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OPERATION OF THE HAPSITE FIELD PORTABLE GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) (TRIAD GC/MS - Based on EPA/TO-15A)

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1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the operation of the Inficon HAPSITE field-portable gas chromatograph/mass spectrometer (GC/MS) used by the Scientific, Engineering, Response and Analytical Services (SERAS) to analyze volatile organic compounds (VOCs) in air samples collected in Tedlar bags or SUMMA canisters, or in water and soil samples using headspace injection. The HAPSITE is capable of providing on-site results with quality comparable to conventional laboratory GC/MS methods. The instrument is capable of measuring sample concentrations in the parts per billion (ppb) to parts per million (ppm) range.

The data generated using this SOP meets the Screening Data objective for a quick, preliminary assessment of site contamination, and provides analyte identification and quantification. Screening data without associated confirmation data are generally not considered to be data of known quality. The field screening data may be definitively confirmed by the analysis of additional samples collected in SUMMA canisters and sent to a laboratory to be analyzed using methods and quality control (QC) criteria associated with definitive data.

2.0 METHOD SUMMARY

The methods for air analysis, air monitoring and headspace analysis in water/soil samples are summarized below. These methods employ internal standards and a multi-level calibration curve for quantitation of VOCs. The methods are applicable for both HAPSITE 1 and HAPSITE 2. The reporting limits (RLs) are 80 parts per billion by volume (ppbv) for air, 10 ppb or micrograms per liter ($\mu\text{g/L}$) for water, and 40 ppb or micrograms per kilogram ($\mu\text{g/kg}$) for soil samples.

2.1 Air Analysis

A minimum of 250 milliliters (mL) of a canister sample is transferred to a clean 1-liter (L) Tedlar bag. The bag is then connected to the probe of the HAPSITE. An aliquot of the sample is transferred from the Tedlar bag through the probe's heated transfer line into a gas sampling loop where it is mixed with the built-in internal standard mix. The contents of the sample loop are subsequently injected onto the GC column for compound separation. Detection of the analytes is performed using a quadrupole mass spectrometer. The target analytes and two internal standards typically used for air analysis are listed in Table 1, Appendix A.

2.2 Air Monitoring

This operation is set up from the HAPSITE instrument's front panel. Air monitoring is performed by directing the probe toward the areas of interest for detection that is performed directly in the quadrupole mass spectrometer. After the monitoring is completed, the acquired data is downloaded to an external computer for processing.

2.3 Headspace Analysis

The HAPSITE is interfaced with a Headspace Sampling Unit (HSU) for the analysis of waters and soils. Approximately 20 milliliters (mL) of a water sample or 5 grams (g) of a soil sample dissolved in 20 mL of



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deionized (DI) water are transferred to a 40-mL screw cap glass vial and spiked with an internal standard mix. The vial is sealed with a Teflon-lined septum and placed in the HSU heat chamber at 60 degrees Celsius (°C) to equilibrate for at least 22 minutes. The sample is transferred using a nitrogen carrier gas, displacing the gas phase in the vial through a heated transfer line into a gas sampling loop. The contents of the sample loop are then injected onto the GC column. Detection of the analytes is performed using a quadrupole mass spectrometer. Typical target analytes and internal standard/surrogate are listed in Table 2, Appendix A.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Air Samples

Air samples are collected in one-L Tedlar bags in accordance with SERAS SOP #2102, *Tedlar Bag Sampling* or in SUMMA canisters using SERAS SOP #1704, *SUMMA Canister Sampling*.

Tedlar bag samples are stored in either a clean cooler or an opaque plastic bag at ambient temperature to prevent photodegradation. Samples analyzed by the HAPSITE on-site should be accompanied by a chain of custody (COC) record detailing sample locations, sample numbers, date collected, sample matrix, and sample volumes. The COC record should agree with the information recorded on the Tedlar bag labels. Samples must be analyzed within 24 hours, preferably within 12 hours, following sample collection since VOCs may escape or become altered.

Canister samples must have the canister valve closed and the sampling port capped after the sample is collected. Samples analyzed by the HAPSITE on-site should be accompanied by a COC record detailing the sample locations, sample numbers, date collected, sample matrix, and sample volumes. The COC record should agree with the information recorded on the attached identification tag. Canister samples do not require refrigeration. The holding time is compound dependent but most VOCs can be recovered from canister samples near their original concentrations for up to 30 days.

3.2 Water Samples

Water samples are collected in triplicate in 40-mL VOC vials in accordance with SERAS SOP #2007, *Groundwater Well Sampling* or SOP #2013, *Surface Water Sampling*. The vials must be completely filled with no visible air bubbles. Samples are stored in a cooler on ice and maintained at 4°C for up to seven days until analysis. Once an aliquot is transferred to a clean 40-mL vial for headspace injection, the sample should be analyzed within 12 hours.

3.3 Soil Samples

Soil samples are collected according to SERAS SOP# 2012, *Soil Sampling*. Samples are stored in a cooler on ice and maintained at 4°C for up to seven days until analysis. Once a portion of sample is transferred to a clean 40-mL vial for headspace injection, the sample should be analyzed within 12 hours.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS



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Structural isomers having co-eluting retention times and identical mass spectra will be reported as a total concentration (e.g., meta-xylene and para-xylene reported as m&p-xylenes). The HAPSITE is limited to the determination of VOCs with sufficient volatility to be removed from the sample based on the headspace technique. Compounds such as 1,2,4- and 1,2,3-Trichlorobenzene cannot be determined using this instrument. The standard chromatographic run time needs to be extended for HAPSITE 1 for the determination of 1,3,5-Trichlorobenzene due to isothermal GC oven used.

5.0 EQUIPMENT/APPARATUS

- HAPSITE Field Portable GC/MS, controlled internally from the front panel or externally from a PC. The instrument can be attached to a service module (turbo pump) for long term operation.
- Headspace Sampling Unit, interfaced with the HAPSITE GC/MS
- Tedlar bags, 1-L capacity (SKC, Inc. or equivalent)
- Canisters, passivated, 6-L (Restek or equivalent)
- Glass septum vials, 40-mL with screw top (Supelco or equivalent)
- Syringes, 10, 25, 50, 100, 500, 1000 microliter (μ L) and 25-mL gas-tight (Hamilton or equivalent)
- Balance, top-loading, capable of weighing solid samples to 0.01 g, calibrated prior to use in field

6.0 REAGENTS

6.1 Air Analysis

- Internal Standard Mix, commercially available from the manufacturer, containing 1,3,5-tris-(trifluoromethyl)benzene (TRIS) and bromopentafluorobenzene (BPF) at approximately 5 and 10 parts per million by volume (ppmv), respectively, for tuning and quantitation
- External Tuning Standard, 4-bromofluorobenzene (BFB) at a nominal 1.0 ppmv in a canister
- Calibration Standards, minimum of three concentrations, typically at 0.2, 0.5, and 1.0 ppmv, prepared in canisters
- Nitrogen, ultra high purity (Praxair, Inc. or equivalent), used as carrier gas and for sample dilution

6.2 Headspace Analysis

- Stock Internal Standard/Surrogate (IS/Surr) Mix, 5000 ppm, containing fluorobenzene, dibromofluoromethane, chlorobenzene-d5 and 4-BFB as internal standards and 1,2-dichloroethane-d4, toluene-d8, and 1, 4-dichlorobenzene-d4 as surrogates (Ultra Scientific or equivalent).



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- Working IS/Surr Mix, 200 ppm. - Make a 1:25 dilution of the stock IS/Surr mix in methanol.
- Stock Calibration Standards, EPA 624 Standard Mix A, B and C, 1000 to 2000 ppm (Ultra Scientific or equivalent). Other additional VOCs of interest may be added at the client's request.
- Working Calibration Standards, 200 ppm - Prepare from the stock calibration standard solution in methanol as follows:

	Stock Conc. (ppm)	Volume of Stock Std. (μ L)	Volume of Methanol (μ L)
Mix A/B/C/Others	2000/1000	100/200	900/800

Each of the working solutions is prepared in a 2-mL GC vial with screw top and stored in a freezer with minimal headspace when not in use.

- Purge water, commercially available, suitable for VOC analysis
- Methanol, purge and trap quality
- Working Matrix Spike (MS) Standard, 250 ppm, containing 1,1-dichloroethene, benzene, trichloroethene, toluene and chlorobenzene (AccuStandard or equivalent), independent of the source used for calibration. Spike 4 μ L of this solution to two separate aliquots of a sample for the matrix spike/matrix spike duplicate (MS/MSD) analysis.

7.0 PROCEDURE

Detailed procedures for air and headspace analyses using the HAPSITE can be found in Appendix B.

7.1 Startup and Equilibration

1. For air analysis, connect the HAPSITE probe. Refer to HAPSITE User Manual to startup the instrument. Refer to Appendix B.1.1.
2. Wait for at least 40 minutes to allow the HAPSITE to equilibrate.

7.2 Tuning

Both TRIS and BPFB are used for internal tuning of the HAPSITE. Refer to Appendix B.1.2 to perform the tuning. After the internal tuning is passed, BFB is used to check the HAPSITE tuning.

1. For air analysis, transfer BFB to a Tedlar bag. Refer to Appendix B.1.3 to setup this acquisition.



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2. For headspace analysis, refer to Appendix B.2.2 to prepare the IS/Surr mix of 250 ppb (ng/mL) in duplicate. Refer to Appendix B.2.4 to perform the BFB tune check.

7.3 Air Analysis Procedure

1. Refer to Appendix B.1.3 to setup acquisition. Run a system blank by injecting nitrogen that has been placed in a Tedlar bag.
2. If the result of the system blank indicates no interference found in the instrument, setup and generate a three-point initial calibration.
3. Concentrations at 0.2, 0.5, and 1.0 ppmv of the calibration standards are used for full scan. Refer to Appendix B.1.3 to run the three standards.
4. Once the initial calibration is acceptable, refer to Step 1 of Appendix B.1.4 to run samples and Appendix B.3 to process the results.
5. Record all sample acquisitions in the HAPSITE injection log. Use the equation in Section 8.1 to calculate analysis results.
6. To continue to run samples after 12 hours, the calibration standard of 0.5 ppmv is used as a continuing calibration check (CCC). Refer to Appendix B.1.4 to run CCC, followed by acquiring samples.

7.4 Headspace Analysis

1. Refer to Step 7.2.2 to inject BFB to tune the HAPSITE. The BFB must be injected every 12 hours during sample analysis.
2. While both the HAPSITE and the HSU are under equilibrium, prepare the working solutions of IS/Surr mix, standard mix A, B, C, and any other VOCs of interest from the individual stock solutions.
3. Once the BFB tune criteria passes, prepare five levels of calibration standards based on the following table to generate an initial calibration:

Calibration Level	Calibration Standard Concentration (ppb)	Stock Standard Concentration (ppm)	Volume of Stock Standard (μ L)	Volume of IS/Surr Mix (μ L)	IS/Surr Mix Concentration (ppb)
1	10	200	1.0	5.0	50
2	20	200	2.0	5.0	50



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3	50	200	5.0	5.0	50
4	100	200	10	5.0	50
5	200	200	20	5.0	50

4. Run a system blank by transferring 20 mL of DI water into a 40-mL vial (no IS/Surr mix is added). If the result of the system blank indicates that no target analytes are found, refer to Appendix B.2.5 to run the five calibration standards. Refer to Section B.3 to generate the initial calibration report (Figure 13).

5. Sample Analysis

Once the initial calibration passes, refer to Appendix B.2.6 to analyze samples. Record all sample acquisitions in the HAPSITE instrument log. Refer to Appendix B.3 to process sample data files and print out sample quant reports. Use the equation in Section 8.2 to calculate analysis results. To continue running samples after 12 hours, use the 50 ppb calibration standard as the continuing calibration check (CCC). Target analytes in samples are quantitated based on the average response factors in the initial calibration.

The Service Module is used for continuous operation of HAPSITE. Refer to the HAPSITE User Manual for installation and operation. When the operation is finished, refer to HAPSITE User Manual to shutdown the instrument.

- 7.5 Air Screening

Refer to HAPSITE User Manual for conducting air screening in field operations.

8.0 CALCULATIONS

- 8.1 Air Analyte Concentration

VOC concentration in air are calculated based on the following equation:

$$C, \text{ ppmv} = C' \times \text{DF}$$

where:

C' = Concentration calculated by the HAPSITE
DF = Dilution factor (SUMMA canister dilution to create a positive pressure)

- 8.2 Water Sample Concentration

$$\text{Analyte Concentration, } \mu\text{g/L} = C_s \times \text{DF}$$

where:



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C_s = Concentration, $\mu\text{g/L}$, calculated on the Chemstation quant report
DF = Dilution factor (20 mL/volume (mL) of sample used)

8.3 Soil Sample Concentration

For direct injection:

$$\text{Concentration, } \mu\text{g/kg} = C_s \times 20 \text{ mL}/W_t$$

where:

C_s = Concentration, $\mu\text{g/L}$ or ng/mL , calculated by the ChemStation quant report
DF = Dilution factor (See B.2.6.10 in Appendix B)
 W_t = Sample weight (e.g., 5g)

For methanol extractions:

$$\text{Concentration, } \mu\text{g/kg} = C_s \times 20 \text{ mL} \times \text{DF}/W_t$$

where:

C_s = Concentration, $\mu\text{g/L}$ or ng/mL , calculated by the ChemStation quant report
DF = Dilution factor (See B.2.6.10 in Appendix B)
 W_t = Sample weight (e.g., 5g)

8.4 Surrogate Spike Recoveries

Calculate surrogate standard recovery on all samples, blanks, and spikes by the following equation:

where:

Q_D = Quantity determined by analysis
 Q_A = Quantity added to sample

Alternatively, surrogate recoveries on all blanks, spikes, and samples are reported from the Chemstation quant report.

8.5 Matrix Spike/Matrix Spike Duplicate Recoveries

Spike recoveries and relative percent difference (RPD) of each matrix spike compound in the MS/MSD are calculated according to the following equations :



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where:

SSR =	Spike	sample result
SR =	Sample	result
SA =	Spike	added

where:

PD = Relative percent
difference

MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

NOTE: RPD is always expressed as a positive value.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Air Analysis

1. The HAPSITE will be tuned by the built-in internal standard mix (i.e., TRIS and BPFB) until the criteria is passed (Figure 3).
2. A minimum of a three-point initial calibration must be generated before sample analysis. The acceptable %RSD of the 3-point initial calibration is $\leq 30\%$.
3. A method blank is a Tedlar bag containing nitrogen. It must be run after acquiring the initial calibration or CCC to evaluate the cleanliness of the HAPSITE. A system blank may be analyzed after a highly contaminated sample is run to check for carryover. The method blank must be free of any target analytes prior to running any samples.
4. The percent difference (%D) for the CCC must be $\leq 25\%$ for the analysis to continue. The initial response factors of target analytes are used for subsequent sample quantitation. Refer to Section



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B.3, Appendix B.

5. It is advisable that ten percent (10%) of all samples received are to be analyzed in duplicate. The RPD should be <30%.
6. The typical RL for air analyses is 0.08 ppmv.

9.2 Headspace Analysis

1. After the internal tuning passes, BFB prepared at 250 ppb is used to check the HAPSITE tuning. The BFB criteria must be passed before running an initial calibration or CCC.
2. A five-point initial calibration must be generated before sample analysis. The acceptable %RSD of the 5-point initial calibration curve is $\leq 30\%$. Target analytes in samples are quantitated based on their average response factors from the initial calibration.
3. A method blank is an aliquot of 20 mL deionized water spiked with the IS/Surr standard mix. It is run after generating initial calibration or the CCC to verify the absence of any target compounds. The method blank must be free of any target analytes prior to running any samples. A system blank may be run to check for carryover after a highly contaminated sample is analyzed.
4. The percent difference (%D) for the CCC must be $\leq 25\%$ for the analysis to continue. The initial response factors of target analytes are used for subsequent sample quantitation. Refer to Section B.3, Appendix B.
5. A MS/MSD may be run to evaluate the accuracy and precision of analysis, including possible sample matrix effects. The spike solution must be the second source different from the calibration standards.
 - One MS/MSD must be prepared every 20 samples.
 - The matrix spike solution specified in Section 6.2 must be used.
 - The quality control limits for recovery and RPD are listed below. The QC limits are advisory at this time and no further action is required if the recovery fall outside the limits.

<u>Compound</u>	<u>% Recovery</u>	<u>RPD</u>
1,1-Dichloroethene	70-130	30
Benzene	70-130	30
Trichloroethene	70-130	30
Toluene	70-130	30
Chlorobenzene	70-130	30

6. It is advisable that ten percent (10%) of all samples are analyzed in duplicate. The RPD should be <30%.



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6. The typical RL for headspace analysis is the lowest calibration standard at 10 ppb (10 $\mu\text{g/L}$) for water or 40 ppb ($\mu\text{g/kg}$) for soil.

9.3 Maintenance

No daily maintenance is needed for the HAPSITE instrument. Check the pressure of the internal standard can. Replace with a new can if the pressure falls in the red area. The total pressure (vacuum) of the HAPSITE is usually around 3.0×10^{-3} Pa. As the NEG pump consumes, total pressure starts to rise. If the total pressure exceeds 8.0×10^{-3} Pa, replace the NEG pump.

In the event that the HAPSITE response drops significantly, contact the manufacturer about replacing GC column or/and other instrument troubleshooting.

9.4 Manual Integrations

Manual integration of all target analytes, surrogates, and internal standards will be submitted for review. The manual integration results will be flagged with a “M” using the instrument software and verified during the data review process. Documentation of the manual integration of quantitation ion peaks must be included in the data package. Refer to SERAS SOP #1001, *Chromatographic Peak Integration Procedures*.

9.5 Initial Demonstration of Capability

Initial proficiency in VOC analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or for instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicates of a well-mixed reference standard is analyzed using the procedures outlined in this SOP. Calculate the average mean and the standard deviation (s). The Quality Assurance Officer (QAO) will tabulate the results from all of the analysts per matrix per parameter, and calculate control limits.

9.6 Work Assignment Field Change Form

A Work Assignment Field Change Form must be generated to initiate any on-site change in the scope of a project. This form must document the original scope of work that is being changed, the new scope and the signatures of the SERAS Task Leader or their designee and the Work Assignment Manager.

10.0 DATA VALIDATION

The data is reviewed using the Quality Assurance/Quality Control considerations listed in Section 9.0 by the analyst prior to submittal to the client to ensure that the instrument has been operated in accordance with this SOP and manufacturer’s recommendations and that all QA/QC checks have been performed. The Screening data objective requires that the calibration and detection limits be evaluated. All field analytical reports must be reviewed in accordance with Administrative Procedure (AP) #22, *Peer Review of SERAS Deliverables*.



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11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow EPA, OSHA, and laboratory health and safety practices. More specifically, Tedlar bag samples should be stored in cool place, away from direct sunlight and analysis area; and water/soil samples should be refrigerated until analysis. In addition, analysis area should have adequate ventilation.

12.0 REFERENCES

United States Environmental Protection Agency, 1999. *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. EPA /625/R-96/010b, Office of Environmental Research and Development, Compendium Methods TO-15.

National Environmental Laboratory Accreditation Committee (NELAC), *Quality Systems*, current approved version.

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APPENDIX A
Tables
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TABLE 1. VOC Target Analytes for Air Analysis

	<u>Analyte</u>	<u>Ret Time*</u>	<u>Quant Ion</u>
1	Chloromethane	1:10.19	50
2	Vinyl chloride	1:14.84	62
3	Chloroethane	1:15.13	64
4	Trichlorofluoromethane	1:21.72	101
5	1,1-Dichloroethene	1:24.00	61
6	Methyl chloride	1:24.14	49
7	trans-1,2-Dichloroethene	1:25.85	61
8	1,1-Dichloroethane	1:30.97	63
9	Trichloromethane	1:39.94	83
10	1,2-Dichloroethane	1:45.44	62
11	1,1,1-Trichloroethane	1:48.81	97
12	Benzene	1:53.74	78
13	Carbon tetrachloride	1:55.00	117
14	Dibromomethane	2:03.35	174
15	Bromodichloromethane	2:05.99	83
16	Trichloroethene	2:09.64	130
17	1,1,2-Trichloroethane	2:32.20	97
18	Toluene	2:40.38	91
19	Tetrachloroethene	3:22.41	166
20	Ethylbenzene	4:03.24	91
21, 22	m+p-Xylene	4:13.47	91
23	Styrene	4:38.02	104
24	1,1,2,2-Tetrachloroethane	4:45.67	83
25	o-Xylene	4:46.00	91
26	1,3,5-Trimethylbenzene	7:17.02	105
27	1,2,4-Trimethylbenzene	8:34.00	105
Internal Standards			
1	1,3,5-Tris(trifluoro methyl)-benzene (TRIS)	1:58.00	69, 75**
2	Bromopentafluorobenzene (BPFB)	3:47.00	117, 167**

* For reference only

** The secondary ions of the internal standards are used when their primary ions (i.e., 69 & 117) are interfered by sample matrix.



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TABLE 2. VOC Target Analytes and Internal Standards/Surrogates For Water/Soil Analysis

Analyte	Ret Time*	Quan Ion
1 Chloromethane	1:12.32	50
2 Vinyl chloride	1:13.50	62
3 Bromomethane	1:15.00	94
4 Chloroethane	1:15.13	64
5 Trichlorofluoromethane	1:22.72	101
6 1,1-Dichloroethene	1:23.30	61
7 Methyl chloride	1:23.49	49
8 trans-1,2-Dichloroethene	1:29.85	61
9 1,1-Dichloroethane	1:32.97	63
10 Chloroform	1:40.33	83
11 1,2-Dichloroethane	1:47.86	62
12 1,1,1-Trichloroethane	1:50.81	97
13 Benzene	1:54.82	78
14 Carbon tetrachloride	1:57.09	117
15 1,2-Dichloropropane	2:05.35	63
16 Bromodichloromethane	2:07.90	83
17 Trichloroethene	2:07.94	130
18 cis-1,3-Dichloropropene	2:23.00	75
19 trans-1,3-Dichloropropene	2:33.00	75
20 1,1,2-Trichloroethane	2:38.40	97
21 Toluene	2:47.38	91
22 Dibromochloromethane	2:59.00	129
23 Tetrachloroethene	3:22.00	166
24 Chlorobenzene	3:50.00	112
25 Ethylbenzene	4:10.00	91
26,27 m+p-Xylene	4:20.00	91
28 Bromoform	4:24.00	173
29 Styrene	4:44.70	104
30 1,1,2,2-Tetrachloroethane	4:51.00	83
31 o-Xylene	4:52.67	91
32 1,3-Dichlorobenzene	8:46.30	111
33 1,4-Dichlorobenzene	9:02.00	111
34 1,2-Dichlorobenzene	10:16.70	111



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TABLE 2.(cont) VOC Target Analytes and Internal Standards/Surrogates For Water/Soil Analysis

Internal Standards/Surrogates

1	Dibromofluoromethane (IS)	1:42.00	111
2	1,2-Dichloroethane-d4 (Surr)	1:46.00	67
3	Fluorobenzene (IS)	1:58.00	96
4	Toluene-d8 (Surr)	2:01.00	98
5	Chlorobenzene-d5 (IS)	3:48.00	117
6	4-Bromofluorobenzene (IS)	5:27.00	95
7	1,4-Dichlorobenzene-d4 (Surr)	9:00.00	115

* For reference only



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APPENDIX B
Operating Procedures
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B.1.1 Instrument Startup and Equilibration

1. Open the HAPSITE front control panel and insert the internal standard (IS) can into the lower slot. The pressure gauge should fall in the green area. Turn the power on and close the panel.
2. After the instrument initiation is complete, press [3] and [SEL] to activate the remote control (from the PC).
3. Go to a PC (e.g., a Dell laptop) that already displays programs on the screen. Double click the icon **HAPSITE** to open the main menu (Figure 1).
4. Double click the icon, **STARTUP**, and a control box appears. Click [**Startup**] to activate the HAPSITE temperature parameters (Figure 2). Wait for at least 40 minutes for the instrument to equilibrate.
5. After the instrument is equilibrated, click **Membrane Isolation to Open** and click **Filament/Multiplier to On**, followed by clicking [**Exit**] and [**Yes**] to exit the program.

B.1.2 Tuning Calibration

1. Go to the main menu and double click the icon, **TUNE**, to open the program. The bottom of the menu displays the internal standard pressure, carrier gas pressure, total pressure, and temperature of column, membrane, GC feed, and IS feed that should be around 60 °C.
2. Go to **File** and click **Read Tune/Cal File** to select a tune file (e.g., B020904).
3. Go to **Actions** and click **Calibrate** to calibrate the tune.
4. After the tune calibration is complete, the result box appears (Figure 3).
5. Wait for 20 minutes then go to **Actions** and click **Calibration Mass Plots**. A box, **Mass Plots**, appears.
6. Click mass 117 and [**Enter**] and the plot appears. The ion abundance of 117 should be at least 2,500,000.
7. Refer to HAPSITE User Manual to increase mass 117 abundance.
8. Go to **Actions** and click **Calibrate** to continue calibration of the tune. If all the mass ions status are OK and peak % of all mass ions is close to that shown in Figure 3, click [**Print Screen**].
9. Open a word program (e.g., **Word**) and click **Paste** to copy the tune. Indicate the date of the tuning and print out the copy. Click [**No**] to exit the Word program.
10. If some of the mass ions status are not OK, refer to HAPSITE User Manual to adjust each individual mass ion ratio.
11. Return to the **TUNE** program and open **File**. Click **Exit** and [**Yes**] to save the tune file before exiting the program.
12. Refer to HAPSITE User Manual for tuning when used in the SIM mode.

B.1.3 BFB Check

1. Go to **ACQUIRE**, and a box, **Acquire for HAPSITE**, appears. At this time, go to the terminal screen and open **My Computer**. Go to C:\Program files\hpsrun\data and create a new folder. Rename the new folder (e.g., 020105), before exiting **My Computer**.
2. Enter file names as follows:
Tune/Cal: current tune file (e.g., B020904)
Full Method: hdspace.fsm, and



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- Inlet Method:** to14.gch (for HAPSITE 1) or ai_nt_tp (for HAPSITE 2)
- Click **Data** and a second small **Acquire for HAPSITE** box appears. Open the new folder (e.g., 020105), followed by entering a new data file (e.g., ai020101, ai stands for air). A box, **Data File Title**, appears. Enter "BFB, 1 ppmv" on **Title Lane 1**, other information on **Title Lane 2**, and analyst's name on **Title Lane 4**. Click [OK]. A data file displays next to **Data**.
 - Connect the BFB Tedlar bag valve directly to the probe tip of the HAPSITE and open the valve.
 - Click **Startup Sequence** and then click **Start Run**.
 - After 1 minute of the runtime, close the Tedlar bag valve. Be sure to run this step in every acquisition.
 - Go to the main menu and open **PACQHAPS**. A program, **PACQ for HAPSITE**, appears.
 - Go to **File** and click **Open Datafile**. Click the data file in acquisition and click [OK].
 - PACQHAPS is used to monitor real-time acquisition.
 - After the BFB peak appears (the third peak), go to **Modes** and click **Zoom scan range** to zoom-in the BFB peak.
 - Go to **Modes** and click **Plot spectrum**. Move the arrow cursor to the apex of the peak and left-click the mouse. A mass spectrum appears.
 - Go to **Spectrum** and click **Specification Table**. A box, **Select Specification File**, appears. Click **bfb.spt** and then [OK].
 - The result of **Select Specification File** appears. If the status of all mass ions is pass (Figure 9), the HAPSITE passes the BFB tune check.
 - Disconnect the BFB Tedlar bag and prepare to run the three initial calibration standards.
 - After the run is finished, refer to B.3 to convert the data file to Chemstation file.
 - If the status of some mass ions is flagged (e.g., low, high, etc.), close the spectrum and plot spectra at other scans (e.g., M±1, M is the scan number at apex of the peak) and repeat Steps 10 through 12. If the status of some of the mass ions still is not pass, wait until the run finishes.
 - Exit **ACQUIRE** and go to **TUNE**. Repeat B.1.2 to do HAPSITE internal tuning.
 - The following table shows ion masses for the internal tune relates to ion massed in BFB tune. Adjust the ion masses % in internal tune relates to ion masses in BFB tune that fail to pass.

Ion mass in Internal Tune	Ion Mass in BFB Tune
50	50
74	75
167	174
193	176

- After the second internal tune is completed, exit **TUNE** and go to **ACQUIRE** to run the second BFB check.
- If neither of the two BFB runs pass the tune criteria, contact the HAPSITE manufacturer for assistance.

B.1.3 Standard/Sample Acquisition Setup

- Once the BFB check is done, go to the main menu and double click the icon, **ACQUIRE**, and a box, **Acquire for HAPSITE**, appears (Figure 4). At this time, go to the terminal screen and open **My Computer**. Go to C:\Program files\hapsrun\data and create a new folder. Rename the new folder (e.g.,



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- 20000301), before exiting **My Computer**.
2. Return to **Acquire for HAPSITE** and enter the file names for **Tune/Cal** (e.g., B020904), **Full Method** (e.g., to14.fsm) and **Inlet Method** (e.g., to14.gch). These files are created and modified from their default files.
3. Click **Data** and a second small **Acquire for HAPSITE** box appears. Open the new folder (e.g., 20000301), followed by entering a new data file (e.g., ai030101, ai stands for air). Enter the standard/sample information on **Data File Title** and click **[OK]**. A data file appears next to **Data**.
4. When all three calibration standards have run, refer to B.3 for using **AMDIS** to convert the HAPSITE data files to Chemstation data files for data processing (Figure 5).
5. Use the Chemstation to generate an initial calibration (Figure 6).
6. After the initial calibration is generated from the Chemstation, check that the %RSD of each analyte is $\leq 30\%$. If some of the analytes $> 30\%$, examine which calibration level contributes the high %RSD and rerun that calibration standard. Rerun the standard from a new canister, if necessary.
7. If the instrument is acquiring samples collected in Tedlar bags, connect the bag valve directly to the probe tip of the HAPSITE and open the valve. If it is acquiring standards/samples from canisters, transfer approximately 250 mL to a Tedlar bag before hooking up to the probe tip. (NOTE: Unless the canister pressure is at atmospheric pressure, the sample must be transferred to a Tedlar bag.)
8. Return to the program and click **Startup Sequence** and then click **Start Run**. A message, “**Wait for External Run Signal**” next to **Delay** on the top of the program is blinking. A message, “**PRESS RUN TO ACQUIRE**” also appears on the probe’s display panel. Press **RUN** button to start acquisition.
9. After a sample run is finished, enter a new data file on **Data** to continue another acquisition.
10. When sample acquisition is completed, click **Exit** and **[Yes]** to close the program.
11. Use the Chemstation to generate a quant report for each sample (Figure 7).
12. To acquire samples on SIM, follow Steps 2 through 7 but switch **Tune/Cal** to **1 msec** and switch **SIM Method** to a different file name (e.g., to14sim.fsm). The **Inlet Method** remains unchanged.
13. Information on the **Full method**, **Inlet Method**, and **SIM Method** is listed in Appendix D.1.

B.1.4 Continuous Sample Acquisition

1. If the analysis is continued the next day or later, run a mid-level calibration standard (e.g., 0.5 ppmv) as the continuing calibration check (CCC).
2. Refer to B.1.1 through B.2.4 to equilibrate and tune the HAPSITE. Refer to B.2.5 to run CCC.
3. Run **AMDIS** to convert the CCC data file to Chemstation data file.
4. Use the Chemstation to generate CCC report.
5. If the CCC passes the criteria ($\leq 25\%$), then continue to run a new batch of samples
6. If the CCC falls outside the criteria, re-run the second 0.5 ppmv calibration standard.
4. If the second CCC is still unacceptable, rerun the remaining two calibration standards (i.e., 0.2 and 10 ppmv) to generate a new initial calibration.
8. When the new initial calibration is acceptable, continue to run method blank and samples.

B.2 Headspace Analysis

B.2.1 Instrument startup and equilibration

Disconnect the probe and connect the HSU transfer line to the HAPSITE. Insert the internal standard can to the



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HAPSITE and turn the power on. After the HAPSITE initiation is complete, press [3] and [SEL] to switch to remote mode.

1. Go to the HAPSITE main menu on the PC and double click the **STARTUP** program. A box, **STARTUP for HAPSITE** appears.
2. Click **Configure Startup Sequence** and a program, **HAPSITE Startup Sequence Configuration**, appears (Figure 8). Click **Enable** in **HS Enable** and [OK].
3. The program returns to **STARTUP for HAPSITE**. Go to **HAPSITE Target Settings** and enter 60 for both **Oven Temp** and **Xfer Line Temp**.
4. Click **Startup** and wait for equilibration.

B.2.2 BFB Preparation

5. Use a 25-mL syringe to withdraw about 22 mL of DI water from a glass container.
2. Invert the syringe so that any air bubbles move to the top. Push the plunger slowly to purge the air bubbles out of the barrel.
3. Continue to push the plunger until it reaches the 20-mL mark. Pull the plunger back slightly.
4. Use a 10- μ L syringe to aliquot 1 μ L of the stock IS/Surr mix (i.e., 5000 ppm) and inject into the water in the syringe.
5. Transfer this solution to a 40-mL vial. Close the vial with an open-hole screw cap and a septum.
6. Repeat Steps 1 through 4 to prepare a duplicate. The concentration of the IS/Surr mix in water will be 250 ppb.
7. Load the two vials into the HSU heat chamber in a clockwise order (i.e., the first vial goes to Post#1 and the second goes to Post#2, as shown in Figure 9). Close the yellow cap and wait for at least 22 minutes for equilibrium.

B.2.3 Tuning

1. Internal tuning – After the HAPSITE is warmed up and equilibrated, follow B.1.2 to perform the internal tuning. Continue to finish the tuning and print out the report.
2. External tuning check – Use 4-BFB in the IS/Surr mix to tune the HAPSITE. When the IS/Surr mix is in equilibrium, go to the next section (B.2.4).

B.2.4 BFB Check

1. Go to **ACQUIRE**, and a box, **Acquire for HAPSITE**, appears. At this time, go to the terminal screen and open **My Computer**. Go to C:\Program files\hapsrun\data and create a new folder. Rename the new folder (e.g., 20000301), before exiting **My Computer**.
2. Enter file names as follows:
Tune/Cal: current tune file (e.g., B020904)
Full Method: hdspace.fsm, and
Inlet Method: hdspace.gch (for HAPSITE 1) or hs_nt_tp (for HAPSITE 2)
3. Click **Data** and a second small **Acquire for HAPSITE** box appears. Open the new folder (e.g., 20000301), followed by entering a new data file (e.g., hs030101, hs stands for headspace). Enter standard/sample information on **Data File Title** and click [OK]. A data file displays next to **Data**.



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4. Lift the needle assembly out of the empty vial and turn to Post #1 to pierce into the vial.
5. Click **Startup Sequence** and then click **Start Run**.
6. After 2 minute of the runtime, open the yellow cap and lift the needle assembly from the solution vial and return to the empty vial. Be sure to practice this step in every acquisition.
7. Go to the main menu and open **PACQHAPS**. A program, **PACQ for HAPSITE**, appears.
8. Go to **File** and click **Open Datafile**. Click the data file in acquisition and click **[OK]**.
9. PACQHAPS is used to monitor real-time acquisition.
10. After the BFB peak appears, go to **Modes** and click **Zoom scan range** to zoom-in the BFB peak.
11. Go to **Modes** and click **Plot spectrum**. Move the arrow cursor to apex of the peak and left-click the mouse. A mass spectrum appears.
12. Go to **Spectrum** and click **Specification Table**. A box, **Select Specification File**, appears. Click **bfb.spt** and **[OK]**.
13. The result of Select Specification File appears. If the status of all mass ions is pass (Figure 9), the BFB check passes.
14. Take out the second IS/Surr solution from the HSU heat chamber and start to run the five calibration standards.
15. If the status of some mass ions is flagged (e.g., low, high, etc.), close the spectrum and plot the spectra at other scans (e.g., $M \pm 1$, M is the scan number at apex of the peak) and repeat Steps 10 through 12. If the status of some mass ions is not pass, wait until the run finishes.
16. Exit **ACQUIRE** and go to **TUNE**. Repeat B.1.2 to do HAPSITE internal tuning.
17. The following table shows mass ion in internal tune relates to mass ion in BFB tune. Adjust the mass ions' % in internal tune relates to mass ions in BFB tune that fail to pass.

Mass ion in Internal Tune	Mass ion in BFB Tune
50	50
74	75
167	174
193	176

18. After the second internal tune is complete, exit **TUNE** and go to **ACQUIRE** to run the second IS/Surr solution.
19. If neither of the two BFB runs pass the tune criteria, contact the HAPSITE manufacturer for assistance.

B.2.5 Calibration Standard Acquisition

1. Follow Steps 1 through 5 of B.2.4 to setup acquisition for the five calibration standards.
2. Load the first three levels (i.e., 10, 20, and 50 ppb) of the calibration solutions into the HSU heat chamber in a clockwise order. Wait for at least 22 minutes for equilibrium. Place the remaining two calibration standard solutions next to the HSU.
3. When the solutions in the well are in equilibrium, follow Step 1 to run these standard solutions.
4. When the first calibration standard (i.e., 10 ppb) is complete, take out the vial and move the second calibration standard (i.e., 20 ppb) to post#1, 50 ppb to post#2, and 100 ppb to post#3. Continue to run the second calibration standard as per Step 1.



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5. Repeat Step 4 to finish all five calibration standards.
6. Refer to B.3 for using **AMDIS** to convert HAPSITE data files to HP Chemstation data files for data processing.
7. After the initial calibration is generated from the Chemstation (Figure 10), check that the %RSD of each analyte is $\leq 30\%$. If some of the analytes $> 30\%$, examine which calibration level contributes the high %RSD and rerun that calibration standard. Reprepare the standard from the working standard (200 ppm), if necessary.
8. If the initial calibration report is acceptable, prepare to run a method blank, which is 20 mL of DI water spiked with 5.0 μL of the 200 ppm IS/Surr mix.
9. To acquire samples on SIM, follow Step 1 except using **Tune/Cal** as 1 msec and switch **SIM Method** to another file (e.g., 624sim.fsm). The **Inlet Method** is unchanged.
10. Information for the **Full method**, **Inlet Method**, and **SIM Method** is listed in Appendix D.2.

B.2.6 Sample Acquisition

8. If the blank result is clean, start to run samples.
9. For a liquid sample, aliquot 20 mL into the syringe, followed by spiking 5.0 μL of the 200 ppm IS/Surr mix. Load the vial to the HSU heat chamber and wait for 22 minutes.
10. Refer to Steps 1 through 5 of B.2.4 to setup acquisition
11. After finish the run, use **AMDIS** to convert the data file.
12. For a solid sample, weigh approximately 5.0 g ($\pm 0.01\text{g}$) into a 40-mL vial.
13. Withdraw 20 mL of DI water into the syringe, followed by spiking 5.0 μL of the 200 ppm IS/Surr mix.
14. Transfer the water solution into the solid sample vial. Close the cap and vortex the vial vigorously for 1 minute before loading into the HSU heat chamber for acquisition.
15. Go to **PACQHAPS**. to monitor each sample chromatogram.
16. If the sample requires dilution, aliquot the amount (mL) of the sample according to the dilution factor. For example, a 1:10 dilution would be 2 mL of the sample + 18 mL of deionized water. Be sure to spike the sample with the IS/Surr mix for all injections.
17. For soil samples containing high concentrations of analytes, methanol extraction is used for analysis.
 - Weigh approximately 5 g ($\pm 0.01\text{g}$) of a sample in a 40-mL vial.
 - Add 5 mL of methanol to the vial, followed by 1 minute of vortex. Let the vial sit still until the soil settles.
 - Withdraw 15 mL of deionized water into the syringe and spike with 5.0 μL of the 200 ppm IS/Surr mix.
 - Transfer the water solution to another clean 40-mL vial.
 - Based on the dilution factor ($DF = 5/\text{volume (mL) of methanol extract}$), transfer the amount of methanol extract from the sample extraction vial to the water vial, followed by adding water to make up a total volume of 20 mL. For example, if $DF=10$, transfer 1 mL of methanol extract to the vial, followed by adding 4 mL of DI water. The maximum amount of methanol extract to be used is 5 mL. For $DF > 100$, the amount of sample methanol extract is negligible; therefore add 20 mL of DI water.
 - Refer to Steps 1 through 5 of B.2.4 to setup acquisition.

B.2.7 Continuous Sample Acquisition



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1. If the analysis is continued the next day or later, run a mid-level calibration standard (e.g., 50 ppb) as CCC.
2. Refer to B.2.1 through B.2.4 to equilibrate and tune the HAPSITE. Refer to B.2.5 to run CCC.
3. Run **AMDIS** to convert the CCC data file to Chemstation data file.
5. Use the Chemstation to generate the CCC report.
6. If the continuing calibration check passes the criteria ($\leq 25\%$), continue to run a new batch of samples.
7. If the CCC falls outside the criteria, re-run the second 50 ppb calibration standard.
5. If the second CCC is still unacceptable, rerun the remaining four calibration standards (i.e., 10, 20, 100, and 200 ppb) to generate a new initial calibration.
9. Once an acceptable initial calibration is obtained, continue to run the method blank and samples.

B.3 Using AMDIS

1. The **AMDIS** program converts HAPSITE data files to Agilent Chemstation data file for generating initial calibration, continuous calibration check report and sample quant reports from the Chemstation program.
2. When operating HAPSITE 1 to run samples, go to **PACQHAPS** and open data file of each acquisition.
3. Click **Chro** and click **Switch to AMDIS**. The program will process the conversion
4. When the conversion is finished, click **Done** and click **Yes** and click **Keep** to save the conversion data file (Figure 14) and exit the program.
5. When operating HAPSITE 2, it has an optional of **AMDIS** conversion automatically whenever the acquisition is finished.
6. Repeat B.3 step 4 to exit the program. Refer to Chemstation to process data files.



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APPENDIX C
Figures
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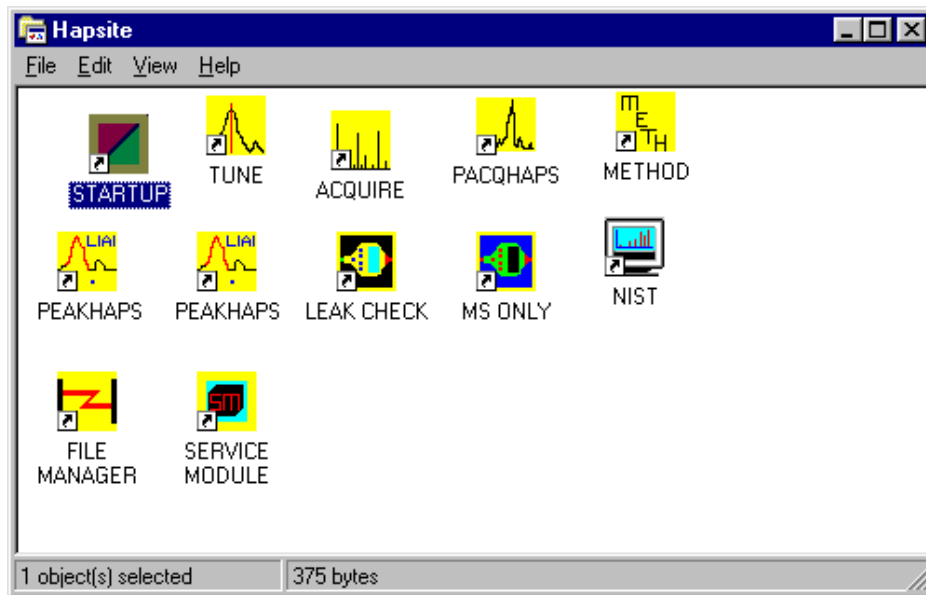


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FIGURE 1. HAPSITE Main Menu



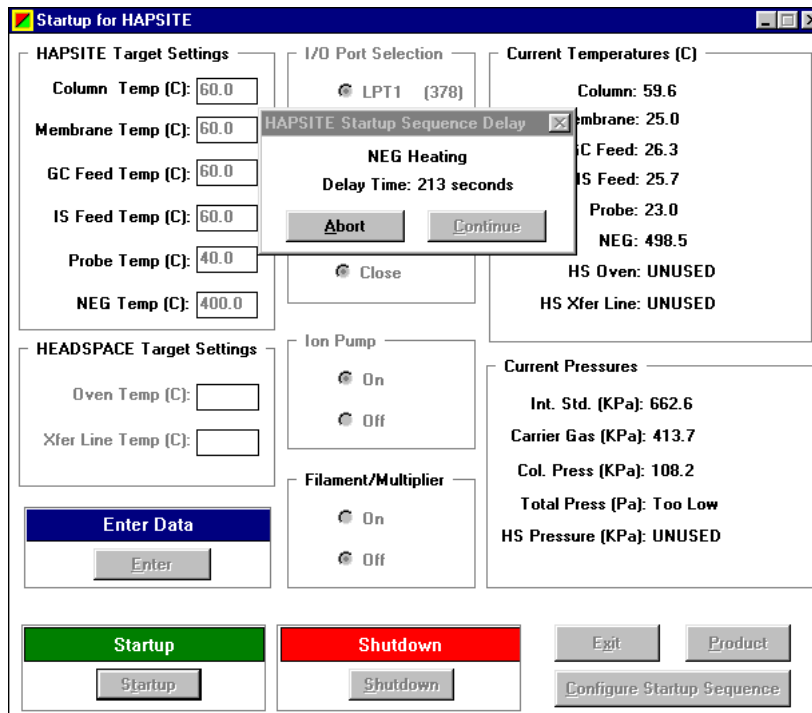


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FIGURE 2. The STARTUP program



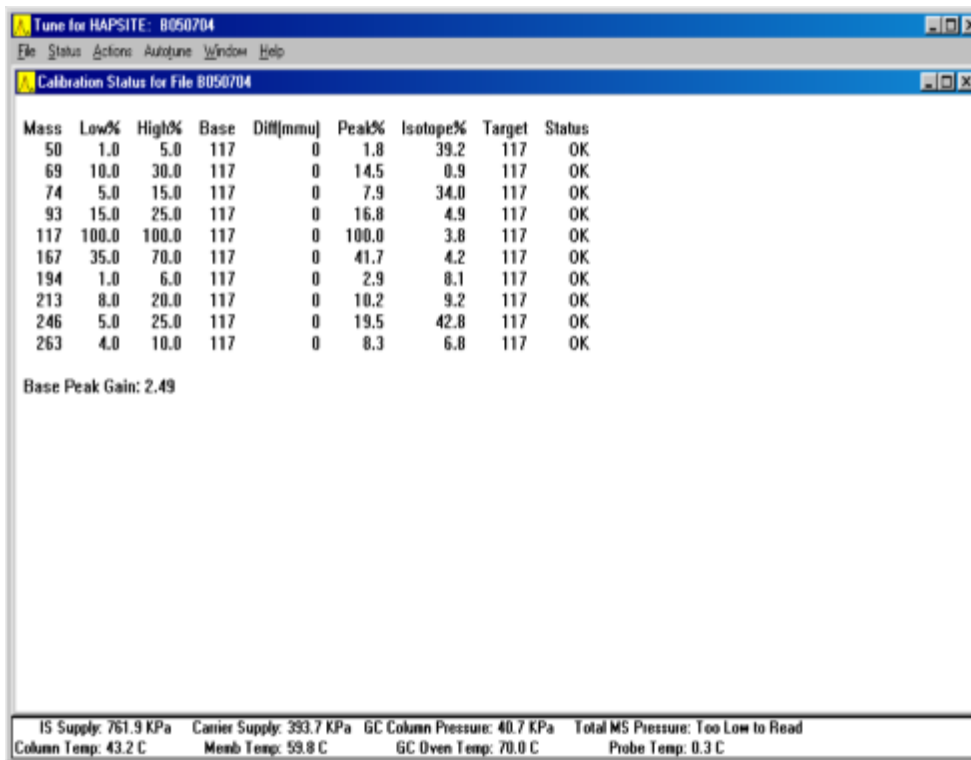


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FIGURE 3. Internal Tuning Calibration Result



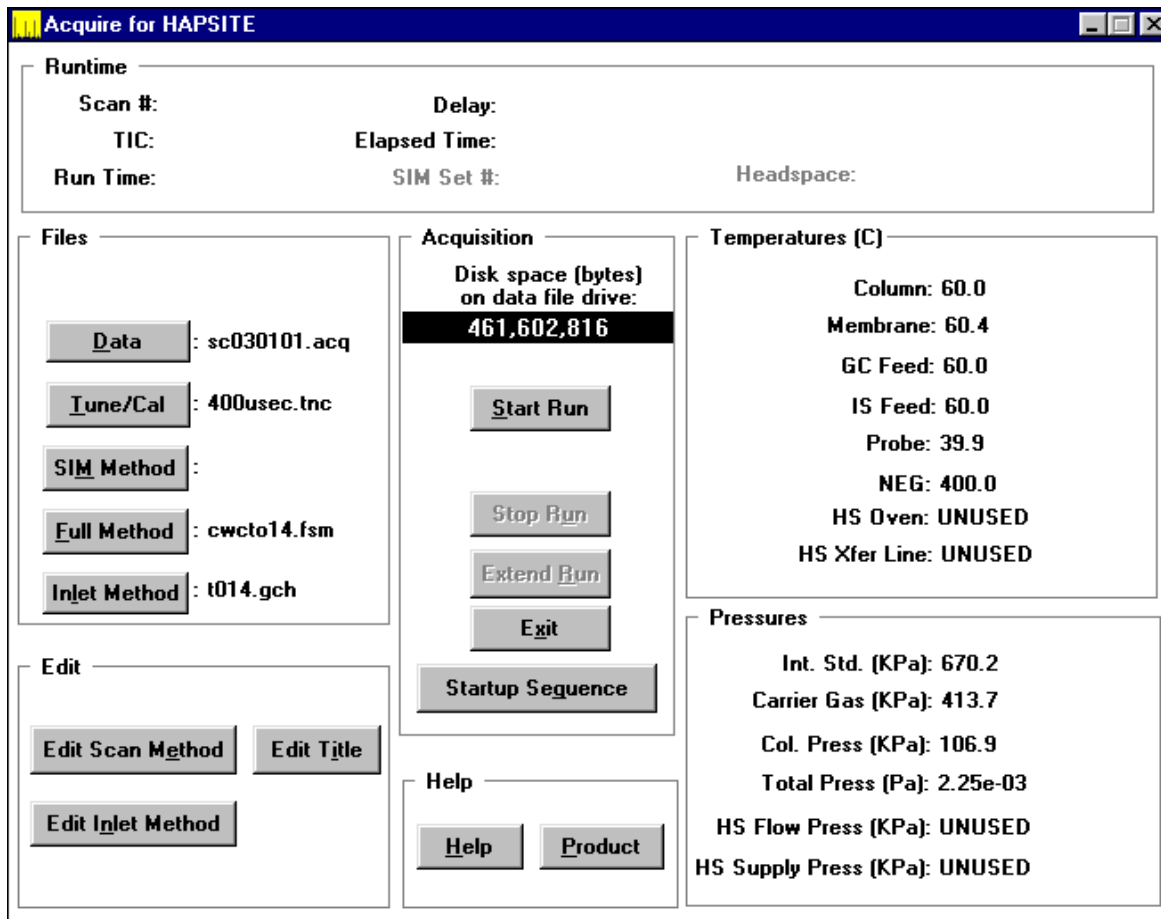


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FIGURE 4. The ACQUIRE Program



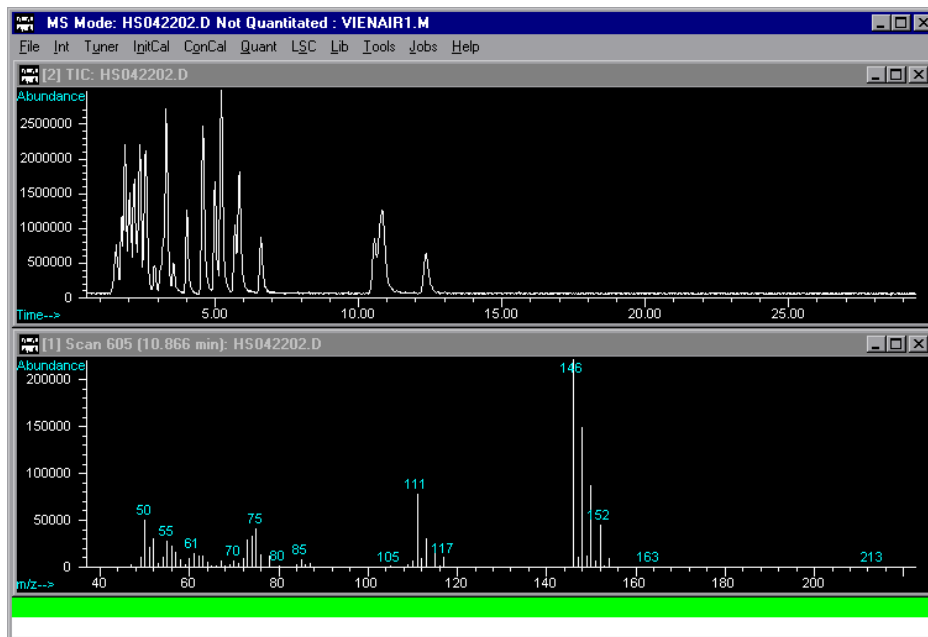


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FIGURE 5. HP Chemstation Data Analysis Program





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FIGURE 6. Initial Calibration Report for Air Samples

MultiVu - [C:\VHPCHEM\1\METHODS\WIENAIR2.M]\CALRPT.TXT

File Edit Search Window

Title : Hapsite VOCs Analysis
 Last Update : Wed Jul 03 09:06:52 2002
 Response via : Initial Calibration

Calibration Files
 0.2 -VIA62704.D 0.5 -VIA62703.D 1.0 -VIA62705.D

Compound	0.2	0.5	1.0	Avg	%RSD
-----ISTD-----					
1) ISTDTRIS_69					
2) Chloromethane	1.387	1.310	1.069	1.255	13.23
3) Vinyl chloride	1.457	1.520	1.174	1.384	13.34
4) Chloroethane	1.640	1.865	1.455	1.654	12.43
5) Trichlorofluoromethan	2.586	2.506	2.603	2.565	2.01
6) 1,1-Dichloroethene	2.574	2.997	2.696	2.755	7.91
7) Methylene chloride	3.811	3.280	3.477	3.523	7.61
8) trans-1,2-Dichloroeth	5.072	5.173	4.341	4.862	9.34
9) 1,1-Dichloroethane	5.895	7.073	7.259	6.742	10.97
10) cis-1,2-Dichloroethen	5.786	5.140	4.120	5.015	16.75
11) Trichloromethane	4.567	4.829	4.837	4.744	3.24
12) 1,2-Dichloroethane	7.640	8.104	7.081	7.608	6.74
13) 1,1,1-Trichloroethane	3.908	3.979	3.796	3.894	2.37
14) Benzene	6.263	7.049	6.032	6.448	8.27
15) Carbon tetrachloride	6.926	5.435	5.068	5.810	16.94
16) Dibromomethane	3.960	4.467	3.788	4.072	8.67
17) Bromodichloromethane	5.839	5.770	5.601	5.737	2.14
18) Trichloroethene(TCE)	6.690	6.935	6.124	6.583	6.32
19) 1,1,2-Trichloroethane	1.815	2.287	2.143	2.081	11.63
20) Toluene	6.033	7.727	6.325	6.695	13.53
21) Tetrachloroethene(PCE)	5.894	5.979	5.495	5.789	4.47
-----ISTD-----					
22) ISTD BPFB					
23) Ethylbenzene	1.375	1.412	1.495	1.427	4.32
24) m&p-Xylenes	1.122	1.339	1.297	1.253	9.18



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FIGURE 7. Air Sample Quant Report

MultiVu - [D:\DATAFI~1\2002DF\VIA62703.D\EPA TEMP.TXT]

File Edit Search Window

Data File : D:\DATAFI~1\2002DF\VIA62703.D Vial: 1
Acq On : 27 Jun 02 13:19 am Operator: C
Sample : TO-15 Std., 0.5 ppm Inst :
Misc : Vienna Wellfield, R1A00066 Multiplr: 1
Quant Time: Jul 3 8:57 19102 Quant Results File:

Quant Method : D:\HPCHEM\1\METHODS\VIENAIR1.M
Title : Hapsite VOCs Analysis
Last Update : Wed Jun 26 11:04:58 2002
Response via : Initial Calibration
DataAcq Meth : VIENAIR

Internal Standards	R.T.	QIon	Response	Conc	Unit
1) TRIS_69	1.72	69	1634984	10.00	pp
2) BPFB	3.96	117	5660096	5.00	pp

System Monitoring Compounds

Target Compounds

2) Chloromethane	1.24	50	107065	0.43	pp
3) Vinyl chloride	1.24	62	124287m	0.56	pp
4) Chloroethane	1.32	64	152492m	0.55	pp



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FIGURE 8. Headspace Analysis Startup Sequence Configuration

Section	Parameter	Value
Duty Cycle	Max NEG duty cycle (%)	80
	Max COLUMN duty cycle (%)	80
	Max MEMB duty cycle (%)	80
	Max GC FEED duty cycle (%)	100
	Max IS FEED duty cycle (%)	40
	Max HS Oven duty cycle (%)	50
	Max HS Xfer Line duty cycle (%)	50
HS Enable	Enable	<input checked="" type="checkbox"/>
NEG Control	By Temperature	<input checked="" type="radio"/>
	By Power	<input type="radio"/>
Heatup	Probe Heatup Time (sec)	300
	NEG Heatup Time (sec)	900
	NEG Heatup Power (Watts)	30
	GC Heatup Time (sec)	480
	Temp Stabilization Time (sec)	300
Pressure	Column Pressure Setpoint (KPa)	102
	Pressure Stabilization Time (sec)	10
	Max Manifold Pressure (Pa)	1.06e-02



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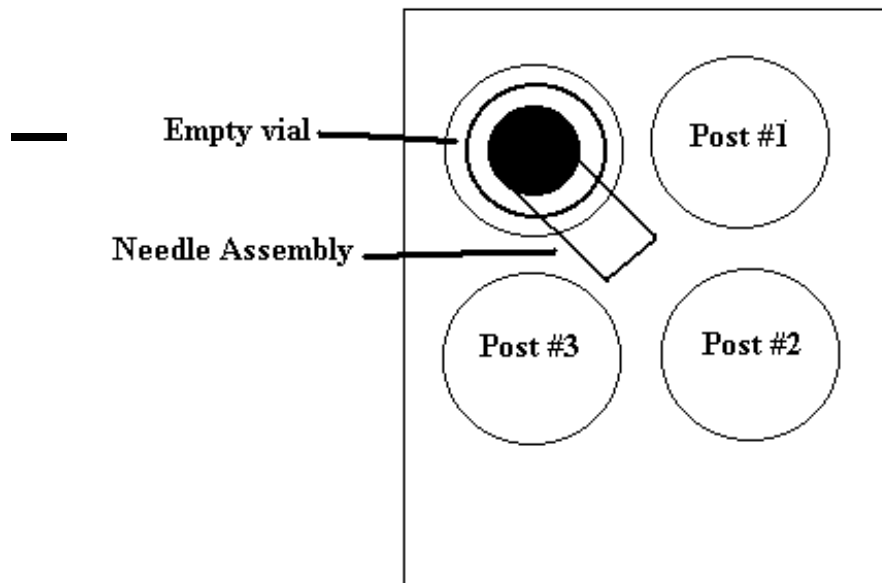


FIGURE 9. HSU Heat Chamber

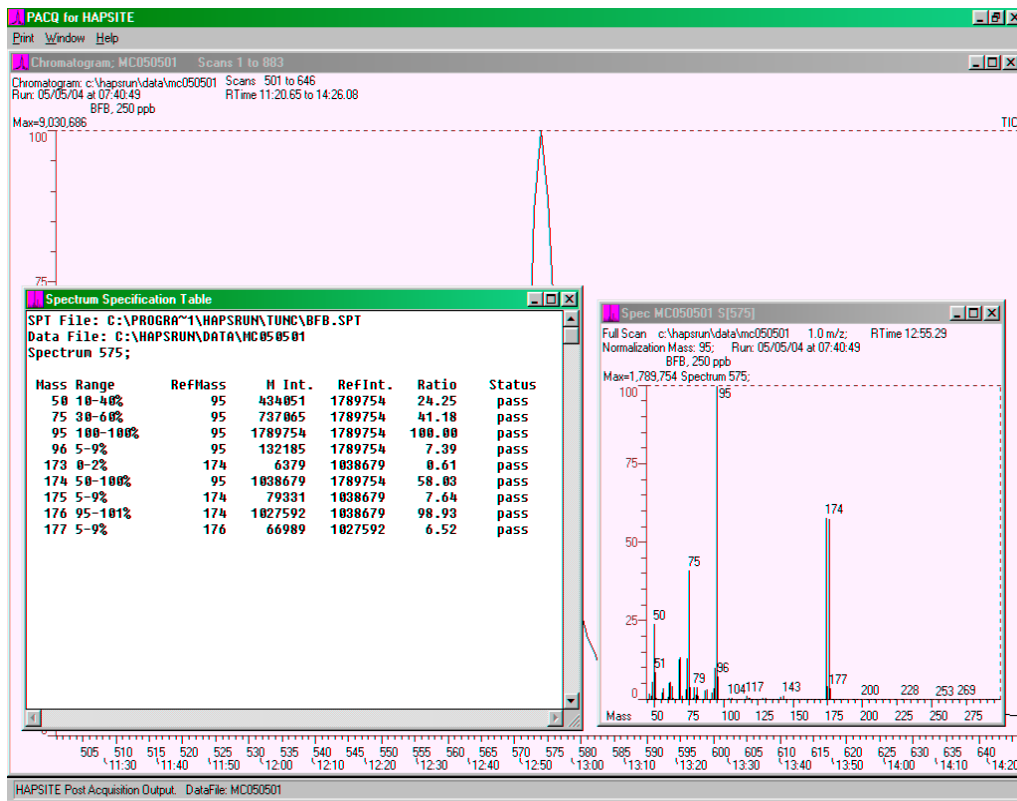


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FIGURE 10. BFB result in the PACQHAPS program



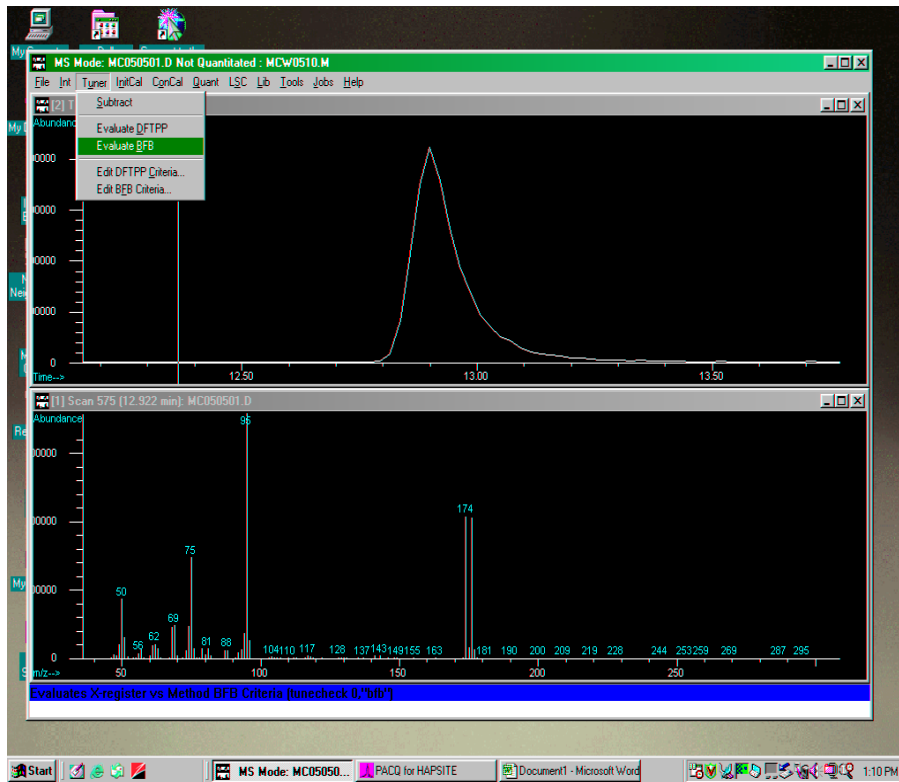


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FIGURE 11. Generate BFB Report in the Chemstation





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FIGURE 12. BFB report from the Chemstation

The screenshot shows two windows from the Chemstation software. The top window is a dialog box titled "Select BFB Evaluation" with four radio button options: "Evaluate BFB to Screen", "Evaluate BFB to Printer" (selected), "Autofind BFB to Screen", and "Autofind BFB to Printer". Below the options are "OK", "Cancel", and "Help" buttons.

The bottom window is titled "MultiVu - [D:\DATAFI~1\2003\LABFIL~1\HS043001.D\TUNEEVAL.TXT]" and displays a BFB report. The report includes acquisition details, method information, and a table of spectrum data for Scan 369.

Report Data:

Data File : D:\DATAFI~1\2003\LABFIL~1\HS043001.D Vial: 1
Acq On : 30 Apr 03 09:54 am Operator:
Sample : BFB, 250 ppb Inst :
Misc : Multiplr: 1.00

Method : D:\HPMETH~1\VIENAIR1.M
Title : Hapsite VOCs Analysis

Spectrum Information: Scan 369

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	30.9	385344	PASS
75	95	30	60	32.1	400512	PASS
95	95	100	100	100.0	1246720	PASS
96	95	5	9	7.0	86744	PASS
173	174	0.00	2	0.5	5025	PASS
174	95	50	100	83.4	1039168	PASS
175	174	5	9	7.1	74048	PASS
176	174	95	101	99.5	1034112	PASS
177	176	5	9	6.5	66784	PASS



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FIGURE 13. Initial Calibration Report for Water/Soil Standards

MultiVu - [C:\HPCHEM\1\METHODS\VOC0323.M\CALRPT.TXT]

File Edit Search Window

Response Factor Report

Method : C:\HPCHEM\1\METHODS\VOC0323.M
 Title : Hapsite VOCs Analysis
 Last Update : Wed Apr 28 14:19:57 2004
 Response via : Initial Calibration

Calibration Files
 10 -HS032402.D 20 -HS032403.D 50 -HS032404.D
 100 -HS032405.D 200 -HS032406.D

Compound	10	20	50	100	200	Avg	%RSD
-----ISTD-----							
1) I Dibromofluoromethane							
2) Vinyl chloride	0.449	0.475	0.367	0.401	0.365	0.412	11.96
3) Bromomethane	0.319	0.319	0.277	0.273	0.255	0.289	9.98
4) Chloroethane	0.427	0.434	0.400	0.398	0.406	0.413	3.95
5) Trichlorofluoromethan	0.471	0.502	0.496	0.532	0.492	0.499	4.42
6) 1,1-Dichloroethene	1.073	1.035	0.956	1.027	1.019	1.022	4.15
7) Methylene chloride	1.062	1.054	0.936	0.943	0.932	0.985	6.73
8) trans-1,2-Dichloroeth	1.727	1.685	1.640	1.636	1.638	1.665	2.41
9) 1,1-Dichloroethane	2.192	2.075	1.980	2.031	1.966	2.049	4.43
10) cis-1,2-Dichloroethen		2.075	1.980	2.031	1.966	0.000	-1.00
11) Chloroform	1.527	1.480	1.443	1.371	1.368	1.438	4.79
12) SURR1,2-Dichloroethane-d4	1.107	1.140	1.136	1.165	1.244	1.158	4.50
13) 1,2-Dichloroethane	1.654	1.400	1.372	1.330	1.278	1.407	10.36
14) 1,1,1-Trichloroethane	1.253	1.279	1.229	1.171	1.160	1.218	4.25
-----ISTD-----							
15) I Fluorobenzene							
16) Benzene	0.922	0.856	0.872	0.868	0.886	0.881	2.88
17) Carbon tetrachloride	0.513	0.462	0.466	0.463	0.471	0.475	4.57
18) 1,2-Dichloropropane	0.314	0.261	0.275	0.267	0.267	0.277	7.71
19) Bromodichloromethane	0.394	0.342	0.335	0.333	0.340	0.349	7.29
20) Trichloroethene	0.543	0.502	0.503	0.517	0.540	0.521	3.78
21) cis-1,3-Dichloroprope	0.282	0.253	0.254	0.236	0.257	0.256	6.36
22) trans-1,3-Dichloropro	0.270	0.197	0.211	0.196	0.209	0.217	14.17
23) 1,1,2-Trichloroethane	0.261	0.255	0.246	0.256	0.257	0.255	2.22
24) SURRToluene-d8	1.112	1.085	1.102	1.127	1.137	1.113	1.83
25) Toluene	1.344	1.268	1.385	1.474	1.541	1.403	7.64
26) Dibromochloromethane	0.298	0.223	0.247	0.247	0.250	0.253	10.75



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FIGURE 14. Water/Soil Sample Quant Report

Quantitation Report (QT Reviewed)

Data File : C:\HAPSRUN\DATA\MCW0504.D Vial: 1
Acq On : 05 May 04 10:56 am Operator:
Sample : B19002, GW1A 20 mL Inst :
Misc : Multiplr: 1.00
Quant Time: May 5 11:15 19104 Quant Results File: MCW0504.RES

Quant Method : C:\HPCHEM\1\METHODS\MCW0504.M
Title : Hapsite VOCs Analysis
Last Update : Wed May 05 09:42:05 2004
Response via : Initial Calibration
DataAcq Meth : HDSPACE

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) Dibromofluoromethane	2.84	111	895230	50.00	ppb	0.00
15) Fluorobenzene	3.91	96	1352992	50.00	ppb	0.02
27) Chlorobenzene-d5	9.98	117	2393376	50.00	ppb	0.01
34) 4-Bromofluorobenzene	12.88	95	874264	50.00	ppb	0.00
System Monitoring Compounds						
12) 1,2-Dichloroethane-d4	3.12	65	799835	46.72	ppb	0.00
Surrogate Spike 50.000			Recovery =	93.44%		
24) Toluene-d8	6.91	98	1245569	46.98	ppb	0.00
Surrogate Spike 50.000			Recovery =	93.96%		
36) 1,4-Dichlorobenzene-d4	15.94	150	1458777	23.08	ppb	-0.01
Surrogate Spike 50.000			Recovery =	46.16%		
Target Compounds						
32) 1,2,3-Trichloropropane	12.28	75	3067948	527.41	ppb	93

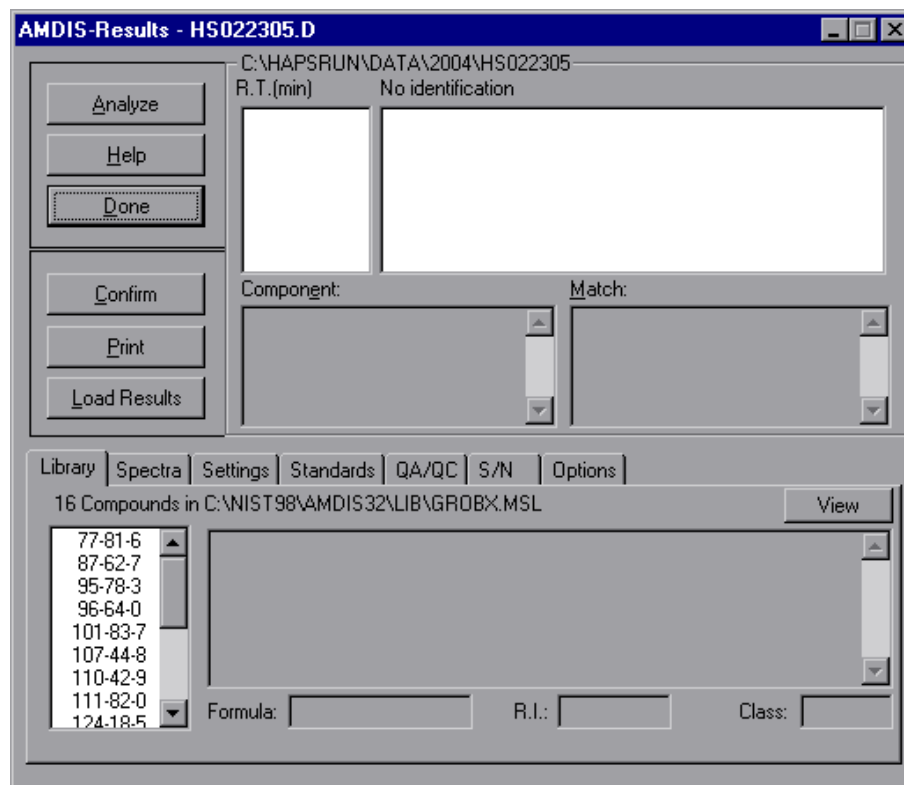


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FIGURE 15. The AMDIS Pro gram





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APPENDIX D
Method Information
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D.1 Air Analysis

D.1.1 Inlet Method (Scan or SIM)

HAPSITE1 Setup

Temperature	(°C)	Event	Elapsed Time (sec)	State	V1	V2	V3	V4	V5	SP	MI	IS	IC	IV	CS
Column	75.1	1	0.0	Loop fill	-	-	-	-	-	X	X	X	X	X	X
Membrane	60	2	30.0	Inject	X	-	X	-	-	-	X	-	-	X	X
GC feed	60	3	35.0	Fore flush	-	X	X	-	-	-	X	-	-	X	X
IS feed	60	4	150	Back flush	-	-	-	-	-	-	X	-	-	-	X
Probe	45														
NEG	400*														
Event to Start Run	2														

HAPSITE 2 Setup

Temperature	(°C)	Event	Elapsed Time (sec)	State	V1	V3	V4	V5	V6	V8	MI	SP	VS	FN	TN
Column	42	1	30.0	Loop fill	X	-	X	X	-	X	X	X	X	-	X
Membrane	60	2	---	Inject	-	-	-	-	-	-	-	-	-	-	-
GC feed	60	3	35.0	Fore flush	X	-	X	-	-	-	X	-	X	-	X
IS feed	60	4	330.0	Back flush	X	-	-	-	-	-	X	-	X	-	X
Probe	45														
NEG	400*														
Event to Start Run	2														

* blank out when the service module is being used

D.1.2 Full Method

Start mass	45	Mass defect	0
End mass	300	Peak width	1
Run time (min)	15	Filament delay (sec)	40
Probe "Run" start	Yes	Computer integration time	0.4
Scan time (sec)	1.02	(done by the computer automatically)	



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D.1.3 SIM Method

Set #1

Integration time (ms)	1	Begin time	1 min 0 sec
Filament delay (sec)	40	End time	1 min 46 sec

Round trip time should be around 1000 ms, the computer assigns this value and it is based on the integration time value.

Masses	Extra measurement	Masses	Extra measurement
49	10	64	10
50	10	65	10
52	10	84	10
57	10	96	10
61	10	98	10
62	10	101	10
63	10	103	10

Set #2

Integration time (ms)	1	Begin time	1 min 46 sec
		End time	3 min 00 sec

Masses	Extra measurement	Masses	Extra measurement
49	10	97	10
51	10	99	10
62	10	117	10
69	10	119	10
75	10	130	10
78	10	132	10
83	10	174	10
85	10	176	10
91	10		

Set #3

Integration time (ms)	2	Begin time	3 min 00 sec
		End time	11 min 00 sec

Masses	Extra measurement	Masses	Extra measurement
47	10	105	10
51	10	106	10



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61	10	117	1
91	10	120	10
92	10	166	10
97	10	167	1
104	10		

D.2 Headspace Analysis

D.2.1 Inlet Method (Scan or SIM)

HAPSITE 1 Setup

Temperature (°C)	Event	Elapsed Time (sec)	State	<----- GC -----><--HEADSPACE-->																	
				V1	V2	V3	V4	V5	SP	MI	IS	IC	IV	CS	V1	V2	V3	SP	MS		
75.1	1	0.0	Loop fill	-	-	-	-	-	X	X	-	-	X	X	X	X	X	X	X	X	X
60	2	8.0	Inject	X	-	X	-	-	-	X	-	-	X	X	-	-	-	-	-	-	-
60	3	14.0	Fore flush	-	-	-	-	-	-	X	-	-	-	-	-	-	-	-	-	-	-
60	4	180.0	Back flush																		
---	5	120.0	Purge	-	-	-	-	-	X	X	-	-	-	-	X	-	-	X	X		
NEG	400*																				
Event to Start Run	2																				

* blank out when the service module is being used

HAPSITE 2 Setup

Temperature (°C)	Event	Elapsed Time (sec)	State	<----- GC -----><--HEADSPACE-->																	
				V1	V3	V4	V5	V7	V8	MI	SP	VS	FN	T N	V1	V2	V3	SP	HS		
42	1	12.0	Sample(HS)	X	-	-	-	-	X	X	X	X	-	X	X	X	X	X	X	X	
60	2	---	Inject	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
60	3	360.0	Fore flush	X	X	-	-	-	-	X	-	X	-	X	-	-	-	-	-	-	
60	4	992.0	Back flush	X						X		X		X							
---	5	120.0	Purge	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
NEG	400*																				
Event to Start Run	2																				

* blank out when the service module is being used



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D.2.2 Full Method

Start mass	45	Mass defect	0
End mass	200	Peak width	1
Run time (min)	18	Filament delay (sec)	55
Probe "Run" start	---	Computer integration time	0.4
Scan time (sec)	1.02	(done by the computer automatically)	

D.2.3 HAPSITE 2 Temperature Program

Initial temp (°C)	42	2nd Ramp (°C/min)	30
Initial hold (min)	5.5	Final temp (°C)	200
1st Ramp (°C/min)	5.5	Final hold (min)	4.14
Final temp (°C)	110		
Final hold (min)	0		

D.2.4 SIM Method

Set #1

Integration time (ms)	1	Begin time	1 min 0 sec
Filament delay (sec)	40	End time	1 min 46 sec

Round trip time should be around 1000 ms, the computer assigns this value and it is based on the integration time value.

Masses	Extra measurement	Masses	Extra measurement
49	10	94	10
50	10	96	10
61	10	98	10
62	10	101	10
63	10	103	10
64	10		



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Set #2

Integration time (ms)	1	Begin time	1min 46 sec
		End time	3min 00 sec

Masses	Extra measurement	Masses	Extra measurement
62	10	96	10
63	10	97	10
67	10	98	10
78	10	111	10
83	10	117	10
95	10	119	10

Set #3

Integration time (ms)	2	Begin time	3 min 00 sec
		End time	20 min 00 sec

Masses	Extra measurement	Masses	Extra measurement
75	10	112	10
83	10	117	10
91	10	129	10
95	10	146	10
97	10	150	10
98	10	166	10
104	10	173	10