August 11, 2011

Ms. Janet Rosati
USEPA SFD-6-2
75 Hawthorne St.
San Francisco, CA 94105

SUBJECT: Freescale Semiconductor, Inc. Sub-Slab & Indoor Air Sampling Work Plan of Residential Structures Neighboring the Former Motorola 52nd St. Facility

Please find enclosed a final work plan for collecting sub-slab soil gas samples and indoor air samples in selected structures in neighborhoods proximal to the former Motorola 52nd St. Facility. This plan incorporates modifications in response to EPA's comments on the work plan provided in its conditional approval letter dated July 22, 2011. Hartman Environmental Geoscience (HEG) prepared this work plan in association with Clear Creek Associates, PLC (Clear Creek) on behalf of Freescale Semiconductor, Inc. (Freescale). This work plan is being prepared in accordance with the Administrative Settlement Agreement and Order on Consent (Consent Order) between Freescale and U.S. EPA Region 9 (EPA) for the Soil Gas and Vapor Intrusion to Indoor Air Evaluation in the Operable Unit 1 Area of the Motorola 52nd Street Superfund Site.

As part of the soil gas to indoor air evaluation, Clear Creek is conducting a soil gas investigation in accordance with an EPA approved Soil Gas Sampling Work Plan (Clear Creek Associates, December 3, 2010, amended March 17, 2011). During the investigation, Clear Creek installed temporary soil gas implants at depths of 5 and 15 feet below ground surface and collected soil gas samples for analysis of select volatile organic compounds (VOCs) by a modified EPA Method TO-15 analysis. In preparing this work plan, HEG has reviewed the soil gas data collected and validated through May 23, 2011. The initial assessment program described here is the first phase of the indoor air assessment program. Depending upon the results of this first phase, additional indoor air assessment phases may be necessary. In addition, since final information on building construction and access is still evolving at the time of this writing, modifications to this work plan may be necessary.

This Work Plan has been prepared pursuant to Section 2.2 of the Statement of Work (SOW) attached to the Consent Order. The Standard Operating Procedures (SOPs) attached to this Work Plan will serve as the Field Sampling Plan for the indoor air investigation required pursuant to Section 3.1.2 of the SOW. This work plan

incorporates by reference Appendix B – Quality Assurance Project Plan (Clear Creek Associates, November 17, 2010, amended March 17, 2011) and Appendix C – Site Safety and Health Plan (Clear Creek Associates, October 29, 2010, Table 1 amended March 17, 2011), except as modified herein.

Project Objective

The purpose of the proposed work is to collect additional data to assess the potential of vapor intrusion above acceptable risk levels into selected structures located near the above-referenced site. The on-going soil gas investigation has detected the presence of VOCs, primarily trichloroethylene (TCE) and tetrachlorethylene (PCE), above the residential Soil Gas Human Health Screening Levels (SGHHSLs) EPA has adopted for the site (Clear Creek, 2011). In accordance with the Consent Order, the presence of VOCs in soil gas at concentrations above EPA's residential SGHHSLs triggers the need to conduct an indoor air investigation.

Site Background

The Motorola 52nd Street Superfund Site is located in the City of Phoenix, Maricopa County, Arizona. The Site has been divided by EPA into three adjoining Operable Units (OUs): OU1, OU2, and OU3. OU1, approximately 1,000 surface acres, is the easternmost OU and contains the former Motorola 52nd Street Semiconductor Plant (Facility). The Facility is currently owned and operated by ON Semiconductor, Inc. The OU1 boundaries are 52nd Street to the east, Palm Lane to the north, Roosevelt Street to the south, and 44th Street to the west.

Motorola Semiconductor Products Sector (Motorola) owned and operated the Motorola Facility from 1956 to 1999. As part of its electronics manufacturing operation, Motorola used solvents, including trichloroethene (TCE) and 1,1,1-trichoroethane (TCA), to clean and degrease parts and equipment. In 1982, a solvent leak was discovered in an underground storage tank at the facility. The 1983 Preliminary Report (Gutierrez-Palmenberg, Inc., 1983) indicated that groundwater contamination was present at the Motorola Facility and to the west. The Preliminary Report also identified sources of contamination in the Courtyard, Acid Treatment Plant (ATP), and Southwest Parking Lot (SWPL) areas. Multiple soil, soil gas, and groundwater investigations were performed at all the potential source areas on the Facility. These investigations are documented in the Remedial Investigation Report for the Motorola 52nd Street Site (Dames and Moore, 1987). In September 1988, ADEQ and EPA selected an interim remedy in the OU1 Record of Decision (1988 ROD) consisting of soils cleanup at the Motorola Facility and groundwater containment near the 46th Street alignment. On October 4, 1989, EPA placed the Motorola 52nd Street Superfund Site on the National Priorities List (NPL). In July 1989, a Consent Order was executed between Motorola and ADEQ for the design and implementation of the remedy for OU1. Pursuant to its separation agreement with Motorola, Freescale has agreed to implement the remedial actions on behalf of Motorola.

In August 2010, Freescale entered into an AOC with EPA to conduct a Soil Gas and Vapor Intrusion to Indoor Air Evaluation in the residential area west of the Facility. Previously, prior to Freescale assuming responsibility for the remediation, soil gas surveys were conducted by Motorola in November 1984; February – March 1985; January 1989; March and October – November 1991; and January 1992 to study the overall distribution of VOCs in soil gas at the Site including the residential area west of the Facility. ADEQ also conducted a soil gas study in March and July 1992 in the residential area to the west of the Facility to confirm and update the previous work conducted by Motorola.

The available soil gas data were used by the Arizona Department of Health Services (ADHS) to evaluate both the indoor and outdoor air exposure pathways as part of the Baseline Risk Assessment for the Motorola 52nd Street Facility (ADHS, 1992). ADHS determined that, based on the then-current understanding of TCE and PCE health risks, both the indoor and outdoor air exposure pathways were below health-based risk levels for all locations.

An additional soil gas survey in the residential neighborhood was performed by Motorola in November - December 1995. Pursuant to the Consent Order, Freescale has been conducting a soil gas investigation in the residential area to the west of the former Motorola 52nd Street facility. The study area is shown on Figure 1. The soil gas investigation is still on-going. The available validated soil gas data from this current investigation are shown on Figure 2 (TCE, 5 and 15-foot intervals) and Figure 3 (PCE, 5 and 15-foot intervals). For sampling locations with unvalidated data, the locations are color coded to designate whether the preliminary results for TCE and PCE are non-detect, below residential SGHHSLs, or above residential SGHHSLs.

Initial Sub-slab/Indoor Air Sample Locations Based Upon the Recent Soil Gas Data

The structures proposed for sampling in the first phase have been selected based upon the areas of interest with the highest concentrations of TCE and/or PCE in the soil gas samples that most exceed EPA's residential SGHHSLs. To be conservative, initial sampling locations have been based upon the 15-foot soil gas data rather than the 5-foot data. In accordance with Section 1.2.1 of the SOW attached to the Consent Order, an inventory of buildings within the identified areas of interest was conducted as part of the effort to develop the scope of the indoor air investigation set forth in this Work Plan.

Soil gas concentrations over EPA's residential SGHHSLs have been observed primarily in three areas of interest within the soil gas investigation study area:

1) To the west of 48th street under an apartment complex bounded by E. McDowell Rd. to the north, E. Willetta St. to the south and Rt. 143 to the west

2) The western side of the school property property along 48th Street.

3) The area bounded by 48th St. on the west, 50th St. on the east, E. McDowell Rd. to the north, and E. Culver St. to the south (henceforth referred to as the "Central Zone").

These three areas are identified on Figure 4. The majority of the structures in these areas are single-story, multi-unit residential structures, built on a common cement slab. To minimize disruption to the residents, a sub-set of the structures will be sampled during the initial phase of the indoor air investigation. Depending upon the results, future sampling of additional units in any of these structures might be necessary.

Proposed structures and number of sample locations in each of these three areas are as follows:

Apartment Complex

Soil gas concentrations guiding the selection of the indoor air sampling locations were observed in the western portion of this area towards the Old Cross Cut Canal. In this phase, the two western-most buildings will be sampled, lying immediately east of soil gas points SV-57 and SV-58. Depending upon access, sample locations will be selected on the ground floor near each end of the building and near the middle of each building, for a total of three sample locations at each building. At each location, sub-slab and indoor air samples will be collected.

Western School Property

The soil gas concentrations surrounding the school (locations SV21, SV22, and SV23) are either non-detect or below EPA's SGHHSLs, so the primary school building does not need to be tested.

The indoor air in the building on the western portion of the school property will be tested if access is allowed as the soil gas concentrations at locations SV48 and SV49 would fall within the range guiding selection of the initial indoor air sampling locations. A minimum of three samples will be collected inside the structure. Sample locations will be selected during a pre-sampling on-site building evaluation. The on-site visit will evaluate the HVAC system, potential preferential pathways, variations in ventilation rates in different zones, and occupancy areas (eg., administrative offices) when selecting sampling locations. Since the occupants mostly at risk are the school faculty and staff, indoor air collection will be for an 8 to 10-hour collection period. Sub-slab data will not be collected under this building since any detected values would be difficult to interpret due to the large size of the building.

Central Zone

The following residential structures within the Central Zone are proposed to be sampled during this first phase:

- Five (5) parcels with quad-plexes that are located along N. 50th Street directly east of soil gas points SV26, SV29, and SV34. In each of these structures, one or two sub-slab and indoor air samples will be collected near the ends of each building.

- A single-family residence located on N. 50th Street, south of sampling location SV02.

- Two tri-plexes identified below located between soil gas points SV27 and SV33. In each of these structures, one or two sub-slab and indoor air samples will be collected depending upon the construction/partitioning of the buildings.

- Two duplexes and 1 quad-plex along E. Willetta Street located just south of soil gas points SV13 and SV31. In each of these structures, one or two sub-slab and indoor air samples will be collected depending upon the construction/partitioning of the buildings.

- Four structures along E. Willetta Street, located just north and south of soil gas points SV20 and SV44. In each of these structures, one or two sub-slab and indoor air samples will be collected depending upon the construction/partitioning of the buildings.

The number and specific locations of the structures to be sampled will be adjusted based on access and occupancy considerations. Additionally, there will be a preference to sample structures with central cooling systems versus evaporative (or "swamp") coolers due to the lower air exchange rate associated with central cooling systems. Locations of outdoor air samples will also be chosen in areas with adequate security to minimize the risk of theft of the canisters.

Proposed Work

Access

Freescale understands that EPA will take the lead, with assistance from Freescale, in securing access from property owners and tenants for conducting the indoor air evaluation. Access to each of the identified properties, or an appropriate alternative as determined by EPA and Freescale, will be secured prior to initiating the indoor air evaluation.

Pre-Sampling Interview & Survey of Structures

Prior to sample collection, an interview will be conducted with the residents at each sampling location explaining the indoor air and sub-slab sampling procedures. During the interview, a survey of the structure's interior will be performed, type of ventilation system will be noted, and any products known to potentially contain the target VOCs will be identified and requested to be removed from the premises approximately two (2), but

no less than one (1) day prior to sample collection. The residents will also be provided a list of activities to refrain from during the indoor air sample collection period. A survey and products inventory form will be completed (see Attachment A).

Indoor Air & Outdoor Air Samples

Indoor air samples will be collected near or at the same locations as the sub-slab samples by Dr. Blayne Hartman with assistance by Clear Creek. Indoor air samples will be collected immediately prior to the sub-slab sample collection to ensure no interferences are produced from the sub-slab sampling effort. Sample collection will be performed in accordance with Hartman Environmental's "Standard Operating Procedures, Indoor Air Sampling, Passivated Canisters by Method TO-15 for Vapor Intrusion Assessments" (2011) (Attachment B). Sample collection devices will be placed in the residence in a location of highest use as allowed by the residents. Samples will be collected as close to the center of the room as possible, away from any cooling system registers, and at a height between 3 feet to 7 feet above the floor (breathing zone). Samples will be collected in individually certified-clean Summa canisters for a period of 24 hours. Meteorological conditions and any unusual residential conditions will be noted.

Outdoor air sample collection will be started approximately one hour before the start of collection of the indoor air samples and collected over approximately the same time period as the indoor air samples. Samples will be collected at four (4) locations bounding the neighborhoods in general locations as shown on Figure 4. Exact locations will be selected based upon access and security issues.

The procedures set forth herein for the collection of indoor and outdoor air samples replace the procedures set forth in Section 6.1 of the QAPP. The field staff will endeavor to document the following conditions when possible:

- Type of ventilation system (evaporative coolers, central cooling, window air conditioner) used in the structure;
- Indoor air (when available) and outdoor air temperatures at the time of sample collection; and
- Type of sample (indoor air or outdoor air).

This information will be provided to EPA along with the analytical results as supporting data that characterize conditions during sampling so that the indoor air data can be properly evaluated and put into context.

Indoor and outdoor air samples will be shipped to Calscience.Environmental Laboratory (Garden Grove, CA) and analyzed by method TO-15 SIM in accordance with CalScience's Standard Operating Procedure, Volatile Organic Compounds in Air by GC/MS SIM (EPA Methods TO--15-SIM) (8/1/2011) (Attachment C). The samples will be analyzed for the nine VOCs listed below, which are the compounds detected in the soil gas samples. Quality assurance criteria can be found in Attachment C. Calscience

holds a NELAC certification for the TO-15 analysis. A five day turn-around-time will be requested.

Table 1	**Calscience Method TO-15 SIM Reporting Limits**	
Compound	**CalScience PQL ($\mu g/m^3$)**	**EPA Regional Screening Level (RSL) for Residential Air (1e-6 risk)**
Trichloroethene	0.2	1.2
Tetrachloroethene	0.2	0.41
1,1,1-Trichloroethane	0.2	5200*
1,1,2-Trichlorotrifluoroethane (Freon 113)	0.2	31000
1,1-Dichloroethene	0.2	210
Chlorobenzene	0.2	52*
cis-1,2-Dichloroethene	0.2	63**
Chloroform	0.2	0.4
Bromodichloromethane	0.2	0.066

* Non-cancer Hazard Index = 1

** No established RSL. Trans-1,2-dichloroethylene used as surrogate.

Sub-Slab Samples

Sub-slab soil vapor samples will be collected immediately after the indoor air sample collection devices are collected and removed from the structure. Sub-slab samples will be collected by H&P Mobile Geochemistry (H&P) and/or Clear Creek with on-site oversight and assistance by Dr. Blayne Hartman following the procedures described in HEG's "Standard Operating Procedures, Sub-Slab Soil Gas Sampling Procedures for Vapor Intrusion Assessments" (June 2011) (Attachment D). These procedures replace the procedures set forth in Section 6.1 of the QAPP. Briefly, a small diameter hole (<1") will be drilled through the slab and a small diameter tubing inserted. The tubing will be sealed into the hole, purged of residual air, and a soil gas sample withdrawn into a 1-liter tedlar bag. The tedlar bag will be tested for the leak-check compound and if leak-free, the sample will be immediately transferred into a small volume (400 cc to 1000 cc) passivated canister. Helium or carbon dioxide will be used as a tracer compound to detect any leaks in the sampling procedure. Analysis of the tracer compound will be made on-site using calibrated meters. An equipment blank sample will also be collected to ensure the sampling equipment used for sub-slab sampling (probe, sample train, and canister) is contaminant free. Duplicate sub-slab samples will be collected at a frequency of 1 every 10 samples. After sampling is complete, the sub-slab sampling probe will be removed from the slab and the hole sealed with cement

The field staff will endeavor to document the following conditions when possible:

- Type of ventilation system (evaporative coolers, central cooling, window air conditioner) used in the structure;

- Indoor air (when available) and outdoor air temperatures at the time of sample collection; and
- Type of sample (sub-slab).

This information will be provided to EPA along with the analytical results as supporting data that characterize conditions during sampling so that the sub-slab data can be properly evaluated and put into context.

Soil vapor samples will be shipped to H&P Mobile Geochemistry (Carlsbad, CA) and analyzed for the same VOCs listed previously by method TO-15 at the practical quantitation level shown in Table 2. Quality assurance criteria can be found in H&P Mobile Geochemistry's "Standard Operating Procedures (SOPs), EPA Method TO-14A, TO-15 (Rev. 6, February 2011) and "Standard Operating Procedures 8260 SV SOP for Determining VOC's in Soil Vapor (June 2011) (Attachment E). H&P holds a NELAC certification for the TO-15 analysis. If the sub-slab samples are at concentrations greater than 5000 ug/m^{3,} the samples may be analyzed by EPA method 8260 to prevent contamination of the TO-15 analytical system..

Table 2	**H&P Method TO-15 Reporting Limits – 400 cc samples**	
Compound	**H&P PQL ($\mu g/m^3$)**	**Sub-slab Screening Level (att factor = 0.1)**
Trichloroethene	5.5	120
Tetrachloroethene	6.9*	4.1
1,1,1-Trichloroethane	5.5	52000
1,1,2-Trichlorotrifluoroethane (Freon 113)	7.7	310000
1,1-Dichloroethene	4.0	2100
Chlorobenzene	4.7	520
cis-1,2-Dichloroethene	4.0	630
Chloroform	5.0*	4.1
Bromodichloromethane	6.8**	0.66

*MDLs are below sub-slab screening levels – refer to table in Attachment E
**MDL 0.95 ug/m3 – refer to table in Attachment E

Samples for Radon Analysis

At each location, an additional sub-slab sample and indoor air sample will also be collected in tedlar bags for possible radon analysis. The purpose of this analysis is to determine a foundation-specific attenuation factor to aid in the interpretation of any detected levels of VOCs in the sub-slab samples. The use of radon as a surrogate for determining a foundation-specific attenuation factor is described in the ITRC toolkit in the ITRC vapor intrusion guidance document (ITRC 2007). The decision to analyze a sample for radon will depend upon the sub-slab soil gas concentrations. If the sub-slab concentrations are very low (<50 times the allowed indoor air values) or very high (>5000 times the allowed indoor air values), the radon analysis is unlikely to provide any benefit.

Radon analysis will be conducted by Dr. Douglas Hammond at the University of Southern California (Los Angeles, CA) using alpha scintillation counting in accordance with established USEPA protocols (USEPA, Indoor Radon and Radon Decay Product Measurement Device Protocols. EPA 402-R-92-004, July 1992 revised). Analytical precision is +/- 15%.

Data Management and Evaluation,

The data reported by the laboratories will be provided in an electronic data deliverable (EDD) format and entered in an electronic database that is Microsoft Access or Microsoft Excel compatible. The database will maintain the integrity of the data while facilitating the retrieval of the data for analysis in tables and figures.

Data collected as part of this vapor intrusion evaluation will be assessed using a tiered approach as described below:

- Tier 1: Sub-slab sample results will be compared to the indoor air concentrations to determine if a potential indoor air pathway is present.

- Tier 2: Indoor air sample results will be compared to outdoor air sample results to determine if the indoor air results may be affected by sources unassociated with vapor intrusion.

- Tier 3: The indoor air results that can be attributed to vapor intrusion will be compared to risk based screening criteria.

If indoor air results that can be attributed to vapor intrusion exceed Tier 3 criteria, Freescale will contact EPA and develop an appropriate follow-up course of action.

Reporting

1) Notification of Initiation of Field Work & Notification of Completion of Field Work

Freescale will notify EPA with a Notification of Initiation of Field Work at least seven (7) days before initiating any physical work in the field and will endeavor to notify EPA fourteen (14) days in advance if possible. The notification will include the planned dates for field activities so that EPA may adequately schedule oversight tasks. Within five (5) days of completion of all field activities associated with the indoor air investigation, Freescale will notify EPA in writing with a Notification of Completion of Field Work.

2) Daily Field Activity Reports

Upon initiation of field work, Freescale will provide Daily Field Activity Reports in accordance with the requirements in the AOC. These Daily Field Activity Reports will include: (1) a description of the actions which have been taken to comply with the AOC

during that day; (2) all the results of sampling and tests and all other data received by Freescale; (3) the work planned for the next day with schedules relating this work to the overall project schedule; and (4) a description of all problems encountered and any anticipated problems, any actual or anticipated delays, and solutions developed and implemented to address any actual or anticipated problems or delays. Daily Field Activity Reports will be provided via electronic mail each day that field work is being conducted.

3) Monthly Progress Reports

In accordance with the AOC, Freescale will provide Monthly Progress Reports. At a minimum the Monthly Progress Reports will: (1) describe the actions during that month; (2) include all results of sampling and tests and all other data (both validated and unvalidated) received by Freescale; (3) describe work planned for the next two months with schedules relating such work to the overall project schedule; and (4) describe all problems encountered and any anticipated problems, and actual or anticipated delays, and solutions developed and implemented to address any actual or anticipated problems or delays. The Monthly Progress Reports will be submitted via electronic mail and are due on the 10th day of each month for the preceding month's work.

4) Vapor Intrusion to Indoor Air Evaluation Report

Within 45 days of receipt of all validated data from the second seasonal sampling event of this initial phase of the indoor air investigation, Freescale will prepare a Vapor Intrusion to Indoor Air Evaluation Report in accordance with the terms of the Consent Order. _The report will be revised in accordance with EPA comments and a final report will be submitted for EPA's review and approval within 30 days of receiving EPA's comments on the draft report.

Quality Control (QC) Elements

Freescale will conduct the investigation in accordance with the general and applicable provisions of the Quality Assurance Project Plan (QAPP) prepared by Clear Creek Associates for the soil gas investigation (December 3, 2010, amended March 17, 2011). Specific modifications to the QAPP for the indoor air investigation will be detailed in this work plan.

Project Team Organization

Section 2.1, Project Team Organization, of the QAPP is modified by this section. A project organization chart is presented on Figure 5. Contact information for each project team member is provided in Attachment F. Descriptions of the specific roles and responsibilities of team members are presented below.

Project Coordinator and Site Manager

Ms. Jenn McCall, Strategic Programs Manager for Freescale Semiconductor, Inc., will serve as the Project Coordinator and Site Manager and will oversee the administration of all tasks implemented pursuant to the AOC. Her responsibilities include defining the project objectives and establishing project policies and procedures that will be followed by all members of the Project Team. The Project Coordinator will review project tasks to ensure compliance with the requirements set forth in the AOC, approve all reports prior to submission to EPA, and represent Freescale at meetings.

Subslab/Indoor Air Sampling Investigation Project Management

Dr. Blayne Hartman of Hartman Environmental Geoscience will serve as Project Manager for Hartman Environmental and will prepare the work plan and direct the indoor air investigation with the assistance of Clear Creek Associates. Mr. Thomas Suriano, R.G., of Clear Creek Associates will serve as Project Manager for Clear Creek Associates. Both will report to the Project Coordinator/Site Manager. Both Clear Creek and Hartman Environmental will work together to maintain the approved QAPP, direct project activities, ensure project schedules are met, prepare project documents, ensure that the work meets AOC requirements and represent project contractors at meetings.

QA Coordinator

Mr. Todd Cruse, R.G., of Clear Creek Associates will serve as QA Coordinator. The QA Coordinator reports to the Clear Creek Project Manager to provide organizational independence from the personnel generating the environmental data. The QA Coordinator also ensures access to the appropriate level of management in order to plan, assess, and improve the quality system. The QA Coordinator has responsibility for evaluating data collection efforts to verify that the appropriate QA/QC measures have been implemented in accordance with the QAPP and with coordinating with third party data validators (the QA Subcontractor).

QA Subcontractor

The QA Subcontractor, Innovative Technical Solutions, Inc., will provide data validation, laboratory management, and QA services. The QA Subcontractor will review, verify and/or validate analytical data, and notify the QA Coordinator of any issues relating to the quality or validity of the data.

Clear Creek and Hartman Environmental Field Teams

The Field Teams will be composed of trained personnel from Hartman Environmental, H&P Mobile Geochemistry and Clear Creek Associates. Field team members will report to their respective Project Managers. The Field Teams will be responsible for the performance of all field activities and will follow all instructions and procedures detailed in the SOPs attached to this work plan.

Laboratory Subcontractors

Two laboratories will be used for this project. The laboratory subcontractor H&P Mobile Geochemistry will provide TO-15 analysis of the sub-slab samples utilizing its California laboratory. They will also subcontract radon analyses through the University of Southern California. CalScience Environmental Laboratories will analyze the indoor/outdoor air samples by Method TO-15 SIM in its California laboratory. The labs will be overseen by both Hartman Environmental and Clear Creek.

Section 2.2 of the QAPP is modified to require that the project manager has either a relevant professional registration (engineering or geology) or is experienced in conducting indoor air investigations. Project staff will be required to have experience in collecting soil gas and/or air samples.

Quality Control Elements

This section presents QC requirements relevant to the collection and analysis of sub-slab and ambient air samples. The purpose of the QC program is to produce data of known quality that satisfy the project DQOs and that meet or exceed the requirements of the standard methods of analysis.

Field Documentation

Section 2.3.1 of the QAPP is amended to require the following amended or additional information:

- Type of ventilation system (evaporative coolers, central cooling, window air conditioner) used in the structure.
- Indoor air and outdoor air temperatures at the time of sample collection.
- Type of sample (sub-slab, indoor air, outdoor air).

Data Quality Objectives

The seven-step DQO process described below was followed to determine the appropriate data quality level needed for the project.

Step 1: State the problem	A soil gas study being conducted in the study area has detected soil gas concentrations in excess of risk-based screening levels. An indoor air investigation is necessary to evaluate the soil gas to indoor air pathway.
Step 2: Identify the goal of the study	There are two principal study questions: Is there a completed pathway from soil gas to indoor air? If there is a completed pathway, do indoor air concentrations exceed the risk-based screening levels contained in Table 1 of

	the Work Plan?
Step 3: Identify the inputs to the decision	Sub-slab, indoor air, and outdoor air samples will be collected in summa canisters at identified sampling locations within the study area and analyzed for the target VOCs on Table 1 (air) and Table 2 (sub-slab) of the Work Plan.
Step 4: Definition of study boundaries	The AOC defines the study area as the residential neighborhood between the Motorola 52nd Street Facility and the Old Crosscut Canal. Areas of interest for conducting an indoor air investigation were identified based on the results of the soil gas investigation. The areas of interest are described in this Work Plan.
Step 5: Develop the analytical approach	If the validated data show that observed indoor air concentrations from both sampling events are below the risk-based levels, then no further work will be required for those locations. If validated indoor air data from either sampling event at any sampling location exceed the applicable risk-based level, then a risk management evaluation will be conducted and mitigation or other measures will be considered.
Step 6: Specify Performance or Acceptance Criteria	The performance or acceptance criteria are established by the Data Quality Indicators set forth in the QAPP as amended by this Work Plan.
Step 7: Develop the plan for obtaining data	In accordance with the AOC, indoor air samples will be collected in the residential neighborhood between the Facility and the Old Crosscut Canal. As specified in the Work Plan, sub-slab and indoor air samples will be collected at locations identified based on the soil gas sampling results obtained pursuant to the AOC. Indoor air samples will be collected immediately prior to the sub-slab sample collection to ensure no interferences are produced from the sub-slab sampling effort. Sample collection will be performed in accordance with the SOPs in Attachment B. Sample collection devices will be placed in the residence in a location of highest use as allowed by the residents. Samples will be collected as close to the center of the room as possible, away from any cooling system registers, and at a height between 3 feet to 7 feet above the floor (breathing zone). Samples will be collected in individually certified-clean Summa canisters for a period of 24 hours. Meteorological conditions and any unusual residential conditions will be noted. Outdoor air sample collection will be started approximately 1 hour before the start of collection of the indoor air samples and collected over approximately the same time period as the indoor air samples.

	For sub-slab sampling, a small diameter hole (<1”) will be drilled through the slab and a small diameter tubing inserted. The tubing will be sealed into the hole, purged of residual air, and a soil gas sample withdrawn into a 1-liter tedlar bag. The tedlar bag will be tested for the leak-check compound and if leak-free, the sample will be immediately transferred into a small volume (400 cc to 1000 cc) passivated canister. Helium or carbon dioxide will be used as a tracer compound to detect any leaks in the sampling procedure. Analysis of the tracer compound will be made on-site using calibrated meters. An equipment blank sample will also be collected to ensure the sampling equipment used for sub-slab sampling (probe, sample train, and canister) is contaminant free.

Quality Control Procedures

This section modifies Section 5.1 of the QAPP. The chemical data to be collected may be used to determine if the vapor intrusion pathway is complete. Consequently, strict QA/QC procedures will be adhered to. These procedures include:

- Adherence to strict protocols for field sampling and decontamination procedures – refer to attached sampling SOPs
.
- Collection and analysis of appropriate field blanks to monitor for contamination of samples in the field or the laboratory; and

- Collection and analysis of duplicate samples to evaluate analytical precision and accuracy.

Equipment Decontamination

- All sub-slab probe materials will be new tubing. None of the probe materials will be reused.

- A new tedlar bag will be used for each sub-slab sample.

- The transfer piece used to downfill samples from the tedlar into the canister will be reused and decontaminated between samples by flushing with at least 50 internal dead volumes of outdoor air.

Calibration Standards

Standards used for calibration of laboratory equipment will be from a traceable, certified supplier with an expiration date established by the supplier. Standards will be labeled with expiration dates and will reference primary standard sources or vendor certifications.

Sample Canisters & Flow Controllers

Canisters for sub-slab samples will be batch tested (1 every 10 canisters) at the 5 ug/m3 PQL since allowable screening values for sub-slab samples are higher than for indoor air. The batch will be certified to the reporting limit for the relevant constituents but detections will be reported down to the Method Detection Limit (MDL). For the canister that is tested for the batch, any detections above the MDL and below the PQL would be reported as J flagged for "estimated.".

Canisters and flow controllers for indoor and outdoor samples will be individually tested prior to shipping to ensure they are contaminant free at the 0.2 ug/m3 PQL.

All flow controllers will be checked for proper flow rate and adjusted as necessary prior to shipment by the supplying lab to ensure nominal collection time of approximately 24 hours.

Holding Time Compliance

Sample analysis will be completed well within the allowed analysis holding time of 30 days. Holding time begins at the time of sample collection.

Quality Assurance and Quality Control (QA/QC) Samples

This section modifies Section 5.2 of the QAPP. Quality assurance and quality control samples will be collected as part of the overall QA/QC program. The purpose of this QA/QC program is to produce data of known quality that satisfy the project DQOs and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Trip Blanks

Trip blanks are analyzed to ensure that the sampling canisters are contaminant free prior to sample collection. The trip blank consists of sending an evacuated canister out in the same shipment as the other canisters and returning it to the laboratory for analysis unfilled. One trip blank will be collected during this program.

Sub-Slab Sample Equipment/Procedure Blank

Equipment/procedure blanks are performed to ensure that the supplies used for the sub-slab probes and for the sample collection are contaminant free. The equipment blank consists of passing outdoor air through the probe materials, collecting the air into a tedlar bag, and transferring the sample from the tedlar bag to the canister following the same procedure as with the sub-slab samples. The canister is then analyzed by the laboratory. One equipment/procedure blank will be collected during this program.

Field Duplicate Samples

Field duplicate samples will be collected and analyzed to evaluate sampling and analytical precision. Field duplicates are collected and analyzed in the same manner as the primary samples. Agreement between duplicate sample results will indicate good sampling and analytical precision. Field duplicates will be collected at a frequency of approximately one field duplicate for every ten (10%) sub-slab samples collected and one sample for every ten indoor air samples collected. The duplicate sample will be analyzed for all laboratory analyses that were requested for the primary sample. The precision goal for field duplicate analyses will be a RPD that is less than or equal to 50% for subslab samples and a RPD of 25% for indoor air samples.

Sampling and Analytical Procedures

Section 6.0 (Sampling Procedures) of the QAPP is modified by Hartman Environmental's "Standard Operating Procedures, Indoor Air Sampling, Passivated Canisters by Method TO-15 for Vapor Intrusion Assessments" (2011) and "Standard Operating Procedures, Sub-Slab Soil Gas Sampling Procedures for Vapor Intrusion Assessments" (June 2011) attached to this work plan (Attachments B and D). Section 7.0 (Analytical Procedures) of the QAPP is modified by CalScience's Standard Operating Procedure, Volatile Organic Compounds in Air by GC/MS SIM (EPA Methods TO-15-SIM) (8/1/2011) (Attachment C), and H&P Mobile Geochemistry's "Standard Operating Procedures (SOPs), EPA Method TO-14A, TO-15 (Rev. 6, February 2011) (Attachment E) and "Standard Operating Procedures 8260 SV SOP for Determining VOC's in Soil Vapor (June 2011) attached to this Work Plan (Attachment E).

Data Validation

Results collected from the indoor air investigation will be submitted to ITSI for third-party data validation in accordance with the QAPP.

Site Safety and Health Plan

Field work under this work plan will be performed in accordance with the general portions of the Site Safety and Health Plan prepared by Clear Creek for the soil gas investigation (October 29, 2010, amended March 17, 2011). Sections 5.2 and 5.3, and

Tables 1 through 4 of that plan have been modified to apply to the sub-slab/indoor air investigation and are attached to this Work Plan as Attachment G. The work performed by subcontractors will be performed under their own respective health and safety plans.

Proposed Schedule

Freescale is prepared to begin the field program as soon as approvals are given by EPA and access for all proposed sampling locations is granted. As of this writing, a tentative schedule is as follows (this schedule may change depending upon access):

- Week 1 (July 11): Conduct interviews with residents.
- Week 2 (July 25): Conduct field work.

It is anticipated that three working days are needed to complete the interviews with residents and 5 working days are needed to complete the indoor air/sub-slab sampling program at the structures identified in this work plan

After access approvals have been secured for the indoor air evaluation, advanced notice with specific dates of the field activities will be provided to EPA in accordance with the Consent Order.

FIGURES

Air Photo Base from Google Earth

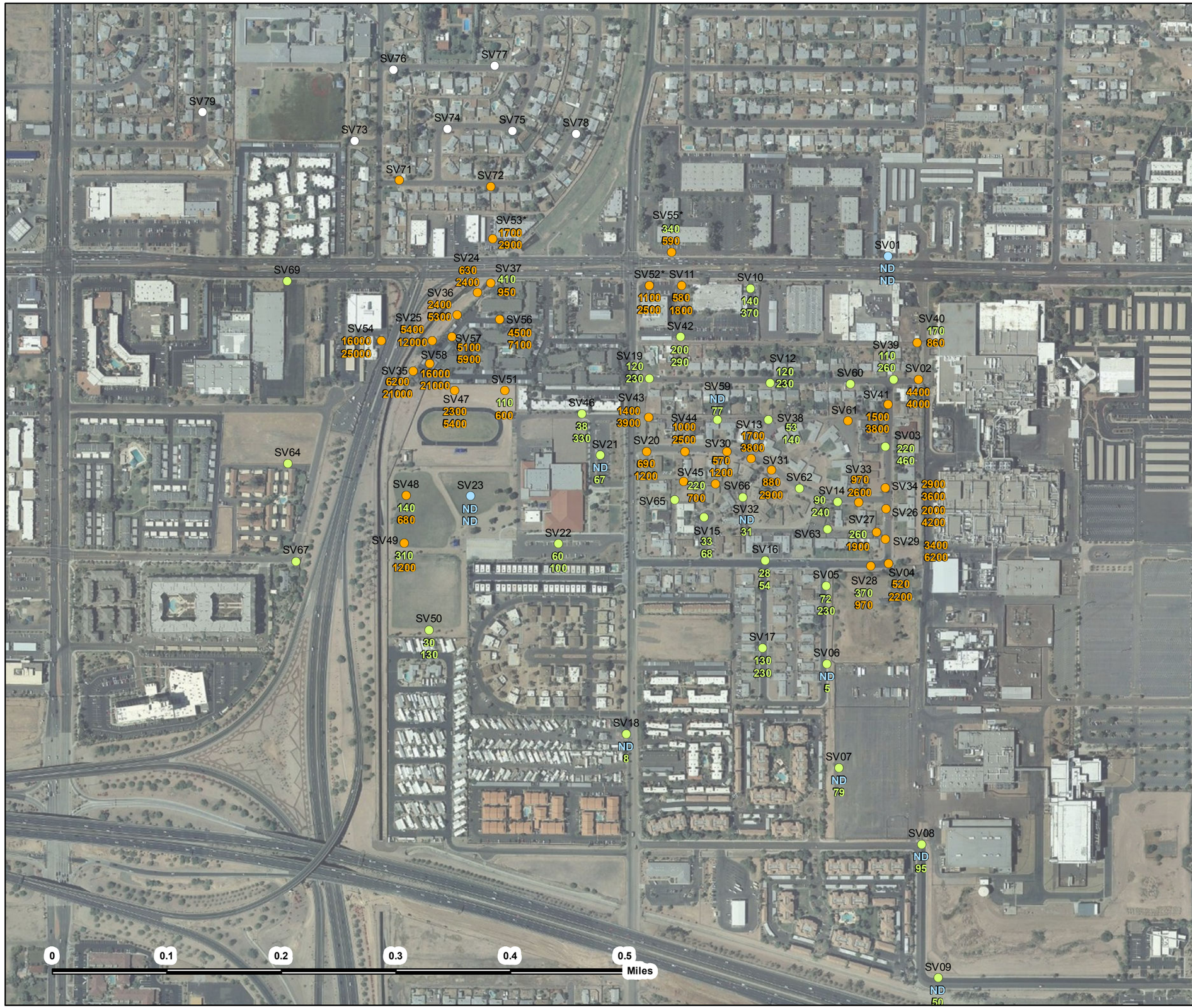
Scale

0 1,000 ft

N

Figure 1
Study Area Location
Operable Unit 1
Motorola 52nd Street Superfund Site
Phoenix, Arizona

CLEAR CREEK ASSOCIATES



Color Designation Based on Maximum Detection for Each Location Posted

- Not Yet Sampled
- TCE Not Detected
- TCE Detected Below Residential SGHHSL
- TCE Detected at or Above Residential SGHHSL

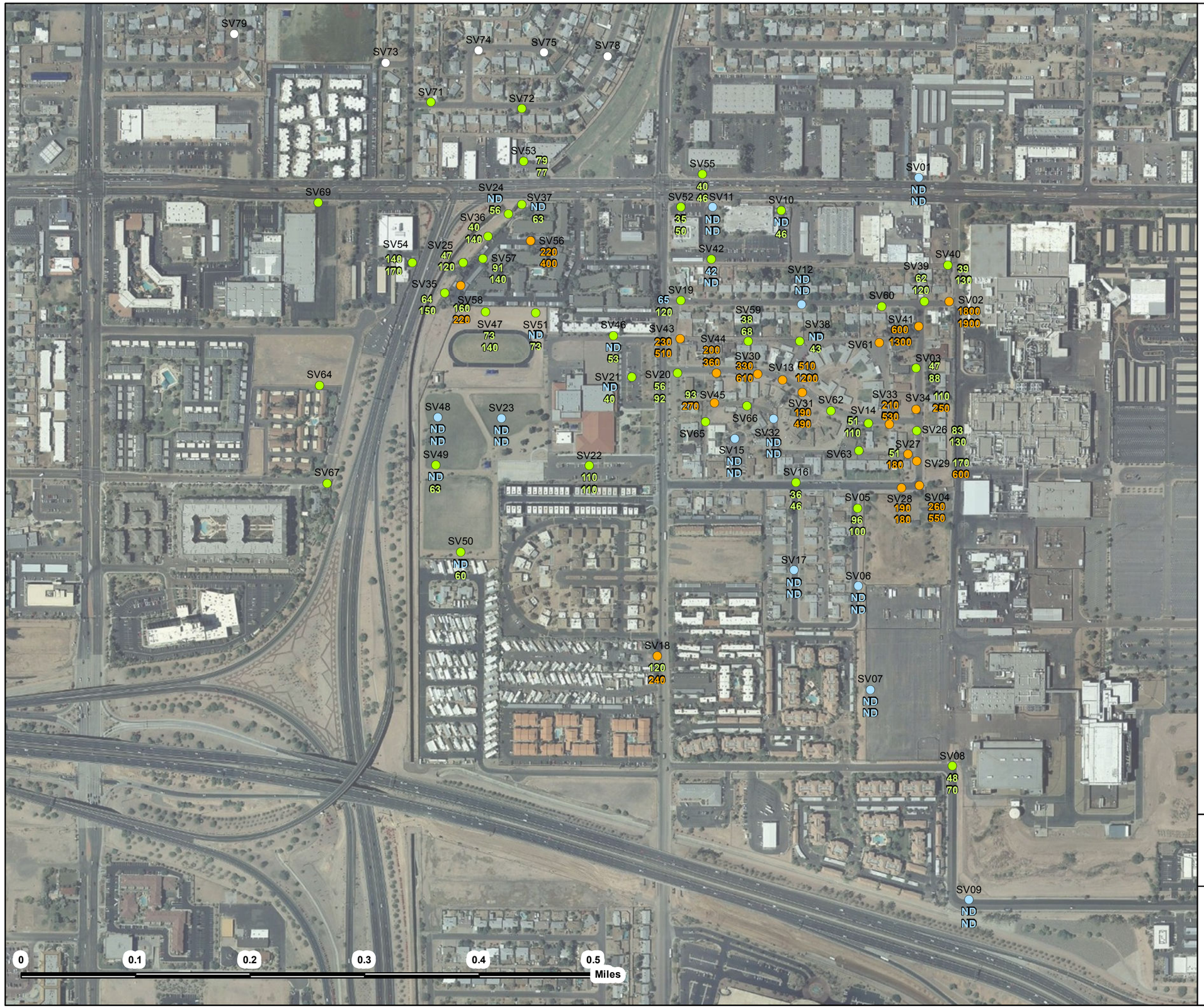
Site ID
● Sample Location
51 TCE Concentration at 5 foot Interval Below Ground Surface
110 TCE Concentration at 15 foot Interval Below Ground Surface

Notes:
1) Sample location symbol color is determind by highest analytical result reported in either 5 foot or 15 foot sample
2) Validated results shown with numeric value and color coded number
3) Preliminary unvalidated results shown color coded symbol only
4) TCE = Trichloroethene
5) ND = Not Detected
6) g/m^3 = micrograms per cubic meter
7) SGHHSL = Soil Gas Human Health Screening Level
8) TCE SGHHSL = 520 ug/m^3
9) * exceeds residential SGHHSL but below commercial SGHHSL
10) SV68 and SV70 locations are not shown; these were contingent locations that will not be sampled

CLEAR CREEK ASSOCIATES

Figure 2

Preliminary TCE Results (ug/m^3)
5 Foot and 15 Foot Intervals
Below Ground Surface
OU1 Soil Vapor Sampling, April/May 2011



Color Designation Based on Maximum Detection for Each Location Posted

- Not Yet Sampled
- PCE Not Detected
- PCE Detected Below Residential SGHHSL
- PCE Detected at or Above Residential SGHHSL

Site ID
● Sample Location
51 PCE Concentration at 5 foot Interval Below Ground Surface
110 PCE Concentration at 15 foot Interval Below Ground Surface

Notes:
1) Sample location symbol color is determind by highest analytical result reported in either 5 foot or 15 foot sample
2) Validated results shown with numeric value and color coded number
3) Preliminary unvalidated results shown color coded symbol only
4) PCE = Tetrachloroethene
5) ND = Not Detected
6) ug/m^3 = micrograms per cubic meter
7) SGHHSL = Soil Gas Human Health Screening Level
8) PCE SGHHSL = 180 ug/m^3

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Figure 3

Preliminary PCE Results ug/m^3)
5 Foot and 15 Foot Intervals
Below Ground Surface
OU1 Soil Vapor Sampling, April/May 2011

Outdoor Air Sampling Location

Area of Interest

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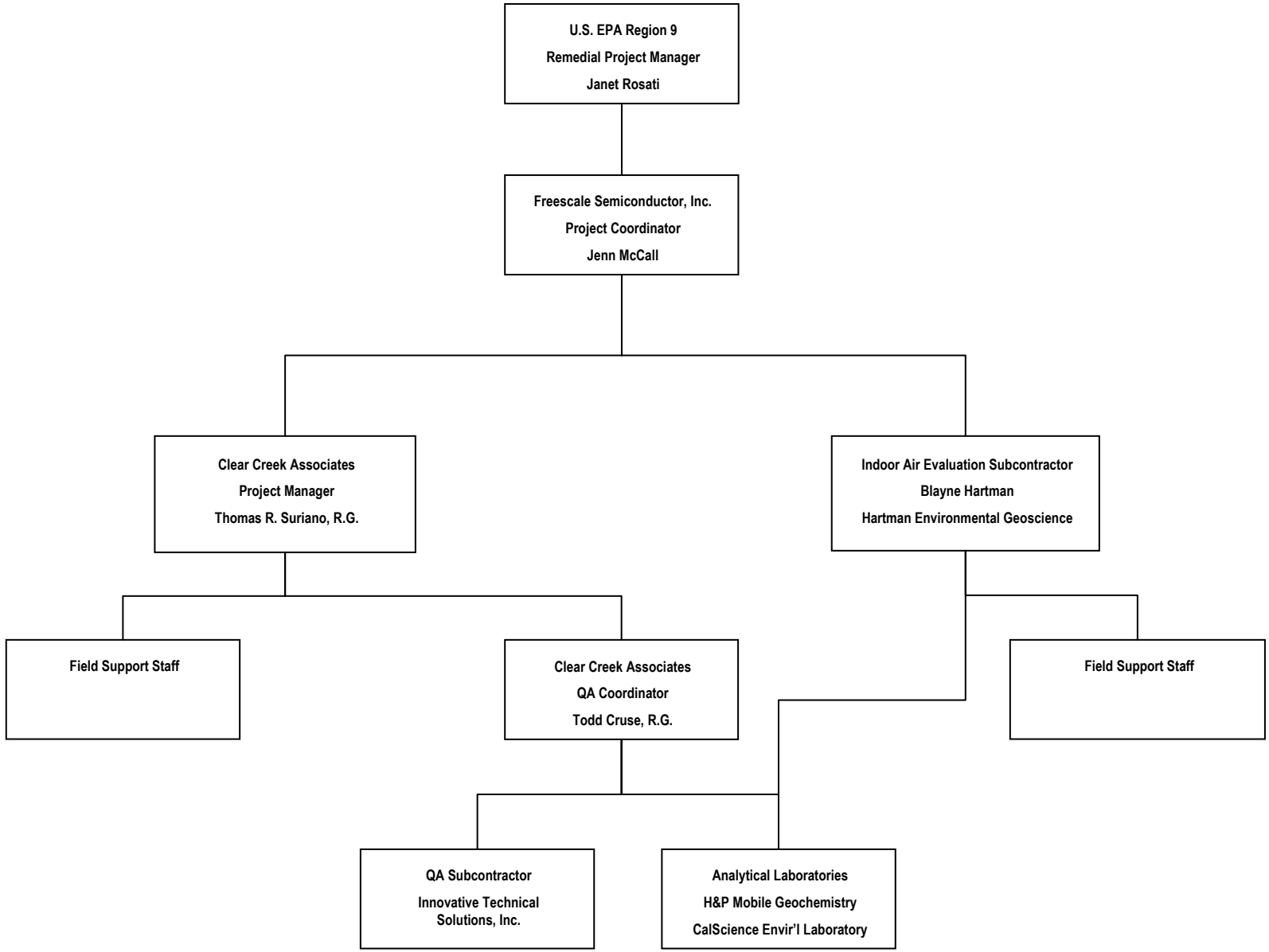
Figure 4

Areas of Interest and Outdoor Air Sampling Locations
OU1 Indoor Air Investigation, July 2011



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HARTMAN ENVIRONMENTAL GEOSCIENCE

Sub-slab and Indoor Air Sampling Investigation Organization Chart
Freescale Semiconductor, Inc.

Figure 5

July 2011

ATTACHMENT A

APPENDIX K - BUILDING SURVEY FORM

This form must be completed for each building involved in an indoor air investigation.

Preparer's name _____ Date prepared _____

Preparer's affiliation _____

Telephone number _____

1. **OCCUPANT**

Name _____

Address _____

_____

City _____

Home telephone number _____

Office telephone number _____

2. **OWNER OR LANDLORD**

Name _____
(If different than occupant)

Address _____

_____

Telephone number _____

A. Type of Building Construction

Type (circle appropriate responses): Single Family Multiple Dwelling Commercial

Ranch	Two-family
Raised Ranch	Duplex
Split Level	Office
Colonial	Warehouse
Mobile Home	Strip Mall
Apartment Building:	Number of Units _____

Other _____

Building Age _____ Number of stories _____

Area of the Building (square feet) _____

Is the building insulated? yes / no How sealed is the building? _____

Number of elevators in the building _____

Condition of the elevator pits (sealed, open earth, etc.) _____

General description of building construction materials _____

_____

B. Foundation Characteristics (circle all that apply)

1. Full basement, crawlspace, slab on grade, other _____
2. Basement floor description: concrete, dirt, wood, other _____
   a. The basement is: wet, damp, dry _____
   b. Sump present? yes / no _____Water in sump? yes / no ____
   c. The basement is: finished, unfinished _____
   d. Is the basement sealed? Provide a description _____

   _____
3. Concrete floor description: unsealed, painted, covered; with _____
4. Foundation walls: poured concrete, block, stone, wood, other _____
5. Identify all potential soil gas entry points and their size (e.g., cracks, voids, pipes, utility ports, sumps, drain holes, etc.). Include these points on the building diagram.

C. Heating, Ventilation, and Air Conditioning (circle all that apply)

1. The type of heating system(s):

Hot Air Circulation	Heat Pump
Hot Water Radiation	Unvented Kerosene Heater
Steam Radiation	Wood Stove
Electric Baseboard	Other (specify) _____

2. The type of fuel used: Natural Gas, Fuel Oil, Electric, Wood, Coal, Solar

   Other (specify) _____
3. Location of heating system: _____
4. Is there air-conditioning? yes / no Central Air or Window Units?

Specify the location _____

5. Are there air distribution ducts present? yes / no

6. Describe the supply and cold air return duct work including whether there is a cold air return and comment on the tightness of duct joints.

_____

7. Is there a whole house fan? yes / no _____
   What is the rated size of the fan? _____

8. Temperature settings inside during sampling. Note day and night temperatures.
   a. Daytime temperature(s) _____
   b. Nighttime temperature(s) _____
   (Note times if system cycles during non-occupied hours during the day)

9. Estimate the average time doors and windows are open to allow fresh outside air into the building. Note rooms that frequently have open windows or doors.

_____

D. Potential Indoor Sources of Pollution

1. Is the laundry room located inside the home? yes / no

2. Has the house ever had a fire? yes / no

2. Is there an attached garage? yes / no

3. Is a vehicle normally parked in the garage? yes / no

4. Is there a kerosene heater present? yes / no

5. Is there a workshop, hobby or craft area in the residence? yes / no

6. An inventory of all products used or stored in the home should be performed. Any products that contain volatile organic compounds or chemicals similar to the target compounds should be listed. The attached product inventory form should be used for this purpose.

7. Is there a kitchen exhaust fan? yes / no Where is it vented? _____

8. Is the stove gas or electric? _____ Is the oven gas or electric? _____

9. Is there an automatic dishwasher? yes / no

10. Is smoking allowed in the building? yes / no

11. Has the house ever been fumigated or sprayed for pests? If yes, give date, type and location of treatment.

_____

E. Water and Sewage (Circle the appropriate response)

Source of Water

Public Water Drilled Well Driven Well Dug Well Other (Specify) _____

Water Well Specifications

Well Diameter _____ Grouted or Ungrouted _____

Well Depth _____ Type of Storage Tank _____

Depth to Bedrock _____ Size of Storage Tank _____

Feet of Casing _____ Describe type(s) of Treatment _____

Water Quality

Taste and/or odor problems with water? yes / no If so, describe _____

Is the water chlorinated, brominated, or ozonated? yes / no _____

How long has the taste and/or odor problem been present? _____

Sewage Disposal: Public Sewer Septic Tank Leach Field Other (Specify) _____

Distance from well to septic system _____ Type of septic tank additives _____

F. Plan View

Sketch each floor and if applicable, indicate air sampling locations, possible indoor air pollution sources, preferential pathways and field instrument readings.

G. Potential Outdoor Sources of Pollution

Draw a diagram of the area surrounding the building being sampled. If applicable, provide information on the spill locations (if known), potential air contamination sources (industries, service stations, repair shops, retail shops, landfills, etc.), outdoor air sampling locations, and field instrument readings.

Also, on the diagram, indicate barometric pressure, weather conditions, ambient and indoor temperatures, compass direction, wind direction and speed during sampling, the locations of the water wells, septic systems, and utility corridors if applicable, and a statement to help locate the site on a topographical map.

APPENDIX L – HOUSEHOLD PRODUCTS INVENTORY FORM

Occupant of Building _____

Address _____

City _____

Field Investigator _____ Date _____

Product Description (commercial name, dispenser type, container size, manufacturer, etc.)	Volatile Ingredients in the Product	Field Instrument Reading

Comments:

_____

_____

ATTACHMENT B

Standard Operating Procedures
Indoor Air Sampling
Passivated Canisters by Method TO-15

For Vapor Intrusion Assessments

Prepared by:
Blayne Hartman, Ph.D.
Hartman Environmental Geoscience
Solana Beach, California

Sampling Procedures

Pre-Sampling Interview & Survey of Structures

Prior to sample collection, an interview will be conducted with the residents at each sampling location explaining the indoor air sampling procedures. During the interview, a survey of the structure's interior will be performed and any products known to potentially contain the target VOCs will be identified and requested to be removed from the premises approximately two (2), but no less than one (1) day prior to sample collection. The residents will also be provided a list of activities to refrain from during the indoor air sample collection period. A survey and products inventory form will be completed (example attached at end of this SOP).

Sample Hardware

One to six liter canisters with flow choke to match desired collection period. Flow chokes calibrated by the supplying laboratory and if necessary, rechecked on-site.

Sample Collection Times

Nominally 8 hours for commercial receptors and 24 hours for residential receptors. Collection times may vary depending upon project DQOs and site conditions.

Sample Collection Height

Nominally in typical breathing zone extending to approximately 3-feet to 7-feet above the ground surface.

Sample Location

Nominally in the room of most common use, unless site conditions or local regulations specify an alternative location. In structures with basements, samples are typically collected in the basement and on the first floor.

Outdoor Air Collection Period

Nominally to begin approximately 1 hour prior to indoor air collection. Canisters also deployed in breathing zone if logistically possible.

Check on Collection Rate

Approximately 4 hours after sample collection commences, if access exists, the canisters are re-checked to ensure filling at proper rate.

Field Records

The field technician maintains a log sheet summarizing:

- Sample identification
- Identity of samplers
- Date and time of sample collection start
- Location in residence
- Photo of canister in location
- Canister & flow controller IDs
- Initial & final vacuums
- Sample collection stop time
- Weather conditions
- Any unusual conditions in the structure before and after sample collection

Quality Control Procedures

This section presents QC requirements relevant to the collection and analysis of ambient air samples. The purpose of the QC program is to produce data of known quality that satisfy the project DQOs and that meet or exceed the requirements of the standard methods of analysis.

Compliance with Standards

Sampling and analytical procedures comply with the EPA method TO-15 and any local State or agency guidance.

Sample Canisters & Flow Controllers

Canisters and flow controllers for indoor and outdoor samples are typically individually tested (certified clean) prior to shipping to ensure they are contaminant free at the required detection levels.

Trip Blanks

Trip blanks are performed to ensure that the sampling canisters are contaminant free prior to sample collection. The trip blank consists of sending an evacuated canister out in the same shipment as the other canisters and returning it to the laboratory for analysis unfilled.

Duplicate Samples

Field duplicate samples are collected and analyzed to evaluate sampling and analytical precision. Field duplicates are collected and analyzed in the same manner as the primary samples. Agreement between duplicate sample results will indicate good sampling and analytical precision. Field duplicates will be collected at a frequency of approximately one field duplicate for every twenty (20%) primary samples collected. The duplicate sample will be analyzed for all laboratory analyses that were requested for the primary sample. The precision goal for field duplicate analyses will be a RPD that is less than or equal to 50%.

Corrective Action

Corrective action is taken when canisters arrive without proper vacuum, fill at the incorrect rate, parts don't fit, etc.

Instructions for Collecting Air Samples

Indoor, Outdoor, or Ambient Air Sampling

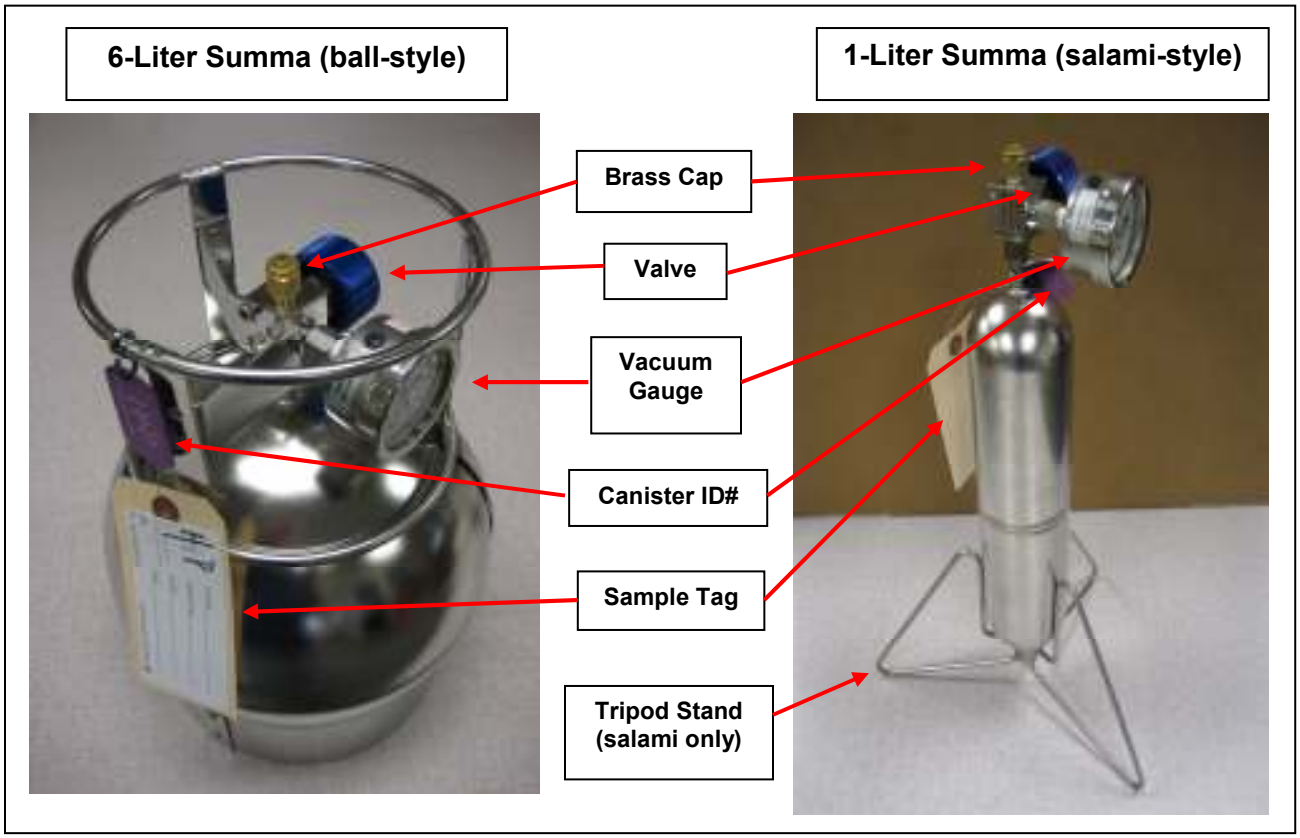
There are two modes of indoor air sampling that can be performed, "Time Integrated" or "Grab" sampling. "Time Integrated" sampling requires a calibrated flow controller to be attached to the summa canister. The flow controllers are calibrated to fill a specific volume summa canister in a specific amount of time. For "Grab" sampling, the sample is collected into the summa canister without a flow controller, allowing the canister to fill in <1 minute, providing an instantaneous sample.

Recommended Tools

- Open end 9/16" wrench
- Crescent wrench
- Ball point pen

Sample Hardware Provided

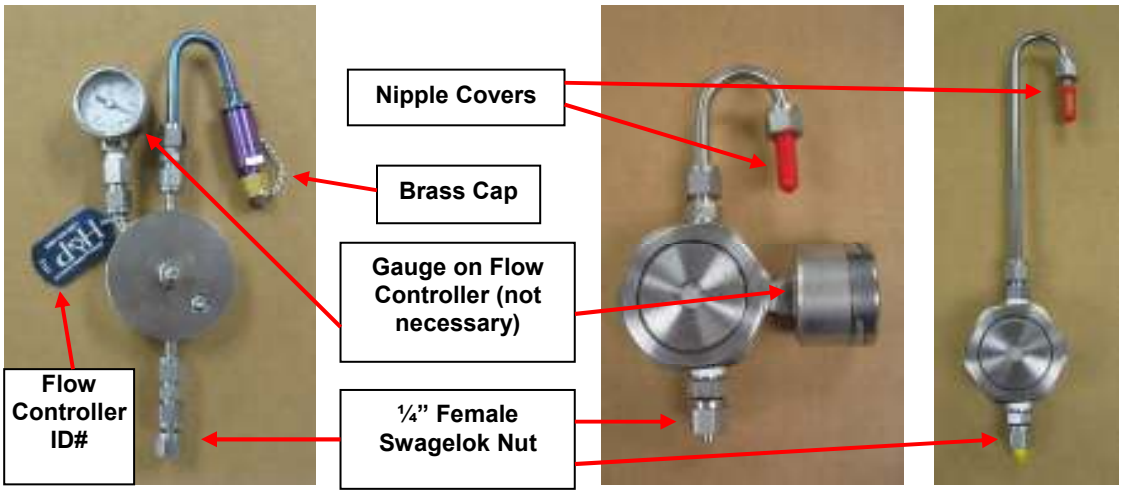
- Summa canister with Vacuum Gauge
  - *The summa will either be a 1-Liter or 6-Liter size and may be in the shape of a ball-style canister or a salami-style canister.*
- Flow controller with critical orifice & fine particulate filter
  - *There may also be a gauge on the flow controller, but this is not required.*
- If using salami-style summa canister, a canister tripod stand is included.



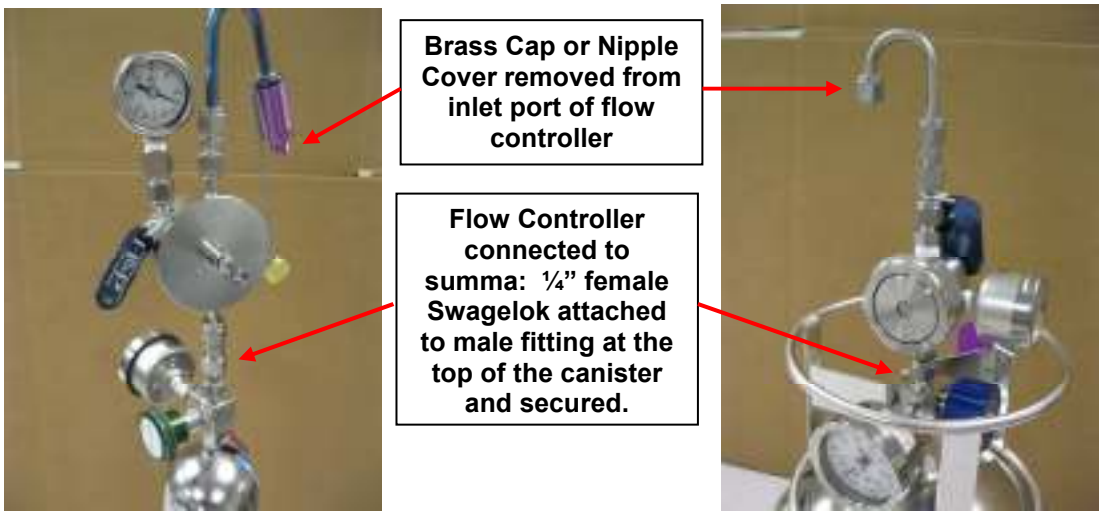
Time Integrated Sampling Procedures (Steps 1-4)

1. **Inspect the summa canister before using:** Ensure that valve is fully closed (completely clockwise). Record vacuum from gauge attached to summa canister. NOTE: Initial vacuum reading should be greater than 25" Hg. If vacuum is <25", contact H&P for replacement canister.
2. **Attaching the flow controller to the summa canister:** *If a salami-style summa is being used, place it in the tripod stand now.* Remove the brass cap on the summa canister. Attach the ¼" female Swagelok nut at the base of the flow controller to the male fitting on the top of the summa. Hand-tighten the ¼" Swagelok nut and then tighten with a 9/16" wrench.

Flow Controller Styles



Flow Controllers Attached to Summa Canisters



Time Integrated Sampling Procedures (Steps 1-4), continued…

3. **To begin sampling:** First remove brass cap or nipple cover from inlet port of flow choke (as shown in photos on page two). Open valve on canister by slowly turning counterclockwise until valve is fully open, approximately 1 to 2 full turns. Record time on log sheet as start time.
    - *A sample log sheet is provided with your shipment. It is recommended that you monitor the progress of the integrated sampling event by periodically recording the time and the vacuum reading throughout the event.*

4. **To finish sampling:** At the end of the sampling period, close valve on the canister by turning clockwise until snug. Do not over tighten. Record time on log sheet as end time. Complete the sample tag on the canister with the ID and the sample information.
    - *When collecting a time integrated air sample, it is recommended to stop the sampling and close the canister when the vacuum in the canister reaches ~-5" Hg. This procedure helps to ensure that the canister filled accordingly for a known amount of time and an accurate ending time is recorded.*

ATTACHMENT C

Title : **EPA METHOD TO-15, VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS) IN AIR USING SUMMA® CANISTERS AND SELECTIVE ION MONITORING (SIM)**

CONFIDENTIAL

Document No. : SOP-M393
Revision No. : 0.0
Supersedes : M380 6.4 – SIM-Related Sections

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MANAGEMENT **DATE**

_____ 07-28-11
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. METHOD IDENTIFICATION

1.1. EPA Method TO-15 Volatile Organic Compounds by Gas Chromatography / Mass Spectrometry in Air using Summa® canisters and Selective Ion Monitoring (SIM).

2. APPLICABLE MATRICES

2.1. This method is applicable for indoor and ambient air samples collected in 6-liter summa canisters. There can be exceptions, but be advised of the following:

2.1.1. The use of Tedlar bags for TO-15 SIM analysis is not recommended due to the low quantitation limits required and are considered to be a deviation to the TO-15 method.

2.1.2. The use of smaller summa canisters, i.e. 1-liter, may not yield sufficient volume to achieve the low detection limits reported by this method.

2.1.3. The use of the TO-15 SIM method for soil gas or sub-slab air samples may yield elevated detection limits due to the nature of the samples collected.

3. DETECTION LIMITS

3.1. The estimated quantitation limits (EQLs) generated for the compounds determined using the SIM method are approximately 0.025 ppbv, but may be higher for some analytes. The EQLs will be proportionally higher for samples that require dilution.

3.2. Please refer to Appendix 2 for standard TO-15 SIM analyte, reporting and method detection limits.

4. SCOPE AND APPLICATION

4.1. The TO-15 SIM method is used to determine the concentration of a select number of volatile organic compounds (VOCs) having a vapor pressure greater than 10^{-1} torr at 25°C at standard pressure. The low detection and quantitation limit for these analytes is determined by operating the instrument in selective ion monitoring (SIM) mode. Samples are collected in 6 liter Summa® canisters.

4.2. The following compounds can be routinely determined by this method:

Benzene	1,1-Dichloroethene	Toluene
Bromodichloromethane	c-1,2-Dichloroethene	Tetrachloroethene
Carbon tetrachloride	t-1,2-Dichloroethene	Trichloroethene
Chlorobenzene	Ethylbenzene	Trichlorofluoromethane
Chloroethane	4-Ethyltoluene	1,1,1-Trichloroethane
Chloromethane	Hexachloro-1,3-butadiene	1,1,2-Trichloroethane
Chloroform	Methyl-t-Butyl Ether (MTBE)	1,3,5-TrimethylBenzene
Dibromochloromethane	Methylene chloride	1,1,2,2-Tetrachlorethane
Dichlorodifluoromethane	o-Xylene	1,2,4-TrimethylBenzene
1,1-Dichloroethane	p/m-Xylene	Vinyl Chloride
1,2-Dichloroethane	1,1,2-Trichloro-1,2,2-Trifluoroethane	1,1-Difluoroethane*

4.3. Additional analytes that may be analyzed for using TO-15 SIM (by request only):

Acetone	Hexane	Isopropanol*
Benzyl Chloride	Cyclohexane	Naphthalene
Bromoform	Heptane	1,4-Dioxane
Bromomethane	1.2.4-trichlorobenzene	2-butanone
Carbon Disulfide	1.2-dibromoethane	2-hexanone
Ethanol	Dichlorotetrafluoroethane	4-methyl-2-pentanone
Styrene	1.2-dichlorobenzene	1,3-Butadiene
c-1,3-dichloropropene	1.3-dichlorobenzene	1.2-dichloropropane
t-1,3-dichloropropene	1.4-dichlorobenzene`	

* leak check compound

4.4. This method is restricted to use by or under the supervision of analysts and supervisors who are experienced in the use of GC/MS and skilled in the interpretation of mass spectra both in scan and SIM mode.

5. METHOD SUMMARY

5.1. EPA Method TO-15 SIM describes chromatographic procedures that will allow for the separation of volatile organic compounds and their qualitative and quantitative analysis by mass spectrometry.

5.2. A known volume of sample is directed from the Summa® canister to a multi-module (empty trap, tenex, cryofocuser) concentrator. The sample is first dehydrated by passing through a cold, empty trap, and then cryogenically concentrated on Tenax. Post concentration, the VOC's are thermally desorbed onto a gas chromatographic column for separation prior to detection.

5.3. Detection is achieved using a mass selective detector and quantitation is performed using the selective ion monitoring mode. With the selected ion monitoring technique, the mass spectrometer is not scanned over all masses; instead, the instrument jumps from one selected mass to another allowing the MS to spend more time at a given mass, thus improving the signal-to-noise ratio at a mass and therefore the sensitivity at that mass.

6. DEFINITIONS

6.1. Abundant: An ion that is easy to detect at low concentrations

6.2. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.3. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.4. Analytical Batch: Samples, which are analyzed together as a group with the same process and personnel, using the same reagents and can exceed 20 samples.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the calibration standards should bracket the range of expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Dwell: The amount of time (in msec) spent monitoring each m/z value during SIM. A dwell time can be specified for each group.

6.8. Group: The set of m/z values monitored during a given SIM cycle. The maximum group size is 30 ions. Fifty groups of up to 30 ions can be created.

6.9. Holding Times: The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.10. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.11. Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory precision and bias and to assess the performance of the measurement system.

6.12. Method Blank: A sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.13. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.14. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.15. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.16. Selective ion Monitoring (SIM): A sensitivity enhancement technique, where the mass spectrometer is programmed to scan for only those ions that are pertinent to the compounds of interest (2-3 mass ions scanned per compound) while ignoring non-essential ions.

6.17. Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for QC purposes.

7. INTERFERENCES

7.1. Contamination by carryover can occur whenever high and low concentration level samples are analyzed sequentially. Suspected high level samples should be diluted and then analyzed at the end of the sequence to prevent carryover contamination. The analysis of blanks after high concentration samples is encouraged.

7.2. Interference can also occur when "dirty" samples leave residue in the column. The column can be "baked" after such samples.

7.3. The following provides information regarding possible interferences and target analyte anomalies during analytic processing:

7.3.1. Very volatile compounds such as Chloromethane and Vinyl Chloride can display peak broadening and co-elution with other species if the compounds are not delivered to the column in a small volume or carrier gas. Refocusing of the sample after collection on the primary trap, either on a separate focusing trap or at the head of the column, mitigates this problem.

7.3.2. Interferences in canister samples may result from improper use or from contamination of: 1) the canisters themselves due to poor manufacturing, 2) the canister cleaning apparatus, or 3) the sampling or analytical system. Attention to the following details will help to minimize the possibility of contamination of canisters.

7.3.2.1. New canisters are cleaned per SOP T-016, filled with high-purity Nitrogen and then analyzed to determine cleanliness. Pending and post analysis, canisters should be stored in a contaminant-free location.

7.3.2.2. Impurities in the calibration dilution gas and carrier gas, organic compounds out-gassing from system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system itself must be demonstrated free of contamination by the periodic analysis of humidified zero air blanks.

7.3.3. Best efforts must be taken to maintain the analytical areas free of all contaminants that include target analytes that are common solvents or reagents in other areas of the laboratory. This can be minimized by restricting entry of these solvents or solvent-contaminated items (e.g., laboratory coats) into the VOC laboratory and proper management / maintenance of laboratory ventilation.

7.3.4. Other major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-Polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber BUNA-N components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should locate and remove the source of contamination before proceeding with calibration and analysis.

7.3.5. Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from chromatographic grade stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during SVOC extraction procedures may contribute to sample contamination.

7.4. Summa Canister Cleaning is referenced under SOP T-016.

8. SAFETY

8.1. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats should be worn in the air laboratory. Protective gloves shall be worn when handling liquid or solid chemicals.

8.2. The following compounds covered by this method have been tentatively classified as known or suspected human carcinogens: benzene, methylene chloride, chloroform, Bromoform, Bromodichloromethane, Chlorodibromomethane, and Vinyl Chloride. Primary standards of these toxic compounds must be prepared in a hood.

8.3. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. EQUIPMENT AND SUPPLIES

9.1. Gas Chromatograph:

9.1.1. Agilent 6890 Gas Chromatograph configured with a PC based data system - Chemstation or equivalent.

9.2. Entech 7016 Autosampler or equivalent

9.3. Entech 7100 Cryogenic Concentrator or equivalent

9.4. Mass Spectrometer:

9.4.1. Agilent 5973 Mass Selective Detector (MSD) or equivalent capable of operating in Selective Ion Monitoring (SIM) mode.

9.4.2. The MSD must be capable of producing a mass spectrum for BFB that meets all of the criteria in Section 12.1 below, when 100ppbv of BFB is injected.

9.5. Chromatographic Column:

9.5.1. J&W DB-1 or Restek RTX-1, 60M, 0.32mm ID, 1.0μ film or equivalent.

9.6. 6-liter Summa® or Silonite Polished Canisters, or equivalent – certified clean to the MDL or RL as needed, for samples, QC and Standards.

9.7. Environics Series 2000 Computerized Multi-Component Gas Mixer.

9.8. 2-stage regulators capable of handling pressures up to 4000psi and 200psi

9.9. Stainless steel or inert teflon tubing with Swagelock fittings, or equivalent

9.10. Gas Tight Syringes:

9.10.1. Hamilton 702N gas tight Leurlock type w/ on-off valves or equivalent, sizes 1mL, 2.5mL, 5mL, 25mL, 50mL, and 100mL.

10. REAGENTS AND STANDARDS

10.1. REAGENTS

10.1.1. Zero Air

10.1.2. Reagent grade water for humidifying Summa canisters

10.2. STANDARDS

10.2.1. A tuning standard containing 100ppbv of 4-bromofluorobenzene (BFB) made up in a tedlar bag. The tuning standard also contains the internal and surrogate standards in the same amount as samples, QC and standards.

10.2.1.1. 64mL of the tuning standard is injected onto the instrument

10.2.2. Internal and Surrogate standards – 1 canister:

10.2.2.1. Internal Standards: Bromochloromethane, Chlorobenzene-d_5 and 1,4-Difluorobenzene each at a nominal concentration of 2.0ppbv.

10.2.2.2. Surrogates: 1,4-Bromoflurobenzene (also used as the tuning standard), 1,2-Dichloroethane-d_4 and Toluene-d_8 each at a nominal concentration of 2.0ppbv.

10.2.2.2.1. 100mL of Internal/Surrogate standard is analyzed with every sample, QC sample, and method blank. This is equivalent to a 0.5ppbv standard.

10.2.3. Primary Calibration Standards in 2 canisters containing each target analyte at a nominal concentration of 1000ppbv.

10.2.4. Intermediate Calibration Standard – 25ppbv

10.2.4.1. The 25ppbv intermediate calibration mix (Ethanol at 400ppbv) is made by blending 20ml/min of each canister with 760ml/min of zero air in an evacuated Summa® canister.

10.2.4.2. Press RECALL and 04 followed by START or UPDATE on the Environics Series 2000 Computerized Multi-Component Gas Mixer. The Primary standard canisters are hooked up to the gas mixer and are ready for use. Input the appropriate volumes of the standards and the zero air to make up the working standards.

10.2.5. Working Initial and Continuing Calibration Standards

10.2.5.1. Two working calibration standards are made by direct dilution from the 25ppbv intermediate calibration standard – 0.25ppbv and 1.0ppbv. The other four calibration standards are made by dilution of, or injecting lesser volumes of, the two working calibration standards.

10.2.5.1.1. The 0.25ppbv working calibration standard is made by blending 10mL of the 25ppbv intermediate standard with 990ml/min of zero air in an evacuated 6 liter Summa® canister.

10.2.5.1.2. The 1.0ppbv working calibration standard is made by blending 40mL of the 25ppbv intermediate standard with 960ml/min of zero air in an evacuated 6 liter Summa® canister.

10.2.5.2. The 0.025ppbv calibration standard is made by injecting 40mL of the 0.25ppbv working calibration standard onto the instrument.

10.2.5.3. The 0.05ppbv calibration standard is made by injecting 80mL of the 0.25ppbv working calibration standard onto the instrument.

10.2.5.4. The 0.10ppbv calibration standard is made by injecting 160mL of the 0.25ppbv working calibration standard onto the instrument.

10.2.5.5. The 0.25ppbv calibration standard is made by injecting 100mL of the 1.0ppbv working calibration standard onto the instrument.

10.2.5.5.1. Alternately, 400mL of the 0.25ppbv working calibration standard may be injected used.

10.2.5.5.2. This standard is also used for the daily CCV.

10.2.5.6. The 0.50ppbv calibration standard is made by injecting 200mL of the 1.0ppbv working calibration standard onto the instrument.

10.2.5.7. The 1.0ppbv calibration standard is made by injecting 400mL of the 1.0ppbv working calibration standard onto the instrument.

10.2.6. Second Source Verification Standard (ICV/LCS) - 2 canisters:

10.2.6.1. The second source standard is made from two different canisters (a separate source from the ICAL standard) by blending 20ml/min of each second source canister with 760ml/min of zero air in a Summa® canister to create a 25ppbv standard.

10.2.6.1.1. Press RECALL 06 followed by START or UPDATE on the Environics Series 2000 Computerized Multi-Component Gas Mixer. The secondary source standard canisters are hooked up to the gas mixer and are ready for use. Input the appropriate volume of the standard and the zero air to make up the working ICV/LCS standard.

10.2.6.1.2. Attach and fill the evacuated LCS 6 liter Summa® Canister.

10.2.6.2. The 25ppbv standard is then used to create a 0.25ppbv second source ICV/LCS standard by blending 40ml/min of the 25ppbv standard with 960ml/min of zero air to create a 1.0ppbv standard.

10.2.6.2.1. 100mL of the 1ppbv standard is injected onto the instrument.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Air samples collected in Summa® canisters must be analyzed within 30 days of collection. Preservation is not required (store at ambient temperature).

11.1.1. Regulatory agencies may require shorter hold times than 30 days. It is the responsibility of the Client to relay differing requirements as appropriate to Calscience. From there it is the responsibility of the Calscience Project Manager to relay this information to the Air group.

11.2. Air samples collected in Tedlar™ bags must be analyzed with 3 days of collection. Preservation is not required (store at ambient temperature).

11.2.1. When analyzed in Tedlar™ bags, the report results will include a comment noting a deviation to the TO-15 method as the EPA TO-15 method makes no reference to Tedlar™ bags as a sampling medium.

12. QUALITY CONTROL

12.1. Hardware Tuning

12.1.1. An acceptable tune demonstrates satisfactory hardware performance. A tune meeting the acceptance criteria is required and MUST be recorded prior to any ICAL, CCV, QC or sample analyses.

12.1.2. Prior to running the calibration standard(s) or CCVs, the GC/MS BFB tuning standard must be analyzed and meet the following acceptance criteria:

Mass	Ion Abundance Criteria
50	15 - 40% of mass 95
75	30 - 60% of mass 95
95	Base peak, 100% relative abundance[Note 1]
96	5 - 9% of mass 95
173	< 2% of mass 174
174	> 50% to 120% of mass 95
175	5 - 9% of mass 174
176	Greater 95% but less than 101% of mass 174
177	5 - 9% of mass 176

Note 1: All ion abundances must be normalized to mass 95, the nominal base peak, even though the ion abundance of mass 174 may be up to 120% of mass 95.

12.1.3. These criteria must be demonstrated initially and, at a minimum, every 24 hours thereafter, prior to any other analyses.

12.1.4. If a tune does not meet the acceptance criteria, correct the problem and re-tune the system.

12.1.5. Whenever invasive maintenance of the GC/MS hardware is performed, the system must be re-tuned.

12.2. Initial Calibration

12.2.1. A minimum of a five-point, usually six-point, initial calibration must be established prior to the processing of samples.

12.2.2. The initial calibration is deemed valid if:

12.2.2.1. The %RSD for the RF for each target analyte is <30% with, at most, two exceptions up to a limit of 40%.

12.2.2.2. The %RSD for the RRT for each target analyte at each concentration level is within 0.06 RRT units of the mean RRT of the target analyte.

12.2.2.3. The internal standard area response Y at each calibration level must be within 40% of the mean Y over the initial calibration range.

12.2.2.4. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

12.3. Initial Calibration Verification (ICV)

12.3.1. Immediately following establishment of a valid initial calibration, an ICV standard must be analyzed. The ICV is the second source standard used for the LCS/LCSD.

12.3.1.1. The ICV is deemed valid if the %D for each target analyte is ±30%.

12.3.1.2. If these criteria are not met, the ICV/LCS is deemed unacceptable for sample analysis to resume. Reanalyze the ICV/LCS.

12.3.1.2.1. If the ICV/LCS criteria remain unacceptable, effect corrective action and re-tune/recalibrate. If acceptable, proceed with sample analysis.

12.3.1.3. The internal standard area responses and retention times in the ICV must be evaluated after data acquisition. If the retention time for any internal standard changes by more than 20 seconds from the mean retention time in the initial calibration, the chromatographic system must be inspected for malfunctions and corrective action must be effected.

12.3.1.4. If the area response for any internal standard changes by +/- 40% from the mean area response in the initial calibration, the system must be inspected for malfunctions and corrective action effected. Following corrective action, reanalyze the ICV. If still out, recalibrate.

12.4. Continuing Calibration Verification (CCV)

12.4.1. Following establishment of a valid initial calibration and every 24 hours thereafter, a CCV standard must be analyzed following the tune standard.

12.4.1.1. The CCV is deemed valid if the %D for each target analyte is ±30%.

12.4.1.2. If these criteria are not met, the CCV is deemed unacceptable for sample analysis to resume. Reanalyze the CCV. If the CCV criteria remain unacceptable, effect corrective action and re-tune/recalibrate.

12.4.1.3. The internal standard area responses and retention times in the CCV must be evaluated after or during data acquisition. If the retention time for any internal standard changes by more than 20 seconds from the last CCV (24 hours), the chromatographic system must be inspected for malfunctions and corrective action must be effected.

12.4.1.4. If the area response for any internal standard changes by more than +/- 40% (Range: 60% to 140%) from the mean area response of the initial calibration, the system must be inspected for malfunctions and corrective action effected. Following corrective action, re-analysis of samples analyzed while the system was malfunctioning is required.

12.4.1.5. If these criteria are not met then all samples analyzed since the last acceptable CCV should be invalidated, corrective action effected, and the affected samples re-analyzed. If a failed CCV is the first of the day, corrective action must be effected prior to analyzing any samples.

12.5. Event Based Quality Control (MB, BB and LCS/LCSD)

12.5.1. Method Blank: (MB):

12.5.1.1. The Method Blank is an instrument blank analyzed directly from the autosampler. In the processing of the MB, the surrogates and internal standards identical to those for actual samples are used.

12.5.1.2. Additionally, a summa blank is also analyzed to monitor the autosampler process.

12.5.1.2.1. The summa blank is pressurized with humidified Zero Air and is processed concurrently with the associated field samples.

12.5.1.2.2. One method blank is required every 20 samples.

12.5.1.3. Ideally, the concentration of target analytes in a MB should be less than the respective reporting limits (RLs). (Less than ½ the RL for DOD work) If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs is as follows:

12.5.1.3.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

12.5.1.3.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-analyzed.

12.5.1.4. Internal Standard Retention Times (RT) must be +/- 0.33 minutes.

12.5.1.5. The area response for each internal standard in the method blank must be within ±40% (Range: 60% to 140%) of the mean area response of the internal standards in the most recent initial calibration.

12.5.2. Tedlar Bag Blank (BB):

12.5.2.1. Although unlikely, samples received in Summa canisters for SIM analysis may require dilution to account for high levels of hydrocarbons, non-target or target analytes. When the dilution cannot be performed directly from the summa canister, the dilutions are performed in tedlar bags.

12.5.2.2. To monitor potential contamination from the tedlar bags used for dilution, a Bag Blank (BB) is analyzed along with the standard method blank.

12.5.2.3. The acceptance and reporting criteria for the bag blank are as follows.

12.5.2.3.1. If no target analytes are detected, batch all sample analyses with the MB and file the BB raw data with the QC paperwork.

12.5.2.3.2. If target analytes are detected in the BB but not in the Tedlar bag dilution samples, batch all sample analyses with the MB and file the BB raw data with the QC paperwork.

12.5.2.3.3. If target analytes are detected in the BB and in the corresponding Tedlar bag sample dilutions, batch the dilutions with the BB and file the BB raw data with the QC paperwork.

12.5.2.3.3.1. Create separate batches for the MB and the BB. Summa analyses are batched with the MB. Tedlar bag dilutions are batched with the BB. Flag results as needed.

12.5.2.4. Internal Standard Retention Times (RT) must be +/- 0.33 mins.

12.5.2.5. The area response for each internal standard in the method blank must be within ±40% (Range: 60% to 140%) of the mean area response of the internal standards in the most recent initial calibration.

12.5.3. Lab Control Sample (LCS/LCSD):

12.5.3.1. The LCS is a known gaseous matrix containing a known concentration of specific target analytes (0.25ppbv). The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCSD is a duplicate of the LCS.

12.5.3.2. The LCS/LCSD is processed concurrently with the associated samples. In the processing of the LCS/LCSD, reagents and procedures identical to those for actual samples are used. The LCS/LCSD should be run after the CCV and is required for every batch of 20 samples or portion thereof, whichever is more frequent.

12.5.3.3. In addition to assessing the accuracy of the analytical measurement, the LCSD, in combination with the LCS, is used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD).

12.5.3.4. The acceptance criteria for LCS/LCSD compounds vary depending upon historical data. The upper and lower acceptance limits for each LCS/LCSD compound are based upon the historical average recovery ±3S.

12.5.3.5. All LCS/LCSD compounds must be within the current historical control limits as noted in the table in Appendix 2. If one or more LCS/LCSD compounds are not acceptable, the problem must be identified and corrected.

12.5.3.6. Internal Standard Retention Times (RT) must be +/- 0.33 minutes.

12.5.3.7. Internal Standard Retention Areas must be +/- 40% (Range: 60% to 140%) of initial calibration

12.6. Sample Based Quality Control (Surrogates)

12.6.1. The acceptance criteria for surrogate spike compound recoveries vary depending upon historical data. The upper and lower acceptance limits for each surrogate spike compound is based upon the historical average recovery ± 3S.

12.6.2. The % recovery criteria for standard analyte reporting is currently as follows:

Analyte	% Recovery
1,4-Bromofluorobenzene	45-153
1,2-Dichloroethane-d4	37-163
Toluene-d8	73-121

12.6.2.1. If the surrogate compound recoveries are acceptable, report the surrogates and sample data without qualification.

12.6.2.2. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high analyte concentration or a problem may have occurred during extraction.

12.6.2.3. The data alone cannot be used to evaluate the precision and accuracy of individual sample analyses. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

12.6.2.4. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.

12.6.2.4.1. Check the internal standard and surrogate spiking mixtures for degradation and contamination.

12.6.2.4.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate(s) recovery, the sample should be re-analyzed.

12.6.2.4.3. If incorrect procedures or degraded/contaminated spiking mixtures are determined to have not caused the unacceptable surrogate recoveries, the affected sample(s) must be re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

12.6.2.4.3.1. If, upon re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

12.6.2.4.3.2. If the MB surrogates are unacceptable, all associated sample data must invalidated and all associated samples re-analyzed.

12.6.3. Sample Based Quality Control (Internal Standards)

12.6.3.1. Internal Standard Retention Times (RT) must be +/- 0.33 minutes.

12.6.3.2. Internal Standard Retention Areas must be +/- 40% (Range: 60% to 140%) of initial calibration.

12.7. Method Detection Limit (MDL):

12.7.1. An MDL must be established prior to the analysis of samples, whenever major instrument changes are made, or annually following the procedures outlined in SOP-T006, Determination of Detection Limits.

12.7.2. The formula for calculating the MDL is listed Section 15.6.

12.7.3. MDLs should be verified immediately following the establishment of the MDL at >1x - 4x the MDL value for each analyte. MDL's may then be re-verified on a quarterly or annual basis as required by project or client requirements.

12.8. Demonstration of Capability

12.8.1. Performance of this method is restricted to analysts experienced in the use of the instruments and apparatus required in executing this method and interpretation of mass spectra.

12.8.2. Each analyst must demonstrate the ability to generate acceptable results and quality control prior to the analysis of billable samples.

12.8.2.1. Prior to beginning the analysis, analysts are required to initially demonstrate method competence by generating 4 LCSs that meet the acceptance criteria for the method. Additionally, analysts are required to generate continuing demonstrations of capability on an annual basis to show continued proficiency.

12.9. Additional information regarding internal QC checks is provided in SOP-T020.

13. CALIBRATION AND STANDARDIZATION

13.1. Prior to the analysis of samples or QC samples, the GC/MS system must be hardware tuned and an initial calibration established. The acceptance criteria for the tuning parameters are listed in Section 12.1.

13.2. Tuning:

13.2.1. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of the BFB. Do not background subtract part of the BFB peak.

13.2.2. Additionally, baseline separation of Benzene and Carbon Tetrachloride is an indication of acceptable chromatographic performance and must be achieved prior to establishment of the calibration. If not, check or replace the column.

13.3. Initial and Continuing Calibration :

13.3.1. An initial six-point calibration is obtained by injecting 0.025, 0.050, 0.10, 0.25, 0.50 and 1.0ppbv standards into the GCMS.

13.3.1.1. The ICAL is acceptable if the %RSD for the RF for each target analyte is ≤30% with the exception of two target analytes, to a limit of ≤40%.

13.3.2. Prior to the analysis of any samples or QC samples and after obtaining an acceptable initial calibration, the ICAL must be verified by the analysis of an Initial Calibration Verification standard (ICV/LCS).

13.3.2.1. The ICV is deemed valid if the %D for each target analyte is ±30%.

13.3.3. To verify the continued validity of the ICAL, a continuing calibration verification (CCV) must be analyzed prior to analyzing any QC or samples. This is done daily at the start of each batch, following the tuning standard.

13.3.3.1. The CCV is deemed valid if the %D for each target analyte is 30%.

14. PROCEDURE

14.1. Samples are initially analyzed directly from the canister (1x dilution) without pressurization. In the event that matrix effects or high levels of target or non-target analytes are present, dilutions will be required. The reporting and detection limits will be elevated by the dilution factor.

14.2. Samples should be loaded in the following order:

- Tuning Standard
- Continuing Calibration Verification (CCV)
- Laboratory Control Sample (LCS)
- LCS Duplicate (LCSD)
- Method Blank (MB)
- Samples (up to 20)

14.2.1. Summa® canisters are usually analyzed as received from the field (at vacuum) but some may need to be pressurized prior to analysis. Refer to SOP, T-016 for pressurization procedures.

14.3. Attach to the samples to the autosampler "tree" by using the metal Swage-type connections for Summa® canisters.

14.4. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the run logbook.

14.5. Initiate the sequence.

14.6. SIM Analysis – Set-up and Theory

14.6.1. Selected Ion Monitoring (SIM) allows the mass spectrometer to detect specific compounds with very high sensitivity. In SIM mode, the instrument is set to gather data at masses of interest, instead of stepping the mass filter over a wide range of masses. Since the mass spectrometer collects data at only the masses of interest, it responds only to those compounds that possess the selected mass fragments. In essence, the instrument is focused on only the compounds of interest. Also, because only a few masses are monitored, much more time may be spent looking at these masses, with the attendant increase in sensitivity.

14.6.2. SIM also allows the collection of more points across a chromatographic peak, thus enhancing the accuracy and precision of quantitative results. To achieve the potential benefits of SIM, careful attention must be paid to the acquisition parameters. In general, the following items need to be addressed prior to, during and after analysis has been completed:

14.6.2.1. Select appropriate ions to monitor for each target compound.

14.6.2.2. Group signals to be monitored into time programmed SIM groups to minimize the number of ions acquired at any one time.

14.6.2.3. Select a dwell time for each group that yields 15-20 cycles across a peak.

14.6.3. Selecting appropriate ions to monitor:

14.6.3.1. It is common practice to monitor at least two, possibly three ions per surrogate or target analyte. One ion signal is used to quantitate, and the others are used for qualitative information. Since, by definition, internal standards are present in every sample, it is common practice to monitor only one or possibly two ions for the internal standard: the quant ion and possibly one qualifier.

14.6.3.2. Determination of the SIM ions is of utmost importance. For most analytes that are analyzed by the SIM technique, the method has already been established and the ions predetermined. Refer to Appendix 1, Table 1 for the current ions.

14.6.3.2.1. If you are adding new analytes you will need to reference another source for those analytes without predetermined SIM criteria. These references could come from a full list TO-15 scan analysis if available in the lab, or you may need to analyze a scan-mode spectrum of the compounds you plan to analyze by SIM.

14.6.3.3. When examining the spectrum for candidate SIM ions, consider ions that are:

14.6.3.3.1. Unique to the compound (not common in a wide range of compound spectra). If selecting a derivatized compound, do not select ions that represent only the portion of the molecule added by the derivatizing step.

14.6.3.3.2. Higher in mass (usually more specific and separated from interference). Ions of higher mass to charge are usually more unique.

14.6.3.3.3. Abundant. If you chose an ion of very low abundance (albeit very unique), this compound may not be found at low concentrations.

14.6.3.3.4. Characteristic of the compound class (in some cases). For example, if you are looking aromatic hydrocarbons, the molecular ion is typically very abundant (possibly the base peak). This would be an excellent choice for a quant ion or a qualifier.

14.6.3.4. Once you have chosen the fragments to be monitored, it is important to determine the accurate mass (± 0.1 AMU) for the fragments you will monitor. The fragment labeled m/z 371 may be more accurately measured as 371.2 or 371.3. This deviation from whole numbers is due to the mass defect of certain elements, and the measured mass is called the Mass Centroid.

14.6.3.4.1. For optimum sensitivity and stability, you should monitor the Mass Centroid in your SIM Method to ± 0.1 AMU.

14.6.3.5. There are two ways to determine the Mass Centroid and the validity of these measurements depends on an accurately calibrated mass axis. (Insure the mass axis is accurately calibrated before proceeding.)

14.6.3.5.1. Tabulate a full scan spectrum. Prepare a standard sample of sufficient concentration to obtain scan spectra of the compounds of interest. Inject the standard sample then tabulate the mass spectrum for several data points across the apex of the peak in the resulting data file. Use the m/z value shown in the tabulation (to the nearest 0.1 AMU) for the SIM acquisition mass.

14.6.3.5.1.1. If necessary, take the mathematical average of several scans across the apex to the nearest 0.1 AMU.

14.6.3.5.2. Perform a dynamic SIM calibration. Set up a SIM acquisition for several incremental masses around the ion of interest.

14.6.3.5.2.1. For example, if you are looking for the 76 and 91 ions in toluene, do a SIM acquisition for the 75.9, 76.0, 76.1, 76.2, 90.9, 91.0, 91.1 and 91.2 ions.

14.6.3.5.3. Evaluate the mass spectrum and use the strongest ion for each of these fragments in your SIM Method.

14.6.4. Grouping ions into SIM groups:

14.6.4.1. To gain the benefits of SIM, one must limit the number of ions being monitored at any one time. ChemStation allows the monitoring of 50 simultaneous ions, but monitoring more than 10-15 ions at the same time defeats the purpose of SIM and should be avoided.

14.6.4.1.1. Most SIM analyses monitor 3 to 8 ions simultaneously in a given time period or range. If more ions are needed to do your analysis, try to separate them into SIM groups.

14.6.4.2. A SIM group is a group of simultaneously monitored ions. Only one group can be monitored at a given time, and the start time for group 2 becomes the end time for group 1. Using SIM groups, a Method can monitor the important ions for a list of compounds by switching from one group to another at the appropriate time during the chromatographic run. You would only look at the ions of interest, in the time range of interest.

14.6.5. Determining an appropriate dwell time

14.6.5.1. Once the fragments are chosen, the Mass Centroids are determined, and the ions grouped into SIM groups, you will need to set the dwell time for each group.

14.6.5.2. If your groups contain 2-5 ions, try 50msec as a first value for dwell time. To determine what adjustment needs to be made in the dwell time setting, you will need to run the Method once and examine the resulting data. The central goal in adjusting the dwell time is to optimize cycle time to get 15 to 20 points or scans across your chromatographic peak(s). Evaluate the data acquired with the dwell time set to 50msec, and determine what adjustment, if any, is required, as follows:

14.6.5.2.1. Load the data file acquired with your 50msec Method.

14.6.5.2.2. Determine the number of SIM cycles across your peak as follows:

14.6.5.2.2.1. Use the mouse to select a spectrum from the start of the peak (where the signal leaves the baseline).

14.6.5.2.2.2. Note and record the scan number of this spectrum.

14.6.5.2.2.3. Repeat for the end of the peak (where the signal approaches the base line).

14.6.5.2.2.4. The difference in these numbers is the number of SIM cycles over the peak.

14.6.5.2.3. If the number of cycles is less than 10, the dwell time needs to be reduced. If more than 25, increase the dwell time. Your goal should be about 15-20 points across most peaks.

14.7. Data Interpretation for SIM Analysis

14.7.1. The qualitative identification of target analytes determined by this method is based on the elution of the sample component at the same relative retention time (RRT) as the standard component, and the presence of the primary quant ion and at least one secondary qualitative ion.

14.7.2. Target analytes should be identified as present when:

14.7.2.1. The analyte is identified within the defined retention time window.

14.7.2.1.1. The RRT of the sample target analyte is within ±0.06 RRT units of the RRT of the standard target analyte.

14.7.2.2. The intensity of the primary quant ion is 100% and the secondary characteristic ion (and tertiary ion if used) is within the appropriate intensity ratio:

14.7.2.2.1. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

14.7.2.3. Identification is hampered when sample components are not resolved chromatographically, fall outside of the retention time window due to a matrix effect, or are not confirmed by the presence of qualitative ions. Careful examination of the chromatogram by a chemist experienced in SIM analysis is critical.

14.7.3. When a compound has been identified, the quantitation of the compound will be based on the integrated abundance of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

14.7.3.1. Identify and compute the concentration of each target analyte in the sample. The GC/MS data system is programmed to perform these functions.

15. CALCULATIONS

15.1. Response factors are calculated as follows:

$$RF = \frac{(A_x \times C_{is})}{(A_{is} \times C_x)}$$

where:
- RF = response factor for target analyte being measured.
- A_x = area of the characteristic ion for target analyte being measured.
- A_{is} = area of the characteristic ion for the applicable internal standard.
- C_{is} = concentration of the specific internal standard in ng/µL.
- C_x = concentration of the target analyte being measured in ng/µL.

15.2. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the average RFs for the target analyte.
RF_{ave} = mean of the 5 initial RFs for the target analyte.

15.3. The relative retention time (RRT) is calculated as follows:

$$RRT = \frac{RT_c}{RT_{is}}$$

where: RRT = relative retention time of the target analyte.
RTc = retention time of the target analyte.
C_c = retention time of the nearest internal standard.

Note: Retention times must be in equivalent units.

15.4. The RPD between the sample and sample duplicate is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements (C_1 & C_2).
C_1 = concentration of target analyte recovered in measurement 1.
C_2 = concentration of target analyte recovered in measurement 2.

Note: Concentrations must be in equivalent units.

15.5. The recovery of LCS compounds is calculated as follows:

$$\%REC_{LCS} = \left(\frac{C_{recovered}}{C_{added}}\right) \times 100$$

where: $\%REC_{LCS}$ = percent recovery of target analyte in LCS (or LCSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.6. Calculate the MDL as follows.

$$MDL = t \times S_{n-1}$$

where: MDL = method detection limit.
t = student's t value at a 99% confidence level with (n − 1) degrees of freedom. For seven replicate analyses, t = 3.14.
S_{n-1} = standard deviation of the (n − 1) replicate analyses.
n = number of replicate analyses.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13, "Calibration and Standardization," shall be followed.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices. In general, safety glasses, lab coats and gloves should be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. Method Blank:

18.1.1. Ideally, the concentration of target analytes in a MB should be less than the respective reporting limits (RLs). (less than ½ the RL for DOD work) If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs is as follows:

18.1.1.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.1.1.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination.

18.1.1.3. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-extracted and/or re-analyzed.

18.2. LCS/LCSD:

18.2.1. All LCS/LCSD compounds must be within acceptance limits. If one or more LCS/LCSD compounds are not acceptable, the problem must be identified and corrected. Refer to Appendix 2 for current analytes and control limits.

18.2.2. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.

18.3. Internal Standards:

18.3.1. Internal Standard Retention Times (RT) for Bromochloromethane, Chlorobenzene-d_5 and 1,4-Difluorobenzene in all samples and QC must be +/- 0.33 minutes.

18.3.2. The area response for each internal standard in all samples and QC must be within ±40% (Range: 60% to 140%) of the mean area response of the internal standards in the most recent initial calibration.

18.4. Surrogates:

18.4.1. The acceptance criteria for surrogate spike compound recoveries vary depending upon historical data. The upper and lower acceptance limits for each surrogate spike compound is based upon the historical average recovery ± 3S. The acceptance criteria for the surrogate compounds is currently as follows:

Analyte	% Recovery
1,4-Bromofluorobenzene	45-153
1,2-Dichloroethane-d4	37-163
Toluene-d8	73-121

18.4.2. If the surrogate compound recoveries are acceptable, report the surrogates and sample data without qualification.

18.4.3. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high analyte concentration. This data alone cannot be used to evaluate the precision and accuracy of individual sample analyses. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

18.4.4. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.

18.4.4.1. Check the internal standard and surrogate spiking mixture for degradation and contamination.

18.4.4.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate(s) recovery, the same sample or extract should be re-analyzed.

18.4.4.3. If incorrect procedures or degraded/contaminated spiking solutions are determined to have not caused the unacceptable surrogate recoveries, the affected sample(s) must be re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

18.4.4.3.1. If, upon re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

18.4.4.3.2. If the MB surrogates are unacceptable, all associated sample data must be invalidated and all associated samples re-analyzed.

18.5. Additional information regarding internal quality control checks is provided in SOP-T020.

18.6. All concentrations shall be reported in ppb (v/v). Units may be reported in ppm (v/v), ug/l or ug/m^3, on a project specific basis.

18.7. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Assurance Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Out-of-control data are reviewed and verified by the Group Leader of the appropriate department. All samples associated with an unacceptable QC set is then subject to reanalysis, depending upon the QC type in question.

20.1.1. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptability criteria. If the recoveries fail for a given reported compound, the technical director confirms the unacceptable result.

20.1.1.1. If the LCS results are verified as acceptable, no corrective action is required.

20.1.1.2. If the LCS result is verified as out-of-control, and the subject compound is to be reported in samples within that analytical batch, the samples reported with that failed compound must be reanalyzed with a valid LCS recovery for the compound.

20.1.1.3. If the LCS result is verified as out-of-control, and the subject compound is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client or placed into the proper laboratory waste stream, if applicable.

21.2. Each specific laboratory area shall maintain clearly labeled satellite waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste, if applicable.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal, if applicable.

21.4. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly, if applicable.

21.5. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Waste."

22. REFERENCES

22.1. US EPA, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1997, Method TO-15, "The Determination of Volatile Organic Compounds (VOCs) in Air Collected in Summa® Canisters and Analyzed by Gas Chromatography/Mass Spectrometry".

22.2. www.chem.agilent.com/Library/Support/Documents/a05042.pdf - Setting up a SIM acquisition method – MS ChemStation-Agilent

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix 1: Table 1 and Table 2: Ions for Qualitative Identification of Standard and Additional Target Compounds.

23.2. Appendix 2: Standard Analyte, Reporting, Method Detection and Control Limits for TO-15SIM.

APPENDIX 1

Table I : Ions for Qualitative Identification of Standard Target Compounds

Routine Analyte	Qion E	2nd	3rd
Chloromethane	50	52	
1,1-Difluoroethane	51	65	
c-1,2-Dichloroethene	61	96	
t-1,2-Dichloroethene	61	96	
1,2-Dichloroethane	62	98	
Vinyl Chloride	62	64	
1,1-Dichloroethane	63	65	
Chloroethane	64	66	
1,2-dichloroethane-d4 (surr)	65	67	
Methyl-tert-Butyl Ether	73	57	
Benzene	78	77	
Chloroform	83	85	
1,1,2,2-Tetrachloroethane	83	85	
Bromodichloromethane	83	85	
Methylene Chloride	84	86	
Dichlorodifluoromethane	85	87	
Toluene	91	92	
Ethylbenzene	91	106	
p/m-Xylene	91	106	
o-Xylene	91	106	
1,4-Bromofluorobenzene (surr)	95	174	176
1,1-Dichloroethene	96	98	
1,1,1-Trichloroethane	97	63	
1,1,2-Trichloroethane	97	83	
Toluene-d8 (surr)	98	100	
Trichlorofluoromethane	101	103	
1,1,2-Trichloro-1,2,2-Trifluorethane	101	151	
4-Ethyltoluene	105	120	
1,3,5-Trimethylbenzene	105	120	77
1,2,4-Trimethylbenzene	105	120	77
Chlorobenzene	112	77	
1,4-Difluorobenzene (IS)	114	---	
Chlorobenzene-d5 (IS)	117	---	
Carbon Tetrachloride	117	119	
Dibromochloromethane	129	127	
Trichloroethene	130	132	
Bromochloromethane (IS)	130	---	
Tetrachloroethene	166	164	131
Hexachloro-1,3-Butadiene	225	227	223

APPENDIX 1, Cont'd

Table 2 : Ions for Qualitative Identification of Additional Target Compounds

Non-Routine Analyte	Qion E	2nd	3rd
2-Butanone	43	42	72
Ethanol	45	46	43
Isopropanol	45	43	
1,3-Butadiene	54	39	
Cyclohexane	56	84	
Acetone	58	43	
4-Methyl-2-Pentanone	58	85	
2-Hexanone	58	43	
1,2-Dichloropropane	63	62	41
c-1,3-Dichloropropene	75	39	
t-1,3-Dichloropropene	75	39	
Carbon Disulfide	76	44	78
Dichlorotetrafluoroethane	85	135	87
Hexane	57	43	41
1,4-Dioxane	88	58	
Benzyl Chloride	91	106	65
Bromomethane	94	96	
Heptane	100	43	71
Styrene	104	78	
1,2-Dibromoethane	107	109	
Naphthalene	128	127	
1,2-Dichlorobenzene	146	148	
1,3-Dichlorobenzene	146	148	
1,4-Dichlorobenzene	146	148	
Bromoform	173	170	174
1,2,4-Trichlorobenzene	180	182	

APPENDIX 2

Standard Analyte, Reporting, Method Detection and Quality Control Limits

Routine Analyte	CAS #	RL ppbv	MDL ppbv	LCS/LCSD % REC	LCS/LCSD % RPD
Benzene	71-43-2	0.025	0.0007	50-150	0-30
Bromodichloromethane	75-27-4	0.025	0.0013	50-150	0-30
Carbon Tetrachloride	56-23-5	0.025	0.0008	7-187	0-31
Chlorobenzene	108-90-7	0.025	0.0028	50-150	0-30
Chloroethane	75-00-3	0.025	0.0011	50-150	0-30
Chloroform	67-66-3	0.025	0.001	50-150	0-30
Chloromethane	74-87-3	0.025	0.0017	50-150	0-30
Dibromochloromethane	124-48-1	0.025	0.0007	50-150	0-30
Dichlorodifluoromethane	75-71-8	0.025	0.001	50-150	0-30
1,1-Dichloroethane	75-34-3	0.025	0.0022	50-150	0-30
1,1-Dichloroethene	75-35-4	0.025	0.0011	50-150	0-30
1,2-Dichloroethane	107-06-2	0.025	0.0008	28-166	0-40
c-1,2-Dichloroethene	156-59-2	0.025	0.0008	35-165	0-35
t-1,2-Dichloroethene	156-60-5	0.025	0.0007	50-150	0-30
Ethylbenzene	100-41-4	0.025	0.0005	27-153	0-46
4-Ethyltoluene	622-96-8	0.025	0.0006	27-153	0-34
Hexachloro-1,3-Butadiene	87-68-3	0.025	0.0008	50-150	0-30
Methyl-t-Butyl Ether (MTBE)	1634-04-4	0.025	0.0009	50-150	0-30
Methylene Chloride	75-09-2	0.025	0.0012	50-150	0-30
o-Xylene	95-47-6	0.025	0.0005	22-160	0-48
p/m-Xylene	179601-23-1	0.025	0.0011	21-165	0-51
Tetrachloroethene	127-18-4	0.025	0.0009	34-154	0-33
Toluene	108-88-3	0.025	0.0007	28-154	0-42
Trichloroethene	79-01-6	0.025	0.0008	43-139	0-31
Trichlorofluoromethane	75-69-4	0.025	0.001	50-150	0-30
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	0.025	0.0009	50-150	0-30
1,1,1-Trichloroethane	71-55-6	0.025	0.0008	50-150	0-30
1,1,2-Trichloroethane	79-00-5	0.025	0.0008	27-171	0-38
1,3,5-TrimethylBenzene	108-67-8	0.025	0.0007	50-150	0-30
1,1,2,2-Tetrachloroethane	79-34-5	0.025	0.0013	50-150	0-30
1,2,4-TrimethylBenzene	95-63-6	0.025	0.0008	50-150	0-30
Vinyl Chloride	75-01-4	0.025	0.0028	44-140	0-33

TO-15 SIM RL/MDLs and EPA Regional Screening Levels (RSLs)

TC#	METHOD	TESTCODE	COMPOUND	TYPE	CAS	EPA RSL for Resid'l Air (1E-6 risk) (ug/m^3)	RL	MDL	UNIT	LCS CL	LCS RPD
4664	EPA TO-15 SIM	TO-15 SIM	1,1,1-Trichloroethane	A	71-55-6	5200*	0.14	0.0045	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	1,1,2-Trichloro-1,2,2-Trifluoroethane	A	76-13-1	31000	0.19	0.0071	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	1,1-Dichloroethene	A	75-35-4	210	0.099	0.0044	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	Bromodichloromethane	A	75-27-4	0.066	0.17	0.0087	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	c-1,2-Dichloroethene	A	156-59-2	63**	0.099	0.0031	ug/m3	35 - 165	0 - 35
4664	EPA TO-15 SIM	TO-15 SIM	Chlorobenzene	A	108-90-7	52*	0.12	0.013	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	Chloroform	A	67-66-3	0.4	0.12	0.0048	ug/m3	50 - 150	0 - 30
4664	EPA TO-15 SIM	TO-15 SIM	Tetrachloroethene	A	127-18-4	0.41	0.17	0.0060	ug/m3	34 - 154	0 - 33
4664	EPA TO-15 SIM	TO-15 SIM	Trichloroethene	A	79-01-6	1.2	0.13	0.0042	ug/m3	43 - 139	0 - 31

*Non-cancer Hazard Index = 1

**No established RSL. Trans-1,2-dichloroethylene used as surrogate.

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AE-16 Final Report

Air and Emissions Proficiency Testing

Air and Emissions Study

Open Date: 04/25/11

Close Date: 06/09/11

Report Issued Date: 07/15/11

ERA
A Waters Company

July 15, 2011

Larry Lem
Calscience Environmental Laboratories
7440 Lincoln Way
Garden Grove, CA 92841-1427

Enclosed is your final report for ERA's AE-16 Air and Emissions Proficiency Testing (PT) study. Your final report includes an evaluation of all results submitted by your laboratory to ERA.

All analytes in ERA's AE-16 Air and Emissions Proficiency Testing (PT) study have been evaluated using the following tiered approach: ERA has gathered proficiency testing data for air and emissions across many national and international studies. Where available the acceptance criteria have been calculated using these studies' data. Where appropriate, ERA has based the acceptance criteria on the study means and study standard deviations. Where these data were not available, the reported results have been evaluated using the procedures outlined in ERA's Standard Operating Procedure for the Generation of Performance Acceptance Limits (SOP 0260).

Thank you for your participation in ERA's AE-16 Air and Emissions Proficiency Testing (PT) study. If you have any questions, please contact the proficiency testing department, or myself, at 1-800-372-0122.

Sincerely,

Jay R. McBurney
Quality Program Manager

attachments
jrm

6000 West 54th Ave., Arvada, CO 80002 800-372-0122 fax (303) 421-0159 www.eraqc.com

Report Recipient	Contact/Phone Number	Reporting Type
California	Fred Choske / 510-620-3175	All Analytes
DoD EDQW	Fred S McLean / 843-764-7266	All Analytes
EPA Region 9	Andrew Lincoff / 510-412-2330	All Analytes
New York	Dan Dickinson / 518-485-5570	All Analytes
Washington	Alan Rue / (360) 895-6178	All Analytes

AE-16 Definitions & Study Discussion

Study Dates: 04/25/11 - 06/09/11 **Report Issued: 07/15/11**

AE Study Definitions

The Reported Value is the value that the laboratory reported to ERA.

The Assigned Values for ERA's Air and Emissions Proficiency Testing (PT) Standards are equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The assigned values are directly traceable to the commercially prepared starting materials used to manufacture the PT standards.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable.

The Performance Evaluation:

Acceptable	= Reported Value falls within the Acceptance Limits.
Not Acceptable	= Reported Value falls outside the Acceptance Limits.
No Evaluation	= Reported Value cannot be evaluated.
Not Reported	= No Value reported.

The Method Description is the method the laboratory reported to ERA.

AE Study Discussion

ERA's AE-16 Air and Emissions Proficiency Testing (PT) study has been reviewed by ERA senior management.

A full review of all homogeneity, stability and accuracy verification data was completed. All analytical verification data for all analytes in the Air and Emissions study standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003. The acceptance limits were calculated using the procedures outlined in ERA's Standard Operating Procedure for the Generation of Performance Acceptance Limits (SOP 0260, Rev. 2.0).

The data submitted by participating laboratories was also examined for study anomalies. There were no anomalies observed during the statistical review of the data.

ERA's Air and Emissions Proficiency Testing study reports shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratories to claim product endorsement any agency of the U. S. government.

Per DOT shipping requirements, ERA was required to notify laboratories that received the AE-16 Volatiles in Gas Cylinder, catalog #1000, that the cylinder contained 1,1,2-Trichlorotrifluoroethane (Freon 113).

The data contained herein are confidential and intended for your use only.

If you have any questions or concerns regarding your assessment in ERA's Air and Emissions Proficiency Testing program, please contact Jay McBurney, Quality Program Manager, or the proficiency testing department at 1-800-372-0122.

Study: **AE-16**

ERA Customer Number: **C052701**

Laboratory Name: **Calscience Environmental Laboratories**

Inorganic Results

AE-16 Final Complete Report

Larry Lem
Calscience Environmental Laboratories
7440 Lincoln Way
Garden Grove, CA 92841-1427
(714) 895-5494

EPA ID: CA00111
ERA Customer Number: C052701
Report Issued: 07/15/11
Study Dates: 04/25/11 - 06/09/11

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
AE Lead on Filter Paper (cat# 1029)							
1075	Lead	µg/Filter	36.3	36.9	27.7 - 46.1	Acceptable	EPA 6020

Study: **AE-16**

ERA Customer Number: **C052701**

Laboratory Name: **Calscience Environmental Laboratories**

Organic Results

AE-16 Final Complete Report

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EPA ID: CA00111
ERA Customer Number: C052701
Report Issued: 07/15/11
Study Dates: 04/25/11 - 06/09/11

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
AE Volatiles in Gas Cylinder (cat# 1000)							
4375	Benzene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4395	Bromodichloromethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4400	Bromoform	ppbv	22	22.4	17.9 - 24.7	Acceptable	EPA TO-14A GCMS
4950	Bromomethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4410	2-Butanone (MEK)	ppbv	< 1.5	0.00		Acceptable	EPA TO-14A GCMS
5000	tert-Butyl methyl ether (MTBE)	ppbv	< 2.0	0.00		Acceptable	EPA TO-14A GCMS
4455	Carbon tetrachloride	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4475	Chlorobenzene	ppbv	6.2	7.50	4.93 - 8.44	Acceptable	EPA TO-14A GCMS
4575	Chlorodibromomethane	ppbv	19	20.0	13.5 - 26.0	Acceptable	EPA TO-14A GCMS
4485	Chloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4505	Chloroform	ppbv	19	20.3	14.7 - 22.6	Acceptable	EPA TO-14A GCMS
4960	Chloromethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4555	Cyclohexane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4585	1,2-Dibromoethane (EDB)	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4610	1,2-Dichlorobenzene	ppbv	18	23.0	10.5 - 30.1	Acceptable	EPA TO-14A GCMS
4620	1,4-Dichlorobenzene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4625	Dichlorodifluoromethane (Freon 12)	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4630	1,1-Dichloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4635	1,2-Dichloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4640	1,1-Dichloroethylene	ppbv	10	10.8	6.65 - 13.5	Acceptable	EPA TO-14A GCMS
4645	cis-1,2-Dichloroethylene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4655	1,2-Dichloropropane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4680	cis-1,3-Dichloropropylene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
4685	trans-1,3-Dichloropropylene	ppbv	< 1.0	0.00		Acceptable	EPA TO-14A GCMS
4695	1,2-Dichlorotetrafluoroethane (Freon 114)	ppbv	< 2.0	0.00		Acceptable	EPA TO-14A GCMS
4765	Ethylbenzene	ppbv	23	25.2	15.1 - 28.6	Acceptable	EPA TO-14A GCMS
4542	p-Ethyltoluene	ppbv	2.1	2.70	1.67 - 2.98	Acceptable	EPA TO-14A GCMS
4825	n-Heptane	ppbv	17	19.7	12.7 - 21.7	Acceptable	EPA TO-14A GCMS
4835	Hexachlorobutadiene	ppbv	21	26.0	15.5 - 32.4	Acceptable	EPA TO-14A GCMS
4855	n-Hexane	ppbv	5.6	5.90	4.03 - 6.49	Acceptable	EPA TO-14A GCMS
4860	2-Hexanone	ppbv	< 1.5	0.00		Acceptable	EPA TO-14A GCMS
4995	4-Methyl-2-pentanone (MIBK)	ppbv	< 1.5	0.00		Acceptable	EPA TO-14A GCMS

AE-16 Final Complete Report

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EPA ID: CA00111
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
AE Volatiles in Gas Cylinder (cat# 1000) (Continued)							
4836	Propylene	ppbv	< 10	0.00		Acceptable	EPA TO-14A GCMS
5105	1,1,1,2-Tetrachloroethane	ppbv		0.00		Not Reported	
5110	1,1,2,2-Tetrachloroethane	ppbv	< 1.0	0.00		Acceptable	EPA TO-14A GCMS
5115	Tetrachloroethylene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
5140	Toluene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
5155	1,2,4-Trichlorobenzene	ppbv	< 2.0	0.00		Acceptable	EPA TO-14A GCMS
5160	1,1,1-Trichloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
5165	1,1,2-Trichloroethane	ppbv	24	27.8	17.0 - 30.6	Acceptable	EPA TO-14A GCMS
5175	Trichlorofluoromethane (Freon 11)	ppbv	< 1.0	0.00		Acceptable	EPA TO-14A GCMS
5195	Trichlorotrifluoroethane (Freon 113)	ppbv	< 1.5	0.00		Acceptable	EPA TO-14A GCMS
5210	1,2,4-Trimethylbenzene	ppbv	< 1.5	0.00		Acceptable	EPA TO-14A GCMS
5215	1,3,5-Trimethylbenzene	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
5230	Vinyl bromide	ppbv		0.00		Not Reported	
5235	Vinyl chloride	ppbv	< 0.50	0.00		Acceptable	EPA TO-14A GCMS
5260	Xylenes, total	ppbv	< 2.5	0.00		Acceptable	EPA TO-14A GCMS

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4635	1,2-Dichloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-15 GCMS
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4825	n-Heptane	ppbv	17	19.7	12.7 - 21.7	Acceptable	EPA TO-15 GCMS
4835	Hexachlorobutadiene	ppbv	21	26.0	15.5 - 32.4	Acceptable	EPA TO-15 GCMS
4855	n-Hexane	ppbv	5.6	5.90	4.03 - 6.49	Acceptable	EPA TO-15 GCMS
4860	2-Hexanone	ppbv	< 1.5	0.00		Acceptable	EPA TO-15 GCMS
4995	4-Methyl-2-pentanone (MIBK)	ppbv	< 1.5	0.00		Acceptable	EPA TO-15 GCMS

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5115	Tetrachloroethylene	ppbv	< 0.50	0.00		Acceptable	EPA TO-15 GCMS
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5160	1,1,1-Trichloroethane	ppbv	< 0.50	0.00		Acceptable	EPA TO-15 GCMS
5165	1,1,2-Trichloroethane	ppbv	24	27.8	17.0 - 30.6	Acceptable	EPA TO-15 GCMS
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5195	Trichlorotrifluoroethane (Freon 113)	ppbv	< 1.5	0.00		Acceptable	EPA TO-15 GCMS
5210	1,2,4-Trimethylbenzene	ppbv	< 1.5	0.00		Acceptable	EPA TO-15 GCMS
5215	1,3,5-Trimethylbenzene	ppbv	< 0.50	0.00		Acceptable	EPA TO-15 GCMS
5230	Vinyl bromide	ppbv		0.00		Not Reported	
5235	Vinyl chloride	ppbv	< 0.50	0.00		Acceptable	EPA TO-15 GCMS
5260	Xylenes, total	ppbv	< 2.5	0.00		Acceptable	EPA TO-15 GCMS

ATTACHMENT D

Standard Operating Procedures
Sub-Slab Soil Gas Sampling Procedures

For Vapor Intrusion Assessments

Prepared by:
Blayne Hartman, Ph.D.
Hartman Environmental Geoscience
Solana Beach, California

June 2011

Soil Gas Probe/Implant Construction

Materials, nylon tubing, Teflon tubing, or stainless steel tubing

Diameter: nominally 1/8" OD or ¼" OD

Length: Cut to desired length within or just below base of slab

Tip: SS, Al, ceramic, plastic (typically no tip required for sub-slab)

Surface Termination: Swagelok fittings or valve

Refer to Photos 1 and 2 for typical parts.

Notes:

- Stainless steel and nylon tubing are preferred over Teflon due to lower adsorption. Nylon tubing is more flexible and easier to work with than stainless steel tubing.
- Tips (aluminum, ceramic, SS) can be put on the end if desired to give a longer screen interval, but typically are not used for sub-slab samples.
- Various surface terminations are available and the selection often depends on whether the probes are temporary or permanent and whether they need to be installed flush with the surface.

Probe Installation Protocol

1. Ensure all sub-slab utilities (public and building specific) are marked prior to installation.
2. Drill a ½" to 1" OD hole through the slab with a drill and spline bit. Do not use water. If dust prevention necessary, cover the location with a towel/cloth and drill through a pre-cut hole in the cloth (Photo 3).
3. Measure slab thickness. Cut tubing to appropriate length to reach base of slab and to give required type of surface termination (flush, recessed, protruding). If a flush or recessed surface termination is required, a larger diameter hole (1") in the upper 1-inch of the slab may be useful to leave enough room for the fitting on the probe tubing (Photo 4).
4. Insert tubing. Add sand to cover tip with about 1 inch of sand.
5. Grout to the surface using bentonite (if temporary installation) or cement (if permanent installation).
6. Wait 30 to 45 minutes prior to sampling for bentonite to congeal.

Soil Gas Sample Collection

Since sub-slab sampling is from very shallow depths (typically 2” to 6” below surface), minimum purge volumes and low volume samples are preferred to minimize potential breakthrough from the surface. Tracer/leak gas is necessary to ensure breakthrough does not occur.

Materials: 1/8” or ¼” OD nylon or Teflon or stainless steel tubing.

Sample Canister: syringe, tedlar bag, SS canister, gas-tight glass, 3-way valve.

1. Connect 3-way valve to probe tubing. Purge out 150 cc using a 60 cc disposable syringe. 150 cc represents three purge volumes of the volume of the hole created in the slab (~50 cc) during drilling.
2. Place small plastic shroud over hole (Photo 5). Weigh shroud down to maintain seal. Pass tubing through similar diameter hole at top of shroud and connect to purging device. Inject helium, carbon dioxide, or other tracer into shroud through a side port until concentrations are at least 20 times the meter detection limit. Measure shroud concentration using a portable meter. Record on log sheet as shroud initial tracer concentration.
3. Connect 60 cc syringe to sampling train and purge out 3 dead-volumes of the connecting tubing plus the sampling train at a rate not to exceed 0.2 L (200 ml) per minute.
4. Attach tedlar bag to sideport of 3-way valve. Collect approximately 800 cc to 1 liter of sample into a tedlar bag using the gas-tight syringe and 3-way valve (Photo 6).
5. Measure the tracer concentration in tedlar bag. Record on log sheet as sample tracer concentration. If sample concentration less than or equal to 15% of initial concentration in shroud, sample is considered valid. If concentration in tedlar bag is greater than 15% of the shroud concentration, improve probe seal or reinsert new probe and re-sample.
6. If a mobile lab exists, transfer tedlar bag to the mobile lab for analysis. Keep the tedlar out of direct sunlight.
7. To transfer soil gas sample into a canister, check and record canister vacuum (minimum vacuum 27” Hg). Connect tedlar bag up to the canister and open canister valve (Photo 7). Tedlar bag will collapse as sample is transferred. Close valve and ship canister to laboratory.

Notes:

- For larger canisters (> 1 liter), sample flow rates are not to exceed 500 ml/min (200 ml/min for CA-EPA) to minimize potential for vacuum extraction of contaminants from the soil phase.
- If large volume canisters used (3 or more liters), a purge volume test may be required to ensure sample dilution from other zones is not occurring.

Field Records

The field technician maintains a log sheet summarizing:

- Sample identification
- Probe location
- Date and time of sample collection
- Sampling depth
- Identity of samplers
- Weather conditions
- Sampling methods and devices
- Soil gas purge volumes
- Volume of soil gas extracted
- Vacuum of canisters before and after samples collected.
- Apparent moisture content (dry, moist or saturated etc.) of the sampling zone
- Chain of custody protocols and records used to track samples from sampling point to analysis.

Photo 1 – Soil gas sampling materials: sample collection train, vapor implants, mini-can (400 cc), purging syringe.

Photo 2 – Sub-slab vapor implant (1/8” OD) and cap. Nylon tubing can be cut to any length.

Photo 3 – Drilling hole through slab (1/2” OD).

Photo 4 – Surface completions. Left: tubing coiled in hole. Removable plug installed to make flush with surface. Right: cemented flush to surface (hole slightly recessed purposely to enable fitting to be visible in photo).

Photo 5 – Shroud for leak testing.

Photo 6 – Set-up for collecting sub-slab sample with a 60 cc syringe and filling into a tedlar bag (front stem of syringe connects to the probe tube).

Photo 7 – Transferring sample from tedlar bag to a 400 cc evacuated canister..

ATTACHMENT E

H&P Mobile GeoChemistry, Inc
STANDARD OPERATING PROCEDURES (SOPs)

EPA Method TO-14A, TO-15

Revision 6
February 2011

1.0 Scope and Applications

This SOP has been prepared for H&P Mobile GeoChemistry (H&P), Inc. to help ensure consistent analytical protocol and applies to the performance of the EPA Method TO-14A and TO-15. This method is applicable to ambient air and soil vapor samples. The scope of topics discussed in this SOP includes the following:

* Method Summary
* Health and Safety Warnings
* Cautions
* Interferences
* Personnel Qualifications
* Instruments and Materials
* Method Calibration
* Sample Collection
* Handling & Preservation
* Sample Preparation
* Troubleshooting
* Data Acquisition, Calculation and Data Reduction
* Computer Hardware and Software
* Data Management and Records Management
* QA/QC Requirements
* References

The following topics are included as part of this SOP by reference as follows:

* References and Definitions (Appendix 1A of QSM)
* Data Assessment (Section 9.0 of QSM)
* Corrective Action and Contingencies for out-of-control data (Section 8.0 of QSM)

2.0 Summary of Method

H&P uses EPA Method TO-14A/15 to identify more than seventy eight (78) volatile organic compounds in ambient air and subsurface soil gas**.** Samples are introduced into a gas chromatograph by concentrative trapping as described in the methods, either from a container such as mini Summa canister, Tedlar bag or direct collection of air or soil vapor. The analysis is performed on a gas chromatograph (GC) with a quadrupole mass spectrometer (MS).

The volatile organic compounds identified are as follows. Additional compounds possible are also included in the list below.

Main List

1,1-Dichloroethane
1,1-Dichloroethene
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
1,1,2-Trichlorotrifluoroethane (F-113)
1,1,2,2-Tetrachloroethane
1,1,1,2-Tetrachloroethane
1,2,4-Trimethylbenzene
1,2,4-Trichlorobenzene
1,3,5-Trimethylbenzene
4-Ethyltoluene
Acetone
Benzene
Bromodichloromethane
Bromoform
Bromomethane
Carbon disulfide
Carbon Tetrachloride
Chlorobenzene
Chloroethane
Chloroform
Chloromethane
cis-1,2-Dichloroethene
Cis-1,3 Dichloropropene
Dibromochloromethane
Dichlorodifluoromethane (F-12)
Dichlorotetrafluoroethane (F-114)
Ethylbenzene
Hexachlorobutadiene
Methyl ethyl ketone
Methyl isobutyl ketone
Methyl butyl ketone

Methylene Chloride
m,p-Xylene
o-Xylene
Styrene
Tetrachloroethene
Toluene
Trans-1,3-Dichloropropene
trans-1,2-Dichloroethene
Trichloroethene
Trichlorofluoromethane (F-11)
Vinyl Chloride

Additional Compounds
1,3-Butadiene
1,1-Difluoroethane (soil gas leak check compound)
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2-Dibromo-3-chloropropane
1,3-Dichloropropane
2,2-Dichloropropane
2-Chlorotoluene
4-Chlorotoluene
1,4-Dioxane
2,2,4-Trimethylpentane
Benzyl Chloride
Bromobenzene
Cyclohexane
Dibromomethane
Di-isopropyl Ether
Ethanol
Ethyl acetate
Ethyl-tert-Butyl Ether
Isopropyl alcohol
Isopropylbenzene
Methyl t-butyl ether
Naphthalene
n-Butylbenzene
n-Propylbenzene
n-Hexane
n-Heptane
Propene
sec-Butylbenzene
tert-Butyl Alcohol
tert-Butylbenzene

tert-Amyl Methyl Ether
Tetrahydrofuran
Vinyl acetate

Note: Other volatile compounds may be analyzed by this method if a vapor standard is available and calibration is performed according to the procedure described below.

3.0 Health and Safety Warnings

H&P employs conservative safety practices at all times. Smoking is not permitted in the van or lab and eating is not permitted in the analysis area. Fire extinguishers, first aid kits and Material Safety Data Sheets are located in each van or lab. The laboratory equipment may include high-pressure hydrogen which is a fire and explosion hazard. Eye protection is to be worn by all laboratory operators while handling high pressure gas cylinders and liquid N2. Heavy duty gloves (cold resitant insulated) are required to be worn to protect hands from contact with the liquid N2.

4.0 Cautions

The analyst is cautioned that the equipment uses high-pressure gases and cryogenics which, while not flammable nor explosive, are suffocating in large quantities. The analyst must use caution when changing cryogenic tanks since a leak could allow liquid nitrogen and/or carbon dioxide to contact the skin, which could result in permanent and painful skin damage. The analyst should be aware of any potential source of leaking gas and should inspect all plumbing from cylinders to the instruments on a daily basis.

5.0 Interferences

When analyzing for volatile organics, samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment or storage. A trip blank prepared from nitrogen or helium and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

In the Entech 7100/7300 sampler a sample position may become contaminated from a high level sample. A good practice for the analyst is to purge all sampling positions to be used in that day's sequence prior to analysis. This is performed by placing the fitting for nitrogen or helium onto the position to be purged. The Entech sequence table is then used to select that position. A sample volume of 20-50 mls can be selected. No calibration standard volume is selected. Click on the "Flush" icon on the sequence table and select "Go". The Entech will flush and pressurize this position three times. Repeat this step for all positions. After flushing the system it is recommended that the system is baked for 10 minutes before proceeding with analysis.

6.0 Personnel Qualifications

This method is to be performed by a trained analyst in gas chromatographic/mass spectrometric methods. A bachelor's degree in science or equivalent training is the minimum requirement for performance of this method.

7.0 Instruments and Materials

The following subsections discuss the instrumentation and materials needed for this method.

7.1 Instruments

H&P uses Agilent GC/MS systems for EPA Method TO-14A/15 analysis. The labs use Agilent GCs coupled to a Mass Selective Detector (MSD). The concentrator is an Entech Model 7100A concentrator coupled to a 7032A autosampler. The column used is an HP-1column of 60m X 0.32mm X 1.8u film thickness and uses these columns in all systems. The carrier gas He is UHP (Ultra High Purity). The following instrument conditions are typical:

GC

Injector mode	not applicable
Injector temp.	not applicable
Interface temp.	230 degrees C
Column:	60m x 0.32mm x 1.8u HP-1 (or equivalent)
Flow:	3.0 mL/min He initial for desorb
	Programming down to 1.5 mL/min by 4 minutes
Initial temp.	35 degrees C 4 minute hold
Ramp	20 degrees/min
Final temp	230 degrees

MS

SCAN mode	41-300 amu, 2.5 scans/sec
Source temp	230 degrees
Quad temp	150 degrees
Multiplier	+200 relative

Detailed descriptions of the analytical method parameters are to be found in the initial demonstration of proficiency data package, which is kept as part of H&P 's initial lab certification.

The following SOP is written for analysts using the Entech Air concentrator and/or autosampler. The 7100 concentrator can concentrate from a few mL of sample up to as much as 6 liters of air. As standard practice, H&P will assume a standard sample size as being 50 mL.

Method Name: C:\Smart\co2n2napnew.CTD Software ver. 3.19
Report Date/time 11/15/2010 9:44:29 AM

Stream: Sample
Preflush (sec): 10
Trap (cc/min): 60
Volume (cc): 50

Stream: Internal Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 50

Stream: Analytical Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 0

Stream: Sweep/Purge
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 75

Stream: M1 -> M2
Preflush (sec):
Trap (cc/min): 5
Volume (cc): 25

Module1:
Trap temp(C): -35 Preheat? Yes
Preheat temp(C): 10
Desorb temp(C): 10
Bake temp(C): 150
Bake time(Min): 10
Bulk1:
Trap temp(C): 30
Desorb temp(C): 30
Bake temp(C): 150

Module2:
Trap temp(C): -35 Preheat? No
Preheat temp(C): 190
Desorb temp(C): 190
Bake temp(C): 200
Desorb time(C): 3
Bulk2:
Trap temp(C): 30
Desorb temp(C): 100
Bake temp(C): 180

Module3:
Trap temp(C): -180 Focus? Yes

Inject temp(C): 150
Inject time(Min):3
Bake temp(C): 100
Bake time(Min): 3
Bake on EventEx# : 3
Total Time (Min) : 11

Misc:
Sample Xfer temp(C): 80
GC Xfer temp(C): 100
MPOS Valve temp(C): 100
Wait for GC before injecting
Active GC: GC1
Pressure: 100
MPOS Valve temp(C): 100

Air samples may be taken in Tedlar bags, Summa canisters or other approved containers. (see other H&P SOPs for soil vapor and ambient air sampling)

Agilent Chemstation software version G1701DA with Enviroquant software running on Windows XP/Pentium personal computers are used for data acquisition and instrument control. Chromatograms and quantitation reports are printed out on HP Laserjet printers.

The above-mentioned equipment requires 110 VAC as a power source to operate.

Sources of further information can be found in the Agilent GC/MS and the Entech hardware and software manuals, which are kept with each instrument. Each instrument also comes with Agilent maintenance information on CD, which can be viewed on the computer.

7.2 Reagents and Standards

Zero grade air**,** Nitrogen or helium (all UHP) is used for preparation of standards and for preparation of laboratory blanks. Primary standards for analytes, surrogates and internal standards are normally purchased from commercial vendors and are diluted for use. As of 11/10/08, there is only one manufacturer of gas standards for TO-14A/15, Spectra Gases, Branchburg, N.J., although they sell the gases through other supply houses (e.g., Restek). All purchased standards are shipped with certification attesting to their purity and authenticity. Each mobile lab and the fixed-based lab performing TO-14A/15 shall have notebooks to record standards. Refer to H&Ps SOP manual or the individual notebook for nomenclature of standards.

All standards are prepared in 6L or greater Summa canisters and stored at room temperature.

7.2.1 Standard Preparation

Three standards are required to be prepared for Method TO14A/15. The first contains the internal standards (Bromochloromethane, chlorobenzene-d5, 1,4-difluorobenzene) and surrogate compounds (Toluene-d8, 1,2-dichloroethane- d4, 4-bromofluorobenzene). The second two contain the target analytes. Commercial mixes are used to prepare these.

7.2.1.1 Using the Entech can cleaner, clean a 15 liter can and two 6-liter summa canisters. Refer to the Entech literature for guidance. In general ten cycles of evacuation down to 1000 millitorr and filling with high purity nitrogen will be sufficient. Evacuate the cylinder to at least 100 millitorr at the end of the cleaning. Allow cans to completely cool before proceeding.

7.2.1.2 Introduce about 80 ul (For 6 liter, use equivalent for different size summas) of reagent grade water into the quick connects on each of the three summa canisters to be used.

7.2.1.3 Using a gas-tight 100 mL syringe introduce 1500 mL of internal standard/surrogate mix @ 1.0 ppmv into 15-L summa canisters. Then attach summa to nitrogen and pressurize to a stable 14.7 psi (2 ATM). This will yield a vapor standard containing 50 ppbv of internal standard and surrogate. (Note that there is one atmosphere of pressure in the cylinder, thus roughly two atmospheres of diluent has been added to the standard.)

7.2.1.4 Into a clean humidified evacuated 6-liter canister, introduce 120 mL of each of the 1 ppmv primary source standard mixes to be used. Then attach summa to helium or zero air source and pressurize to a stable 14.7 psi (2 ATM).This will result in a standard at 10 ppbv containing the target analytes.

7.2.1.5 Prepare a second source standard as described in 7.2.1.4 using second source gas standards.

7.2.1.6 The primary stock standard purchased has been given an expiration date by the manufacturer of one year. Secondary standards for TO-14A/15 analysis will be prepared and given expiration dates not to exceed the manufacturer's date of expiration on the primary standard or more than 30 days after preparation. Self certification can be performed on the stock mixes to extend the manufacturers original expiration date. The lab operator should monitor response factors to determine whether standards are degrading. In general, response factors will vary with tuning characteristics. However, comparison to response factors from fresh standard is suggested.

7.2.2 Reagents

High pressure ultra pure Helium and Nitrogen are both required. Helium is used as the carrier gas for the GC/MS and the Nitrogen is used to flush the sample inlet on the Entech (5 psi) and

to assist in the desorption of analytes in the cryo chamber (50 psi). Liquid Nitrogen is also required to cool the cryofocuser. This is stored in the portable dewar in the lab.

8.0 Method Calibration

In order to analyze samples the instrument must first be calibrated. The following subsections describe the system start-up and initial calibration procedures.

8.1 Initial System Calibration

Prior to performing analyses, the GC-MS must be tuned and calibrated to ensure system precision and accurate results.

Tuning of the GC-MS is accomplished using the Agilent Target Tune function in the BFB tune mode. The instrument automatically performs the tuning in an attempt to adjust parameters such that the spectrum of the compound 1,4-Bromofluorobenzene will meet the following specifications:

- Mass 50 — 8.0 percent to 40.0 percent of mass 95
- Mass 75 — 30.0 percent to 66.0 percent of mass 95
- Mass 95 — base peak, 100 percent relative abundance
- Mass 96 — 5.0 percent to 9.0 percent of mass 95
- Mass 173 — less than 2.0 percent of mass 174
- Mass 174 — 50.0 to 120.0 percent of mass 95
- Mass 175 — 4.0 percent to 9.0 percent of mass 174
- Mass 176 — 93.0 to 101.0 percent of mass 174
- Mass 177 — 5.0 percent to 9.0 percent of mass 176

Following tuning, 50 ng of BFB or less should be evaluated in the next blank or standard to determine the validity of the tune. Since BFB is one of the surrogate compounds that are included in every analysis, it can be monitored following any analysis. (See section 11.0 for details.) If the above criteria are not met, the instrument must be retuned or the problem found prior to proceeding.

Prior to performing analyses, the GC-MS must be calibrated to ensure system precision and accurate results. To calibrate the GC-MS, standards of the analytes, along with internal standards and surrogates are introduced into the concentrator and measured by the method. A minimum of five concentration levels should be used during calibration for EPA Method TO-14A/15. (For TO-14A, three level are required, for TO-15, five levels are used.) Each standard level is analyzed under the identical parameters as samples will be analyzed. The end result is a calibration curve for each analyte.

To prepare a calibration curve:

1. Turn on the Entech system.

2. Load a Method (e.g.0919TO.mth) into the software. Schedule the autosampler and concentrator for the correct Method (CO2naph.mth).

3. Entech:
Schedule the concentrator and autosampler to analyze standards of 5, 10, 25, 50, 100, 250, 375, 500 and 1000 mL of standard, respectively while delivering 50 mL of internal standard/surrogate mix.

This is equivalent in a 50 mL sample to 1, 2, 5, 10, 20, 50, 75, 100, 200 ppbv while the I.S/surrogate is kept at 50 ppbv. Program the AGILENT software to collect data for the standards. Start the sequence on the AGILENT software and the Entech software.

4. After completing the standards, quantitate and load the standards as the initial calibration. Evaluate the initial curve for linearity and response factors per the current version of EPA Method TO-14A/15 (or project specific DQO's). This is currently that ..

a. All compounds must have RSD<30% with at most two exceptions to be RSD<40%. The RRT (relative retention time) for each target compound at each calibration level must be withing 0.06 RRT units of the mean RRT for the compound. The RRT for each Internal Standard compound at each calibration level must be withing .33 RRT units of the mean RRT for the compound. The area response of each internal standard for each calibration level must be within 40% of the mean area response over the initial calibration range.

b. For certain regulatory requirement such as EPA Region 9 the calibration curve must be verified by a second source standard by analyzing the standard in the same manner as the initial calibration was performed. Acceptance is <or = 35% deviation for all compounds. This criteria will be met only be as required.

8.2 Initial Calibration Filename Format

The initial calibration is saved as part of the Chemstation method in the format MMDDYY##.m where MMDDYY is the date and ## indicates method type. (e.g., 080106TO, indicating an initial calibration performed on August 1, 2006 for a TO-15 Method)

9.0 Sample Collection

It is the responsibility of the field technician and/or field chemist to collect all vapor or air samples in manners which are consistent with protocol as set forth by federal, state and local regulatory agencies. These protocols are described in H&P 's sampling standard operating procedures. All sampling activities must be documented onto a sampling collection log sheet. Care should be taken by the lab operator to insure that samples are not inadvertently contaminated in the lab.

9.1 Soil Vapor/Air Samples

It is the responsibility of the field scientist to ensure that all vapor samples are collected in accordance with regulatory protocol. Samples should be collected in summa canisters, Tedlar bags or other vessels designed to prevent the loss of volatile compounds. Holding times for the various sample vessels should be observed. H&P will observe maximum holding times of 72 hours for Tedlar bags, 30 days for Summa or other passivated canisters unless changed or as required by regulatory decree.

9.2 Laboratory Acceptance of Samples

If the laboratory operator accepts samples from the field or in-house, samples should be delivered with the appropriate chain-of custody (COC) form. The lab operator should immediately inspect the samples against the chain-of-custody. The COC must contain

Unique sample identifier for all samples
Date and time of sampling
Source of sample including name, location and sample type
Analyses required
Name of collector
Pertinent field data such as purge volumes
Serial numbers of custody seals and transport cases (if used)
Custody transfer signatures and dates and times of sample transfer from the field to transporter and the lab
Bill of lading or transporter tracking numbers (if applicable)

The laboratory operator will check the COC for the above criteria in reference to the applicable methods. If any discrepancies are found, the laboratory operator should document those on the COC or in the daily analysis logs. The sample receipt custodian should also immediately inform the laboratory director and/or the project manager. No samples whose name or other information is in question should be analyzed. Once the laboratory director and/or the project manager have solved the problem analysis can begin.

Upon receipt of summa canisters, the sample receipt custodian is to measure and record the vaccum in the summa. The can # used and sample vacuum is to be recorded onto the COC.

10.0 Handling and Preservation

The purpose of this section is to describe the standard procedures used to assure that samples can be related to their data.

10.1 Soil Vapor Samples

As is the case with soil and water samples, it is the responsibility of the field scientist to ensure that all vapor samples are collected in accordance with regulatory protocols. Samples should be collected in summa cannisters, Tedlar bags or other vessels designed to prevent the loss of volatile compounds. Holding times for the various sample vessels should be observed. H&P will observe maximum holding times of 72 hours for Tedlar bags and 30 days for Summa canisters.

11.0 Sample Preparation

Sections 11.1 through 11.5 describe the steps for sample preparation for vapor samples.

11.1 Dilution Factor Calculation

The calibration curve assumes a standard volume of sample is introduced into the analytical instrument. Calculations for sample concentrations must take into account the actual volume of sample analyzed. To achieve lower detection limits or to analyze higher level samples, other sample volumes are required. The factor by which the current sample size differs from the expected sample size of 50 mL is called the "dilution" factor. Dilution factors less than one (or perhaps more precisely "concentration factors) are possible and mean that more than 50 mL was analyzed.

11.2 Laboratory Blank Preparation

Place a cleaned minican or other appropriate sample container on then autosampler. Alternatively, pure gas obtained from a high pressure tank may be used as long as the incoming pressure is less than 25 psi. Laboratory blanks are prepared from zero grade air or nitrogen in the appropriate container

11.3 Soil Vapor/Air Sample Preparation

Prepare soil vapor/air samples using the following steps:

If the sample is contained within a Summa canister, it may be directly placed on the autosampler using the quick connect fittings. Always measure the vacuum of the sample before analysis. If the vacuum is too high , sampling errors may have occurred**.** In this case the sample should be pressurized (diluted) and a larger sample volume analyzed. Screen can for water by pulling out 1 cc of sample while holding can tip down. If water is present, the syringe will show signs of water. If water is present use an adapter to keep water from entering the Entech sampler. Only vapor samples known to be dry may be placed on the autosampler upside down.

12.0 Troubleshooting

If there are problems with instrumentation refer to the appropriate manual for troubleshooting options.

12.1 Preventive Maintenance

Preventive Maintenance (PM) is that set of procedures taken in an effort to assure that sample throughput is continued and that data quality is not degraded by system malfunctions. Although

failure to perform preventive maintenance does not of itself produce poor quality data, the lack of such procedures may lead to earlier degradation of data and slower processing of samples.

This section treats PM in two sections: preparation and instrument PM.

12.1.1 Preparation PM

The primary area where preventable errors can enter throughout the preparation steps is the introduction of contamination. Preventive maintenance in the preparation steps primarily consists of thorough cleaning of canisters, glass syringes and other apparatus.

12.1.2 Instrument PM

There are five pieces of instrumentation involved in the analysis of volatile organics by GC/MS. They are the following:

(1) the Mass Spectrometer
(2) the Gas Chromatograph
(3) the Entech concentrator
(4) the Entech Autosampler
(5) the computer system

H&P follows the manufacturer's recommendations on mass spectrometer PM. Monitoring and changing rough and diffusion pump oil and cleaning of source are performed at regular intervals. A full stock of parts are kept in the lab including but not limited to source filaments and cleaning apparatus.

Gas Chromatograph PM consists primarily of maintaining a full stock of consumable parts. Swagelok nuts, ferrules, septa, etc. allow the operator to repair, change worn parts quickly and to continue operation without loss of time. Tubing should be periodically inspected for cracks and possible leaks. Monitoring of the gas levels and rate of pressure loss may help discover problems.

The concentrator can be monitored for leaks using a leak test program of the manufacturer. The tenax trap usually needs to be replaced every 6 months.

Autosampler(AS) PM is mainly concerned with cleanliness of the system and the checking of leaks. Following high level samples and standards it is desirable to run a blank through the AS.

The Computer system PM consists of keeping dust and dirt out of the components and backing up the data and methods as often as possible.

13.0 Data Acquisition, Calculation and Data Reduction

Once the system has been turned on, the daily calibration checked and a blank analyzed, samples and QA/QC samples may be analyzed.

13.1 Daily Calibration and Blank Analysis

1. Load the method files on concentrator and GC-MS. Use sequencing for multiple GC-MS analyses. Use scheduling to run multiple samples.

2. Standard Analysis

   Analyzed a standard equilvalent to 20 ppbv in a 50 ml sample using 100 mL of the 10 ppbv of standard mix and 50 ml of I.S/surrogate mix. All targets must be within specifications of the method less than 30% deviation in the RRF If they are not, a second calibration can be analyzed**.** The internal standard responses must be within 40% of the mean area response of the IS in the most recent calibration. The relative RRT for each internal standard must be within 0.33 minutes compared to the RRT of the internal standard from the most recent calibration.

   If the second calibration does not meet calibration specifications, troubleshooting may take place and subsequent calibrations attempted. For NELAC certified work, one must achieve two consecutive passing daily calibration prior to processing samples.

3. Analysis of blank air

   Analyzed a clean canister containing zero grade air or helium. Evaluate standard and blank according to QC parameters for CCV and blank. The blank must have all targets < R.L. The internal standard responses must be within 40% of the mean area response of the IS in the most recent calibration. The RRT for each internal standard in the blank must be within 0.33 minutes compared to the RRT of the internal standard from the most recent calibration.

4. Analysis of a sample duplicate (if required)

5. Analysis of LCS and LCSD (if required)

   A LCS is prepared using a second source standard at the same concentration of the primary source. This is performed in duplicate. Evaluate % recovery of each analyte. Recoveries must be 65 – 135%. The internal standard responses must be within 40% of the mean area response of the IS in the most recent calibration. The RRT for each internal standard in the LCS & LCSD must be within 0.33 minutes compared to the RRT of the internal standard from the most recent calibration.

13.2 Soil Vapor/Air Sample Analysis

1. Prior to vapor samples, it is necessary to adjust the multiplication factor in the computer to the appropriate value. For example, if a 50 mL vapor sample is analyzed, the multiplication factor to be entered would be 1. This factor can be adjusted in the Sample log table menu. (e.g., a 20 mL sample would have a dilution (concentration) factor of 50/20 =2.5)

2. After sample analysis , evaluate the internal standard responses, the surrogate recoveries and the target analyte concentrations. The internal standard responses must be within 40% of the mean area response of the IS in the most recent calibration. The RRT for each internal standard in the LCS & LCSD must be within 0.33 minutes compared to the RRT of the internal standard from the most recent calibration. At least 2 surrogates, one aromatic , the other chlorinated must pass. If not, the sample should be rerun either at a dilution or at the same concentration. Otherwise the data should be flagged. The chemist is to contact the QA Officer or lab director for the appropriate flag to use. The target analyte must not exceed the initial calibration range.

13.3 Data Processing

The GC/MS data system will perform peak integration of the target ion chromatograms. If parameters for threshhold and peak width are correctly set (see Agilent manuals), the algorithm used by the Hewlett-Packard instruments should correctly integrate 90% or greater of the ion chromatograms correctly. It is recommended that the operator allow this to occur and to simply view or monitor the result.

Sometimes manual integration is necessary. The following are some common examples in TO-15 where manual integration may be necessary.

1. Gaseous VOCs at low levels
   Due to noise at the top of the peak the program may only choose one half of the peak to integrate.

2. Peak tailing
   Excessive peak tailing can add significant area to a peak. The peak should be cut off such that the peak is as symmetrical as possible.

3. Unresolved peaks
   The program may pick the wrong peak to quantitative. E.g. the internal standard chlorobenzene-d5 is not always completely resolved from chlorobenzene and the latter can be chosen. This may be manually integrated using a drop to baseline method.

4. Interference
   Interference with the target ion may require manual integration. E.g. the surrogate 1,4 bromofluorobenzene is often interfered by fuel hydrocarbons.

5. Noise in the baseline

Noise in the baseline may add area to compounds which may incorrectly increase the amount detected. This is more often a problem at the lower levels. A noisy baseline can be witnessed by several small "bumps" in the ion chromatogram preceding or following the peak of interest. If noise is evident it shall be acceptable to manually integrate from the top of the noise line instead of the "zero" baseline level.

13.4 Qualitative Analysis

For the qualitative identification of each compound the analyst will follow the guidelines in Method TO-15,second edition Jan. 1999 Section 10.8

13.5 Reporting Limits

The normal reporting limits for most compounds are nominally 1-2 ppbv. These limits should be compared annually to the MDL's to make certain that they are appropriate. Depending upon client data quality objectives (DQO's) H&P may report lower than these values. The reporting limits, are determined from the lowest point on the calibration curve. The MDL is calculated at initial instrument checkout and updated yearly or at major instrument overhaul. Results reported that are between the R.L. and the MDL should be flagged with the "J" flag as a quantitative estimate. The "J" flag means the concentration reported is between the MDL and the RL. Reporting limits may be raised to accomplish specific data quality objectives as clients or regulatory agencies dictate with no effect on the data or flags required.

When samples are diluted for TO-14A/15, the reporting limits are raised proportionately. All multipliers must also be applied to the reporting limits as well. Dilutions are recorded on the daily extraction/run logs and are entered into the AGILENT chemstation software for the particular analysis.

13.6 Precision, Bias and Working Range

The applicable working range of the analyses for the EPA Method TO-14A/15 is dependent upon the concentrations of the calibration standards used while developing the calibration curve for the instrument. This calibration curve is discussed in Section 8.0. Generally, the working range of the instrument is between the detection limit for the analyte and the concentration of the high standard used for system calibration (normally 200 ppbv). In the event that sample results are greater than the amount used for the high standard, then sample dilution may be necessary. If the sample result is greater than the high standard then the sample must be diluted and reanalyzed or the "E" flag (estimated value) used to qualify the data.

14.0 Computer Hardware and Software

Agilent Technologies enviroquant software running on Pentium (or equivalent) PC's under Windows XP operating system is the data acquisition system. Chromatograms and quantitation reports are printed out on laser printers.

15.0 Data Management and Records Management

Document control is the process by which the documentation associated with samples and sample data are tracked and monitored.

15.1 Chain of Custody Record

A Chain of Custody Record serves to note the disposition of the samples, from the time of collection to laboratory receipt. This form should be filled out at the time of collection and signed by all persons collecting, relinquishing and receiving samples. All samples must be documented on an appropriate Chain of Custody form.

15.2 Sample Run Logs

H&P maintains a Sample Run Log for all samples analyzed. These logs are kept within a logbook, one of which is located in each data package. A new log is prepared for each work day. The following information is included on the run logs:

Date of operation
H&P job number
Operator name
Instrument No.
Analysis type
Extraction method
Duplicate information
Sample data (sample amount,dilution factor, etc.)
Calibration Standard identification
Sample Analysis data

Also located in each van are instrument and excursion logs. The instrument log is used to document problems and subsequent corrective action for the instrumentation (excessive column bleed, baseline irregularities, interference, etc.). The excursion log documents where and when the particular job takes place, the type of analysis, and operating conditions (weather, temperature, etc.). The excursion log should be filled out for each new job.

15.3 Reporting

15.3.1 The Data Package

The analyst will prepare a data package for Reporting and Review. For EPA Method TO-14A/15 the package will be produced on a daily basis corresponding to the 24-hour window of operations. The package will consist of the following in the order listed or as near as possible to that order:

a) The Original COCs for the samples analyzed during that day
b) The daily run logs

c) The BFB tuning report
d) Edited Quantitation reports from the daily initial Calibration (ICAL) or the passing Continuing Calibration Verification (CCV)
e) The Response factor summary (if ICAL) or the Evaluation of CCV (if daily CCV)
f) Edited quantitation report from the Initial Calibration verification (ICV) (If ICAL)
g) ICV summary, if done showing all compounds <30%D
h) Edited quantitation report from the LCS (if applicable)
i) LCS Summary showing percent recovery and control limits(if applicable)
j) Edited quantitation report from a blank analysis
k) Edited quantitation reports for all analyses and sample duplicate (if applicable).

In addition to the above daily package, other data pertinent to the project should be supplied to the data entry person(s) and the reviewer. Such project specific information as out of control events and associated recommendations for qualifier flags, corrective actions, MDL studies should be added to the above package if required by project DQOs.

The analyst will assure that the data is in the proper format above before passing it on for data entry or review.

15.3.2 Report Review

Data Review will consist of a third party review of the data generated during this project. The data reviewer will confirm the accuracy and completeness of the following items:

1. Folder
   Project Number
   Project Name (site)
   Project date

2. Accounting Information
   Lab confirmation form
   Report to information
   Billing to information

3. Print report on plain paper

4. From Chain of Custody Record confirm:
   a. Project information (client, project No. etc.)
   b. Correct dates
   c. Special instructions or out of control items
   d. Does the COC sample names match the report.

e. Check the COC for the following items:
f. Unique sample identifier for all samples
g. Date and time of sampling
h. Source of sample including name, location and sample type
k. Analyses required
l. Name of collector
m. Pertinent field data
n. Serial numbers of custody seals and transport cases (if used)
o. Custody transfer signatures and dates and times of sample transfer from the field to transporter and the lab
p. Bill of lading or transporter tracking number (if applicable)

5. From Raw Data:
   a. Organize raw data in order of date
   b. Go through the raw data page by page
   c. Date - is the correct date analyzed listed
   d. BFB tuning – does it pass?
   e. Blank - is it clean
   f. ICAL – does it meet method or project QC requirements (if applicable)
   g. ICV - does it meet method or project QC requirements?
   h. CCAL - is it acceptable under method or project QC
   i. LCS – within method or project specifications
   j. Sample - identify each individual set of data

6. Using the final report pages or forms, check that the raw data from above is correctly entered in the proper places on the pages or forms. Apply data qualifier flags to the data as necessary according to project requirements.

7. Edits
   With edited copy of rough draft
   Page by page make all changes
   Print out on letterhead
   Compare final to rough draft

8. Mail (If hardcopy is requested)
   Prepare the report; COC, chromatograms (if needed)
   Make a complete copy for our file

15.3.3 <u>Final Report</u>

The following information should be included in each H&P report on H&P letterhead:

1. client (name and address)
2. client job number / project name

3. site location (location of analysis)
4. test methods
5. dates samples received
6. dates samples collected
7. dates samples analyzed
8. analyst
9. sample results in table form titled Report Summary
10. sample identification number
11. units reported (ug/m3 or ppbv)
12. amount found and units
13. detection limit for each sample
14. QA/QC report for each method used(including deviations)
15. unique laboratory project ID number
16. identification of flagged data (including data outside quantitation levels)
17. chain of custody (including sample dates and time)
18. cover letter (including signature and title page)
19. a brief description of the test method
20. narrative (if needed)

A description of any unusual or notable events or conclusions, including samples that did not meet NELAC sample acceptance requirements (if applicable)

Any analytical testing provided by a subcontractor will be reported by the subcontractor on their letterhead, separate from the H&P report.

After the final laboratory report is issued, the report will remain unchanged. Any amendments to the report will be made as separate reports and will include a statement of amendment or supplement to the original report. H&P will notify the client promptly in writing of any defective measuring or validity problems with data.

H&P takes the necessary steps to ensure the confidentiality of its report, by providing data only to the client by phone, fax, e-mail or mail.

H&P shall certify that the test results meet all the requirements of the applicable regulatory laws (e.g., ELAP**,** NELAC, etc**.**) or shall provide a reasonable explanation as to why they do not.

16.0 Quality Assurance and Quality Control

Quality Assurance (QA) and Quality Control (QC) are a set of procedures and conditions designed to maximize the precision and accuracy of the analytical process. The procedures are also designed to maximize the precision and accuracy of the analytical process. QA/QC is a continuous process requiring verification by inspection and, if necessary, appropriate corrective action. Listed below are key items used to

ensure proper QA/QC. A brief description of each term is included, along with H&P and regulatory requirements.

Initial Calibration
Continuing Calibration
Detection Limits
- Minimum detection limit (MDL)
- Estimated Quantitation Limit (EQL)
- Reporting Limit

Blanks
Percent Recovery Calculation
Percent Relative Difference
Laboratory Control Limits
Sample Run Logs
Chain-of-custody

16.1 Initial Calibration (ICAL)

Prior to performing sample analysis, the GC must be initially calibrated. Following a clean blank analysis, at least five levels (only three are required for some regulatory areas) of target compounds along with surrogates and internal standards are introduced into the GC/MS system and the total area under each peak determined. These values are entered into the software. The computer will construct the calibration curve according to one of several methods. Hardcopy output of the chromatograms should be saved and kept with the instrument throughout the lifetime of the ICAL. The hardcopy output should list the method used to generate the ICAL curve. The ICAL is considered valid until the continuing calibration fails or a major change in the instrument operating condition occurs.

16.2 Tune/Continuing Calibration (CCAL)

The BFB tune as outlined in section 8.1 must be performed prior to analysis of any standards and repeated every 24 hours if samples or standards are to be analyzed. The calibration of the instrument is checked prior to analyzing samples and repeated every 24 hours if samples or standards are to be analyzed. The continuing calibration (CCAL) or continuing calibration verification (CCV)is analyzed to check the validity of the ICAL. Normally a standard corresponding to the midpoint of the curve is chosen. Response of the compounds of interest must be within that specified by the method (<30% RPD) for the continuing calibration check compounds (CCCs) for the curve to be valid. If not all compounds are of interest only those being analyzed need to be chec ked. The analyst may repeat a CCV if the CCV does not pass.

For work performed under the National Environmental Laboratory Accreditation Program (NELAP) If the second CCV does not pass, it will require two consecutive CCV analyses in order to pass the criteria or a new valid initial calibration.

16.3 Detection Limits

16.3.1 Minimum Detection Limit

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these MDLs at least once per twelve-month period. The laboratory shall maintain proof of the MDL demonstrations (i.e., before project samples are analyzed) and upon request in the format specified in Section 8. Results less than or equal to the MDL shall be reported as the MDL value and/or reported as non-detect ("nd") with the reporting limit value clearly shown.

Laboratories participating in the work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the following instructions:

(1) Estimate the MDL using one of the following:
   a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5, or
   b) The concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in helium or zero air, or
   c) The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

(2) Prepare (i.e., extract, digest, etc.) and analyze seven samples of 50 cc vapor containing the analyte of interest at a concentration three to five times the estimated MDL.

(3) Determine the variance (S2) for each analyte as follows:

$$S2 = \frac{1}{n-1}\left[\sum_{i=1}^{n}\left(x_i - \overline{x}\right)^2\right]$$

where xi = the ith measurement of the variable x and $\overline{x}$ = the average value of x

$$\overline{X} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

(4) Determine the standard deviation (s) for each analyte as follows:

s = (S2)1/2

(5) Determine the MDL for each analyte as follows:

$$MDL = 3.14(s)$$

(note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

(6) If the spike level used in step 2 is more than 5 times the calculated MDL, repeat the process using a smaller spiking level.

Where multiple instruments are used, the MDL used for reporting purposes shall represent the least sensitive instrument.

16.3.2 Estimated Quantitation Limit (EQL) formerly called Practical Quantitation limit (PQL).

It should be noted that this detection limit might vary due to low sample amounts or dilutions. In most cases, H&P reports EQL values roughly 5-10 times the MDL. However for TO-15 the PQL is set at the lowest standard in the calibration curve.

16.3.3 Reporting Limit

The reporting limit is a limit below which regulators or clients are uninterested or will not regulate. Thus TO-14/TO-15 compounds that are detected but are below the established reporting limit are reported as non-detect.

16.4 Blanks

A Method Blank is an analyte free matrix sample, run through the sample preparation and analysis procedures of those used for samples. These blanks check the levels of contamination for the method. Blanks must be run at a frequency of one per batch of samples or preparation method. If a contaminate is found the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problems if:

a) the target analytes in a reagent blank are higher than the reporting limit (dilution factors taken into account) in effect at the time

b) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated blank sample batch or the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

16.5 Matrix Spike/Matrix Spike Duplicate and Lab Control Sample

The Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are two aliquots of the same sample, which have been which have been spiked with a known concentration of a target analyte. Together, the results of these two sample spikes are used to indicate method precision and accuracy, and to characterize the effects, if any, of matrix interference. H&P usually analyzes only laboratory control samples and their duplicates since vapor samples typically have little matrix effects. The LCS is spiked directly into a clean minican or Summa Canister with the same analytes at the same concentrations as the ccv and is analyzed with the sample batch. If the results of the Laboratory Control Sample (LCS) fails, the failing analyte is to be qualified. (Certain locales have a different definition of an LCS, e.g., for the LARQCB an LCS is a low level spike that is performed after a batch of samples that have all tested non-detect. This is supposed to prove that the method was functioning during the analysis of the batch.) If matrix spikes are required, the chemist is to consult the laboratory direstor to determine appropriate procedures.

16.6 Percent Recovery Calculation

The values for the LCS and LCSD are used to calculate the Percent Recovery (PR), an estimate of the accuracy of the analytical process. The Average PR can be calculated using the following general equation:

$$\text{Average PR} = 100((\text{LCS} + \text{LCSD})/2(\text{SA})$$

where: PR = Percent Recovery
LCS = Laboratory Control Sample Result
LCSD= Laboratory Control Sample Duplicate Result
SA = Spiked Amount

H&P calculates the PR for each set of spikes (LCS and LCSD) and the resultant value is included in the QA/QC section of the report.

16.7 <u>Relative Percent Difference</u>

The Relative Percent Difference (RPD) is calculated from LCS and LCSD data and is used to estimate overall method precision. The RPD can be calculated using the following general equation:

$$\text{RPD} = 100(\text{LCS} - \text{LCSD})/(\text{LCS} + \text{LCSD})/2$$

This RPD data is calculated for each set of spikes (LCS and LCSD) and is included in the QA/QC section of the report.

16.8 <u>Laboratory Control Limits</u>

Each certified laboratory establishes their own set of Laboratory Control Limits (LCL's). These limits include criteria such as recovery ranges and maximum allowable RPD's. H&P has established the following list of control limits for EPA Method TO-14A/15 :

	Average percent recovery	Relative Percent difference	Marginal Exceedance Limit
All Targets	65 - 135	≤ 30	≤40

16.8.1 LCS Marginal Exceedance

The current typical list of target compounds contains 65 target analytes. If any analyte exceeds the QAQC limit of 65-135% recovery, the analyte is to be qualified in the data report. QAQC limits may be changed due to control chart evaluations.

16.8.2 Marginal Exceedance Randomness.

The LCS marginal exceedance allowance assumes randomness of the exceedance for all compounds. The analyst can monitor the occurrence of LCS compounds using the Laboratory Information Management System OR simply by close observance of their instrument data. In either case a compounds will be deemed to have exceeded the random nature of the marginal exceedance when its frequency of exceedance becomes more than twice that of the next most frequent compounds exceedance. That compound will then be deemed out of control and corrective action must be taken or qualifying flags attached to the data.

Surrogates

4-Bromofluorobenzene	77 - 125
Toluene-d8	78 - 125
1,2-Dichloroethane-d4	76 - 134

Samples where more than 1 surrogate exceed the limits are re-analyzed one time.

17.0 References

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination of Volatile Organic Com=pounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectromtery, Center for Environmental Research Information office of Research and Development U.S.E.P.A Cincinnati,OH, January 1999

Quality Systems Manual, H&P, Inc. most recent version

H&P MOBILE GEOCHEMISTRY, INC.
STANDARD OPERATING PROCEDURES (SOPs)

8260 SV SOP for determining VOC's in Soil Vapor

1.0 Scope and Applications

This SOP has been prepared for H&P Mobile Geochemistry, Inc to help ensure consistent analytical protocol and applies to the performance of the the analysis of Volatile Organic Compounds(VOCs) in soil vapor samples. This method is applicable to soil vapor samples that are collected in summa canisters, syringes and Tedlar bags. The scope of topics discussed in this SOP includes the following:

* Method Summary
* Health and Safety Warnings
* Cautions
* Interferences
* Personnel Qualifications
* Instruments and Materials
* Method Calibration
* Sample Collection
* Handling & Preservation
* Sample Preparation
* Troubleshooting and Maintenance
* Procedure, Data Acquisition, Calculation and Data Reduction
* Computer Hardware and Software
* Data Management and Records Management
* QA/QC Requirements
* Detection Limits
* Pollution Prevention and Waste management
* References and Definitions

2.0 Summary of Method

H&P Mobile Geochemistry, Inc. uses a Purge and Trap system in conjuction with a gas chromatograh and mass selective detector to identify and quantitate seventy (70) volatile organic compounds. Samples are introduced into the chromatograph by Purge and Trap autosampler. Soil vapor samples are introduced into a glass purge chamber that has been prepared with water containing a specified amount of internal standard (I.S.) and surrogates. Alternatively, a large volume of vapor sample may be purged directly onto the trap while the internal standard in being simultaneously purged in another chamber up stream. The analysis is then performed on a gas chromatograph (GC) with a quadruple mass spectrometer (MS).

The volatile organic compounds identified are as follows:

Dichlorodifluoromethane
Chloromethane
Vinyl Chloride
Bromomethane
Chloroethane
Trichlorofluoromethane
1,1-Dichloroethene
Freon 113
Acetone
Methylene Chloride
Methyl t-butyl ether
trans-1,2-Dichloroethene
1,1-Dichloroethane
2-Hexanone
2-Butanone
2,2-Dichloropropane
cis-1,2-Dichloroethene
Chloroform
Bromochloromethane
1,1,1-Trichloroethane
1,1-Dichloropropene
Carbon Tetrachloride
1,2-Dichloroethane
Benzene
Trichloroethene
1,2-Dichloropropane
Bromodichloromethane
Dibromomethane
trans-1,3-Dichloropropene
Methyl Isobutyl Ketone
Toluene
cis-1,3-Dichloropropene
1,1,2-Trichloroethane
2-Hexanone
1,2-Dibromoethane
1,3-Dichloropropane
Tetrachloroethene
Dibromochloromethane
Chlorobenzene
Ethylbenzene
1,1,1,2-Tetrachloroethane
m,p-Xylene
o-Xylene

Styrene
Bromoform
Isopropylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichloropropane
n-propylbenzene
Bromobenzene
1,3,5-Trimethylbenzene
2-Chlorotoluene
4-Chlorotoluene
tert-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
p-Isopropyltoluene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
n-Butylbenzene
1,2-Dichlorobenzene
1-2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene
Diisopropyl ether
Ethyl t-butyl ether
2-methoxy-2-methylbutane
t-Butanol
Ethanol

3.0 Health and Safety Warnings

H&P Mobile Geochemistry, Inc. employs conservative safety practices at all times. Eye protection is to be worn by all laboratory operators handling high pressure gas cylinders and during sample collection. The laboratory operator is not to enter the exclusion zone (nor any other areas designated as exclusive by the field safety officer) at any time. Smoking is not permitted in the laboratory and eating is not permitted in the sample preparation area.. Fire extinguishers, first aid kits and Material Safety Data Sheets are located in each lab. The laboratory equipment may include high-pressure hydrogen which is a fire and explosion hazard.

4.0 Cautions

The analyst is cautioned that the equipment uses high-pressure gases which, while not flammable nor explosive, are suffocating in large quantities. The lab may also contain high pressure hydrogen for other analyses and that there is both a fire and explosion hazard in the event of a leaking system. The analyst should be aware of any potential source of leaking H_2 gas and should inspect all plumbing from hydrogen cylinders to the instruments on a daily basis.

5.0 Interferences

When analyzing for volatile organics, samples can contain high concentrations of petroleum products that contain a high concentration of target and non-target analytes. These analytes may interfere with internal standard and surrogate recoveries. The analyst should attempt to analyze these samples at the lowest dilution factor to obtain the lowest achievable reporting limits but at the same time meeting QAQC requirements. In addition, the purge and trap method is prone to carry over. A blank should be analyzed after any high level sample to ensure that carry over is eliminated. In addition, any glass syringe used in the collection and/or transfer of vapor samples are to be baked before reuse.

6.0 Personnel Qualifications

This method is to be performed by a trained analyst in gas chromatographic/mass spectrometric methods. A bachelor's degree in science or equivalent training is the minimum requirement for performance of this method. In addition, a mobile lab analyst performing this method without on-site supervision must have a minimum of 3 months of GC/MS experience with this method or equivalent.

7.0 Instruments and Materials

The following subsections discuss the instrumentation and materials needed for this method.

7.1 Instruments

H&P uses several GC/MS systems for Method 8260 SV analysis. The gas chromatographs used can be an Agilent model 5890 or 6850 or a 6890, the Mass Selective Detector (MSD) can be an Agilent model 5973 or a 5975, . (See section 12 of the QSM for instrumentation approved for this procedure). The purge and trap is a Tekmar 3000 or 3100 with a Tekmar Solatek 72 autosampler with 72 sample capacity. H&P Mobile Geochemistry, Inc. has had the most success using HP-624 columns of 25m X 0.25mm (.25 mm as well) X 1.8u film thickness and uses these columns in all systems. The carrier gas He is UHP purity. The following instrument conditions are typical:

GC

Split mode	40:1
Injector temp.	200 degrees C
Interface temp.	230 - 250 degrees C
Column:	25m x 0.25mm x 1.8u HP-624
Flow:	0.8 - 1.0 mL/min He

Initial temp.	40 degrees C 2 minute
Ramp	20 degrees/min
Final temp	230 degrees

MS	
SCAN mode	41-300 amu, 2.5 scans/sec
Source temp	230 degrees
Quad temp	150 degrees
Multiplier	+200 relative depends on response

Detailed descriptions of the analytical method parameters are to be found in the initial demonstration of proficiency data package which is kept as part of H&P Mobile Geochemistry Lab initial lab certification.

The Solatek AS has default amounts of 20 mL water and 20 mL vapor. The chemist is to manually spike 20 ml of water with 2.5 ul of 20 ppm I.S/surrogate combo mix or individual I.S. and surrogate mix.

Soil vapor samples are collected in gas-tight 50 mL glass syringes, summa canisters, or tedlar bags. An aliquot of the sample is removed using another glass syringe and immediately transferred (within 15 minutes from sampling) to the autosampler purge apparatus**.** Soil vapor sample must be transferred to the autosampler as soon as possible preferably within 5 minutes, if not possible, the maximum time allowed is 15 minutes. It may be necessary to carry prepared VOAs to the vapor probe so that the vapor sample can be spiked as soon as possible after collection.

HP Chemstation software version G1701BA, or equivalent, with Enviroquant software running on Windows XP/Pentium personal computers are used for data acquisition and instrument control. Chromatograms and quantitation reports are printed out on HP LaserJet printers.

The above-mentioned equipment requires 110 VAC as a power source to operate.

Sources of further information can be found in the Hewlett-Packard GC/MS and the Tekmar Purge and Trap hardware and software manuals, which are kept with each instrument. Each instrument also comes with HP maintenance information on CD, which can be viewed on the computer.

7.2 Reagents and Standards

Purge and Trap grade methanol is used for all preparation and dilutions of standards. Primary standards for analytes, surrogates and internal standards are normally purchased from commercial vendors and are diluted for use. All purchased standards are shipped with certification attesting to their purity and authenticity. All standards are prepared at the Carlsbad facility. Refer to H&P Mobile Geochemistry's QSMl or the individual notebook for nomenclature of standards.

All standards should be stored in the standards refrigerators located within the laboratory and stored separately from samples.

7.2.1 Standard Preparation

If available, primary standards of 2000 ug/mL should be purchased. Normally a solution is diluted to a convenient volume, such as 10 to 1. To prepare a stock standard from a primary solution, place approximately 2 mL Purge & Trap grade methanol in a 20 mL ground glass stoppered volumetric flask. Inject 2 mL of each 2000 ug/mL primary standard into the methanol using a syringe. Fill the flask to volume, stopper the flask, and mix by inverting three times. Calculate the concentration of the stock standard in ug/mL by dividing the primary concentration (2000 ug/mL) by 10, resulting in 200 ug/mL**.** If alternative stocks are used with different concentrations, it will be necessary to adjust the volume spiked. The working stock standard is to result in all VOC's, gases, freon 113, and oxygenates at 20 ug/mL, all ketones at 200 ug/mL, and isopropyl alcohol and ethanol at 800 ug/mL. The oxygenate, tert-butyl alcohol, will be at 200 ug/mL for the first source and 100 ug/mL for the second source working standards**.** Label the storage vial with the proper lab ID, concentration in ug/mL, the expiration date and the initials of the chemist. Transfer the stock standard to the pre-labeled vials and store at 4 degrees C. Each vial is to be labeled with lab ID and distributed to each chemist. Log the standard into the laboratory standard preparation log book. The second source stock must be obtained from a different vendor than the first source stock.

The working standards are prepared at the Carlsbad facility and verified as accurate before distributing to other chemists for use. During the preparation, a second source is prepared as well and also verified before use.

7.2.2 Standard Expiration

Standards for VOC analysis will be prepared and given expiration dates of no longer than those indicated below. Care must be taken that these dates do not exceed the manufacturer's date of expiration on the primary standard.

Stock Standards

Internal Standards and surrogates	- 6 months
Non gas VOCs	- 6 months
Gaseous VOCs	- 1 month

Secondary Standards

Internal Standards and surrogates	- 3 months
All other standards	- 1 month

Once standards are prepared, they are to be certified by a chemist prior to distributing for use. The lab chemist should also monitor response factors to determine whether standards are degrading. If this should occure, the laboratory director is to informed immediately.

8.0 Method Calibration

In order to analyze samples the instrument must first be calibrated. The following subsections describe the system start-up and initial calibration procedures.

8.1 Initial System Calibration

Prior to performing analyses, the GC-MS must be tuned and calibrated to ensure system precision and accurate results.

Tuning of the GC-MS is accomplished using the HP Target Tune function in the BFB tune mode. The instrument automatically performs the tuning in an attempt to adjust parameters such that the spectrum of the compound 1,4-Bromofluorobenzene will meet the following specifications:

- Mass 50 — 15 percent to 40 percent of mass 95
- Mass 75 — 30 percent to 60 percent of mass 95
- Mass 95 — base peak, 100 percent relative abundance
- Mass 96 — 5 percent to 9 percent of mass 95
- Mass 173 — less than 2 percent of mass 174
- Mass 174 — greater than 50 percent of mass 95
- Mass 175 — 5 percent to 9 percent of mass 174
- Mass 176 — greater than 95 percent, but less than 101 percent of mass 174
- Mass 177 — 5 percent to 9 percent of mass 176

Following tuning, 50 ng of BFB must immediately be evaluated in the next blank or standard to determine the validity of the tune. Since BFB is one of the surrogate compounds that is included in every analysis, it can be monitoried following any analysis. If the above criteria are not met, the instrument must be retuned or the problem found prior to proceeding. The area response for the first standard, Fluorobenzene, should be set to have a total area response of around 130,000. If not, the electron multiplier can be increased to accomplish this sensitivity.

Prior to performing analyses, the GC-MS must be calibrated to ensure system precision and accurate results. To calibrate the GC-MS, standards of the analytes, along with internal standards and surrogates are injected into reagent water, and measured by the method. A minimum of five concentration levels must be used during calibration. Each standard solution is analyzed under the identical parameters as samples will be analyzed. The end result is a calibration curve for each analyte. The following section decribes the preparation of a 10-point calibration curve which is commonly used for vapor analysis calibration.

To prepare a calibration curve:

1. Turn on the system.

2. Load a Method (e.g.8260.mth) into the software. Schedule the autosampler for the correct Method (8260.mt).

3. Vendor approved, VOA vials with septa caps are to be used.. Each sample VOA should be heated to 230 degrees C for approximately 15 minutes in a GC oven or in a toaster oven, and allowed to cool until they can be safely handled. When cool, they should be recapped immediately. Each VOA can also be flushed with lab grade N_2 or lab grade He.

4. Transfer 20 mL of reagent water to a syringe. Spike the water with the 50 ng of internal standard standard **(**2.5 uL of 20 ug/ml). Add **1** ng VOC compounds, gases and Oxygenates etc. (.05 uL of 20 ug/mL VOC standard) to the water in the syringe. Transfer the water to a VOA purge tube. Alternatively, a chemist may inject all working stocks directly into a VOA vial containing 20 cc of water. Repeat this step using 0.1 uL of 20 ug/mL VOC standard to prepare the 2 ng VOC standard.

5. Repeat Step 4 for the 5, 10, 20, 50, 100 and 500 ng level by injecting .25 uL, .5 uL, 1.0 uL, 2.5 uL, 5.0 uL and 25 ul of the 20ug/mL VOA standard and the 20 ug/mL surrogate stock respectively. Spike the water with the 50 ng of internal standard standard **(**2.5 uL of 20 ug/ml). Alternatively, a chemist may inject all working stocks directly into a VOA vial containing 20 cc of water.

6. Repeat Step 5 for the 1000 ng and 2000 ng by injecting 50 uL and 100 uL, of the 20ug/mL VOA standard, respectively (do not add surrogate standard). . Spike the water with the 50 ng of internal standard standard **(**2.5 uL of 20 ug/ml). Alternatively, a chemist may inject all working stocks directly into a VOA vial containing 20 cc of water.

7. After completing the standards, quantitate and load the standards as the initial calibration. Evaluate the initial curve for linearity using response factors using internal standard calibration method.

   a. The SPCC compounds must have RF's greater than
      Chloromethane 0.1
      1,1-Dichloroethane 0.1
      Bromoform 0.1
      Chlorobenzene 0.3
      1,1,2,2-Tetrachloroethane 0.3

   b. All analytes must have %RSD less than 30%.

   c. Targets with %RSD greater than 30% may be treated in the following manner:

The analyst may drop either the high or low point of the curve thus achieving an abbreviated or narrower linear range for the curve. If the high point is dropped, this compound will have a lower high point for the curve. If the low point is dropped, the reporting limit must be raised proportionately.

Targets with %RSD greater than 30% may be evaluated using a linear regression of the instrument response versus the concentration of the standards. The algorithms chosen in the software must be "linear regression" not "linear regression – force (0,0)." The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. To use the linear regression algorithm, r must be greater or equal to 0.99. If linear regression is used, the analyst should determine if the daily CCV will pass if quatitated under this curve.

For soil vapor analysis many analytes have to have the lowest calibration standard in the calibration curve set at 2 ng. Vinyl Choride actually requires a 1 ng calibration RL. The acceptance criteria for the % RSD for the calibration curve is 30%. As a result, the calibration curve for water and soils will be different than the one used to process vapor samples.

8. The calibration curve is checked for validity using a calibration standard from a second source. A mid-level (typically 50 ng on column) second source is introduced into the system in the same manner as the curve. In order for validity to be achieved, the second source must pass the requirements of a daily calibration i.e., SPCC's and CCC's must pass per method. In addition, all analytes must be withing 30% recovery. This can be determined by evaluating the second source as a continuing calibration standard using relative response factors. If any linear regression is used, the evaluation based upon calculated concentration is to be used for the effected analytes.

   For some soil vapor projects, the curve must be also validated against a vapor standard. This is performed by injecting 20 cc of a 1 ppmv standard into a prepared VOA vial. The syringe should be warm to the touch when used. The true concentration is to be converted to ppmv and evaluated for recovery. The criteria is the same as listed above.

   The calibration curve must be verified each working day by injecting the first source standard at near the mid point of the calibration curve. SPCCs must have RF's at least equal to the levels specified above. CCC compounds must be less than 20% for the system to be considered still within calibration.

9. If all the above CCC compounds do not meet the 20% criteria several options are available.

   a. The analyst may re-analyze the calibration standard, re-prepare the calibration standard and re-analyze and/or perform instrument troubleshooting and re-analyze.

   b. Target compounds may be individually screened to see if they meet the 20% criteria. If the target analytes necessary to meet the client DQO meet the 20% criteria, analysis may continue.

   c. Data from those compounds which do not pass the 20% criteria may be flagged as estimated.

10 The RRT of each target analyte in each calibration standard must agree within .06 RRT units. The retention times of the internal standards in the CCV must be evaluated. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level in the most recent calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made as required.

11 The EICP area for any of the internal standards in the CCV must fall within -50% to +100% from that in the mid-point standard level of the recent calibration. If not, then the chromatographic and mass spec system must be inspected for malfunctions and corrections must be made as required. Usually adjusting the EM voltage and reanalyzing the CCV will solve this problem.

8.2 Initial Calibration Format

The initial calibration is saved in the format MMDDYY##.m where MMDDYY is the date and ## indicates method type.

9.0 Sample Collection

If the chemist is collecting the soil vapor samples, they are to follow the sampling protocols listed in the project confirmation sheet. Care should be taken by the lab operator to insure that samples are not inadvertently contaminated in the lab.

If the chemist is collecting samples they are to follow H&P Mobile Geochemistry's SOP for soil vapor sample collection and complete the soil vapor collection log sheet. The chemist is to refer to the project confirmation sheet to determine the details of the sampling such as purge tests and purge volume requirements. Samples should be collected in decontaminated glass syringes that are gas tight. Tedlar bags or summa cannisters designed to prevent the loss of volatile compounds can also be used to hold or collect samples respectively . Holding times for the various sample vessels should be observed. H&P Mobile Geochemistry will observe maximum holding times of 72 hours for Tedlar bags, 15 minutes for syringe samples and 30 days for summa canisters.

Once vapor samples have been collected in glass syringes, they should immediately be placed on the purge and trap autosampler or in an appropriate container away from sunlight. Sunlight can affect temperature sensitive or photosensitive compounds. Wrapping foil around the sample container will help protect the samples.

9.1 Laboratory Acceptance of Samples

The laboratory operator may accept samples from the field or perform the sample collection. If acceptabed from a client, samples should be delivered with the appropriate chain-of custody (COC) form. The lab operator should immediately inspect the samples against the chain-of-custody. Alternatively, the chemist can prepare the COC. The COC must contain

Unique sample identifier for all samples
Date and time of sampling
Source of sample including name, location, depth, container and sample type
Analyses required

Name of collector
Pertinent field data
Custody transfer signatures and dates and times of sample transfer from the field to transporter and the lab
Bill of lading or transporter tracking numbers (if applicable)

For on site mobile laboratory, the chemist may fill out the COC and it must contain the following:

Client Project number
Address of site location
Project contact name, phone number and email address
Under Sample Receipt box, be sure to Circle N/A (received on site)

The laboratory operator will check the COC for the above criteria in reference to the applicable methods. If any discrepancies are found, the laboratory operator should document those on the COC or in the chemist log sheet. The custodian should also immediately inform the field personnel of the problem. Samples in which the analysis information is in question should not be analyzed. Once sampling personnel have solved the problem analysis can begin.

10.0 Handling and Preservation

Once vapor samples have been collected, they should immediately be placed in a VOA or a purge chamber on the purge and trap autosampler or in an appropriate container away from sunlight. This should be done within 15 minutes from collection. If necessary, the chemist may carry prepared VOAs to the sampling area and immediately transfer the soil vapor sample to the VOA after collection. The VOA will be stable for at least 8 hours with minimal losses. If this is not possible, the vapor sample is to be placed in a tedlar bag or a summa canister.
If samples are to be shipped after collection they must be in either tedlar bags or summa canisters. The samples must be wrapped using some foam or packaging material to protect from being damaged during shipment. All sample shipments must be given a tracking number. Tedlars should be shipped overnight due to holding time restrictions and Summa canisters must be insured due to their high cost . All vapor samples are to be shipped at ambient temperature and not under refrigeration.

11.0 Sample Preparation

Sections 11.1 through 11.5 describe the steps for sample preparation for both soil and water matrices.

11.1 Dilution Factor Calculation

The calibration curve is based on the mass analyte introduced into the instrument. Calculations for sample concentrations are based on the volume of sample analyzed. If one cc of soil vapor are analyzed, the concentration of the analyte is reported as follows:

$$\frac{\text{Mass}}{\text{Sample mass}} \quad \text{OR} \quad \frac{\text{Mass}}{\text{Sample volume}}$$

To achieve lower detection limits, larger sample masses or volumes are required. If a five cc of soil sample is purged, the results are for mass/5 . To correctly report mass/1 g, the results must be divided by five. The standard soil vapor reporting limits are based upon a 20 cc sample size. To correctly report the concentration the value “on column” must be divided by 20. (i.e. dilution factor = 0.05).

The HP Chemstation software requires the analyst to input a "dilution factor" into the sample sequence to properly report sample concentration. This "dilution factor" is calculated by dividing 1 by the amount of sample used

11.2 Laboratory Blank Preparation

For soil vapor sample analysis, a blank is prepared as follows:

1. Add 20 mL blank water to a 20 mL syringe. Add 50 ng I.S. (2.5 ul of 20 ug/ml I.S). and 50 ng surrogate solution (2.5 uL of the 20 ug/mL surrogate solution.). Transfer to a VOA

2. Using a clean glass air-tight syringe, collect 20 cc of ambient air. Which has been collected outside of laboratory and away from any generators or vehicles. Inject the ambient air into the prepared VOA vial through the septa. Analyze. If high volume analysis is performed using the purge chamber, inject 100 cc of ambient air into the purge chamber when autosampler is in the puge mode.

11.3 Soil Vapor Samples Preparation

Prepare soil vapor samples using the following steps:

1. Transfer 20 mL of water to a VOA purge vessel. Add 50 ng of the I.S./surrogate solution (2.5 uL of the 20 ug/mL solutions).

2. Add 20 mL soil vapor through VOA septa .

3. Analyze the sample**.** If sample is expected to be high level, use smaller sample volume. If the client wants the data reported to the commercial CHHSLs, the chemist should inject either 25 mL or 30 mL or use the high volume injection procedure through the purge chamber on the concentrator. It may be necessary to keep a small volume of reagent water in this purge chamber to minimize carry over. If the client wants the data reported to the residential CHHSLs, the chemist must use the large volume injector and use a 100 cc sample volume.

11.4 Laboratory Control Standard (LCS) Preparation

A LCS is prepared as follows:

Add 20 mL blank water to a 20 mL syringe. Add 50 ng I.S. (2.5 ul of 20 ug/ml I.S). and 50 ng surrogate solution (2.5 uL of the 20 ug/mL surrogate solution.). Add 50 ng of 20 ug/ml second source standard. Analyze.

12.0 Troubleshooting and Maintenance

If there are problems with instrumentation refer to the appropriate manual for troubleshooting options.

12.1 Preventive Maintenance

Preventive Maintenance (PM) is that set of procedures taken in an effort to assure that sample throughput is continued and that data quality is not degraded by system malfunctions. Although failure to perform preventive maintenance does not of itself produce poor quality data, the lack of such procedures may lead to earlier degradation of data and slower processing of samples.

This section treats PM in two sections: preparation and instrument PM.

12.1.1 Preparation PM

The primary area where preventable errors can enter throughout the preparation steps is the introduction of contamination. Preventive maintenance in the preparation steps primarily consists of baking and/or nitrogen flushing of VOAs before use**.** In addition, the baking of syringes before collection and trasfer of vapor samples is essential.

12.1.2 Instrument PM

There are five pieces of instrumentation involved in the analysis of volatile organics by GC/MS. They are the following:

(1) the Mass Spectrometer
(2) the Gas Chromatograph
(3) the Purge and Trap concentrator
(4) the Purge and Trap autosampler
(5) the computer system

H&P Mobile Geochemistry follows the manufacturer's recommendations on mass spectrometer PM. Monitoring and changing rough and diffusion pump oil and cleaning of source are performed at regular intervals. A full stock of parts are kept in the lab including but not limited to source filaments and cleaning apparatus.

Gas Chromatograph PM consists primarily of maintaining a full stock of consumable parts. Swagelok nuts, ferrules, septa, etc. allow the operator to repair, change worn parts quickly and to continue operation without loss of time. Tubing should be periodically inspected for cracks and possible leaks. Monitoring of the gas levels and rate of pressure loss may help discover problems. The chemist should routinely monitor the foreline pressure of the mass spec and document the value that is expected when the instrument is operating properly. Sometime a power flucuation may cause the mass spec transfer line to go OFF. This temperature should also be checked.

The purge and trap concentrator can be monitored for leaks by observing the pressure of purging. In general the purge operates at a purge pressure of 4-5 psi. A pressure less than this may indicate a leak in the purge vessel. To determine if there is a leak in the purge & trap concentrator, measure the flows at the vent outlet on the concentrator for the following:
During purging at time =5 minutes, during desorb, during bake, during standby. The purge flow should be about 40 cc/min, desorb flow should be less than 1 cc/min , bake flow should be 60-100 cc/min and at stanby the flow should be the same as the purge flow. Any descepencies may indicate a leak is present.

The chemist should also keep track of the temperatures of all the listed items in the autosampler and concentrator to ensure they are at the "Set" value. A routine hand check of the transfer lines to determine if they are hot, is suggested.

Autosampler(AS) PM is mainly concerned with cleanliness of the purging system and the checking of leaks. Following high level samples and standards it is desirable to run blank water and purge gas through the AS.

The Computer system PM consists of keeping dust and dirt out of the components and backing up the data and methods as often as possible.

13.0 Procedure, Data Acquisition, Calculation and Data Reduction

Once the system has been turned on, the daily calibration verified and a blank analyzed, samples may be analyzed. After preparing the sample extractions as directed above, load the control file and proceed with analyses.

13.1 System Start-Up - Mobile labs

In the mobile laboratories the mass spectrometer is maintained heated and under vacuum through the use of the inverter/battery system, generator or shore power.

Upon leaving the garage:

1. Shut all AC off. Unplug the electric power cords. If power cords are attached to the laboratory, place them securely in their storage cabinet and secure the door. Pull the lab out of the bay and start the generators. Wait for the delay period to ensure that the system is running on AC.

2. Start air conditioners if needed. Check temperature of lab to ensure the internal temperature is between 70 – 80 degrees F. The analyst must attempt to get internal lab temperature consistent with its calibration temperature. If the outside temperature becomes such that it is impossible to maintain the internal lab temperature within 70 – 80 degrees F, the laboratory director is to be contacted. If approved to proceed with analysis, the analysts is to document on the chemist's notes that non-conforming work was performed and provides details accordingly.

3. Make sure all equipment has transitioned to generator power successfully. Allow laboratory to come to standard operating temperature before proceeding to analyze sample. Equilibration time is typically 15 minutes.

4. Check the vacuum status. Diff. pump should be hot, MS should be around 145-165 degrees (230 source, 150 analyzer for the 5973A) and foreline pressure around 50 mm or less.

13.2 Daily Calibration and Blank Analysis

1. Load the method files on P&T and GC-MS. Use sequencing for multiple GC-MS analyses. Use scheduling to run multiple P&T samples.

2. Standard Analysis

   For the Solatek, 50 ng of target (2.5 uL of the 20 ug/mL target solution) are added by hand into an appropriate amount of reagent water in the syringe. Introduce into the VOA

3. Analysis of blank water

Add an appropriate amount of blank water to syringe. Add 50 ng each of surrogates and internal standards solutions to water. Introduce into the VOA

4. Evaluate the standard and blank according to QC parameters for CCV and blank**.** For soil vapor another blank is to be analyzed containing ambient air collected from the site. This is to be reported as the method blank.

13.3 CCV and Blank Evaluation

CCV Evaluation - SPCC Compounds

All SPCCs must be within specifications of the method. If they are not, a second calibration can be analyzed. If all SPCC compounds do not meet criteria instrument, troubleshooting should be performed.

CCV Evaluation - CCC Compounds

If all compounds are within 20% drift then all analytes are assumed to be within calibration and analysis can continue. Otherwise compound "hits" out of calibration must be flagged (see QSM Appendix 8E for a list of data qualifiers). Approval must be obtained by the laboratory director or QA Officer before proceeding to run client samples. Data quality objectives may be revised on a project by project basis to relax this requirement.

Blank evaluation

The blank must have all targets < R.L.

Once the system has been turned on, the daily calibration checked and a blank analyzed, samples may be analyzed.

13.2 Soil Vapor Sample Analysis

Prior to analyzing vapor samples, it is necessary to adjust the multiplication factor in the computer to the appropriate value. For example, if a 20 mL vapor sample is analyzed, the dilution factor to be entered would be 0.05. This factor can be adjusted in the Sample log table menu.

13.3 QAQC Sample Analysis

Soil vapor batches must have an acceptable a laboratory control sample (LCS) analyzed and pass QAQC criteria for the batch to be considered acceptable. Otherwise all samples in the batch may need to be reanalyzed or flagged. A single LCS is to be analyzed at the end of the analytical run. See section 16 for more information.

13.4 Data Processing

The GC/MS data system will perform peak integration of the target ion chromatograms. If parameters for threshholding and peak width are correctly set (see HP manuals), the algorithm used by the Hewlett-Packard instruments should correctly integrate 90% or greater of the ion

chromatograms correctly. It is recommended that the operator allow this to occur and to simply view or monitor the result.

Sometimes manual integration is necessary. The following are some common examples where manual integration may be necessary.

1. Gaseous VOCs at low levels - Due to noise at the top of the peak the program may only choose one half of the peak to integrate.

2. Peak tailing - Excessive peak tailing can add significant area to a peak. The peak should be cut off such that the peak is as symmetrical as possible.

3. Unresolved peaks - The program may pick the wrong peak to quantitate. E.g. the internal standard chlorobenzene-d5 is not always completely resolved from chlorobenzene and the latter can be chosen. This may be manually integrated using a drop to baseline method.

4. Interference -Interference with the target ion may require manual integration. E.g. the surrogate 1,4 bromofluorobenzene is often interfered by fuel hydrocarbons.

5. Noise in the baseline - Noise in the baseline may add area to compounds which may incorectly increase the amount detected. This is more often a problem at the lower levels. A noisy baseline can be witnessed by several small "bumps" in the ion chromatogram preceding or following the peak of interest. If noise is evident it shall be acceptable to manually integrate from the top of the noise line instead of the "zero" baseline level.

When manual integration is necessary, it must be documented. The operator should follow the following guidelines.

Check to see how the particular compound integration is performed on the standards (ICAL,CCAL). Is it baseline, valley to valley, etc?

Manually integrate the compound in the new data file in a manner as consistent with (a) as possible.

Print copies of the computer integrated (before) and manually integrated (after) ion chromatogram showing the integration. Sign and date both QEDITs and indicate reason for manual integration.

13.5 Qualitative Analysis

For the qualitative identification of each compound the analyst will follow the guidelines in SW-846 Method 8260B Revision 2 Dec 1996 Section 7.6.

When samples are diluted for 8260B the reporting limits are raised proportionately. All multipliers must also be applied to the reporting limits as well. Dilutions are recorded on the daily run logs and are entered into the HP chemstation software for the particular analysis.

The calibration range defines the reporting range for analytes. Any analytes reported below the lowest point of the calibration curve or above the highest point of the calibration curve will be flagged with either a "J" or an "E", as appropriate.

13.6 Quantitative Analysis

Quantitative results are based upon a qualitative determination of analyte presence and a quantitative determination of analyte concentration. The software will identify an analyte hits based upon retention time and mass assignments. It is the responsibility of the analyst to evaluate these results and determine whether the "hit" is real or a false positive. To evaluate if a peak is a real hit, the analyst is to evaluate retention time and the qualifier value. If the retention time is correct, there is still a possibility that the peak is not a hit. This can be determined by the mass ratios and if they are close to what is expected. It is therefore imperative that the analyst updates the retention times and mass assignments when ever they calibrate. It is also possible to obtain a false negatives. This occurs on very large peaks where the concentration of the analyte is so high that the software cannot identify it. It is suggested that the analyst investigate the identity of any large peaks on the chromatogram.

14.0 Computer Hardware and Software

Agilent Technologies (HP) enviroquant software running on Pentium (or equivalent) PC's under Windows XP operating system is the data acquisition system. Chromatograms and quantitation reports are printed out on laser printers. All data and methods are to be backed up on a bi-annual basis. The disks are to be sent to the laboratory director for safe storage.

15.0 Data Management and Records Management

Document control is the process by which the documentation associated with samples and sample data are tracked and monitored.

15.1 Chain of Custody Record

A Chain of Custody Record serves to note the disposition of the samples, from the time of collection to laboratory receipt. This form should be filled out at the time of collection and signed by all persons collecting, relinquishing and receiving samples. All samples must be documented on an appropriate Chain of Custody form.

15.2 Sample Run Logs

H&P Mobile Geochemistry maintains a Sample Run Log for all samples analyzed. These logs are kept within a logbook, one of which is located in each van. A new log is prepared for each work day. The following information is included on the run logs:

Date of operation
H&P Mobile Geochemistry job number
Operator name
Instrument No.
Analysis type
Calibration Standard identification
Sample Analysis data

Also located in each van are instrument and excursion logs. The instrument log is used to document problems and subsequent corrective action for the instrumentation (excessive column bleed, baseline irregularities, interference, etc.). The excursion log documents where and when the particular job takes place, the type of analysis, and operating conditions (weather, temperature, etc.). The

excursion log should be filled out for each new job**.** If the analyst is collecting soil vapor samples then a sample collection log sheet is also to completed by hand.

15.3 Reporting

The Data Package

The analyst will prepare a data package for Reporting and Review. For EPA Method 8260B the package will be produced on a daily basis corresponding to the 12-hour window of operations. The package will consist of the following in the order listed or as near as possible to that order:

a) The Original COCs for the samples analyzed during that day
b) The daily run logs
c) The BFB tuning report
d) Edited Quantitation reports from the daily initial Calibration (ICAL) or the passing Continuing Calibration Verification (CCV)
e) The Response factor summary (if ICAL) or the Evaluation of CCV (if daily CCV)
f) Edited quantitation report from the Initial Calibration verification (ICV) (If ICAL)
g) ICV summary, if done showing all compounds <20%D
h) Edited quantitation report from the LCS
i) LCS Summary showing percent recovery and control limits
j) Edited quantitation report from a blank analysis
k) Edited quantitation reports for all analyses.
l) For mobile lab jobs, the confirmation sheet and the analyte list is to be included**.**

In addition to the above daily package, other data pertinent to the project should be supplied to the data entry person(s) and the reviewer. Such project specific information as out of control events and associated recommendations for qualifier flags, corrective actions, and LOD and LOQ studies should be added to the above package if required by project DQOs.

The analyst will assure that the data is in the proper format above before passing it on for data entry or review.

Report Review

Data Review will consist of a third party review of the data generated during this project. The data reviewer will

1. Folder
   Project Number
   Project Name (site)
   Project date

2. Accounting Information
   Lab confirmation form
   Report to information
   Billing to information

3. Print report on plain paper

4. From Chain of Custody Record confirm:
   a. Project information (client, project No. etc.)
   b. Correct dates

c. Special instructions or out of control items
d. Does the COC sample names match the report.
e. Check the COC for the following items:
   a) Unique sample identifier for all samples
   b) Date and time of sampling
   c) Source of sample including name, location and sample type
   d) Analyses required
   e) Name of collector
   f) Pertinent field data
   g) Custody transfer signatures and dates and times of sample transfer from the field to transporter and the lab
   kh Bill of lading or transporter tracking number (if applicable)

5. From Raw Data:
   Organize raw data in order of date
   Go through the raw data page by page
   Date - is the correct date analyzed listed
   BFB tuning – does it pass?
   Blank - is it clean
   ICAL – does it meet project QC requirements (if applicable)
   ICV - does it meet project QC requirements?
   CCAL - is it acceptable under project QC
   LCS – within specifications
   Sample - identify each individual set of data

6. Using the final report forms, check that the raw data from above is correctly entered in the proper places on the forms. Apply data qualifier flags to the data as necessary according to project requirements.

7. Edits
   With edited copy of rough draft
   Page by page make all changed marked in red
   Print out on letterhead
   Compare final to rough draft

8. Mail
   Prepare the report; COC, chromatograms (if needed)
   Make a complete copy for our file

<u>Final Report</u>

The following information should be included in each H&P Mobile Geochemistry report on H&P Mobile Geochemistry letterhead:

client (name and address)
client job number / project name
site location (location of analysis)
analyte list with reporting limits
dates samples received
dates samples collected
dates samples analyzed
analyst

sample results in table form titled Report Summary
sample identification number
units reported (mg/kg or ug/l)
amount found and units
QA/QC report for each method used(including deviations)
unique laboratory project ID number
identification of flagged data (including data outside quantitation levels)
chain of custody (including sample dates and time)
cover letter (including signature and title page)
a brief description of the test method
narrative (if needed)
a description of any unusual or notable events or conclusions, including samples that did not meet sample acceptance requirements

Any analytical testing provided by a subcontractor will be reported by the subcontractor on their letterhead, separate from the H&P Mobile Geochemistry report.

After the final laboratory report is issued, the report will remain unchanged. Any amendments to the report will be made as separate reports and will include a statement of amendment or supplement to the original report. H&P Mobile Geochemistry will notify the client promptly in writing of any defective measuring or validity problems with data.

H&P Mobile Geochemistry takes the necessary steps to ensure the confidentiality of its report, by providing data only to the client by phone, fax, e-mail or mail.

H&P Mobile Geochemistry shall certify that the test results meet all the requirements of this SOP or shall provide a reasonable explanation as to why they do not.

16.0 Quality Assurance and Quality Control

Quality Assurance (QA) and Quality Control (QC) are a set of procedures and conditions designed to maximize the precision and accuracy of the analytical process. The procedures are also designed to maximize the precision and accuracy of the analytical process. QA/QC is a continuous process requiring verification by inspection and, if necessary, appropriate corrective action. Listed below are key items used to ensure proper QA/QC. A brief description of each term is included, along with H&P Mobile Geochemistry and regulatory requirements.

16.1 Initial Calibration (ICAL)

Prior to performing sample analysis, the GC must be initially calibrated. Following a clean blank analysis, at least five levels of target compounds along with surrogates and internal standards are introduced into the GC/MS system and the total area under each peak determined. These values are entered into the software. The computer will construct the calibration curve according to one of several methods. Hardcopy output of the chromatograms should be saved and kept with the instrument throughout the lifetime of the ICAL. The hardcopy output should list the method used to generate the ICAL curve. The ICAL is considered valid until the continuing calibration fails or a major change in the instrument operating condition occurs.

16.2 Tune/Continuing Calibration (CCAL)

The BFB tune as outlined in section 8.2 must be performed prior to analysis of any standards and repeated every 12 hours if samples or standards are to be analyzed. The calibration of the instrument is checked prior to analyzing samples and repeated every 12 hours if samples or standards are to be analyzed. The continuing calibration (CCAL) or continuing calibration verification (CCV) uses a second source of the standard to check the validity of the ICAL. Normally a standard corresponding to the midpoint of the curve is chosen. Response of the compounds of interest must be within that specified by the method for the continuing calibration check compounds (CCCs) for the curve to be valid. If not all compounds are of interest only those being analyzed need to be checked.

In the case of a non-compliant CCV, corrective action is to be taken. The CCV is to be reanalyzed and if the second CCV is acceptable, analysis may proceed. If the 2nd CCV is not acceptable, the laboratory director or QA Officer is to be contacted for direction.

If samples are analyzed in conjunction with an unacceptable CCV, all sample should be reanalyzed, if possible, unless the CCV is biased high with all affected analytes resulting in non-detects. Otherwise, the client should be notified and the data appropriately flagged. See QSM Appendix 8F for a list of acceptable qualifiers

16.4 Blanks

A Method Blank is an analyte free matrix sample, run through the sample preparation and analysis procedures of those used for samples. These blanks check the levels of contamination for the method. Blanks must be run at a frequency of one per batch of samples or preparation method. If a contaminate is found the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problems if:

a) the target analytes in a reagent blank are higher than the reporting limit (dilution factors taken into account) in effect at the time

b) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated blank sample batch or the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

16.5 Lab Control Sample

For soil vapor samples, a single LCS is required at the end of the day.

16.6 Percent Recovery Calculation

The values for the LCS are used to calculate the Percent Recovery (PR), an estimate of the accuracy of the analytical process. The Average PR can be calculated using the following general equation:

$$\text{Average PR} = \text{LCS}/\text{SA} \times 100$$

where: PR = Percent Recovery
LCS = LCS Result
SA = Spiked Amount

H&P Mobile Geochemistry calculates the PR for each LCS and the resultant value is included in the QA/QC section of the report.

16.7 Laboratory Control Limits

Each certified laboratory establishes their own set of Laboratory Control Limits (LCL's). These limits include criteria such as recovery ranges and maximum allowable RPD's. H&P Mobile Geochemistry has established the following preliminary list of control limits for EPA Method 8260B from the method:

Soil Vapor
All DTSC analytes are to fall with 30% recovery of the true value.

Surrogates

4-Bromofluorobenzene	75-125
dibromofluoromethane	75-125
toluene	75-125
1,2-Dichloroethane-d4	75-125

Samples whose surrogate exceeds the limits are re-analyzed one time. Only 3 surrogates are required and the analyst may choose to drop one if the samples are shown to have interference. Once interference is confirmed, samples may be flagged as such and the analyst need not rerun other samples from the site.

17.0 Detection Limits

17.1 Minimum Detection Limit

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these MDLs at least once per twelve-month period. The laboratory shall maintain proof of the MDL demonstrations (i.e., before project samples are analyzed) and upon request in the format specified in Section 8.

Laboratories participating in this work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the following instructions:

(1) Estimate the MDL using one of the following:
   a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5, or
   b) The concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in reagent water **(Limit of Blank (LOB),** or
   c) The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

(2) Prepare and analyze seven samples containing the analyte of interest at a concentration three to five times the estimated MDL.

(3) Determine the variance (S2) for each analyte as follows:

$$S2 = \frac{1}{n-1}\left[\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2\right]$$

where xi = **is** the measurement of the variable x and

$\bar{x}$ = the average value of x

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

(4) Determine the standard deviation (s) for each analyte as follows:

s = (S2)1/2

(5) Determine the MDL for each analyte as follows:

MDL = 3.14(s)

(note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

(6) If the spike level used in step 2 is more than 10 times the calculated MDL, repeat the process using a smaller spiking level. If the calculated MDL is more than the spike level concentration, repeat the process using a higher spiking level. If the calculated MDL is less than 10% of the spikes level concentration, repeat the process using a lower spiking level.

Where multiple instruments are used, the MDL used for reporting purposes shall represent the least sensitive instrument.

17.2 Limit of Detection

The limit of detection (LOD) is the lowest analyte concentration that can be reliably distinguished from baseline noise. The LOD is to be reported in lieu of the MDL for DoD projects that require “J” flagging. The LOD is matrix dependant and must be verified for each analytical instrument on a quarterly basis.

17.3 Reporting Limit and Limit of Quantitation (LOQ)

The normal reporting limits are nominally 1-10 ng of compound on column. This corresponds to 0.05 to 0.5 ug/L for 20 cc vapor sample. These limits should be compared annually to the MDL's to make certain that they are appropriate. LOD's and LOQ's are to be evaluated quarterly to ensure that bias and precision are acceptable at the reporting limits. Depending upon client data quality objectives (DQO's) the H&P Mobile Geochemistry may report lower than these values. The reporting limits, however, should be no lower than lowest point in the calibration curve. Results reported that are between the LOD and RL (LOQ) should be flagged with the "J" flag as a quantitative estimate.

When samples are diluted for 8260B, the reporting limits are raised proportionately. All multipliers must also be applied to the reporting limits as well. Dilutions are recorded on the daily extraction/run logs and are entered into the HP chemstation software for the particular analysis.

The calibration range defines the reporting range for analytes. Any analytes reported below the lowest point of the calibration curve or above the highest point of the calibration curve will be flagged with either a "J" or an "E", as appropriate

18.0 Pollution Control and Waste Management

Soil vapor samples cannot be disposed of in the same manner as soil or water samples. They also will not have as great of an impact on the environment as do hazardous soils or waters. The chemist will, however, need to use some chemicals for use as leak check compounds. These may include, but are not limited to, 1,1 difluroethane, 1, 1,1,2,-tetrafluoroethane, hexane, and isopropyl alcohol. Care must be taken by the chemist not to spill or drop any containers containing these chemicals. The leak checks, 1,1 difluoroethane and 1,1,1,2-tetrafluoroethane, are stored in aerosol cans but are freons which are harmful to the environment. These should be used in limited quantities to minimize their environmental impact. If a chemist should spill any hexane or isopropyl alcohol, proper cleanup measures are to be used to properly collect and dispose of the spill.

19.0 References & Definitions

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), U.S.E.P.A including Update III, December 1996

Quality Systems Manual, H&P Mobile Geochemistry, Inc. most recent version

For Definitions refer to H&P mobile Geochemistry, Inc. Quality Systems Manual Appendix 1

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EPA Method TO-15
Standard List VOCs

Compound	***400mL RLs*** *Vapor (µg/m³)*	***400mL MDL*** *Vapor (µg/m³)*
1,1,1,2-Tetrachloroethane	7.0	1.19
1,1,1-Trichloroethane	5.5	1.27
1,1,2,2-Tetrachloroethane	7.0	0.77
1,1,2-Trichloroethane	5.5	1.49
1,1,2-Trichlorotrifluoroethane (F113)	7.7	0.70
1,1-Dichloroethane	4.0	0.74
1,1-Dichloroethene	4.0	1.65
1,2,4-Trichlorobenzene	7.5	1.35
1,2,4-Trimethylbenzene	5.0	1.15
1,2-Dibromoethane (EDB)	7.8	0.86
1,2-Dichlorobenzene	12.2	2.08
1,2-Dichloroethane (EDC)	4.1	0.91
1,2-Dichloropropane	9.4	1.74
1,3,5-Trimethylbenzene	5.0	1.30
1,3-Dichlorobenzene	12.2	2.14
1,4-Dichlorobenzene	12.2	2.32
2-Butanone (MEK)	29.9	3.59
2-Hexanone (MBK)	8.3	1.46
4-Ethyltoluene	5.0	1.30
4-Methyl-2-pentanone (MIBK)	8.4	0.46
Acetone	24.1	8.27
Benzene	3.2	0.71
Bromodichloromethane	6.8	0.95
Bromoform	10.5	5.26
Bromomethane	15.8	4.58
Carbon disulfide	6.3	0.98
Carbon tetrachloride	6.4	1.28
Chlorobenzene	4.7	0.56
Chloroethane	8.1	2.28
Chloroform	5.0	0.64
Chloromethane	2.1	0.31
cis-1,2-Dichloroethene	4.0	1.25
cis-1,3-Dichloropropene	4.6	0.88
Dibromochloromethane	8.7	0.78
Dichlorodifluoromethane (F12)	5.0	0.65
Dichlorotetrafluoroethane (F114)	7.1	1.13
Ethylbenzene	4.4	0.93
Hexachlorobutadiene	10.7	1.61
m,p-Xylene	8.8	0.66
Methylene chloride (Dichloromethane)	3.5	0.81
o-Xylene	4.4	1.01

Continued on page 2…

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Continued from page 1…	***400mL RLs*** *Vapor (µg/m³)*	***400mL MDL*** *Vapor (µg/m³)*
Styrene	4.3	0.86
Tetrachloroethene	6.9	0.90
Toluene	3.8	0.61
trans-1,2-Dichloroethene	8.1	1.65
trans-1,3-Dichloropropene	4.6	1.52
Trichloroethene	5.5	0.76
Trichlorofluoromethane (F11)	5.7	1.07
Vinyl chloride	2.6	0.47
Additional Compounds by TO-15		
Naphthalene	5.3	0.80
Leak Check Compound		
1,1-Difluoroethane (LCC)	10 µg/L	

ATTACHMENT F

Attachment F
Project Team Contact List

Project Team Member	Organization	Address	Phone Number	Email Address
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ATTACHMENT G

ATTACHMENT G

5.2 PROJECT HAZARD ANALYSIS

This section provides information regarding potential hazards that might be encountered during field activities and the risk(s) associated with each hazard. Projects are identified by activity numbers as follows:

Activity Number	Job Project
1	Soil Gas Probe installation/Construction
2	Soil Gas Sampling
3	Soil Gas Implant Removal/Abandonment
4	Indoor Air Sampling
N/A	Not applicable to this Project.

The hazard analysis evaluates the possible type of hazards at the site by activity. The analysis is presented in Table 3.

5.3 HAZARD MITIGATION

Procedures that will be used to minimize hazards identified onsite are listed in this section.

Hazards		Procedures to Mitigate Hazards
Physical		
	1,2,3,4	Keep ground clutter in the work area to a minimum to minimize the potential for tripping.
	1,2,3,4	Use caution when the ground surface is slick or uneven.
	1,2,3,4	Clean up liquid spills or use sorbent material (e.g., dry soil) to minimize the potential for loss of footing.
	1,2,3,4	Exercise caution when using hand tools.
	1,2,3,4	Use proper lifting and reaching techniques.
	1,2,3,4	Use hand carts or ask for assistance when lifting or moving heavy loads.
	1,2,3,4	Wear safety vests and be aware of motorized traffic when working in roadways or on road shoulders.
Mechanical		
	N/A	Do not stand near backhoe buckets and earthmoving equipment.
	1,2,3	Establish communication hand signals with heavy equipment operators.

Hazards	Procedures to Mitigate Hazards
1,2,3,4	Verify daily that all equipment and associated tools are in good condition.
1,2,3	Identify the drilling rig kill switches to personnel.
N/A	Do not stand or walk under elevated loads or ladders without appropriate guarding.
1,2,3,4	Do not repair equipment while it is in operation.
1,2,3,4	Immediately remove defective equipment from the work site for subsequent repair.
1,2,3	Wear a seat belt if you are a heavy equipment operator or a motor vehicle occupant.
N/A	Do not ride on the forks of lift truck, backhoe, or front-end loader buckets, or on a load, rigging, hook, or ball.
1,2,3,4	Inspect motor vehicles and heavy equipment before operating.
1,2,3,4	Do not wear loose clothing near operating equipment. Tie back long hair.
1,2,3,4	Consult the DSHO if other mechanical hazards exist.
Trenching/Excavating	
N/A	Review OSHA regulations and CCA Safety and Health Policy and Procedure 6.2 for trenching and excavation.
N/A	Do not stand near unguarded excavations or trenches.
N/A	Do not enter excavations or trenches greater than 5-feet deep that are not properly guarded, shored, or sloped, as specified in 29 CFR 1926, Subpart P, or applicable state regulations.
N/A	Use a protection system if personnel will be entering a trench/excavation greater than 5-feet deep. Provide a description of the system.
1	Locate and mark buried utilities.
1	Locate spoils, other materials, and equipment at least 2-feet from the side of the excavation.
N/A	Ensure that daily inspections of the excavations are performed by a competent person.
N/A	Avoid using an existing wall or structure until it has been determined to be safe.
1	Barricade, cover, or backfill wells, pits, and shafts when the operation is complete.
N/A	Use diversion ditches, dikes, or other means to prevent water from entering the excavation.
N/A	Ensure that work in trenches and excavations are supervised by a qualified person (one knowledgeable in soil conditions and trenching and excavation safety).
N/A	Ensure that trenches 4-feet deep or greater have an adequate means

Hazards		Procedures to Mitigate Hazards
		of exit, such as ladders or steps, with no more than 25-feet of lateral travel.
Electrical		
	1,4	Locate and mark buried utilities before drilling or excavating.
	1,4	Utilities located by Blue Stake/The Locator (Private Locator) on ____. (To be completed by the Site Supervisor when location is complete.)
	1,2,3	Obtain permits, licenses, or right of entry required by local or state authorities.
	1	Maintain at least a 20-foot clearance (or minimum recommended by the local utility company) from overhead power lines. Contact the client for additional requirements.
	1	Contact the utility company for information regarding minimum clearance from high-voltage power lines.
	1,4	If unavoidably close to buried or overhead power lines, have power turned off, with circuit breaker locked and tagged or have the local utility company mask the wires.
	1,2,3,4	Properly ground electrical equipment. Use only three-wire grounded receptacles and extension cords.
	1,2,3,4	Do not stand in water when operating electrical equipment.
	1,2,3,4	If equipment must be connected by splicing wires, be sure connections are properly taped and that the splice is equal to the cord's quality.
	1,2,3,4	Be familiar with specific operating instructions for each piece of equipment.
	1,2,3,4	Consider all wires live until locked and tagged out.
	1,2,3,4	Use ground fault circuit interrupters (GFCIs) if operating outdoor electrical equipment.
Chemical		
	1,2,3,4	Use PPE as indicated in Section 5.4 or as directed by the SSHO.
	1,2,3,4	Conduct direct-reading air monitoring to evaluate respiratory and explosion hazards (list instrument, action level, monitoring location, and action to be taken in Section 5.5).
	1,4	Locate underground pipelines before drilling or excavating.
	1,2,3,4	Do not use spark-ignition equipment when in a flammable or combustible environment.

Hazards		Procedures to Mitigate Hazards
	1,2,3,4	Do not smoke, except in designated areas.
	1,2,3,4	Use fans to disperse airborne contaminants at the work site as necessary.
	1,2,3,4	No sparking or open flame equipment will be permitted inside the work area if there is a potential of reaching the Lower Explosive Limit (LEL) of contaminants present at the site.
	1,2,3,4	Respirators with high-efficiency particulate/air (HEPA) filters should be worn if there is a potential for contaminated dust at the site.
	1,2,3,4	Consult the DSHO or SSHO for personal air monitoring.
Temperature		
Heat stress		
	1,2,3,4	When the temperature exceeds 70 degrees Fahrenheit (°F), take frequent breaks in shaded areas. Use the buddy system.
	1,2,3,4	Unzip or remove coveralls during breaks.
	1,2,3,4	Have cool water or electrolyte replenishment solution available. Drink small amounts of cool water or electrolyte replenishment solution frequently to avoid dehydration.
	1,2,3,4	Count the pulse rate for 30 seconds as early as possible in the rest period. If the pulse rate exceeds 110 beats per minute at the beginning of the rest period, shorten the work cycle by one-third.
	1,2,3,4	If the internal body temperature exceeds 99.6°F, shorten the next work cycle by one third. Personnel wearing Modified D or Level C PPE are not permitted to continue work if their temperature meets or exceeds 100.6°F.
	1,2,3,4	Include salt in your food intake. Salt tablets are not recommended.
Cold stress		
	1,2,3,4	Wear multilayer cold-weather clothing. The outer layer should be wind-resistant fabric.
	1,2,3,4	Limit total work time in 0° to -30°F to four hours. Alternate one hour in and one hour out of the low-temperature area. Below -30°F, consult the DSHO.
	1,2,3,4	Drink warm fluid and take rest breaks in a warm shelter. Use the buddy system. Avoid heavy sweating.
Acoustical		
	1,2,3,4	Post appropriate signs in areas of hazardous noise.

Hazards	Procedures to Mitigate Hazards
1,2,3,4	Use earplugs or earmuffs when noise prevents conversation in a normal voice at a distance of 3 feet. (This is a "rule of thumb" that indicates noise levels are exceeding 85 decibels.) Double protection (e.g., earplugs and muffs) must be used for noises exceeding 104 decibels.
1,2,3,4	Contact the DSHO for a noise survey, as needed.

Confined Spaces

N/A	Confined spaces include trenches, pits, sumps, elevator shafts, tunnels, or any other area that is not designated for human occupancy, where circulation of fresh air is restricted, or where the ability to readily escape from the area is restricted. Consult the DSHO and CCA Safety and Health Policy and Procedures Manual before entering a confined space.
N/A	The SSHO will consult with the DSHO to assess whether a confined space is a permitted or nonpermitted confined space. If it is a permitted confined space, a permit must be obtained from the DSHO before entering.
N/A	Monitor oxygen (O_2), LEL, and organic vapors before entering a confined space. If the following values are exceeded, DO NOT ENTER: O_2 less than 19.5 percent or more than 23.5 percent Total hydrocarbons greater than 5 parts per million (ppm) above background, if all air contaminants are not identified Concentrations of specific contaminants exceeding action levels in Section 5.5.1 if all air contaminants are identified Flammable gases more than 10 percent of the LEL
N/A	Monitor O_2, LEL, and organic vapors continuously while inside a confined space. If Threshold Limit Values (TLVs) cited in Appendix A are exceeded, EVACUATE IMMEDIATELY. Record instrument readings.
N/A	Ensure that at least one person capable of pulling personnel from the confined space is on standby outside the confined space. The observer must be trained in cardiopulmonary resuscitation (CPR) and first aid.
N/A	Use portable fans or blowers to introduce fresh air to confined spaces whenever respirator use is required.
N/A	Do not perform work that involves the use of flame, arc, spark, or

Hazards		Procedures to Mitigate Hazards
		other source of ignition in a confined space that contains a hazardous atmosphere.
Natural		
	1,2,3,4	Wear long sleeves and/or sun block on sunny days.
	1,2,3,4	Cease field activities during severe storms. Seek shelter until the storm has passed.
	1,2,3,4	If a tornado occurs: Seek shelter in the lowest level or interior wall of a substantial building. Do not seek shelter in a trailer or vehicle. Lie flat in the nearest ditch.
	1,2,3,4	If an earthquake occurs while working outside: Stay away from buildings, trees, and power lines. If operating a motor vehicle or heavy equipment, stop immediately but stay in the vehicle or piece of equipment until the tremors have stopped.
	1,2,3,4	See Section 7.0 for further information.
Biological		
	1,2,3,4	Learn to recognize toxic plants, such as poison oak, poison ivy, and poison sumac.
	1,2,3,4	Wear long-sleeved shirts, sturdy trousers, and boots when working near toxic plants to minimize the potential of skin contact.
	1,2,3,4	If exposed to toxic plants, shower as soon as possible with a strong soap.
	1,2,3,4	Do not touch plants that have hairy leaves, milky sap, or thorny leaves, fruit, or seed pods.
	1,2,3,4	Do not approach or agitate animals, especially ones behaving strangely or foaming at the mouth.
	1,2,3,4	Avoid breathing dust in dry dessert or central valley areas (may cause valley fever).
	1,2,3,4	Use insect repellent to avoid contact with ticks, mosquitoes, and other insects (disease carriers or poisonous), as necessary. Use a solid repellent to minimize potential contamination of field samples.
	1,2,3,4	If possible, avoid contact with poisonous snakes or other reptiles by quietly walking away. If bitten, seek medical assistance immediately.
	1,2,3,4	Avoid contact with rodents because they frequently are hosts to fleas, which can carry typhus and the plague. Rodent urine may also contain spirochetes harmful to human health.
	1,2,3,4	Avoid encounters with stinging insects.

Hazards	Procedures to Mitigate Hazards
Fire/Explosion	
1,2,3,4	Use a fire extinguisher only to escape or to fight very small fires. Do not attempt to fight large fires.
1,2,3,4	Field vehicles must have a 5-pound ABC-rated fire extinguisher.
1,2,3,4	Obtain fire extinguisher use training.
NA	Obtain "hot work" permits when appropriate.
1,2,3,4	Do not smoke or operate spark-ignition equipment within 50 feet of explosive or flammable storage or where flammable liquid or vapor is present.
1,2,3,4	Do not use equipment that may generate a spark where the potential presence of explosive gas or vapor is suspected.
NA	Use a combustible gas indicator when working at a site with the potential for explosive gas or when the potential for flammable vapor exists.
NA	Use fans as an engineering control to limit buildup of explosive gas.

TABLE 1
CHEMICAL COMPOUNDS IDENTIFIED ONSITE

NOTE: Delete/add chemical(s) as needed

CHEMICAL	CONCENTRATION OF MAXIMUM DETECTION
Trichloroethene (TCE)	120,000 µg/L in Water
1,1-Dichloroethene (1,1-DCE)	500 µg/L in Water
Tetrachloroethene (PCE)	280 µg/L in Water
cis-1,2 Dichloroethene (cis-1,2-DCE)	250 µg/L in Water
1,1,1-Trichloroethane (1,1,1-TCA)	220 µg/L in Water
Vinyl Chloride (VC)	7.1 µg/L in Water
1,1-Dichloroethane (1,1-DCA)	3 µg/L in Water
Trichloroethene (TCE)	71,000 µg/m^3 in Soil Vapor
Tetrachloroethene (PCE)	19,000 µg/m^3 in Soil Vapor
1,1,1-Trichloroethane (1,1,1-TCA)	570 µg/m^3 in Soil Vapor
1,1,2-Trichlorotrifluoroethane (Freon 113)	6,100 µg/m^3 in Soil Vapor
1,1-Dichloroethene (1,1-DCE)	53,000 µg/m^3 in Soil Vapor
Chlorobenzene	120 µg/m^3 in Soil Vapor
cis-1,2-Dichloroethene (cis-1,2-DCE)	800 µg/m^3 in Soil Vapor
Chloroform	5,900 µg/m^3 in Soil Vapor

NOTES:

µg/L micrograms per liter
mg/kg milligrams per kilogram
mg/L milligrams per liter
ppm parts per million
All chemical concentrations were detected in groundwater samples.

TABLE 2
HAZARD ANALYSIS

Job Project by Activity Number

Hazards	**Activity 1**	**Activity 2**	**Activity 3**	**Activity 4**
Physical	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting	hand tools slip/trip/fall lifting
Mechanical	heavy equipment	N/A	heavy equipment	Hand tools
Trenching/Excavating	N/A	N/A	N/A	N/A
Electrical	Buried underground utilities	N/A	N/A	Buried underground utilities
Chemical	chemicals in vapors, dust and water	chemicals in vapor, dust and water	chemicals in vapor, dust and water	chemicals in vapor, dust and water
Asbestos	N/A	N/A	N/A	N/A
Temperature	heat stress, cold stress	heat stress, cold stress	heat stress, cold stress	heat stress, cold stress
Acoustical	high noise levels	high noise levels	high noise levels	high noise levels
Confined Spaces	N/A	N/A	N/A	N/A
Natural	high winds, weather	high winds, weather	high winds, weather	N/A
Biohazards	plants, insects, animals, reptiles	plants, insects, animals, reptiles	plants, insects, animals, reptiles	insects, animals, reptiles
Fire/Explosion	flammable chemicals released to air	flammable chemicals released to air	flammable chemicals released to air	N/A

N/A Not Applicable
Activity 1 Drilling and Soil Gas Sampling
Activity 2 Soil Gas Sampling
Activity 3 Soil Gas Implant Removal/Abandonment
Activity 4 Indoor Air Sampling

TABLE 3
RISK ASSESSMENT

	Hazard	Exposure[a]	Probability[b]	Consequence[c]
Physical	hand tools	frequent	unusual	minor to serious
	slip/trip/fall	continuous	likely	minor to fatal
	lifting	frequent	unusual	minor to moderate
Mechanical	heavy equipment	continuous	likely	minor to fatal
Trenching/Excavation	N/A	N/A	N/A	N/A
Electrical	power lines	frequent	improbable to likely	moderate to fatal
	generator	frequent	improbable to likely	moderate to fatal
Chemical	chemicals in air	continuous	unusual	minor to chronic
	chemicals in water	continuous	improbable	minor to chronic
	dust	continuous	unusual	minor to chronic
Asbestos	N/A	N/A	N/A	N/A
Temperature	heat stress	continuous	likely	minor to fatal
	cold stress	continuous	likely	minor to fatal
Acoustical	drilling rig,	continuous	unusual	chronic
	generator	continous	likely	minor to chronic
Confined Spaces	N/A	N/A	N/A	N/A
Natural	sunlight	continuous	likely	minor to serious
	storms	occasional	unusual	minor to fatal
Biohazards	plants	seldom	improbable	minor to moderate
	insects	continuous	likely	minor to fatal
	animals	seldom	unusual	minor to fatal
	reptiles	seldom	unusual	minor to fatal
Fire/Explosion	flammable chemicals released to air	frequent	unusual	minor to fatal

N/A Not Applicable

[a] Exposure: the frequency of exposure to the hazardous event
- Continuous = many times daily
- Frequent = once/day or twice/day
- Occasional = once/week to once/month
- Seldom = once/month to once/year

TABLE 3
RISK ASSESSMENT

[b] Probability: the likelihood that an injury will occur after exposure to the hazardous event
- Certain = certain or almost certain
- Likely = not unusual, 50 percent chance of occurring
- Unusual = would happen less often than not
- Improbable = not likely to happen

[c] Consequence: the degree of injury resulting from exposure to the hazardous event if injury occurs
- Fatal = fatality
- Serious = serious injury, including chemical exposure requiring hospitalization
- Moderate = moderate injury, including chemical exposure requiring outpatient medical treatment
- Minor = minor injury, including chemical exposure requiring onsite first aid
- Chronic = chemical acoustical, or other exposure above the Threshold Limit Value (TLV) or other recommended standard that may not produce immediate acute effects (especially chronic toxicants)

TABLE 4
TASK-SPECIFIC PERSONAL PROTECTIVE EQUIPMENT

Level D	Modified D	Level C
Head Hardhat	**Head** Hardhat	**Head** Hardhat
Eye/Face Safety glasses Chemical goggles Face shield	**Eye/Face** Safety glasses Chemical goggles Face shield	**Eye/Face** Safety glasses Chemical goggles Face shield
Ear Ear plugs Earmuffs	**Ear** Ear plugs Earmuffs	**Ear** Ear plugs Earmuffs
Hand (Underglove) N-Dex® Thermax®	**Hand (Underglove)** N-Dex® Thermax®	**Hand (Underglove)** N-Dex® Thermax®
Body Rainsuit Cloth coveralls	**Hand (Outerglove)** Nitrile Neoprene Butyl PVC Viton	**Hand (Outerglove)** Nitrile Neoprene Butyl PVC Viton SilverShield®
Foot STCR boot	**Body** Tyvek® Polycoated Tyvek®	**Body** Tyvek® Polycoated Tyvek® Saranex®
	Foot STCR boot Overboot	**Foot** STCR boot Overboot
		Face/respiratory Full-face PAPR Full-face respirator Half-face respirator Cartridges (specify type)

PAPR Powered Air Purifying Respirator
PVC Polyvinyl chloride
SCBA Self-contained breathing apparatus
STCR Steel-toed, chemical-resistant

* With 5 minute egress bottle.

TYPE OF MONITORING			
Volatile	**Explosimeter**	**Personal**	**Miscellaneous**
PID with 10.2 eV lamp	N/A	N/A	Heat stress meter Thermometer

eV Electron volts
PID Photoionization detector