identify toxic emissions and hazardous substances resulting from chemical terrorism and fire incidents[A27, B12, B13]. In emergency response, the direct sampling inlet with hand control makes it easy to acquire and analyze gas samples at the sampling point every 15 minutes (Figure 7)[A32]. The HAPSITE has been used to detect weapons of mass destruction (WMD), such as nerve agents (Tabun, Soman, Sarin, VX), blood agents (Hydrogen Arsine) and blistering agents (Lewisite, Sulfur Mustard) [A27, B12].

Figure 7 Operation at Sampling Point

The British Special Forces in Afghanistan used HAPSITE to detect chemical weapons as shown in Figure 8 [A32].

Figure 8 Practical Use in Afghanistan

(2) Environmental Monitoring for VOC Contamination in Air, Water and Soil

With the associated headspace sampling accessory, the instrument has the capabilities for on-site analysis of water, soil and sludge samples. The rates of sample throughput for water analysis were 2 to 3 samples per hour, including periodic analysis of blanks and calibration check samples[A4, A5]. The benefits of HAPSITE on-site analysis to support decision making in the field has been well demonstrated in several real-word applications[A4, A5, A20, A33, B13]. The US Navy has used HAPSITE in several site mitigation projects for long term monitoring of VOC contaminations in air and water[A5, A20, A31]. With a consultant on-site, the decision can be made according to the field measurements, the cleanup activities can be continuously carried out without waiting for the laboratory data.

(3) Characterization of Waste Streams

The selective ion monitoring (SIM) mode is for the sensitive detection of specific compounds at industrial sites, homes and wells. The HAPSITE has been used to identify the unknown volatile vapors from the waste disposal drums and pit boreholes at hazardous waste sites[B13].

4.2 Advantages

Providing Confirmatory Data
The GC/MS provides confirmatory information for compound identification. Using a five-concentration calibration curve and appropriate quality control procedures, the data quality can be comparable to fixed laboratory data;

(1) Time and Cost Effective
The on-site analysis provides real time data to carry out the field activities continuously without depending on off-site laboratory analysis, thus significantly saving time and money;

(2) Effective for Emergency Responses
The instrument can be operated at the sampling point instantly identifying unknown chemicals for immediate decision making;

(3) Maintenance of Sample Integrity
The results can be obtained on-site within minutes, eliminating procedures for sample handling, transportation, and the need for sample preservation. This virtually eliminates the process for chain-of- custody and sample contamination problems;

(4) Rugged and Weather Resistant
The HAPSITE is engineered to endure a harsh testing environment for field work.

4.3 Disadvantages

(1) Headspace Analysis

Headspace analysis is limited to the determination of chemicals with sufficient volatility to be removed from their matrices, usually with molecular weight less than 300 amu. Thus, semi-volatile organics and other compounds can not be detected.

(2) Isothermal GC

The isothermal GC oven limits the resolution of several compounds listed in U.S. EPA Method 8260B, such as 1,2-dibromo-3-chloropropane, 1,2,4-trichlorobenzene, hexachlorobutadiene, and 1,2,3 trichlorobenzene, and the analysis of compounds with higher boiling point. The isothermal GC has been replaced with a temperature programmable GC in the improved model of the HAPSITE.

(3) Not as Sensitive as a Laboratory GC/MS

The detection limit is not as low as the laboratory GC/MS, specifically for water and soil analysis. Thus, the applications of HAPSITE would depend on the project-specific objectives. The new HAPSITE model has been shown to greatly improve in sensitivity.

(4) NEG Pump with Limited Lifetime

INFICON claims the NEG pump provides 240 hours of use. However, the useful period can be reduced unless the pump continuously operating in the service mode.

(5) Sample Holding Time

The stability of analyte and sample concentration in the Tedlar bag has been a concern for air analysis. To avoid this problem, the sample holding should be minimized and the feasibility of Tedlar bags for air analysis at low concentrations levels for certain analytes should be evaluated.

4.4 The improved Model of HAPSITE

To overcome the instrument sensitivity, INFICON has incorporated a temperature programmable GC and a sample pre-concentrator into the new generation HAPSITE system. The programmable GC can be heated to 200ºC through three temperature ramps. These modified GC conditions improve the resolution and the range of compound identifications. The microtrap concentrator, Tenax or Carbopack X, used in the improved model extends the detection limits of HAPSITE to the parts per trillion (ppt) range. Figure 9 and 10 show the analysis of tetrachloroethene (PCE) calibration gas by a first generation HAPSITE versus an improved model. As shown in these chromatograms, the responses of PCE at mass 166 in the improved HAPSITE were more than two orders of magnitude greater than the original model[A27]. In addition to increased sensitivity, the improved HAPSITE can also detect organic compounds bordering on the semi-volatile range. The extended range allows analysis of compounds with a boiling point up to approximately 250ºC.The performance of the improved HAPSITE model is out of the scope of this evaluation.

Figure 9 TIC and Ion Chromatogram of PCE – HAPSITE

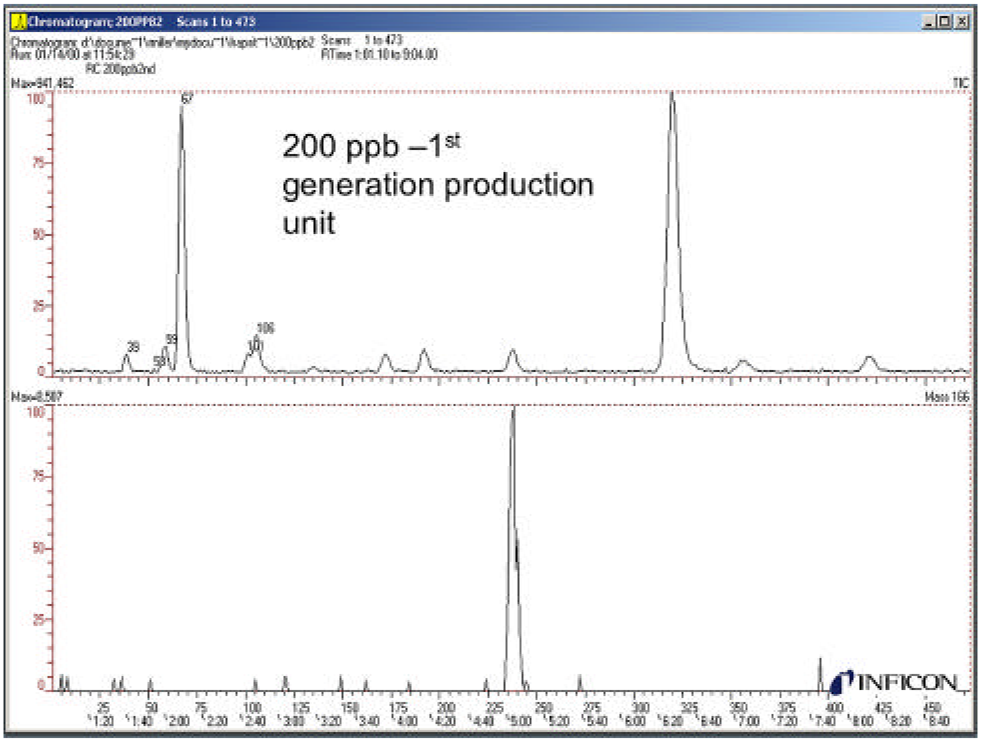
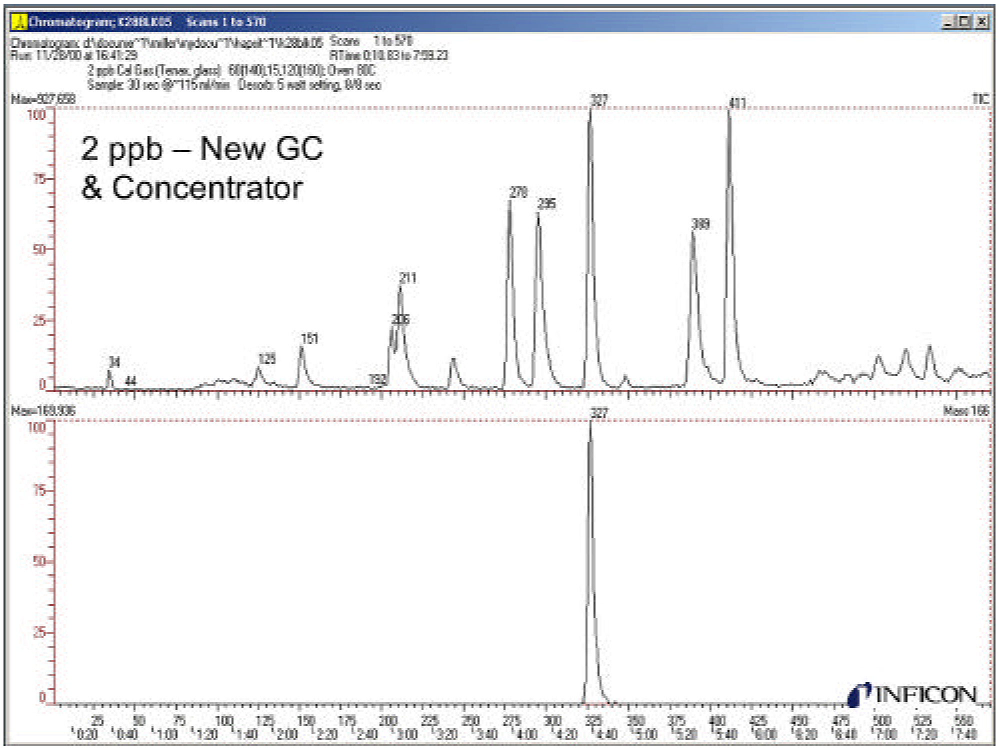


Figure 10 TIC and Ion Chromatogram of PCE – Improved HAPSITE Model



MANUFACTURE - *Quality Assurance in Manufacturing*

INFICON warrants the product manufactured by it, or by an affiliated company and sold by it to be free from defects of materials or workmanship under normal proper use and service[A29]. The product warranty shall not be less than one year from the date of shipment by seller or as specified in the purchase agreement. The HAPSITE is specifically designed for field use to withstand harsh environmental conditions, it is rugged, and easy-to-use.

1. Ruggedness

All the required components of HAPSITE are resiliently mounted to withstand the rough handling, shocks, and temperatures, typical of field work. An insulated jacket for HAPSITE and heavy duty probe insulation are available for the system to sustain field testing in cold temperatures. Figure 11 is a field test by CST in extremely cold temperatures in Alaska for up to 8 hours per day[A32].

The instrument has been used in the hot weather at temperatures over 100ºC in Texas without affecting the analytical results. The instrument can be operated in light rain conditions. The sturdy case, made of impact resistant plastic, is sealed to withstand washing for decontamination. The feasibility of HAPSITE for field analysis has been tested by emergency response personnel and industrial hygiene professionals, such as the HAZMAT and CST teams.

Figure 11 Cold Weather Operation in Alaska

2. Maintenance and Safety Considerations

The HAPSITE requires minimal service. Repairs will normally be carried out at the factory or other INFICON Service Facilities. There is no preventive maintenance required for the HAPSITE analytical module**;** The only attention required is the replacement of consumable items. The operating instructions are described in Chapter 8 of User Guide[A7]. The HAPSITE is designed for use in a contaminated hot zone. However, the instrument must be taken to the decontamination area whenever the instrument is opened. The NEG pump is very hot when it is in operation. For safety reasons, one must follow procedures given by INFICON.

INFICON Inc. certifies that HAPSITE meets the essential safety requirements of the European Union and is placed on the market accordingly. The instrument has been constructed in accordance with good engineering practice in safety matters in force in the community and does not endanger the safety of persons, domestic animals or property when properly installed and maintained and used in applications for which it was made[A7].

3. ISO 9001 Certified Products

INFICON Inc. has been assessed for compliance with provisions of the National Standards Authority of Ireland, I.S. EN ISO 9001. The gas analyzing system and a sorption pump were filed in the US PATENT & TRADEMARK OFFICE in 1995[A13, A14]. The registration No. is M877, dated May 17, 1993, amended on September 12, 2002. This certificate remains valid to December 14, 2005[A11]. Scope of Registration includes the design and manufacture of residual gas analyzers, mass spectrometers, thin film deposition monitor and controllers, halogen leak detectors and vacuum gauges. A copy of QA -91 Quality Assurance Manual signed by Lead Auditor, ISO-9000 is submitted by INFICON for reference[A10]. This manual documents the overall management function, the policy of quality system and the required processed control and testing as required by the accrediting authority.

USER SURVEY

A telephone survey on the uses of HAPSITE was conducted. The questions posed to the users were: (1) How long has the instrument been in use? (2) What were the applications and the objectives of study? (3) What were the environmental media (air, water, soil, etc) measured? (4) What were the target compounds and concentration ranges measured? (5) Does the data quality meet the project objectives? (6) Have the HAPSITE results been confirmed by the laboratory analysis, such as analysis of split samples? (7) What are the advantages and disadvantages of HAPSITE in the environmental or laboratory applications? (8) Are there comments on the instrument performance? and (9) Would you use HAPSITE for the future investigations?

Users from seven organizations were interviewed. These organizations included two fire departments, the US Environmental Protection Agency, the US National Guard, a national laboratory, an industry and a consulting company. The persons that responded to interviews were either project managers or technical staff. The period of use varied from 2 to 5 years at the time of survey. The instrument is predominantly used to test atmosphere conditions for air, organic vapors, and soil gas. However, all these users have a headspace sampling device for soil and water analysis. Depending on the situation, in general, fractions of field samples were split for confirmation by the laboratory. The measuring concentrations range from ppb to high ppm levels. The users claimed that in most cases, the HAPSITE results were consistent with the laboratory analysis and the data quality meeting the project–specific objectives. It is the general consensus among these users that HAPSITE is a cost-effective analytical system capable of generating data to support decision making. All the users responding to this survey used HAPSITE for the field studies. Every user would like to use HAPSITE for the future investigations. The following is a summary of the interviews.

1. Applications

(1) The fire departments use HAPSITE for emergency response to detect and identify toxic emissions and hazardous materials, mainly in air or organic vapors occasionally in soil and water, related to fire accidents. The HAPSITE has been used to detect and identify nerve agents (Tabun, Soman, Sarin, VX) blood agents (Hydrogen cyanide, Arsine), blistering agents (Lewisite, Sulfur Mustard), and other toxic industrial chemicals. In previous cases, the

HAPSITE results had been confirmed by the laboratory without a discrepancy in major findings[B12].

(2) The Department of Defense (DOD) uses HAPSITE as one of the standard pieces of equipment in the Air Force, Army, and Navy, and to support the activities of the National Guard Civilian Support Teams (CSTs). When the military troops are deployed, the CST uses the instrument to detect the presence of chemical warfare agents and their precursors and byproducts to support the civilian and military activities, to assess the area for the safety of operation, and to determine the necessity for evacuating the citizens.

(3) The US Department of Navy and U.S. EPA provided HAPSITE units to contractors for long term monitoring of VOC contaminations in air, water, soil and soil gas. These major applications are for detecting chlorinated and aromatic hydrocarbons at superfund sites, monitoring indoor air quality; site cleanup, remediation and environmental assessment. The main advantage is identifying the environmental unknowns to guide field activities. The US EPA used a certified analyst with no problem in operation.

(4) The national laboratory used HAPSITE for the identification and characterization of organic vapor of waste streams pumping from monitoring wells or from drums at hazardous waste sites. Likewise, the main applications are for identifying VOCs listed in U.S. EPA Method 8260B, mostly for air and liquid samples. Using custom design software linked to the HAPSITE real-time data, the national laboratory developed a method to determine the compliance with exposure limits for mixtures of volatile organic compounds[B17].

2. Users' Comments

The users' comments on the HAPSITE system are mostly consistent with the advantages and disadvantages described in section 4.

2.1 Benefits

(1) The data are dependable and accurate, comparable to that of bench-top GC/MS data (HP 5972);
(2) The HAPSITE analysis saves time and cost in operation;
(3) Relative to FID and PID, HAPSITE can identify more chemicals with more reliable information;
(4) The instrument is small and portable, good for on-site analysis, generating quantitative and qualitative GC/MS data without using a turbo-molecular pump;
(5) The instrument can be used for multi-media analysis for air, water, soil and organic vapor;
(6) The HAPSITE has a great dynamic range to facilitate the analysis of high concentration samples up to percent level without knocking down the mass spectrometer;
(7) HAPSITE uses nitrogen as carrier gas which is less expensive than helium;
(8) In conjunction with the NIST library, the instrument can effectively identify unknown toxic chemicals to guide field activities;

(9) The instrument performs well in extreme heat and cold weather and is sustainable in rough field conditions; and

(10) The instrument has been used effectively for the incident responses.

2.2 Limitations

(1) HAPSITE can only detect chemicals with molecular weigh up to 300 amu;

(2) The operator must continue using instrument to maintain the operating skills;

(3) HAPSITE is not certified by any Environmental Laboratory Accreditation Program (ELAP) and can not be used for regulatory purposes;

(4) It takes time to learn how to use the instrument effectively, taking three and a half days for the initial training and 2 to 4 weeks to be able to modify conditions for the specific applications;

(5) The chemical pump needs to operate periodically to prevent air from leaking into the system;

(6) To make the best use of HAPSITE system, a good work plan is required, it requires a minimal of $5000/year of budget to maintain the system for routine applications;

(7) The chromatogram generated by the HAPSITE can not interact with instruments in the laboratory through network communication. However, the data files can be exported in excel format to other laboratory instruments; and

(8) The detection limit of HAPSITE is not as low as the laboratory instrument for certain applications. The MDL has been improved in the new HAPSITE model.

(9) Analytes in Tedlar bags tend to degrade over time due to permeation,surface effects, humidity, etc.

PROTECTIVENESS CONSIDERATION AND ENVIRONMENTAL BENEFITS

The environmental benefits and protectiveness of the HAPSITE system can be justified in three aspects:

(1) Identification of unknowns to manage emergency situation

The HAPSITE is specifically designed for detecting Hazardous Air Pollutants on-Site. The instant response for environmental assessment is critical for the protection of public health and the environment. The HAPSITE is the only portable GC/MS currently available in the market that allows the analysis of organic vapors at the sampling point to measure below the Immediately Dangerous to Life and Health (IDLH) limits. The benefits of the HAPSITE system in response to emergency cases, such as chemical spills and fire incidents have been well demonstrated by hazardous materials professionals and the National Guard CST.

(2) A cost-effective tool to expedite site mitigation and cleanup activities

In current practice, because of waiting for off-site analytical results and the expensive laboratory analysis, a multi-stage investigative process was required for remedial action. This process has been proven to be very expensive and time consuming. To overcome this multi-stage process, the U.S. EPA OSWER has recently issued *A Guide for Project Managers: Using Dynamic Field Activities for On-Site Decision Making* at contaminated sites[B19]. The HAPSITE provides on-site analysis with effective field data to expedite the site cleanup and remedial activities in a cost-effective and timely manner.

(3) Reducing data uncertainty by providing fast analysis and increasing the number of samples analyzed at a site

As a consequence of inevitable matrix heterogeneity of a site, it becomes clear that the majority of uncertainty in data sets stems from sampling errors rather than analytical errors. The fast on-site analysis provided by the HAPSITE system can reduce data uncertainty by increasing the number of samples analyzed in order to minimize the uncharacterized area.

SUMMARY OF FINDINGS

This evaluation report is based on the documents and publications submitted by INFICON, the data packages obtained from independent investigators and the reports of U.S. EPA Environmental Technology Verification Program[A4] and Innovations in Site Characterization, Technology Evaluation for INFICON HAPSITE[A5] for groundwater analysis.

The operation of HAPSITE is based on the principle of quadrupole GC/MS to identify and quantify volatile organic compounds in environmental media (air, water, and soil). The HAPSITE was originally designed to detect hazardous air pollutants for the air program. In conjunction with a headspace sampling device, the instrument has the capability to use chemical equilibrium concentration in the vapor phase to measure VOCs in liquid and solid samples. The system is lightweight, completely self-contained and portable for the field applications.

The HAPSITE can be operated in three different modes. In the field-portable mode, with a hand control unit, the analysis can be performed at the sampling point for use in the emergency response. In the transportable mode, the HAPSITE mounted on a service module can be operated in a van for on-site analysis. In the stationary mode, the HAPSITE can be set up to use the carrier gas from a high-pressure cylinder in the laboratory as laboratory equipment. The instrument is loaded with software for the automatic instrument calibration and the built-in methods for the sampling and analysis. The analytical procedures for air (including vapor and gas), water, and soil analysis are established by INFICON. On the basis of the submitted documents and the results of these recent studies, the evaluation team finds the INFICON HAPSITE complying with the following performance statements, provided that the instrument is calibrated and maintained according to manufacturer's instructions[A7].

Water Analysis

- *The Practical Quantitation Limits for water analysis were 5 to 20 µg/L for those chemicals listed in Table 3.*

For the MDL studies, INFICON performed seven replicate analyses on water samples spiked at 5µg/L. One study contained 12 commonly found chlorinated and aromatic hydrocarbons and the other containing 36 VOCs. The average recoveries of each spiked compounds ranged from 3 to 5 µg/L and the RSD ranged from 3 to 35%, with exceptions of 1,1,2-trichloroethane at 40% and Bromoform at 50%.

- *Inficon Claims "Under the normal environmental conditions, the HAPSITE precision, as presented by the RSD on replicate analysis, is expected to be = 20% and the recoveries of ± 25% of the spiked values over the instrument calibration range*

The results obtained from the Savanna River site, the McClellan Air Force Base site and the Monterey Peninsula Airport and the associated QC data support this statement. However, the QC study obtained from NAS Alameda Point 25 were not consistent this claim.

(1) Groundwater Analysis at the Savanna River site and the McClellan Air Force Base

A comprehensive QC study was conducted by the U.S. EPA ETV program in which 166 PE samples were analyzed by the participants. The HAPSITE data showed no FP results at this target concentration of 10µg/L (typical regulatory limit) and some FN results associated with the analysis of SRS and MAFB samples (Table 7). The bromoform has relatively lower response than other chlorinated compounds, 9 out of 10 samples were not detected at 10µg/L. Four replicate PE samples at low, mid, and high levels were analyzed to assess the method precision and accuracy at various concentration levels. Thirty three ground water samples collected from 10 monitoring wells were analyzed at each demonstration site. The precision and accuracy of four replicate analyses were found mostly within the specified criteria of RSD =20% and the recovery of 75% to 125% with few exceptions (Table 8, 9, 10, and 11).

(2) On-Site VOC analysis at the Monterey Peninsula Airport

The percent recoveries of surrogates and matrix spikes obtained from the HAPSITE analyses in this project were all within the acceptance criteria of 75 to 125%, with the exception of one duplicate sample at 127% (Table 13 and 14). All the RPDs of duplicates (6 to 17%) and MS/MSD (0 or 1%) were within the range of ±25% (Table14).

(3) VOC analysis at NAS Alameda Site 25

A set of QC samples was designed by the HML to evaluate the current performance of HAPSITE analysis. To evaluate method precision, FPA analyzed four replicate PE samples containing 20 VOCs with certified values. The %RSD for four replicate PE analyses and the duplicate analyses ranged from 5 to 32%, mostly within 20%, but a few exceptions (Table 16). The average recovery for 20 VOCs was 62%, ranging from 42 to 117%. However, the average recoveries calculated on the basis of the certified values were mostly below the acceptance limits. Six target compounds were spiked into a low level groundwater sample at the concentration of 40 µg/L. The recoveries of six spiked compounds in the matrix spike were 55 to 77%, the RPDs of MS/MSDs were 0 to 10%.

In this study, the recoveries of HAPSITE data for both PE samples and MS/MSD were 20 to 30% less than the certified or the HML values. The reasons for these low recoveries could be caused by the incompatible analytical conditions used in the HAPSITE system as compared to the reference method. The headspace equilibrium sampling and the GC analysis at 60ºC isothermal is not as efficient as the purge and trap methods (U.S. EPA Method 5030B) used in the laboratory analysis. This effect could be enhanced when analyzing low level samples than high level samples. In addition, the matrix effect and the operator difference may also contribute to these variations.

- *With an established 5-point calibration curve and QA/QC program, the HAPSITE can generate data comparable to U.S. EPA Method 8260B.*

For the performance evaluation, the laboratory values associated with valid QC data were used as reference to evaluate the HAPSITE performance. Only those compounds having concentrations greater than the PQL of HAPSITE were used for the method comparison.

(1) Groundwater Analysis at the SRS and MAFB Sites

Thirty three ground water samples collected from 10 monitoring wells from the Savannah River Site and the McClellan AFB were split and analyzed for data comparison between HAPSITE analysis and U.S. EPA Method 8260B. The recoveries of HAPSITE analyses are analyte and concentration dependent. At the SRS, the major contaminants in groundwater were TCE and PCE. The HAPSITE data were 61 to 118% of the laboratory values. At the MAFB, the HAPSITE data were 78 to 180 % of the laboratory values. Due to the wide concentration range in these groundwater samples, the data obtained from each site were divided into two groups at the cutoff value of 100 µg/L. The calculated correlation coefficients between the HAPSITE data and the laboratory data were all greater than 0.97 for both low and high level samples collected from either the SRS or the MAFB (Table 12).

(2) On-Site VOC analysis at the Monterey Peninsula Airport

In this project, the HAPSITE GC/MS was used as an “on-site laboratory” to guide the installation of a monitoring network in Monterey Peninsula to determine TCE contaminations in groundwater. The QC data, as specified in the quality assurance project plan, established the data quality of the HAPSITE analysis. Fourteen ground water samples collected from borehole locations were analyzed by the HAPSITE. The ground water samples collected from these fully developed wells were then analyzed for VOCs by a fixed laboratory using SW-846 Method 8260B. The paired data obtained for the boring groundwater and the monitoring well are presented in Table 15. Two major contaminants, TCE and cis-1,2-dichloroethene were consistently identified by the HAPSITE and laboratory analyses. Since these samples were not collected at the same time, (co-located samples instead of split samples), some variations between the HAPSITE and laboratory data were expected. Under this condition, based on the difference of a factor of two, 75% of the data generated by these two analytical systems are consistent for decision making.

(3) VOC analysis at NAS Alameda Site 25

Eleven groundwater samples collected from the existing monitoring wells at the Alameda Site 25 in California were split for analysis between the FPA and the APCL. A set of QC samples, prepared by the HML, was incorporated into the analysis. These QC samples, including a travel blank, a PE sample, MS/MSD and a ground water sample used to prepare MS/MSD, were sent in double blind to the FPA, APCL and HML for the analysis. The HAPSITE performance was evaluated on the basis of HML analysis.

Laboratory Analysis

For the analyses of PE samples, the recoveries of 20 VOCs by the HML were ranged from 69 to 112% with the average of 92%, all within the performance acceptance limits of ± 20% with the exception of carbon tetrachloride (69%) and Tetrachloroethene (70%). The recoveries by the APCL analysis were ranged from 57 to 80% with the average recovery of 74% in which 10 compounds were out of the performance acceptable limits and one compound was not unidentified.

For the analysis of MS/MSD, HML identified low levels of benzene, toluene, and MTBE and very high level of naphthalene and other VOCs in groundwater which was used to prepare the MS/MSD. After background subtraction, the recoveries of HML analyses on six spiked compounds were 74 to 98% and the RPD of 0 to 9.2%. The performance of APCL on the MD/MSD were out of the acceptance: (1) Most of the background contaminants in the groundwater sample were not identified, (2) Three out of six spiked compounds were not identified (missing 1,1-dichloroethene, MTBE and toluene), (3) The recoveries of other three spiked compounds (chlorobenzene, trichloroethene, and benzene) were 56, 68 % and 40%, respectively (Table 18).

Field Analysis

The HAPSITE identified 20 VOCs in the PE sample and six target compounds spiked in the MD/MSD and other VOCs identified by the HML in the groundwater sample. The precision and recovery data of the HAPSITE were discussed in the previous section. The results obtained from these analyses suggested that the HAPSITE data were in general 20% to 30% less than that of the certified or HML data. The results of split sample analyses for eleven well samples were found inconsistent between the HAPSITE data and the APCL data. Because of the unacceptable performance of the APCL analysis on the associated QC samples, the data quality of eleven well samples generated by the APCL can not be validated for data comparison. For this reason, in this study the HAPSITE performance can only compare to the HML QC data, as discussed in the previous section. These QC samples included analysis on PE samples, groundwater P181 MW-46, and the recovery data of MS/MSD

On the basis of studies conducted by the U.S. EPA ETV program and the on-site analysis of VOCs at the Monterey Peninsula Airport, the HAPSITE data are found well correlated with that of the U.S. EPA Method 8260B for water analysis.

Soil Analysis

- *The Proposed VOC Quantitation Limits for Soil Analysis Warrant Further Investigation*

INFICON claims the Practical Quantitation Limits (PQL) for soil analysis are 10 to 40 µg/kg for those chemicals listed in Table 4. These values are approximately one order of magnitude higher than that listed in the U.S. EPA Method 8260B in conjunction with method 5021 (B2). However, the matrix presented in U.S. EPA Method 5021 was based on fortified sand. Since the soil data were prepared by sonication of VOC contaminated soil in water and analyzed the equilibrium headspace for VOC in soil, the same calibration curve was used for both water and soil analysis. No MDL data was provided by INFICON for soil analysis. The variations in partioning of VOCs from soil matrix into water are of concern. The analysis of soil samples spiked with VOCs at the PQL concentrations should be performed to demonstrate the proposed method quantitation limits for soil analysis.

- *Soil analysis results are within the acceptable limits of U.S. EPA Method 8260B*

The FPA participated in two proficiency testings sponsored by RTC for VOC analysis in soil. The HAPSITE values were 100% within the acceptable limits as defined by the RTC. The HAPSITE data were found closer to the mean value of high level soil than that of low level soil. For soil concentrations at the mg/kg level, the HAPSITE data was comparable to U.S. EPA Method 8260B. However, for concentrations at the µg/kg level, the HAPSITE data were in general lower, approximately 50% of the laboratory data at concentrations less than 100 µg/kg.

- *The application of HAPSITE on soil analysis warrants further investigation*

For the analysis of environmental soil from the Hi-Tech Iron Work, the recoveries of HAPSITE analysis on surrogates, Toluene-d8 and 4-bromofluorobenzene, were 84 to 109%, all within the acceptable range of 70 to 130%. No significant levels of VOC were detected by the U.S. EPA Method 8260B or by the HAPSITE for the method comparison (Table 19). Although the results of the proficiency testing for both the high and low concentration soil were satisfactory, because of the heterogeneity and matrix effect of the environmental soil, the applications of HAPSITE on soil analysis warrants further investigations by analyzing a wide range of VOCs in different soil types at various concentration levels.

Air and Soil Gas Analysis

- *The Practical Quantitation Limits for vapor phase analysis of those chemical listed in Table 5 are 0.2 to 0.5 ppmv*

Three set of MDL studies were evaluated, two studies conducted by the FPA, one study by an independent consultant company. These experimental data suggested the PQLs of 0.2 to 0.5 ppmv for air analysis can be achieved for those chemicals listed in Table 5. The MDL of U.S. EPA Method TO-15 is approximately two orders of magnitude lower than these proposed values.

- *The air data of HAPSITE analysis are comparable to the U.S. EPA Method T0-15*

A comparison of air analysis between the HAPSITE system and the U.S. EPA Method TO 15 was performed on a set of QC samples. Six air samples and two QC samples containing high and low VOC concentrations were prepared and split for the analysis between a California State Laboratory and the FPA. These air samples contained eight target compounds with concentrations ranging from 0.42 to 10 ppm (Table 20). The correlation coefficients of data obtained from these two analytical systems are greater than 0.99 for each compound (Table 21). The relative percent differences (RPDs) of duplicate analysis for each analyte were all =12% for the laboratory analysis and =28% for the HAPSITE analysis. The stability of these target compounds in the Tedlar bag was found stable for 2 hrs period under the experimental parameters of this study. All the HAPSITE recoveries are within ± 25% of the spiked concentrations, with the exception of MTBE spiked at 0.43 ppmv. These results meet the acceptance criteria of RPD = 30% and the recoveries of MD/MSD 70 to 130% of the spiked concentrations (Table 27) as specified by the INFICON for air analysis.

- *The soil gas data obtained from HAPSITE analysis are well correlated to that of the U.S. EPA Method T0-14*

(1) Radian Tracy Army Depot Project

HAPSITE on-site analysis was used to detect TCE and PCE contamination in soil gas at the Radian Army Depot in Tracy, CA. In the analysis of a blind audit sample, the HAPSITE data were found comparable to the laboratory data using U.S. EPA Method TO 14. (Table 23). In this project, 47 soil gas samples (10% of the collected samples) were split for the confirmatory analysis using U.S. EPA Method TO 14.

Based on the laboratory data as reference value, the data comparability between the HAPSITE analysis and the U.S. EPA Method TO-14 was assessed by (1) the rates of FP and FN results - (2) the RPD of paired data, and (3) the correlation coefficient of a linear regression analysis obtained from these two analytical systems. Using 0.2 ppmv as the target concentration, out of 47 samples one FN for TCE and one FN for PCE were incorrectly identified by the HAPSITE analysis. For the inter-method comparison, the RPD greater than 100% is considered apparent difference between two methods. Based on this criterion, 95% of the HAPSITE data were consistent with the laboratory data. The correlation coefficient between the laboratory data and the HAPSITE data were 0.85 for the TCE and 0.93 for the PCE (Figure 5 and 6). Relative to the laboratory analysis, the linear regression analysis indicated nearly 50% of the positive bias in the HAPSITE data; it is unclear whether the low values of the laboratory analysis are due to the instrument over saturation on the high concentration samples.

(2) Soil Gas Data Obtained from an Independent Party

The US Department of Navy has used HAPSITE for long term monitoring of VOC contaminations on North Island, San Diego. The intended use of the data was to identify high level VOC contamination for site remediation. The HAPSITE was calibrated only for five chemicals of concern, 1,1,1-trichloroethene, 1,1-dichloroethane, 1,1-dichlorothene, cis-1,2-dichlorothene and trichlorothene, at the reported limit of 1ppmv. On a 10% basis, the soil vapor had been split for the laboratory confirmatory analysis using U.S. EPA method TO 14. The paired data of soil gas analyses by two analytical systems were compared by the RPD. The RPDs of these five major chlorinated compounds were 1.4 to 38%, mostly =25% (Table 26).

Information obtained from users' survey indicated that the instrument can effectively analyze high concentration samples up to the percent level. Surface interactions and permeation losses overtime have been the concern of Tedlar bag sampling. These problems resulted in short sampling holing time in Tedlar bag. However, this issue does not entirely negate its application in VOC analysis. Subsequently, there are several parameters in environmental air analyses that need to be considered: (1) the stabilities of each analyte in a Tedlar bag under the environmental conditions, (2) the method precision at the low ppmv range, (3) the sample holding time, and (4) the interferences for multi-analyte analysis.

INFICON users indicated that the major advantages of HAPSITE are the effectiveness in emergency response to detect and identify toxic emissions in the air; significant savings in time and costs to expedite the site cleanup activities, and the fast sample turnaround time for decision making.

INFICON Inc. is certified under ISO-9001 by the National Standards Authority of Ireland until December 14, 2005. The HAPSITE meets the essential safety requirements of the European Union and is placed on the market accordingly.

CONCLUSIONS AND RECOMMENDATIONS

On the basis of these findings, this evaluation team supports the vendors' claims that the HAPSITE analytical system can be used as field instrument as well as laboratory instrument for water, soil and air analysis. For environmental applications, the detection limit or quantitation limit, and method bias must be evaluated to ensure meeting project-specific objectives. The surrogate compounds must be added to the environmental medium to evaluate the matrix effects and to validate the method performance. For air and vapor analysis, the stability of chemicals and concentrations in the Tedlar bag and the sample holding time must be evaluated before environmental applications. Due to the heterogeneity of environmental soil and sample matrix, the applications of soil analysis based on equilibrium sampling warrants further investigations that analyze a wide range of VOCs in different soil types at various concentration levels. The analysis of blank samples must be performed as necessary to minimize cross-contamination. The appropriate quality control procedures for VOC analysis for air water and soil must be followed to ensure the defensibility of the data.

As with all programs regardless of the fixed laboratory or field technique used, it is recommended that 10% of key samples be collected for confirmatory analysis against a reference method, specifically for samples with concentrations around the action level. It is the responsibility of a user to evaluate independently the factors or interference that are specifically associated with each case and to modify the operating parameters as necessary to meet data quality objectives for the intended applications.

The improved HAPSITE model with a temperature programmable GC and a micro-concentrator has been reported by users to greatly improve the VOC detection limits. The evaluation team recommends certification of the INFICON HAPSITE for use as an alternative to laboratory instrument for the air, water and soil analysis provided the conditions as described above are met.

MATERIALS FOR EVALUATION

A. Data Package Submitted by INFICON

1. Preliminary Eligibility Request Review sent in February 2001.
2. Eligibility Request with Attachment and Application Note submitted by Carol Thielen undated.
3. Application for certification signed by Carol Thielen, Territory Manager, dated May 18, 2001
4. U.S. EPA Environmental Technology Verification Report, Field-Portable Gas Chromatography/ Mass Spectrometer, INFICON, Inc. HAPSITE, EPA/600/R-98/142, November 1998.
5. U.S. EPA, Innovations in Site Characterization, Technology Evaluation: Real-time VOC Analysis Using a Field Portable GC/MS, EPA 542-r-01-011, August 2001.
6. INFICON Publications:
   - HAPSITE Field-Portable headspace Sampling System, dated 1999
   - ON THE MOVE "Taking the Lab out in the Field", a reprint from Steven Publication, Environmental Protection, management and problem solving for environment by Charles Sadowski, dated February 1999.
   - ON-Site VOC analysis in minutes, dated 1999
   - HAPSITE Portable GC/MS Designed for site seeing, undated
   - HAPSITE, information at website:www.HAPSITE.com
7. HAPSITE Portable GC/MS, User's Guide, part number 074-256.
8. INFICON HAPSITE Portable GC/MS Analytical System, Key/Basic Operator Training Section I Training Modules, undated.
9. INFICON HAPSITE Portable GC/MS Analytical System, Key/Basic Operator Training Section III Application/Procedures.
10. INFICON QA-91 Quality Assurance Manual, dated 09-19-00.
11. NSAI, National Standards Authority of Ireland, Certificate of Registration of Quality System to I.S. EN ISO 9001:1994; Registration No: M877, Registration date: May 17, 1993, last amended on September 12, 2002; this certificate remains valid to December 14, 2003.
12. Two PT results of soil testing received from RTC. Sample 0011-605 VOAs, commencement date: May 7, 2001; Sample: 0109-6-8 VOAs – high Level, and 0109-609 VOAs – low level, commencement date: August 16, 2001.
13. US Patent & TradeMark Office, United States Patent 5,426,300, Voss, et. al., Portable GCMS system using getter pump, Appl. No:123755, file: September 1993, Current U.S. Class: 250/288; 250/289; Intern'l Class: Hoij049/04; H0ij049/24; Field of search: 250/288, 288A, 289.
14. US Patent & Trademark Office, United States Patent 5,401,298, Voss; Sorption pump, Appl. No. 123140; Field: September 17, 1993; Current U.S. Class: 96/134; 96/147; 417/48; Intern'l Class: Boid 053/04; Field of Search: 95/43, 45 96/4,134,147,152 417/48-51.
15. Analysis GC/MS Analysis of Water by Equilibrium Headspace, Standard Operating Procedure SOP #8 Rev. 0, Effective date May18th, 2001.
16. GC/MS Analysis of Soil by Equilibrium Headspace, Standard Operating Procedure SOP #9 Rev. 0, Effective date May18th, 2001.

17. VOC Soil Gas Analysis by Field-Portable GC/MS of by, Standard Operating Procedure SOP #7 Rev. 0, Effective date May18th, 2001.
18. Data package of Alameda Site 25 groundwater analysis run on May 30, 2001 with calibration and continuing calibration file and chromatogram.
19. Data package of Alameda Site 25 groundwater analysis run on June 1, 2001 with calibration and continuing calibration file.
20. Interlaboratory Comparison of soil Gas Radian Tracy Army Deport Project, 47 samples, received from Craig Crume on August 10, 01.
21. Records of instrument run log pages, received on September 5, 2001.
22. Water QC data received from DTSC Hazardous Material Laboratory.
23. Groundwater data of Alameda Site 25, provided by IT Corporation with the approval of US Department of the Navy. Analysis performed by a Navy contracted laboratory APCL.
24. Method Detection Limit studies for water and soil gas samples, 7 pages, received from Field Portable Analytical on Jan. 3, 2002.
25. Study on 1,2-Dibromo-3-Chloropropane, received by e-mail on February 11, 2002 sent by Carol Thielen of INFICON.
26. HAPSITE Field-Portable GC/MS, The next generation of on-site Analysis, a brochure on new HAPSITE.
27. A list of Warfare Chemical Agents that can be detected by HAPSITE, provided by Carol Thielen.
28. Remedial Investigation Work Plan for Operable Unit-5, Alameda Point, Alameda, California, prepared for U.S. Department of the Navy by Neptune and Company, Inc. June 4, 2001.
29. INFICON ^{TM} Terms and Conditions of Sale.
30. HAPSITE Portable GCMS, Emergency Response technology Program (ERT Program) Robert C Byrd at national Technology Transfer Center.
31. Data set received from IT OHM, three pages of soil gas data collected from January to April 2002, results of spiked sample analysis with QC data.
32. Photos and presentations provided by INFICON.

B. Relevant Literature

1. U.S. EPA SW 846 Method 3810 Headspace, September 1986.
2. U.S. EPA SW 846 Method 5021 Volatile organic compounds in soil and other solid matrices using equilibrium headspace analysis. Revision 0, December 1996.
3. U.S. EPA SW 846 Method 5030B Purge-and-Trap for aqueous samples, Revision 2, December 1996.
4. U.S. EPA SW 846 Method 5035 Closed-system Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December 1996.
5. U.S. EPA SW 846 Method 8260B Volatile Organic Compounds by gas Chromatography Mass Spectrometry, Revision 2, December 1996.
6. APCL Standard Operation Procedure F- 63 VOCs by GC/MS 8260B, version 8.0 Revision dated 08/2000.
7. Demonstration Plan for Wellhead Monitoring Technologies, prepared by Environmental Characterization and Monitoring Department Sandia National Laboratories and Savannah

River technology Center, sponsored by Consortium for Site Characterization Technology, NERL, Office of Research and Development, EPA.

8. Analysis of Volatile Organics by Field-Portable GC/MS, by Craig Crume, Environmental Testing & Analysis. May/June 2001 p. 25-26.
9. A Basic Primer on Membrane Introduction Mass Spectrometry, by Scott Bauer, Environmental Testing & Analysis March/April, 2001 p.17-23, .
10. Environmental and Forensic Applications of Field-Portable GC-MS: An Overview, B.A. Eckenrode, J. Am. Soc. Mass Spectrum Vol. 12, No.6, p 682-693, 2001.
11. Comparison of Data Quality Produced by an on-site field GC/MS and off-site permanent laboratory GC/MS: support of a cleanup action at an inactive drum recycling facility. S. P. Schuetz, P. J. Solinske, D. B. Mickunas, A.M. Humphrey, and R. D. Turpin, J. of Hazardous Materials Vol. 43, 67-75, 1995.
12. User's comments from Mike Walsh, San Antonio Fire Department.
13. Using a Field-Portable Gas Chromatography/Mass Spectrometer (GC/MS) for organic Vapor Identification and Quantitation. 14 pages, provided by Kirk Meekin Industrial Hygiene and Safety Group, Los Alamo National Laboratory.
14. U.S. EPA Compendium Method TO-14A Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specifically Prepared Canisters With Subsequent Analysis By Gas Chromatography, January 1997.
15. U.S. EPA Compendium Method TO-15 Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specifically Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS).
16 Relationship Between Soil Vapor and Soil Matrix Measurements for Trichloroethene, Alan D. Hewitt, Environmental Testing & Analysis Vol. 8, No. 3 May/June 1999.
17 Determination of Compliance with Exposure Limits for Mixtures of Volatile Organic Compounds with a Field Portable GC/MS.
18 Guide for the Selection of Chemical and Toxic Industrial Material Detection Equipment for Emergency First Responders, NIJ Guide 100-00 Volume I and II, June 2000, US Department of Justice.
19 Using Dynamic Field activities for On-Site Decision Making, EPA/540/F-03/011, OSWER 9200.1-47FS May 2003.

EVAUATION TEAM AND REPORT AVAILABILITY

The professional team of this evaluation include Ruth R. Chang, Ph.D. (Project Manager), Cynthia Digman, B.S. (QA officer), Diamon Pon, M.S. (Chemistry) at the Department of Health Services, Environmental Health Laboratory, and Garbin Orlando, B.S. (Chemistry) and Greg Williams, P.E. (Program Manager). The team members acknowledge Bart P. Simmons, Ph.D. and Bruce E. Labelle, Ph.D. for their comprehensive and critical reviews of this evaluation report.

Copies of this evaluation report are available from the California Department of Toxic Substances Control's, Office of Pollution Prevention and Technology Development, 1001 I Street, P.O. Box 806, Sacramento, California 95812-0806, or from the project manager.

The following text was published in the California Regulatory Register on March 19, 2004, Register Volume No. 12 - Z page 368 – 371. Certificate Number (04-01-042) became effective upon this publication for a period of three (3) years unless revoked in accordance with Section 68070 of Title 22 of the California Code of Regulations.

CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY

DEPARTMENT OF TOXIC SUBSTANCES CONTROL

Final Decision to Certify a Hazardous Waste Environmental Technology

The California Environmental Protection Agency, Department of Toxic Substances Control (DTSC) hereby certifies the following company's hazardous waste environmental technology:

INFICON®, INC. HAPSITE® Portable Gas Chromatograph Mass Spectrometer, a Field and Laboratory Instrument for the Measurement of Volatile Organic Compounds.

California Health and Safety Code section 25200.1.5 authorizes DTSC to certify the performance of hazardous waste environmental technologies. Hazardous waste environmental technologies are certified pursuant to regulations found in Title 22 of the California Code of Regulations (CCR 22), Chapter 46, section 68000 et seq. Only technologies that are determined not to pose a significant potential hazard to the public health and safety or to the environment when used under specified operating conditions may be certified. The purpose of the certification program is to provide an in-depth, independent review of technologies to facilitate regulatory and end-user acceptance and to promote and foster growth of California's environmental technology industry.

DTSC makes no express or implied warranties as to the performance of the manufacturer's product or equipment. The end-user is solely responsible for complying with the applicable federal, state, and local regulatory requirements. Certification does not limit DTSC's authority to require additional measures for protection of the public health and the environment.

By accepting certification, the manufacturer assumes, for the duration of certification, responsibility for maintaining the quality of the manufactured equipment and materials at a level equal or better than was provided to obtain certification and agrees to be subject to quality monitoring by DTSC as required by the statute under which certification is granted.

DTSC's proposed decision to certify was published in the California Regulatory Notice Register, Volume 2004, No. 4-Z, pages 95-97, of January 23, 2004 and has been subject to public review and comment. Written comments were not received.

An Evaluation Report supporting the Department's decision is available for review at the Hazardous Materials Laboratory and can be emailed upon request to Dr. Ruth Chang (see below) or can be obtained from DTSC web site (http://www.dtsc.ca.gov/ScienceTechnology/index.html).
California Environmental Protection Agency, Department of Toxic Substances Control, Hazardous Materials Laboratory, 700 Heinz Avenue, Berkeley CA 94710 - 2737, Attn.: Dr. Ruth R. Chang (510) 540-2651, rchang@dtsc.ca.gov.

A description of the technology to be certified, the proposed certification statement, and the certification limitations for the technology of the company listed above follow.

CERTIFICATION PROGRAM FOR HAZARDOUS WASTE ENVIRONMENTAL TECHNOLOGIES

TECHNOLOGY CERTIFICATION

Technology:

HAPSITE® Portable Gas Chromatograph Mass Spectrometer, a Field and Laboratory Instrument for the Measurement of Volatile Organic Compounds.

Manufacturer:

INFICON®, Inc., Two Technology Place, East Syracuse, NY 13057, Tel. 800-223-0633, http://www.INFICON.com

Technology Description

HAPSITE technology is based on the principle of quadrupole GC/MS, using high-energy electron impact ionization. The sample components are separated by a gas chromatograph (GC) column and passed into a mass spectrometer (MS) via a membrane interface. The selective membrane is permeable for volatile organic compounds (VOCs), but excludes inorganic constituents, such as nitrogen gas, from the MS. Compound identifications are based on matching ion spectra in the National Institute for Standards and Technology (NIST) library. The HAPSITE is designed to analyze volatile organics in a gas phase. In conjunction with a headspace equilibrium sampling accessory, the instrument has the capability to detect the chemical equilibrium concentration in the vapor phase to measure VOCs from liquid and solid samples. The technique applies to chemicals typically with molecular weights of 45 to 300 amu, and with boiling points approximately from -50ºC to +180ºC. The internal standard gas is used as mass calibrator for compound identification and quantitation. The HAPSITE system is lightweight, completely self-contained and portable for field applications. In the field-portable mode, with a hand control unit, the analysis can be performed at the sampling point for emergency response. In the transportable mode, the HAPSITE mounted on a service module can be operated in a van for on-site analysis. In the stationary mode, the HAPSITE can be set up as laboratory equipment by using the carrier gas from a high-pressure cylinder. The instrument is loaded with software for automatic instrument calibration and with methods for sampling and analysis. The analytical procedures for air (including vapor and gas), water, and soil analysis are established by INFICON for environmental applications.

Certification Statement

Under the authority of section 25200.1.5 of the California Health and Safety Code, the Department hereby certifies the performance of the HAPSITE Portable Gas Chromatograph - Mass Spectrometer manufactured by INFICON, Inc, as a Field and Laboratory Technology for the measurement of volatile organic compounds in environmental media as specified herein. According to the standard operating procedures established by the manufacturer, the HAPSITE system is capable of measuring most of the compounds listed under EPA Method 8260B in air, water, soil and soil gas.

The HAPSITE Practical Quantitation Limits (PQLs) are compound and matrix specific. INFICON defines the Practical Quantitation Limit as the lower bound of the calibration range and represents a peak-to-peak signal to noise ratio of 10:1. For those chemicals specified by INFICON, the HAPSITE Practical Quantitation Limits are 5 to 20 µg/L for water analysis, 0.2 to 0.5 ppmv for vapor phase analysis, and 10 to 40 µg/kg for soil analysis. Under normal environmental conditions, the relative standard deviation (RSD) of replicate analysis is expected to be = 20% and the recoveries expected to be ± 25% of the spiked values over the instrument calibration range. With an established 5-point calibration curve and appropriate quality control and quality assurance (QA/QC) program, the groundwater data obtained from HAPSITE analysis are comparable to that of EPA Method 8260B. The air QC study and the soil gas analysis of VOC contaminated sites indicated the HAPSITE data were well correlated to that of EPA Method TO-14 and Method TO-15. Relative to laboratory methods, GC and GC/MS, the HAPSITE has greater dynamic range to analyze samples up to ppm or percent level without over-saturating the instrument. For soil analysis, the HAPSITE reported values obtained from proficiency testing for the high and low level soil were within the acceptable limits established by Resource Technology Corporation (RTC), the proficiency test sample provider approved by the National Voluntary Laboratory Accreditation Program (NVLAP). However, due to the heterogeneity of environmental soil, the applications of HAPSITE on soil analysis based on equilibrium sampling warrants further investigations that analyze a wide range of VOCs in different soil types at various concentration levels. The HAPSITE measurement system has been demonstrated to be a viable cost effective technology to support site characterization, cleanup and remediation activities.

The HAPSITE is specifically designed for field use. With the advantages of fast on-site analysis, the INFICON HAPSITE significantly improves the sample turnaround time to generate data in a timely manner for the protection of public health and the environment.

Limitations of Certification

The Department makes no express or implied warranties as to the performance of the manufacturer's product or equipment. The Department has not conducted all the bench or field tests to confirm the manufacturer's performance data. Nor does the Department warrant that the manufacturer's product or equipment is free from any defects in workmanship or material caused by negligence, misuse, accident, or other causes.

The Department believes, however, that the manufacturer’s product or equipment can achieve performance levels set out in this Certification. Said belief is based on a review of the data submitted by the manufacturer and other information (See “Basis for Certification” below), and is also based on the use of the product in accordance with the manufacturer’s specifications.

This certification is subject to the regulations found in Title 22 of the California Code of Regulations (CCR 22), Chapter 46, section 68000, which include the duration of the Certification, and the procedures for certification amendments and decertification.

By accepting this Certification, the manufacturer assumes for the duration of the Certification, responsibility for maintaining the quality of the manufactured materials and equipment at a level equal or better than was provided to obtain this Certification and agrees to be subject to quality monitoring by the Department as authorized by the law under which this Certification is granted.

Specific Conditions

INFICON shall follow their established QA/QC program to ensure that the materials used in manufacturing and the quality of instrument meet the standards certified under ISO-9001.

INFICON shall maintain their standards for ensuring that users receive appropriate training in operation and maintenance of the instrument. For environmental applications, the method detection limit or quantitation limit, precision, and bias of the HAPSITE technology must be evaluated to ensure meeting the project-specific requirements. The surrogate compounds must be added to the environmental medium to evaluate the matrix effects and to validate the instrument performance. The analysis of blank samples must be performed as necessary to minimize cross-contamination. The quality control samples must be included in the operation as specified in the quality assurance project plan.

Through updates of user guides, the manufacturer shall inform the user of environmental and experimental parameters which potentially affect the performance of the system, as they become known to the manufacturer.

Users should follow the manufacturer's instructions for installation, operation, and maintenance of the instrument. Users should develop and follow a plan in accordance with their facility's quality management system for validating the system at appropriate intervals according to the guidance set for the HAPSITE system.

Basis for Certification

The proposed certification of this technology is based on a comprehensive evaluation conducted by the Hazardous Materials Laboratory (HML) in the California Department of Toxic Substances Control. HML reviewed instrument performance data submitted by the INFICON and field data generated by independent third parties. In addition, HML participated in independent studies evaluating the system's performance in air and soil analyses. HML staff also contacted end users to obtain additional information on performance and reliability. An evaluation report prepared by HML provides details of the evaluation.

Recommended Applications

The INFICON HAPSITE Portable Gas Chromatograph-Mass Spectrometer is intended for the measurement of volatile organic compounds in the field and in the laboratory. The HAPSITE technology operating in accordance with conditions established by the manufacturer can serve as a viable alternative for the measurement of volatile organic compounds in the environment. Applications include: (1) long term environmental monitoring of the chlorinated and aromatic hydrocarbons in air, water and soil; (2) detection and identification of toxic chemicals and hazardous materials released from industrial incidents; (3) fast on-site analysis to expedite site cleanup activities and to increase the number of sample analyses of a site to reduce data uncertainty.

Regulatory Implications

DTSC's certification does not change the regulatory status of field and laboratory measurements for volatile organic compounds in air, water, and soil matrices. This certification is intended, however, to facilitate and encourage the acceptance of this technology where a project's data quality objectives can be met by its use. To this end, regulatory programs are encouraged to consider the Department's findings regarding this technology, depending on each program's objectives and constraints. State-regulated facilities may contact state permitting officers regarding the use of the technology for the analysis of volatile organic compounds in the field and laboratory. Other local and state government permitting authorities may take this certification under consideration when making their permitting decisions. Project managers may consider using this technology where its use can contribute to the project.

Duration of Certification

Unless amended or revoked for cause, this certification will remain in effect for three years from the date of issuance.

APPENDIX A - MDL Studies for Water and Air Analysis

(Attachment)

APPENDIX B - Composition and Concentration of ETV PE Samples

(Attachment)

Appendix C - Procedures for the Preparation of QC samples

(Alameda Site 25 study)

Preparation of QC Samples

The VOC-free water was prepared by the Sanitation & Radiation Laboratory Branch in the DHS. Tap water was boiled for 1 to 2 hours with simultaneous purging with helium. The bottle was then sealed and kept in a refrigerator for preparing the trip blank and PE samples.

PE samples were prepared from quality control standards obtained from Environmental Resource Associates (ERA), Catalog No.710 for volatiles (Lot No. 608). Table 16 shows the certified values of 20 VOCs in the PE sample. To prepare sufficient numbers of PE samples for the study, 25 µl of standard solution were diluted to 500-mL in a volumetric flask containing VOC – free water. After thoroughly mixing, each sample was taken by filling to the top of a 40-mL VOC vial. In preparation for the unexpected situation, each sample was provided in duplicate. A total of 12 PE samples were taken from this preparation for the following distribution: one duplicate to HML, one duplicate to APCL and 4 duplicates to FPA for the analyses.

The MS and MSD were prepared by spiking a mixture of VOA and MTBE standard solutions (composition and concentration provided below) into a low level groundwater sample collected from Site 25, P181-MW46. The spiked concentration was 40 µg/L. In this preparation, 20 µl of VOA mix and 10 µl of MTBE standards were added to 500 ml of groundwater in a volumetric flask. After thoroughly mixing, each water sample was taken by filling to the top of a 40-mL VOC vial. Each sample was provided in duplicate. Twelve MS samples were taken from this sample preparation. Two sets of MS/MSD were sent to HML, APCL, and FPA for analysis.

Composition and concentration of spiked standards:

VOA Matrix Spike Mix (Supelco, Cat. No: 48102) contains Benzene, Chlorobenzene, Toluene, Trichloroethylene, and 1, 1-dichloroethylene with concentrations varied from 944 to 988 µg/ml ± 0.5% in methanol.

Methyl Tert-Butyl Ether (Supelco, Cat. No. 48483) contains 2000 µg/ml ± 0.5% in methanol.

Appendix D - PT Results of Soil Analysis

(Attachment)