SITE MANAGEMENT PLAN

277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

Prepared For:
US Environmental Protection Agency
75 Hawthorne Street
San Francisco, CA 94105

Prepared by:
ENGEO Incorporated

May 5, 2016

Project No:
10216.001.000
Phase 002, Task 002

Copyright © 2016 By ENGEO Incorporated. This Document May Not Be Reproduced In Whole Or In Part By Any Means Whatsoever, Nor May It Be Quoted Or Excerpted Without The Express Written Consent Of ENGEO Incorporated.

Project No.
10216.001.000
Phase 002, Task 002

May 5, 2016

Ms. Alana Lee
Project Manager, Superfund Division
US Environmental Protection Agency, Region 9
75 Hawthorne Street
San Francisco, CA, 94105

Subject: 277 Fairchild Drive, 228/236 Evandale Avenue
Mountain View, California

SITE MANAGEMENT PLAN

Dear Ms. Lee:

As requested, ENGEO has prepared this Site Management Plan (SMP) for the subject property (Property) in Mountain View, California. This SMP provides details regarding soil management, vapor emissions and air monitoring, groundwater management, and other management procedures for the proposed construction activities at the Property.

If you have any questions or comments regarding this report, please call and we will be glad to discuss them with you.

Sincerely,

ENGEO Incorporated

REGISTERED PROFESSIONAL ENGINEER
DIVYA BHARGAVA
No. 82024
CIVIL
STATE OF CALIFORNIA

Divya Bhargava, PE
db/sm/dt

CERTIFIED HYDROGEOLOGIST
SHAWN MUNGER
No. HG 413
CERTIFIED HYDROGEOLOGIST
STATE OF CALIFORNIA

Shawn Munger, CHG

TABLE OF CONTENTS

Letter of Transmittal

Page

1.0 INTRODUCTION 1

1.1 SITE LOCATION AND DESCRIPTION 1
1.2 SITE HISTORY 1
1.3 PROPOSED DEVELOPMENT 2

2.0 SITE MANAGEMENT PLAN OVERVIEW 3

2.1 PURPOSE 3
2.2 OVERVIEW OF REQUIREMENTS 3
2.3 SEQUENCE OF WORK 5
2.4 CONSTRUCTION ACTIVITIES SCHEDULE 6
2.5 CONTACT INFORMATION 6

3.0 VAPOR EMISSIONS MONITORING AND CONTROL 8

3.1 BACKGROUND 8
3.2 WORK OPERATIONS 8
3.2.1 Reporting 8
3.2.2 Environmental Infrastructure Protection 8
3.3 EPA REGION 9 GUIDANCE ON TCE EXPOSURE LIMITS 9
3.4 OVERVIEW OF VAPOR MONITORING PLAN 10
3.5 REAL-TIME SCREENING IN WORKER BREATHING ZONE 10
3.5.1 Real-Time Screening Equipment 10
3.5.2 Real-Time Screening Procedures 11
3.6 PERIMETER AMBIENT AIR MONITORING 11
3.7 CONSTRUCTION RESPONSE ACTIONS 12
3.7.1 Trench Worker Response Actions 12
3.7.2 Perimeter Air Monitoring Response Actions 13

4.0 SOIL MANAGEMENT 14

4.1 OVERVIEW 14
4.2 VISUAL OBSERVATION AND PID SCREENING DURING EXCAVATION WORK 14
4.3 SOIL SAMPLING AND LABORATORY TESTING 14
4.4 STOCKPILE MANAGEMENT 15
4.5 SOIL DISPOSAL 15

5.0 GROUNDWATER MANAGEMENT 16

6.0 OTHER SITE MANAGEMENT PROCEDURES 16

6.1 HEALTH AND SAFETY PLAN 16
6.2 MEETINGS AND COMMUNICATION 17

TABLE OF CONTENTS (Continued)

6.3 DUST CONTROL.....17
6.4 STORMWATER MANAGEMENT PLAN.....18
6.5 FINAL REPORT 18

ACRONYMS
FIGURES
APPENDIX A – Dust Control Plan
APPENDIX B – Sampling and Analysis Plan
APPENDIX C – Quality Assurance Project Plan (QAPP)
APPENDIX D – Standard Operating Procedures (SOPs): Field Monitoring of VOCs by Modified Method TO-14
APPENDIX E – Health and Safety Plan

ACRONYMS

BMP	Best Management Practices
COC	Chain-of-Custody
DTSC	California Department of Toxic Substances Control
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSP	Health and Safety Plan
MEW	Middlefield-Ellis-Whisman
PID	Photoionization detector
PPE	Personal Protective Equipment
QA/QC	Quality Assurance/Quality Control
SAP	Sampling and Analysis Plan
SMP	Site Management Plan
TCE	Trichloroethylene
EPA	United States Environmental Protection Agency
VOC	Volatile Organic Compounds

1.0 INTRODUCTION

This Site Management Plan (SMP) has been prepared by ENGEO on behalf of our client, Warmington Residential California for the proposed construction activities associated with the 1.6-acre Property located at 277 Fairchild Drive and 228/236 Evandale, in Mountain View, California (Figure 1). This SMP has been prepared to comply with requirements of the United States Environmental Protection Agency (EPA) and the City of Mountain View (City).

1.1 SITE LOCATION AND DESCRIPTION

The Property is located at 277 Fairchild Drive and 228/236 Evandale Avenue in Mountain View, California. The approximately 1.6-acre Property consists of Assessor's Parcel Numbers (APN) 160-07-011, 160-07-012, and 160-07-013 (Figures 1 and 2). A vacant motel and mini-mart currently occupy the Property with approximately 25 vacant single-story units located along the western and eastern edges of the Property.

1.2 SITE HISTORY

Review of historical records found that the Property was used for growing wheat or hay prior to 1939 and buildings were first constructed at the Property between 1939 and 1948.

The Middlefield-Ellis-Whisman regional groundwater plume extends beneath the Property. Three groundwater monitoring wells and conveyance piping for an offsite groundwater extraction system are located near Fairchild Drive, toward the northern boundary of the Property. The wells are sampled to monitor concentrations of trichloroethene (TCE) in groundwater at various depths. In addition, an operational groundwater extraction well is situated in the northern area of the Property, along Fairchild Drive.

Volatile organic compounds (VOCs), primarily TCE, have been detected in groundwater beneath the Property. Figure 5 provides the most recent groundwater concentrations for the subject Property. As presented on Figure 5, grab groundwater collected from the Property exhibited TCE concentrations up to 7,300 micrograms per liter (ug/L).Concentrations of TCE up to 130,000 ug/L have been observed south of the Property, along Evandale Avenue.

Elevated levels of VOCs in soil vapor have also been detected at the Property. TCE has been detected at a maximum concentration of 49,000 micrograms per cubic meter ($\mu g/m^3$) in a soil gas sample collected from the central portion of the Property. Figure 6 presents the most recent TCE concentrations in soil gas at the Property and in the vicinity of the Property.

The MEW Superfund Study Area (or MEW Site) is comprised of three National Priorities List (NPL) or Superfund sites: Fairchild Semiconductor Corp. – Mountain View Superfund site; Raytheon Company Superfund site; and Intel Corp. – Mountain View Superfund site; several other facilities; and portions of the former Naval Air Station (NAS) Moffett Field Superfund site. The "MEW Superfund Study Area" itself is not listed on the National Priorities List (NPL).

The primary chemicals of concern at the MEW Site are TCE and its degradation products, cis-1,2-dichloroethene and vinyl chloride. During operations, some of the chemicals leaked or were otherwise released to the ground, impacting soil, soil vapor, and groundwater.

We reviewed the 2014 Annual Summary Report prepared by Geosyntec for the MEW groundwater remediation/monitoring. Specifically, we reviewed the most current data for the onsite extraction and groundwater monitoring wells. The following table summarizes the relevant data for the three permanent monitoring wells and one extraction well.

Existing Wells

Well	Depth (ft)	Aquifer	Screen Interval	Gw Depth (ft)	TCE (uG/L)[1]
79A	24	Upper	13 to 23 feet	11.71	52 ug/L
12B1	72	Intermediate	25 and 75 feet	12.01	2000 ug/L
43B2	94	Lower	75 to 100 feet	9.12	<0.50 ug/L
REG-3B[2]	75	Intermediate	57 to 72 feet	19.43	140 ug/L

Geosyntec performed a pilot in-situ chemical oxidation (ISCO) program in November 2013. As part of the ISCO program, nine wells were installed in the southern portion of the Property (see Table II). Based on our review of the Pilot Study Implementation Report, Evandale Avenue Sources, dated February 27. 2015, while the ISCO program appeared effective, further ISCO remediation was not feasible due to logistical constraints, including utilities, limited site access, traffic and other issues. We understand that EPA is likely to require one or more additional extraction wells to be installed along Evandale Avenue, or possibly within the southern portion of the Property. The EPA may also require additional monitoring wells to address the secondary Evandale Avenue TCE "hot spot."

The existing three, long-term monitoring wells and extraction system will be retained for future monitoring and operation, as required by the EPA. If necessary to accommodate the Property development plan, one or more of the long-term wells may require abandonment and re-construction. We understand that five of the nine temporary monitoring wells have been abandoned. Out of the four remaining on the Property, two of the monitoring wells will be abandoned and two the other two wells may remain to serve as monitoring stations for the additional extraction well described above. We understand the MEW consultants will be developing specific plans for the additional extraction well, conveyances, and monitoring wells.

1.3 PROPOSED DEVELOPMENT

The proposed development consists of 22 row homes and four single-family homes with appurtenant utilities, parking, and landscaping (Figure 3 and 4). Proposed residential

[1] September 2014
[2] Extraction Well

development of the Property will include the demolition/removal of existing structures, paving, and utilities. Minimal site grading will occur to achieve residential pad grades. Utility trenching, up to 6 feet in depth, will also be conducted as part of site development.

2.0 SITE MANAGEMENT PLAN OVERVIEW

2.1 PURPOSE

The purpose of this SMP is to comply with EPA and City requirements by presenting a detailed plan on how demolition and construction activities at the Property will be conducted in a manner that:

- Addresses potential worker and public exposures to potential VOCs that may be encountered during site development activities.
- Includes routine soil monitoring for contaminated soil that may be encountered and a response soil management protocol.
- Includes vapor emissions monitoring, routine air monitoring and, a response plan to protect workers and the public.
- Includes measures to control dust.

2.2 OVERVIEW OF REQUIREMENTS

As part of project approvals, EPA and the City have issued requirements that address environmental issues at the Property. The City of Mountain View Conditions of Approval (COA) includes *Mitigation Measure HAZ-2a*, which states the following:

The SMP shall include the protocols, means and methods to address the following during construction:

- *Prior to construction activities, the project applicant shall implement a Site Management Plan, including a Soil Management and Air Monitoring Plan, that establishes management practices for handling contaminated soil, soil vapor, groundwater or other materials during construction. During construction, the applicant shall coordinate work activities with U.S. EPA and the MEW responsible parties, including identifying conditions that could affect the implementation and monitoring of the vapor intrusion remedy.*
- *Site control procedures to control the flow of personnel, vehicles and materials in and out of the site.*

- *Monitoring of vapors during the removal of the existing buildings' slab and underground wastewater piping as well as any other underground features. An Environmental Professional shall be present to observe soil conditions, monitor vapors with a hand held meter, and determine if additional soil sampling should be performed. Protocols and procedures shall be presented for determining when soil sampling and analytical testing will be performed. If additional sampling is performed, a report documenting sampling activities (with site plans and analytical data) shall be provided to the City and U.S. EPA.*

- *Minimization of dust generation, storm water runoff and off-site tracking of soil.*

- *Minimization of airborne dust during demolition activities.*

- *Management of groundwater discharges during excavation dewatering, if required. Protocols shall be prepared to evaluate water quality and discharge/disposal alternatives. The pumped water shall not be used for on-site dust control or any other on-site use.*

- *Management of groundwater during long-term dewatering, if required, including protocols for extraction, treatment, and disposal of groundwater.*

- *Management of site risks during earthwork activities in areas where impacted soil, soil vapor and/or groundwater are present or suspected. Worker training requirements, health and safety measures and soil handling procedures shall be described.*

- *Decontamination to be implemented by the Contractor to reduce the potential for construction equipment and vehicles to release contaminated soil onto public roadways or other offsite transfer.*

- *Perimeter air monitoring at the site during any activity the substantially disturbs site soil (e.g., mass grading, foundation construction excavation or utility trenching). This monitoring shall be used to document the effectiveness of dust control measures.*

- *Contingency measures for previously unidentified buried structures, wells, debris, or areas of impacted soil that could be encountered during site development activities.*

- *Characterization and profiling of soil suspected of being contaminated so that appropriate disposal or reuse alternatives can be implemented. Soil in contact with groundwater shall be assumed contaminated. All soil excavated and transported from this site shall be appropriately disposed at a permitted facility.*

- *Segregation of "clean" and "impacted" soil stockpiles.*

- *Evaluation and documentation of the quality of any soil imported to the site. Soil containing chemicals exceeding residential (unrestricted use) screening levels or typical background concentrations of metals shall not be accepted.*

- *Monitoring of excavations and trenches for the potential presence of VOC vapors.*

- *Evaluation of the residual contaminants to determine if they will adversely affect the integrity of below ground utility lines and/or structures (e.g., the potential for corrosion).*

- *Measures to reduce soil vapor and groundwater migration through trench backfill and utility conduits. Such measures shall include placement of low-permeability backfill "plugs" at specified intervals on-site and at all locations where the utility trenches extend off-site. In addition, utility conduits that are placed below groundwater shall be installed with water-tight fittings to reduce the potential for groundwater to migrate into the conduits.*

- *Measures to prevent intrusion of contaminated water into stormwater control features including the stormwater detention pond. A Civil Engineer shall design the bottom and sides of the stormwater features to be lined with a minimum 10-mil3 heavy duty plastic to help prevent infiltration.*

- *Methods to mitigate the potential for vapor intrusion of VOC vapors into the planned structures.*

- *Prior to the start of any construction activity that involves below ground work (e.g., mass grading, foundation construction, excavating or utility trenching), information regarding site risk management procedures (e.g., a copy of the SMP) shall be provided to the Contractors for their review, and each Contractor shall provide such information to its Subcontractors.*

- *The project applicant's Environmental Professional shall assist in the implementation of the SMP and shall, at a minimum, perform part-time observation services during demolition, excavation, grading and trenching activities. Upon completion of construction activities, the Environmental Professional shall prepare a report documenting compliance with the SMP; this report shall be submitted to the City of Mountain View, the US EPA, and the Santa Clara County Department of Environmental Health.*

2.3 SEQUENCE OF WORK

The sequence of work at the Property is expected to be generally as follows:

- Demolition of existing above-ground structures that does not require soil disturbance.

- Demolition of at-grade and below-grade features that requires soil disturbance, such as foundations, building slabs, utilities, pavement, and trees.

- Construction of at-grade and below-grade features that requires soil disturbance, such as new building foundations and slabs, utilities, and site grading.

- Construction of above-grade structures that do not require soil disturbance.

2.4 CONSTRUCTION ACTIVITIES SCHEDULE

The following table details the assumed project construction schedule:

Task #	Task Name	Duration	Start	Finish	SMP Required
1	Demolition: Removal of existing structures and surface improvements	9 days	Wed 6/1/16	Mon 6/13/16	No
2	Demolition: Removal of existing remnant underground utilities	5 days	Tue 6/14/16	Mon 6/20/16	Yes
3	Overexavation and rough grading of the project site	7 days	Tue 6/21/16	Wed 6/29/16	Yes
4	Sewer System	10 days	Mon 7/4/16	Fri 7/15/16	Yes
5	Storm Drain System	5 days	Mon 7/18/16	Fri 7/22/16	Yes
6	Water System	7 days	Mon 7/25/16	Tue 8/2/16	Yes
7	On-site Joint Trench	12 days	Mon 8/1/16	Tue 8/16/16	Yes
8	Off-site Joint Trench	10 days	Wed 8/17/16	Tue 8/30/16	Yes
9	Prepare subgrade	2 days	Wed 8/31/16	Thu 9/1/16	No
10	Rock subgrade	4 days	Mon 9/5/16	Thu 9/8/16	No
11	Curb, gutter and Sidewalk	6 days	Mon 9/12/16	Mon 9/19/16	No
12	Pavement	2 days	Mon 9/19/16	Tue 9/20/16	No
13	Adjust utility covers	2 days	Tue 9/20/16	Wed 9/21/16	No
14	Start Foundations and Vertical Construction	TBD	Mon 9/26/16	TBD	No

This schedule may be subject to revisions, based on permitting and city requirements.

2.5 CONTACT INFORMATION

Property Owner and Consultant

Mr. Donald Babbit
Warmington Residential California
2400 Camino Ramon, Suite 234
San Ramon, CA 94583
925-249-7945
donald.babbitt@warmingtongroup.com

Mr. Shawn Munger
ENGEO
2213 Plaza Drive
Rocklin, CA 95765
916-580-6535
smunger@engeo.com

Regulatory Oversight

U.S. Environmental Protection Agency, Region 9
Alana Lee
75 Hawthorne Street
San Francisco, California 94105
415-972-3141
lee.alana@.epa.gov

City of Mountain View
Eric Anderson
Planning Division
500 Castro Street
Mountain View, California 94039
650-903-6306
Eric.Anderson2@mountainview.gov

County of Santa Clara
Jennifer Kaahaaina
Hazardous Materials Program Manager
Department of Environmental Health
1555 Berger Drive, Suite 300
San Jose, California 95112
408-918-4795
Jennifer.Kaahaaina@deh.sccgoc.org

MEW Contact and Consultants

Virgilio Cocianni
Schlumberger Technology Corporation
205 Industrial Blvd.
Sugar Land, Texas 77478
cocianni-v@slb.com

Elie Haddad
Haley & Aldrich
2107 N. 1st Street, Suite 380
San Jose, CA 95131-2028
(408) 961-4806
EHaddad@haleyaldrich.com

Eric Suchomel
Geosyntec Consultants
595 Market Street, Suite 610
San Francisco, CA 94105(510) 285-2786
ESuchomel@Geosyntec.com

3.0 VAPOR EMISSIONS MONITORING AND CONTROL

3.1 BACKGROUND

VOCs in groundwater and soil gas at the Property may result in exposure of workers to vapors, especially those working in newly excavated areas. The construction phase activities are expected to require workers in trenches or excavations. This section describes vapor monitoring for worker exposure to soil vapors and response protocols as needed for worker safety.

An environmental professional will conduct vapor monitoring at the Property during construction activities. He or she will perform the vapor monitoring activities defined in this section and will notify the construction superintendent of the need to implement any response actions.

3.2 WORK OPERATIONS

Work activities will be conducted Monday through Friday between 7:00 AM and 6:00 PM. Excavation will be performed using a combination of scrapers, backhoes, track-mounted excavators and/or loaders. The contractor will adhere to OSHA guidelines. If excavations require shoring, it will be provided by the contractor.

The planned approach is for all excavation and construction work to be performed by workers who have 40-hour HAZWOPER training per 29 CFR 1910.134, i.e., they would be working under Level D personal protective equipment (PPE). Air monitoring will be performed prior to the start of work to confirm that conditions are safe. The air monitoring would continue during work to ensure that workers will continue under safe conditions. Construction response actions will be implemented as necessary (Section 3.7).

3.2.1 Reporting

Daily field reports will be provided via e-mail to the EPA and the City of Mountain View detailing the following:

- Construction Progress
- Air Monitoring Results
- Remedial Measures
- Soil Screening/Sampling Results
- Health & Safety Procedure Summary
- SMP Compliance

3.2.2 Environmental Infrastructure Protection

There are currently five existing groundwater monitoring wells and one extraction well (Figure 5). A workplan to address a future extraction/monitoring well network will be prepared

by MEW consultants. In addition, we understand that one or more additional extraction wells and several new monitoring wells will be required on or immediately adjacent to the Property.

All appropriate measures will be undertaken during construction to protect the existing wells and conveyances, as well as future extraction/monitoring wells and conveyances. Prior to any construction activities, the existing environmental features will be visibly marked in the field and reviewed by the owner's project manager, construction superintendent, and the field monitor. The locations of the field markings will be communicated to the equipment operators on each day when activities in those areas are scheduled.

The existing five monitoring wells and the extraction well will be protected using physical measures such as bollards or monuments. Three future utility lines will be constructed beneath the existing extraction well discharge pipe, near the northeast Property corner (Figure 4). The discharge pipe lies at an elevation of approximately 36.5 feet above mean sea level (msl). The sewer, storm, and water lines will be installed beneath the discharge pipe at elevation of 33.38, 35.44, and 35.5 feet msl, respectively. The future utility trenches in this area will be hand dug, without the use of trenching equipment.

Advanced 48-hour notice will be provided to EPA and the MEW representative prior to conducting work activities that may impact any of the groundwater remedy infrastructure.

In the event any of groundwater remedy infrastructure is damaged or disturbed, the following procedures will be implemented:

- Work within the area of the damaged equipment/infrastructure will be stopped.
- The MEW PRP representative will be notified so that a field inspection can be performed.
- EPA will be verbally notified within 24 hours of the incident, with follow-up notification to EPA in writing within 48 hours.
- Follow-up notification, describing the incident and potential impacts, interim response actions taken, and schedule for follow-up actions.

3.3 EPA REGION 9 GUIDANCE ON TCE EXPOSURE LIMITS

To provide for construction worker safety for projects in the MEW area that require soil disturbance, EPA Region 9 recommends the following TCE exposure limits as guidance for short-term construction worker exposure scenarios:

Accelerated Response Level for TCE

- 8 hour work day: Not to exceed 8 µg/m3
- 10 hour work day: Not to exceed 7 µg/m3

Urgent Response Level for TCE

- 8 hour work day: Not to exceed 24 µg/m3
- 10 hour work day: Not to exceed 21 µg/m3

In the context of the EPA guidance document, the Accelerated Response Level triggers response actions to reduce levels of exposure while work continues. The Urgent Response Level triggers evacuation of workers from the area to prevent additional exposure.

3.4 OVERVIEW OF VAPOR MONITORING PLAN

The vapor emissions monitoring and response plan consists of the following:

- *Perimeter Ambient Air Monitoring:* Ambient air monitoring will be conducted at four specific Property locations on an hourly basis (Figure 7). See Section 3.6 for detailed procedures.

- *Real-Time Screening in Worker Breathing Zone:* While workers are present in the excavated areas, volatile organic compound (VOC) monitoring, speciated specifically for trichoroetheylene (TCE) will be used for real time screening to identify potential hazards and take immediate action needed to protect workers. See Section 3.5 for detailed procedures.

3.5 REAL-TIME SCREENING IN WORKER BREATHING ZONE

At any time that workers are working in an excavation, vapor monitoring will be conducted in the breathing zone of workers for the presence of VOCs using a gas chromatograph (SRI 8610) with an electron capture detector (ECD). This unit allows real-time monitoring of TCE at a detection limit at or below 0.5 ug/m3. Air samples will be recovered using 50 cc glass syringes (KDS 78-0877, or equivalent) Specifics regarding the instrumentation are provided in Appendix D. The following action levels and response protocols will be implemented for workers during soil disturbing activities:

3.5.1 Real-Time Screening Equipment

The GC-ECD monitor allows measurements specific to TCE at a minimum detection limit of 0.5 ug/m3. In accordance with manufacture's specifications, the unit will be calibrated daily.

- Fixed based laboratory verification for VOCs (TO-15)

Specific details regarding equipment calibration and fixed-based laboratory verification are provided in Appendix D.

3.5.2 Real-Time Screening Procedures

During the work day, the field monitor will record TCE readings in the breathing zone of workers on a continuous basis (at least one reading every 30 minutes) while work is underway. The following real-time screening levels are used to determine if workers can enter the area and if response actions are required.

- If the TCE readings in the work area are non-detect, workers can enter the area and no or mitigation actions are required.

- If any TCE reading in the work area is detected, but does not exceed the EPA Region 9 recommended commercial guidance levels of 7 $\mu g/m^3$, workers can continue work in the area; however, the field monitor will continue to collect TCE readings at a minimum of 15-minute intervals to determine if the elevated levels persist in the work area.

- If any TCE reading in the work area exceeds 7 $\mu g/m^3$ for two consecutive 15-minute readings, work will stop and all workers will be immediately evacuated from the area. At no time will workers occupy the area when high readings persist.

- If a TCE reading in the area exceeds 7 $\mu g/m^3$, mitigative actions will be taken to lower the TCE concentrations, as detailed in Section 3.7.

- After mitigation is implemented and TCE testing are reviewed, the field monitor, utilizing proper levels of personal protective equipment per the Health and Safety Plan, shall collect readings at a minimum of 15-minute intervals to determine if the vapor levels in the work area have been reduced or eliminated. At no time will workers occupy the area when high readings persist.

- At the end of each workday, the field monitor will ensure that all data has been secured and stored for later use.

3.6 PERIMETER AMBIENT AIR MONITORING

Ambient air monitoring will be conducted whenever:

- Soil trenching is taking place.
- Workers are working in excavated areas.
- Work is taking place at temporary storage areas for excavated soils.

The field monitor will collect ambient air TCE readings using the GC-VOC at four locations along the Property boundaries as shown on Figure 7. These locations were selected, as they are proximate to adjacent residential dwellings. Samples will be recovered before work begins and hourly throughout the day. The field monitoring equipment will not exceed 0.5 $\mu g/m^3$ as a detection limit. The perimeter air samples will be recovered using 50 cc glass syringes (KDS 78-0877, or equivalent). In addition, time-weighted daily ambient air samples will be

recovered using 1-liter summa canisters with 24-hour flow regulators. The perimeter samples will be submitted to a fixed-based laboratory for TO-15 SIM analysis. Reporting/detection limits are detailed in Appendix D. Initially, the daily perimeter samples will be analyzed on a 24-hour turnaround basis. Once the air monitoring baseline and field equipment calibration has been verified, perimeter sampling will be reduced to weekly samples with 72-hour turnaround.

Additional ambient air monitoring will also be conducted under the following circumstances:

- Work begins at a different location that was not previously monitored that day.
- Change in weather that may affect monitoring expected monitoring results.
- Change in site conditions as may be noted by visual observation or sense of smell.

For perimeter readings, a threshold of 1.0 $\mu g/m^3$ TCE, or the daily pre-construction background level, will be used as a trigger for construction response actions (Section 3.7).

The daily time-weighted ambient air analysis results will be compared to the following EPA response action levels:

- Accelerated Response Level for TCE: 2 $\mu g/m^3$. An exceedance of this threshold will trigger construction response actions (Section 3.7).

- Urgent Response Action Level for TCE: 6 $\mu g/m^3$. An exceedance of this threshold will trigger re-evaluation of construction and excavation practices.

Any exceedances will be immediately reported to the EPA verbally, via e-mail, and through daily field monitoring reports (Section 6.2).

3.7 CONSTRUCTION RESPONSE ACTIONS

There are several methods by which the levels of vapor emissions can be eliminated or reduced as described below. These methods will be implemented should the applicable thresholds be exceeded as detailed in Sections 3.3 and 3.6. Some of these response actions can be deployed routinely in advance to avoid events of vapor emissions and thus reduce lost time due to vapor emissions events. Based on each particular field circumstance, the construction superintendent and the contractor's health and safety office will decide which method to use. Tiered response actions are provided for both trench worker and perimeter exposure scenarios. Depending on field readings and field circumstances, multiple response actions may apply.

3.7.1 Trench Worker Response Actions

In the event a TCE exceedance is noted in accordance with Section 3.5.2, the following response actions will be implemented in order, depending on the results of follow-up trench atmosphere monitoring. The field monitor will collect readings at a minimum of 15-minute intervals to determine if the vapor levels in the work area have been reduced or eliminated. At no time will workers occupy the area when high readings persist.

- **Natural Ventilation:** Delay work or discontinue work in an area for a sufficient time to allow vapor levels to be reduced to acceptable levels.

- **Application of Water:** Application of water will often reduce vapor levels. Water trucks will be on site to provide water for dust control and soil conditioning for compaction and can also be used to mitigate vapor emissions.

- **Application of Foam:** Application of foams can often reduce vapor levels. For example, Rusmar Foam Technology offers several foams products for different uses and associated application equipment.

- **Higher Level PPE:** Higher levels of worker protection per the Health and Safety Plan can be implemented, such as Level C or B personal protective equipment per 29 CFR 1910.134.

- **Mechanical Ventilation:** In the event the above response actions do not result in attenuated TCE levels, mechanical ventilation using large industrial fan units will be used to ventilate trenches and direct air flow off the Property to the north towards Fairchild Drive, away from existing residential structures.

3.7.2 Perimeter Air Monitoring Response Actions

In the event a TCE exceedance is noted in accordance with Section 3.6, the following response actions will be implemented in order, depending on the results of follow-up trench atmosphere monitoring. The field monitor will collect readings at a minimum of 30-minute intervals at the specific perimeter location, to determine if the ambient TCE levels are reduced below the criteria presented in Section 3.6.

- **Natural Ventilation:** Delay work or discontinue work in an area for a sufficient time to allow vapor levels to be reduced to acceptable levels.

- **Trench Excavation Covering:** Exposed trenches exhibiting elevated TCE/VOC levels will be covered with plastic sheeting, or trench plates. Trench covering will be maintained until VOC levels are shown to have dissipated.

- **Application of Water:** Application of water will often reduce vapor levels. Water trucks will be on site to provide water for dust control and soil conditioning for compaction, and can also be used to mitigate vapor emissions.

- **Application of Foam:** Application of foams can often reduce vapor levels. For example, Rusmar Foam Technology offers several foam products for different uses and associated application equipment.

- **Higher Level PPE:** Higher levels of worker protection per the Health and Safety Plan can be implemented, such as Level C or B personal protective equipment per 29 CFR 1910.134.

- **Mechanical Ventilation:** In the event the above response actions do not result in attenuated TCE levels, mechanical ventilation using large industrial fan units will be used to ventilate trenches and direct air flow off the Property to the north towards Fairchild Drive, away from existing residential structures.

4.0 SOIL MANAGEMENT

4.1 OVERVIEW

No impacts to soil have been previously identified at the Property. The following sections establish procedures for sampling, handling, and management of impacted soil, if encountered during the construction activities at the Property.

4.2 VISUAL OBSERVATION AND PID SCREENING DURING EXCAVATION WORK

ENGEO will provide full-time observation services during demolition, excavation, and grading work at the Property. Soils encountered during construction activities will be observed for discoloration/staining or olfactory evidence of VOC impacts. In addition, the GC-VOC unit will be used to screen excavations for VOCs, as well as ambient air during excavation work. The specific locations of air monitoring will be field-adjusted based on potential access and safety limitations, but will generally be performed within the excavation areas, along with the perimeter of the excavations. In the event impacted soils are identified from visual of GC-VOC readings, soils will be screened using a photoionization detector in sealable plastic bags for presence of TCE and other VOCs. The soil screening shall be performed with a photo-ionization detector (PID) that measures down to the 10 parts per billion by volume (ppbv) range. Screening frequency shall be at the rate of at least one soil sample for every 5 cubic yards of excavated soil, or every 10 lineal feet along an excavation/trench. Excavated soil that has continuous PID readings of 500 ppbv or greater for at least 10 seconds shall be designated as "potentially contaminated soil."

4.3 SOIL SAMPLING AND LABORATORY TESTING

If impacted soils (containing elevated concentrations of TCE) are encountered and removed, the following protocols will be followed:

The trenches from which the potentially contaminated soil originated shall be tested by obtaining soil samples from the sidewalls of the trench. Soil sampling shall be performed at a minimum rate of every 10 lineal feet within the area suspected of contamination. Unless otherwise indicated by soil screening, samples shall be collected at a depth midway to the bottom of the trench.

The sampling and analysis procedures for VOCs are required to follow EPA Method 5035 and EPA Region 9's Guidelines for Sampling and Analysis of VOCs in Soils (Appendix B). The

following soil target cleanup levels (TCLs) shall be used to assess the need for further sampling and removal:

a) MEW site soil cleanup levels for TCE (500 micrograms per kilogram) and other MEW site chemicals of concern.

b) For non-VOCs and VOCs for which site cleanup levels do not exist, the most current version of EPA Region 9 Regional Screening Levels (RSLs) for residential soils or the most current version of the California Human Health Screening Levels (CHHSLs) (California EPA, 2005), whichever is more stringent (i.e., lower), with consideration for established background levels for metals.

If soil concentrations exceeding this cleanup criteria are verified by analysis, then notify EPA within 24 hours.

4.4 STOCKPILE MANAGEMENT

Impacted soils, if encountered, will be stockpiled onsite approximate 50- to 100-cubic-yard volumes. The soil stockpiles will be covered with 10-mil plastic sheeting and secured to prevent dust or runoff during storm events. All appropriate dust control and stormwater best management practices (BMPs) will be implemented during the soil mitigation activities.

The soil stockpiles will be profiled in accordance with applicable regulations and the receiving facility's requirements. The specific laboratory profile will be determined prior to excavation activities; however, it is anticipated as a minimum, the stockpile samples will be analyzed on a 10 cubic yard basis for VOCs (EPA 5035/8260B). Depending on the results of laboratory testing, the stockpiles will be transported for landfill disposal, or if appropriate, reused as engineered fill.

Upon completion of excavation work and confirmation sampling, the excavation will be backfilled with clean import fill. Import fill will be tested in general accordance with CAL-EPA DTSC requirements, prior to acceptance. Import soil will be placed in accordance with the project Geotechnical Engineer's recommendations.

4.5 SOIL DISPOSAL

Based on a review of analytical results, the following options will be considered:

- If the initial analytical testing (one per 10 cubic yards) indicates that the tested soil does not contain VOCs above applicable action levels, the soil can remain on Property.

- If VOCs are detected in soil at concentrations exceeding the screening levels, the soil will be removed from the Property and disposed at an appropriately licensed and certified disposal or treatment facility.

- All transportation will be accomplished in accordance with applicable federal, state, and local laws, including providing the necessary packaging, labeling, placarding, and manifesting. The manifests will be signed by the generator, transporter(s), and the acceptance facility. Copies of the fully executed manifests will be returned to the generator and kept on record as required by applicable law.

- All details and records will be included in the Final Report.

5.0 GROUNDWATER MANAGEMENT

Groundwater at the Property has been encountered at approximately 10 to 15 feet below ground surface. Since the maximum excavation depth is 6 feet for the sanitary sewer, no construction dewatering will be necessary.

6.0 OTHER SITE MANAGEMENT PROCEDURES

This section addresses the following additional site management topics:

- Access control
- Health and Safety Plan
- Meetings and communication
- Dust control
- Stormwater management plan
- Final Report

6.1 HEALTH AND SAFETY PLAN

All contractors will be responsible for operating in accordance with the most current requirements of State and Federal Standards for Hazardous Waste Operations and Emergency Response (Cal. Code Regs., title. 8, section 5192; 29 CFR 1910.120). Onsite personnel are responsible for operating in accordance with all applicable regulations of the Occupational Safety and Health Administration (OSHA) outlined in the State General Industry and Construction Safety Orders (Cal. Code Regs., title. 8) and Federal Construction Industry Standards (29 CFR 1910 and 29 CFR 1926), as well as other applicable federal, state and local laws and regulations. All personnel shall operate in compliance with all California OSHA requirements.

In addition, California OSHA's Construction Safety Orders (especially Cal. Code Regs., title 8, sections 1539 and 1541) will be followed as appropriate. A Health and Safety Plan (HASP) for the Property will be prepared by both the General Contractor and the Environmental Professional and submitted separately.

6.2 MEETINGS AND COMMUNICATION

The construction superintendent will facilitate coordination among all necessary parties by pre-construction meetings, daily and/or weekly progress meetings, and special meetings as may be required. In all cases, he or she shall maintain sufficient documentation as to the planned discussion and outcomes of the meetings. As necessary, the appropriate regulatory agencies will be contacted.

As indicated in Section 3.2.1, daily field reports will be provided via e-mail to the EPA and the City of Mountain View at the end of each workday, which require SMP implementation. In addition, the daily field reports will be posted to a community web site maintained by Warmington Residential. The field reports will detail the following:

- Construction Progress
- Air Monitoring Results
- Remedial Measures
- Soil Screening/Sampling Results
- Health & Safety Procedure Summary
- SMP Compliance

The daily field reports will also identify any exceedances of dust or TCE monitoring criteria and detail the mitigation measures implemented. In addition to the field reports, all real-time and TO-15 verification air sampling data for perimeter locations will be maintained daily on the community web site.

6.3 DUST CONTROL

A dust control plan is presented as Appendix A. Basic construction mitigation measures recommended by the Bay Area Air Quality Management District (BAAQMD) will be implemented to reduce fugitive dust emissions. These will likely include such activities as the following:

- A stabilized construction entrance/exit consisting of aggregate material will be provided.

- All exposed surfaces (including parking areas, staging areas, soil piles, graded areas, and unpaved access roads) will be watered two times per day.

- All haul trucks transporting soil, sand, or other loose material off-site will be covered.

- All visible mud or dirt track-out onto adjacent public roads will be removed using wet power vacuum street sweepers at least once per day.

- Limiting vehicle speed on site to minimize generation of dust.

- Minimizing drop heights for loading and unloading of materials.
- Suspending dust-producing activities during period of high wind speeds.
- Cleaning vehicle wheels by rubble strips or wheel washes to minimize track out of soil from the Property.
- Sweeping of public streets near the Property to remove dust and soil that has been tracked from the Property by vehicles.
- A publicly visible sign will be posted on the Property with the telephone number and person to contact at the lead agency regarding dust complaints. This person will respond and take corrective action within 48 hours. The BAAQMD's phone number will also be visible to ensure compliance with applicable regulations.

6.4 STORMWATER MANAGEMENT PLAN

The construction activities at the Property are subject to permitting under the National Pollutant Discharge Elimination System (NPDES) of the Federal Clean Water Act. The procedures for the "Construction SWPPP (Construction Stormwater Pollution Prevention Plan) will be implemented through the California Regional Water Quality Control Board, utilizing the General Permit provisions. The contractor will implement the SWPPP measures. A SWPPP, along with the construction sediment and erosion control plan will be prepared under separate cover.

6.5 FINAL REPORT

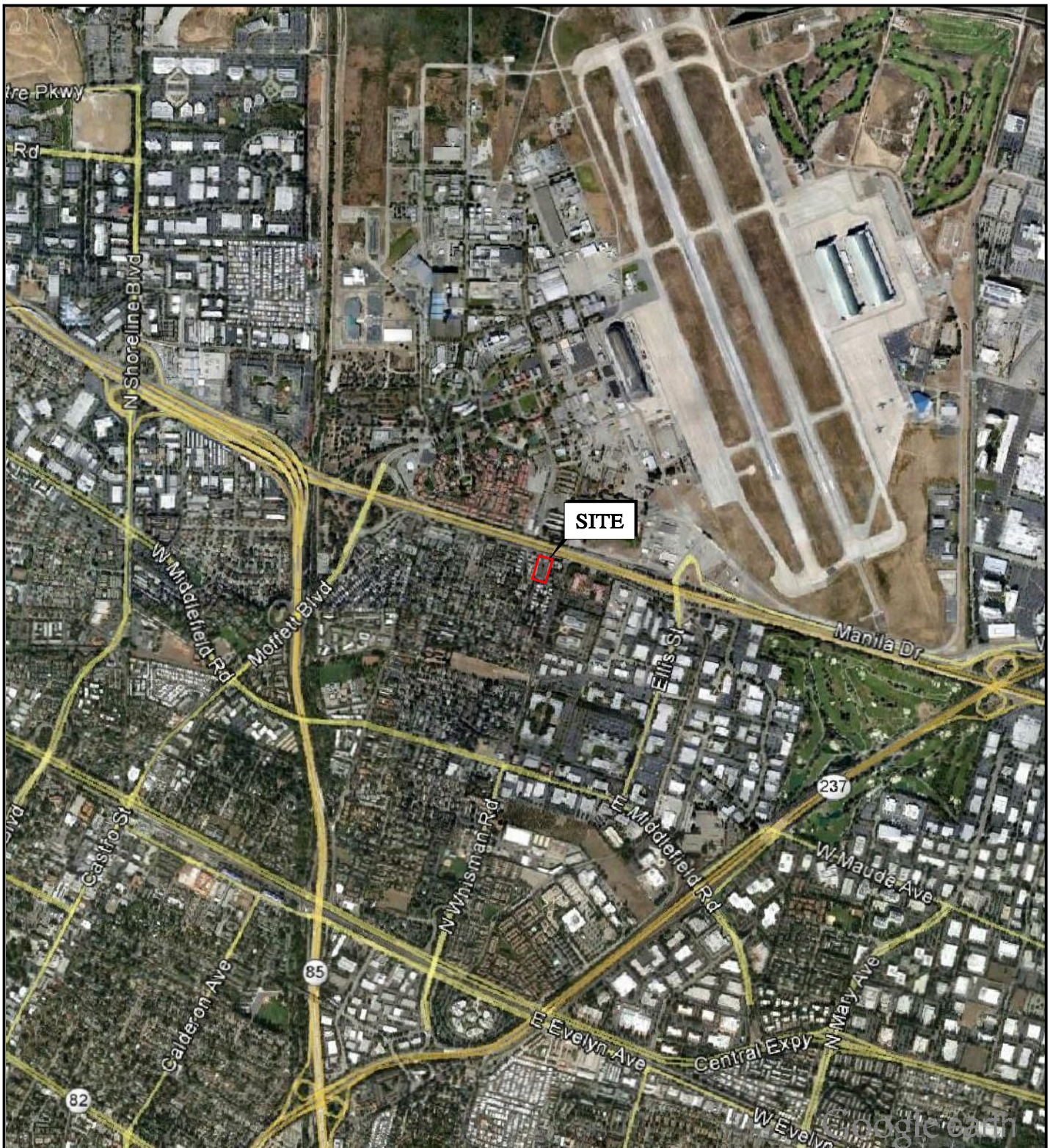
Upon completion of construction activities, a final Site Management Report will be prepared documenting field activities, sampling, stockpile management, soil mitigation, vapor monitoring, groundwater management activities, and health and safety. The Report will also include a summary of all pre-construction screening and construction phase monitoring. The Report will be submitted to EPA and the City within 60 days after completion of the SMP construction activities.

LIST OF FIGURES

Figure 1 – Vicinity Map
Figure 2 – Site Plan
Figure 3 – Proposed Development Plan
Figure 4 – Proposed Utility Plan
Figure 5 – TCE Concentrations in Groundwater
Figure 6 – TCE Concentrations in Soil Gas
Figure 7 – Proposed Air Monitoring Stations

FIGURES

COPYRIGHT © 2013 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.



N

0 FEET 2000

0 METERS 1000

BASE MAP SOURCE: GOOGLE EARTH PRO MAPPING SERVICE

ENGEO Expect Excellence

VICINITY MAP

277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE

MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.:	10216.001.000
SCALE:	AS SHOWN
DRAWN BY: DB	CHECKED BY: SM

FIGURE NO. 1

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-1-VicMap-1015.dwg Plot Date: 2-22-16 llee

ORIGINAL FIGURE PRINTED IN COLOR

© 2015 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.

N

0 FEET 50
0 METERS 25

BASE MAP SOURCE: GOOGLE EARTH PRO MAPPING SERVICE

COPYRIGHT

ENGEO
Expect Excellence

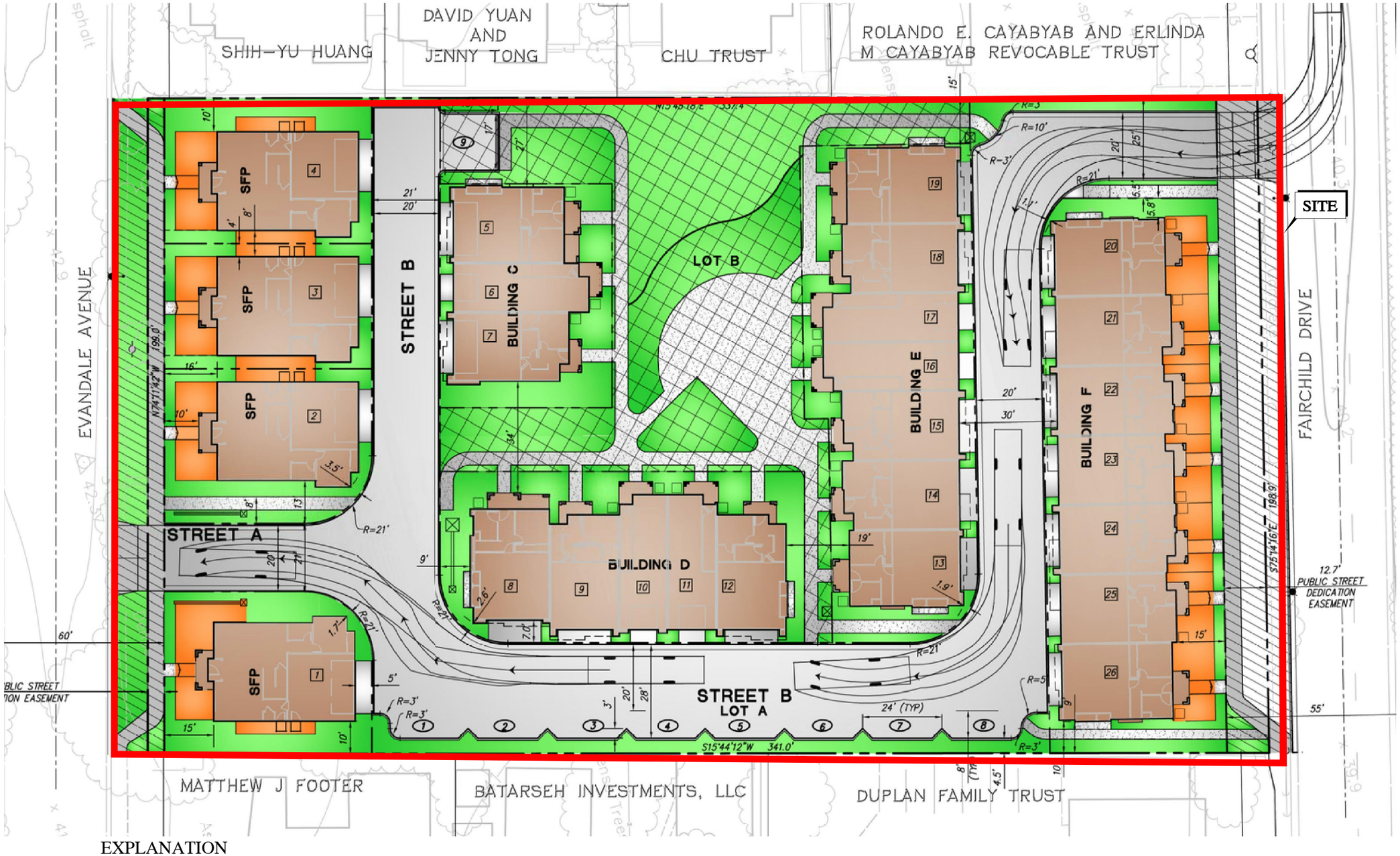
SITE MAP
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000		FIGURE NO.
SCALE: AS SHOWN		2
DRAWN BY: DB	CHECKED BY: SM	

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-2-SiteMap-1015.dwg Plot Date: 2-22-16 llee

ORIGINAL FIGURE PRINTED IN COLOR

COPYRIGHT © 2015 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.



EXPLANATION

ALL LOCATIONS ARE APPROXIMATE

- OPEN SPACE LANDSCAPING
- COMMON OPEN SPACE (LANDSCAPE/WALKWAYS)
- PRIVATE OPEN SPACE (LANDSCAPE/WALKWAYS)
- BUILDING FOOTPRINTS
- OPEN SPACE WALKWAYS
- STREETS/DRIVEWAYS
- PUBLIC SIDEWALK
- PUBLIC LANDSCAPE

N

0 FEET 30
0 METERS 15

BASE MAP SOURCE: RJA, 2015

ENGEO Expect Excellence

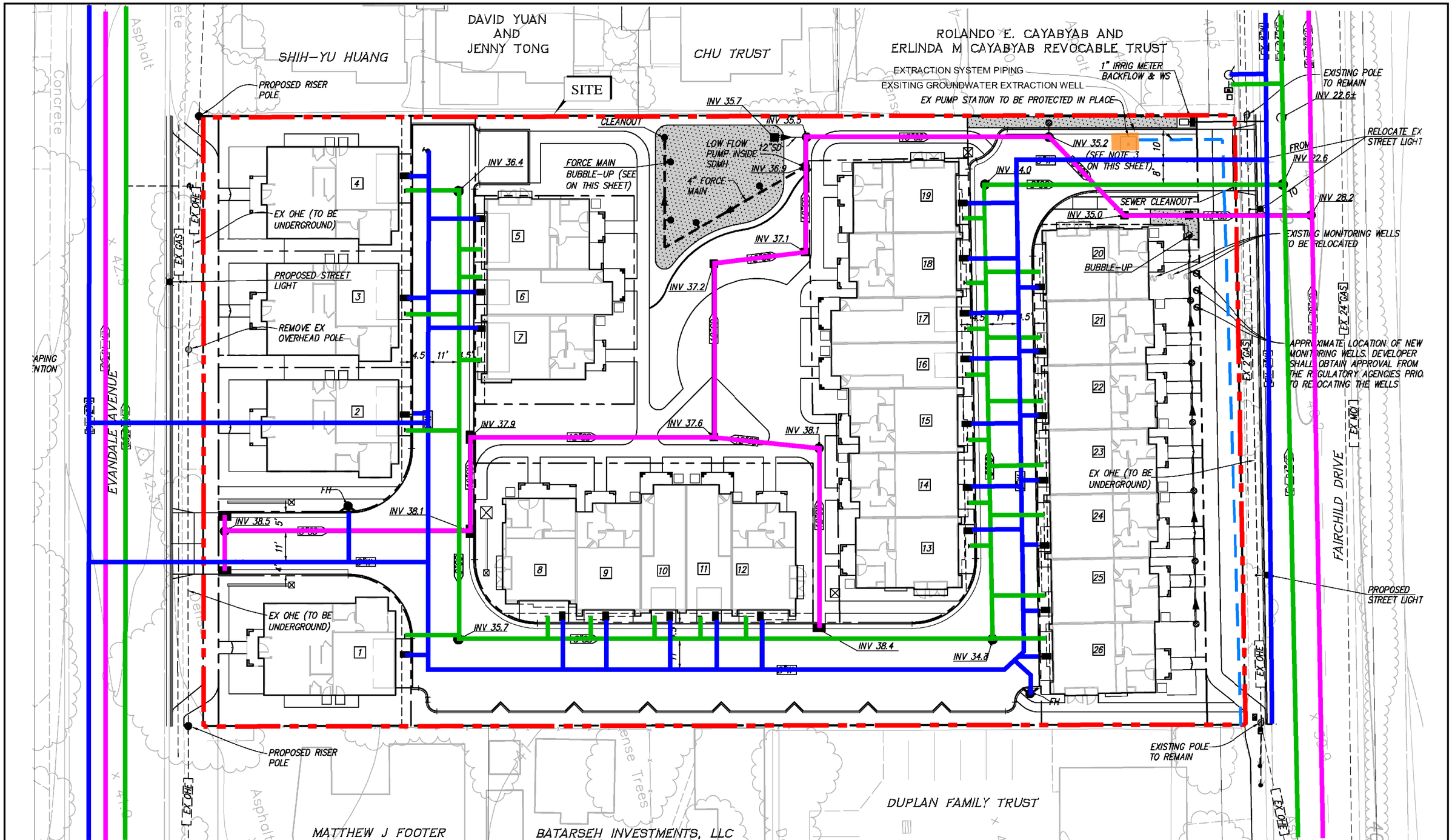
PROPOSED DEVELOPEMENT PLAN
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000		FIGURE NO. 3
SCALE: NO SCALE		
DRAWN BY: LL	CHECKED BY: SM	

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\10216001000-ESA-3-ProposedDevPlan-0915.dwg Plot Date: 9-08-15 llee

ORIGINAL FIGURE PRINTED IN COLOR

COPYRIGHT © 2015 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.



PLANNED UTILITY DEPTHS

WATER: 18"X42"
STORM: 24"X48"
SEWER: 18"X72"

BASE MAP SOURCE: RJA, 2015

ENGEO Expect Excellence

PRELIMINARY UTILITY PLAN
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000
SCALE: AS SHOWN
DRAWN BY: LL
CHECKED BY: SM

FIGURE NO. 4

ORIGINAL FIGURE PRINTED IN COLOR

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-4-PrelimUtilityPlan-1015.dwg Plot Date: 2-23-16 llee

COPYRIGHT © 2016 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-5-TCEConcentrations-1015.dwg Plot Date:4-11-16 llee

EXPLANATION

ALL LOCATIONS ARE APPROXIMATE

- RECOVERY WELL, WITH AQUIFER ZONE INDICATED (2014)
- MEW GRAB GROUNDWATER, WITH AQUIFER ZONE INDICATED (2012/2013)
- TEMPORARY ISCO WELLS (2013)
- EPA GRAB GROUNDWATER, WITH AQUIFER ZONE INDICATED (2013)
- GROUNDWATER MONITORING WELL AND AQUIFER SAMPLING, WITH DEPTH SHOWN (SAMPLING CONDUCTED IN 2014)

NOTE: TCE CONCENTRATIONS SHOWN ARE IN ug/L

N

0 FEET 30
0 METERS 15

BASE MAP SOURCE: GOOGLE EARTH PRO MAPPING SERVICE

ENGEO Expect Excellence

TCE CONCENTRATIONS IN GROUNDWATER
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000		FIGURE NO.
SCALE: AS SHOWN		5
DRAWN BY: LL	CHECKED BY: SM	

ORIGINAL FIGURE PRINTED IN COLOR

COPYRIGHT © 2016 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-6-TCEConcentrations-1015.dwg Plot Date:2-22-16 llee

EXPLANATION

ALL LOCATIONS ARE APPROXIMATE

SOIL GAS SAMPLE

NOTE: RESULTS SHOWN IS TCE CONCENTRATIONS IN ug/m3; SAMPLE DEPTH IN FEET IS SHOWN IN PARENTHESIS. RESULTS ARE FROM SEPTEMBER 2013

ND = NOT DETECTED

BASE MAP SOURCE: GOOGLE EARTH PRO MAPPING SERVICE

N

0 FEET 30
0 METERS 15

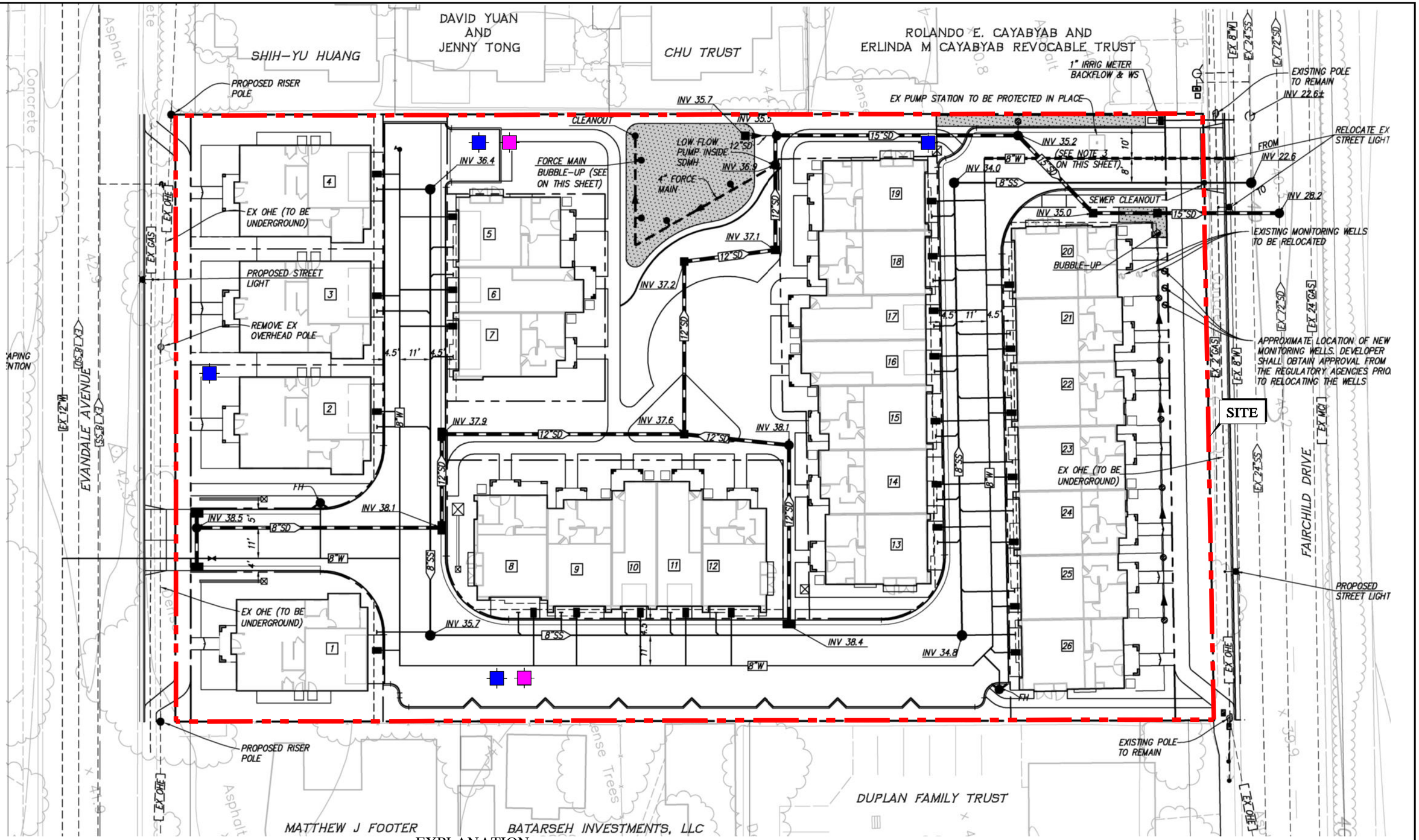
ENGEO Expect Excellence

TCE CONCENTRATIONS IN SOIL GAS
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000	FIGURE NO. 6
SCALE: AS SHOWN	
DRAWN BY: LL	CHECKED BY: SM

ORIGINAL FIGURE PRINTED IN COLOR

COPYRIGHT © 2015 BY ENGEO INCORPORATED. THIS DOCUMENT MAY NOT BE REPRODUCED IN WHOLE OR IN PART BY ANY MEANS WHATSOEVER, NOR MAY IT BE QUOTED OR EXCERPTED WITHOUT THE EXPRESS WRITTEN CONSENT OF ENGEO INCORPORATED.



EXPLANATION

ALL LOCATIONS ARE APPROXIMATE

- PROPOSED FIXED-BASED LABORATORY CONFIRMATION TESTING LOCATION (TO-15 SIM)
- PROPOSED REAL TIME GC-ECD AIR MONITORING LOCATION

BASE MAP SOURCE: RJA, 2015

ENGEO — Expect Excellence

PROPOSED AIR MONITORING STATIONS
277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE
MOUNTAIN VIEW, CALIFORNIA

PROJECT NO.: 10216.001.000	FIGURE NO. 7
SCALE: AS SHOWN	
DRAWN BY: LL	CHECKED BY: SM

G:\Drafting\DRAFTING2_Dwg_10000 to 12999\10216\001\ESA\10216001000-ESA-7-AirMonitoringLocation-1015.dwg Plot Date: 12-09-15 llee

ORIGINAL FIGURE PRINTED IN COLOR

APPENDIX A

Dust Control Plan

APPENDIX A

DUST CONTROL PLAN

277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE

MOUNTAIN VIEW, CALIFORNIA

TABLE OF CONTENTS

Page

1.0 DUST CONTROL PLAN 1
2.0 DUST MITIGATION 1
3.0 STOCKPILE AND VEHICLE MANAGEMENT 1
4.0 METEOROLOGICAL STATION 1
5.0 DUST MONITORING 2
6.0 RECORD KEEPING 2

1.0 DUST CONTROL PLAN

This section details potential dust control measures that the Contractor will implement to minimize dust emissions during the removal action. Dust emissions may result from activities during removal action and from wind erosion. These sources are most effectively controlled using wet suppression. A high wind threshold will also be established to minimize wind erosion during extreme meteorological conditions. Stockpiles will be covered unless being loaded, water will be sprayed on areas, which have already been excavated and are subject to wind erosion. Dust control measures, including air monitoring will be performed in accordance with applicable Bay Area Air Quality Management District standards.

2.0 DUST MITIGATION

The main mechanism for the control of fugitive dust emissions from construction activities and wind erosion is by watering, which leads to the formation of a surface crust to reduce the available reservoir of dust. In addition to water a wide variety of chemical dust suppressants are available to enhance the formation of a surface crust. The effectiveness of wet suppression is dependent on the type of activities occurring, the frequency of watering, and the meteorological conditions. The watering schedule will be determined by an evaluation of the air monitoring and meteorological data, site conditions, and site activities. Watering shall also occur if there are visible dust emissions and/or an exceedance of the air monitoring action level discussed in Section 5.0 below.

3.0 STOCKPILE AND VEHICLE MANAGEMENT

As necessary, based on meteorological and Site conditions, stockpiles will be covered with 10-mil plastic sheeting. All stockpiles will be placed on paved areas when possible or will be placed on 10-mil plastic sheeting. All vehicles onsite will be limited to a maximum speed of 5 mph. Prior to departure from the Site to the surface streets, all vehicles will be checked for material residue and cleaned, if necessary. The public paved roadways surrounding the Site will be checked for any material possibly tracked out, despite mitigation efforts. The Contractor will take all reasonable measures to clean the roadways of this material within an hour of observation.

4.0 METEOROLOGICAL STATION

A meteorological station will be deployed at the Site to monitor wind speed and direction. Measurements will be conducted every 30 minutes to verify conditions and adjust dust monitoring locations. If the wind speed rises to greater than 25 miles per hour (mph), operations will cease. Wind direction measurements from the station will be used to determine the optimum locations for dust monitors.

5.0 DUST MONITORING

A MiniRAM dust meter or equivalent will be used to measure real-time dust levels at a minimum of one upwind and two downwind locations. The meters will be mounted on surveyor's tripods approximately five feet above the ground surface. Dust meters will be equipped with data recorders, which will be periodically downloaded. Meters will be checked hourly to record PM10 readings. The location of the monitors will be adjusted throughout the day based on wind direction data from the on-site meteorological station. Based on the calculations presented in Appendix D, the action level for the project will be based on a 50 □g/m^3 differential between upwind and downwind measurement stations. If the action level is exceeded for a period greater than 30 minutes, work operations will cease until adequate dust mitigation measures can be implemented.

6.0 RECORD KEEPING

The removal action contractor will be responsible for maintaining a field logbook, which will serve to document meteorological conditions, dust monitor readings and dust mitigation measures implemented. This documentation will be included in the final removal action completion report.

APPENDIX B

Sampling and Analysis Plan

APPENDIX B

Sampling and Analysis Plan

277 Fairchild Drive, 228/236 Evandale Avenue

Mountain View, California

TABLE OF CONTENTS

Page

1.0 INTRODUCTION 1
2.0 SAMPLING OBJECTIVES 1
2.1 TRENCH EXCAVATION SOIL SAMPLING 1
2.2 STOCKPILE SOIL CHARACTERIZATION AND OFFSITE DISPOSAL 1
3.0 SAMPLE LOCATIONS AND FREQUENCY 2
3.1 TRENCH EXCAVATION SOIL SAMPLING 2
3.2 STOCKPILE SOIL SAMPLING 2
4.0 SAMPLE DESIGNATION 2
4.1 STOCKPILE SOIL SAMPLES 2
5.0 SAMPLING EQUIPMENT AND PROCEDURES 2
5.1 TRENCH EXCAVATION SAMPLING 3
5.2 STOCKPILE SOIL SAMPLING 3
5.3 DECONTAMINATION PROCEDURES 3
6.0 SAMPLE LABELING, DELIVERY, AND CHAIN-OF-CUSTODY 4
6.1 SAMPLE LABELING 4
6.2 SAMPLE DELIVERY 4
6.3 CHAIN-OF-CUSTODY 5
7.0 FIELD QUALITY ASSURANCE/QUALITY CONTROL 5
7.1 FIELD DUPLICATES 6
7.2 EQUIPMENT RINSATE SAMPLES 6
8.0 SITE MANAGEMENT AND RECORD KEEPING 6

1.0 INTRODUCTION

The purpose of this Sampling and Analysis Plan (SAP) is to provide field sampling procedures and data gathering methods that will be used to support the removal actions for the Property. This SAP will be used by field personnel as a reference for sampling and analysis of soil during the remedial activities.

2.0 SAMPLING OBJECTIVES

Sampling will generally be associated with two activities:

- Potential trench excavation soil sampling (if necessary)
- Stockpile soil characterization

2.1 TRENCH EXCAVATION SOIL SAMPLING

ENGEO will provide full-time observation services during demolition, excavation, and grading work at the Property. Soils encountered during construction activities will be observed for discoloration/staining or olfactory evidence of VOC impacts. In addition, a gas chromatogram-based volatile organic compounds (GC-VOC) monitoring unit will be used to screen excavations for VOCs, as well as ambient air during excavation work. The specific locations of air monitoring will be field-adjusted based on potential access and safety limitations, but will generally be performed within the excavation areas, along with the perimeter of the excavations. In the event impacted soils are identified from visual or GC-VOC readings, soils will be screened using a photoionization detector in sealable plastic bags for presence of TCE and other VOCs. The soil screening shall be performed with a photo-ionization detector (PID) that measures down to the 10 parts per billion by volume (ppbv) range. Screening frequency shall be at the rate of at least one soil sample for every 5 cubic yards of excavated soil, or every 10 lineal feet along an excavation/trench. Excavated soil that has continuous PID readings of 500 ppbv or greater for at least 10 seconds shall be designated as "potentially contaminated soil."

2.2 STOCKPILE SOIL CHARACTERIZATION AND OFFSITE DISPOSAL

The objective of stockpile sampling is to characterize the excavated materials and assist in making a determination of how these materials should be managed and disposed. It is anticipated that the excavated soils will placed into 50 to 100 cubic yard stockpiles. The soil stockpiles will be managed, sampled, and characterized in accordance with this SAP and applicable regulatory guidelines.

Non-hazardous soil will be transported to a Class II/III landfill for disposal. Additional analytical testing may be required to satisfy the profiling and waste acceptance criteria of the receiving facility.

3.0 SAMPLE LOCATIONS AND FREQUENCY

This section discusses the locations and frequency of soil samples that may be collected for analytical testing.

3.1 TRENCH EXCAVATION SOIL SAMPLING

Soils will be screened using a photoionization detector in sealable plastic bags for presence of TCE and other VOCs. The soil screening shall be performed with a photo-ionization detector (PID) that measures down to the 10 parts per billion by volume (ppbv) range. Screening frequency shall be at the rate of at least one soil sample for every 5 cubic yards of excavated soil, or every 10 lineal feet along an excavation/trench. Excavated soil that has continuous PID readings of 500 ppbv or greater for at least 10 seconds shall be designated as "potentially contaminated soil."

3.2 STOCKPILE SOIL SAMPLING

The excavated soil will be stockpiled in approximate 50 to 100 cubic yard volumes. The soil stockpiles will be covered with 10-mil plastic sheeting and secured to prevent dust or runoff during storm events. Stockpiles will be managed in accordance with the Dust Control Plan. The soil stockpiles will be profiled for landfill disposal on a one 4-point composite sample per 50 to 100 cubic yard basis. Depending on soil acceptance criteria of specific treatment facilities and/or landfills; laboratory analyses will be performed to meet profiling requirements.

4.0 SAMPLE DESIGNATION

Samples sent to an analytical testing laboratory will be assigned a unique sample identification number according to the conventions described below. Sample numbers will be recorded in a dedicated field logbook, and on the chain-of-custody at the time of sample collection. A complete description of the sample, sample circumstances/conditions, date and time of sampling, and the location of the sample will be recorded in the dedicated field logbook.

4.1 STOCKPILE SOIL SAMPLES

Stockpile soil samples will be assigned a unique number that includes the stockpile location. For example, sample SP1 indicates the soil sample was collected from stockpile #1. One 4-point composite sample will be recovered from each 50 to 100 cubic yard stockpile. The sample location will be indicated and on the stockpile soil map prepared in the field, will be referenced to the sample number and description in the dedicated field logbook.

5.0 SAMPLING EQUIPMENT AND PROCEDURES

This section describes sampling equipment and procedures associated with the potential trench excavation sampling and stockpile soil sampling. This section also includes a discussion of equipment blank sampling and decontamination procedures for sampling equipment.

5.1 TRENCH EXCAVATION SAMPLING

, If impacted soils (containing elevated concentrations of TCE) are encountered and removed, the following protocols will be followed:

The trenches from which the potentially contaminated soil originated shall be tested by obtaining soil samples from the sidewalls of the trench. Soil sampling shall be performed at a minimum rate of every 10 lineal feet within the area suspected of contamination. Unless otherwise indicated by soil screening, samples shall be collected at a depth midway to the bottom of the trench.

The sampling and analysis procedures for VOCs are required to follow EPA Method 5035 and EPA Region 9's Guidelines for Sampling and Analysis of VOCs in Soils. The following soil target cleanup levels (TCLs) shall be used to assess the need for further sampling and removal:

a) MEW Site soil cleanup levels for TCE (500 micrograms per kilogram) and other MEW Site chemicals of concern;

b) For non-VOCs and VOCs for which site cleanup levels do not exist, the most current version of EPA Region 9 Regional Screening Levels (RSLs) for residential soils or the most current version of the California Human Health Screening Levels (CHHSLs) (California EPA, 2005), whichever is more stringent (i.e., lower), with consideration for established background levels for metals.

If soil concentrations exceeding this cleanup criteria are verified by analysis, then notify EPA within 24 hours.

5.2 STOCKPILE SOIL SAMPLING

Soil samples collected from stockpiles will be collected using clean 2-inch diameter, 6-inch long stainless steel tubes, sealed with Teflon sheets and plastic end caps. Samples will be recovered at depths of 1 to 3 feet below the surface of the stockpile. The samples will be placed in a cooler maintained at 4 degrees Celsius (Cº) with ice. Sample labeling, delivery, and chain-of-custody documentation will be completed per Sections 6.1 through 6.3.

5.3 DECONTAMINATION PROCEDURES

Sampling equipment will be decontaminated to prevent cross contamination between samples. Sampling equipment will be decontaminated by washing with a non-phosphate detergent such as Liquinox™. Decontamination water will be collected and placed in a 55-gallon drum or wastewater holding tank. The following steps will be followed for decontamination of non-disposable sample equipment:

- Wash with a non-phosphate detergent and water solution. This step will remove visible contamination from the equipment. Fill a 5-gallon bucket approximately 3/4 full and dilute with a non-phosphate detergent as directed by the manufacturer. Use a dedicated long-handled brush to assist with cleaning.

- Rinse with potable water. This step will decrease the gross contamination and reduce the frequency of changing of the non-phosphate detergent and water solution. Fill a 5-gallon bucket, 3/4 full with water. Use a dedicated long-handled brush to assist with cleaning of equipment. Frequent changing of this water will increase its effectiveness.

- Rinse with de-ionized water. Fill a 5-gallon bucket approximately 3/4 full of water and use a dedicated long-handled brush to assist with cleaning. Periodic changing of this water is required.

6.0 SAMPLE LABELING, DELIVERY, AND CHAIN-OF-CUSTODY

This section describes how samples will be labeled, picked up, delivered, and tracked.

6.1 SAMPLE LABELING

Sample labels will be completed using indelible, black ink, and affixed to each sample container. Sample containers will be placed into resealable plastic bags to protect the sample from moisture during transportation to the laboratory. Each sample container will be labeled at a minimum with the following:

- Unique sample identification number
- Sample collection date (month/day/year)
- Time of collection (24-hour clock)
- Project number
- Sampler initials
- Analyses to be performed; and preservation, if any

6.2 SAMPLE DELIVERY

This section applies to samples that will be picked up by the analytical testing laboratory or samples delivered to the offsite analytical laboratory. Samples may be picked up in the field or at the Field Geologist/Engineer's office by the analytical testing laboratory. The samples will be maintained at 4° Celsius. The chain-of-custody documentation will be completed and signed by the laboratory- assigned courier. The samples may then be relinquished to the courier for transportation to the laboratory. Each cooler will contain a temperature blank. A temperature blank is a sample container filled with tap water and stored in the cooler during sample collection and transportation. The laboratory will record the temperature of the temperature blank immediately upon receipt of the samples.

6.3 CHAIN-OF-CUSTODY

A chain-of-custody is a vital tool for tracking samples and is a written record of sample possession from the time the sample is collected until it is analyzed. The following will be recorded on the chain-of-custody forms:

- Project name.
- Project location.
- Project number.
- Project contact.
- Client.
- Project Manager.
- Sample identification.
- Date and time sample was collected.
- Sample type.
- Number of sample containers.
- Required analytical test methods.
- Remarks/observations specific to the sample.
- Number of samples to be relinquished to the analytical laboratory.
- Transfer signatures associated with relinquishing samples (the sampler will initiate the chain-of-custody procedure).
- Courier/laboratory representative signature (for commercial carrier, record air bill number) Date/time of custody transfers.
- Comments regarding the condition of the samples, (e.g., cooled with ice, etc.).
- Additional comments.
- Written request for electronic file for all samples analyzed.
- Information regarding sample storage/disposal.
- Turn-around-time requirement; Sampler signature.
- Courier signature.

7.0 FIELD QUALITY ASSURANCE/QUALITY CONTROL

Field Quality Assurance/Quality Control (QA/QC) samples will be collected and analyzed during the post- excavation confirmation and stockpile soil sampling to assess the consistency

and performance of the sampling program. Field QC samples for this project will include field duplicates and equipment rinsate samples.

7.1 FIELD DUPLICATES

Field duplicates consist of a sample of the same matrix as the primary sample collected. Duplicate samples will be collected, if available, at the same time and location as the primary sample, using the same sampling techniques. The purpose of field duplicate samples is to evaluate the precision of the overall sample collection and analysis process. Field duplicates for the trench excavation samples, if necessary, will be collected at a frequency of one per 10 samples and will be analyzed using the same method as the primary sample. Field duplicate sample numbers will be similar to the post-excavation sample nomenclature; however, minor adjustments in the numbering system will be made to ensure that the identities of the duplicate samples are “blind” to the analytical laboratory. Locations of duplicate samples and their identifications will be recorded in the dedicated field logbook and on the appropriate excavation map.

As the landfills do not require duplicate samples, this procedure will not be implemented for stockpiles.

7.2 EQUIPMENT RINSATE SAMPLES

If appropriate, equipment rinsate samples will only be collected once every day with the use of non-disposable sampling equipment. Rinsate samples consist of distilled water collected from the final rinse of the decontamination process. Subsequent to equipment decontamination, distilled water will be decanted over the sampling equipment in the appropriate containers. Rinsate samples will be collected, placed in appropriate pre-cleaned containers supplied by the analytical laboratory, and analyzed for the same constituents as the field samples. Equipment rinsate samples evaluate the effectiveness of the decontamination procedure and possible cross-contamination during sampling events.

8.0 SITE MANAGEMENT AND RECORD KEEPING

Sampling information will be recorded on chain-of-custody forms, in a dedicated field logbook, and on the appropriate excavation or stockpile map/plan. These documents will be completed in the field at the time of sample collection. Entries will be legible and recorded in indelible black ink. A dedicated bound field logbook with consecutively numbered pages will be assigned to this project. If it is necessary to transfer the logbook to another person, the person relinquishing the logbook will sign and date the last page used and the person receiving the logbook will sign and date the next page to be used. At a minimum, the logbook will contain the following information:

- Project name and location.
- Date and time of entries.

- Personnel in attendance, including any visitors to the site; General weather conditions.
- Work performed on a daily basis.
- Field observations.
- Sampling information (including sample identification, sample location, sample description/type, and analytical testing).
- Field measurements data (including air monitoring results, instrument calibration records, and problems, if encountered).
- Descriptions of deviations from the SAP, if applicable; Problems encountered and corrective action taken; QC-related activities and identification of field QC samples.
- Detailed record of oral and/or written requests by the regulatory agencies, client, subcontractor.
- Any other events that may affect the sampling and analyses.

METHOD 5035A

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260. The following compounds are appropriate for this sample preparation technique:

Compound	CAS No.[a]	Response	Stability
Acetone	67-64-1	ht	hs
Acetonitrile	75-05-8	pp	nd
Acrolein (Propenal)	107-02-8	pp	ms
Acrylonitrile	107-13-1	pp	hs
Allyl alcohol	107-18-6	ht	nd
Allyl chloride	107-05-1	c	ms
t-Amyl ethyl ether (TAEE)	919-94-8	c / ht	nd
t-Amyl methyl ether (TAME)	994-05-8	c / ht	hs
Benzene	71-43-2	c	hs
Benzyl chloride	100-44-7	c	nd
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd
Bromoacetone	598-31-2	pp	nd
Bromochloromethane	74-97-5	c	hs
Bromodichloromethane	75-27-4	c	ms
Bromoform	75-25-2	c	hs
Bromomethane	74-83-9	c	hvs
n-Butanol	71-36-3	ht	nd
2-Butanone (MEK)	78-93-3	pp	hvs
t-Butyl alcohol	75-65-0	ht	nd
Carbon disulfide	75-15-0	pp	hvs
Carbon tetrachloride	56-23-5	c	hvs
Chloral hydrate	302-17-0	pp	nd
Chlorobenzene	108-90-7	c	hvs
Chlorodibromomethane	124-48-1	c	nd
Chloroethane	75-00-3	c	ms

(continued)

Compound	CAS No.[a]	Response	Stability
2-Chloroethanol	107-07-3	pp	nd
2-Chloroethyl vinyl ether	110-75-8	c	ls
Chloroform	67-66-3	c	hs
Chloromethane	74-87-3	c	hvs
Chloroprene	126-99-8	c	nd
Crotonaldehyde	4170-30-3	pp	nd
1,2-Dibromo-3-chloropropane	96-12-8	pp	ms
1,2-Dibromoethane	106-93-4	c	hs
Dibromomethane	74-95-3	c	hs
1,2-Dichlorobenzene	95-50-1	c	hs
1,3-Dichlorobenzene	541-73-1	c	ms
1,4-Dichlorobenzene	106-46-7	c	ms
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd
trans-1,4-Dichloro-2-butene	110-57-6	pp	ls
Dichlorodifluoromethane	75-71-8	c	hs
1,1-Dichloroethane	75-34-3	c	hs
1,2-Dichloroethane	107-06-2	c	hs
1,1-Dichloroethene	75-35-4	c	hvs
cis-1,2-Dichloroethene	156-59-4	c	hs
trans-1,2-Dichloroethene	156-60-5	c	ms
1,2-Dichloropropane	78-87-5	c	hs
1,3-Dichloro-2-propanol	96-23-1	pp	nd
cis-1,3-Dichloropropene	10061-01-5	c	ls
trans-1,3-Dichloropropene	10061-02-6	c	ls
1,2,3,4-Diepoxybutane	1464-53-5	c	nd
Diethyl ether	60-29-7	c	nd
Diisopropyl ether (DIPE)	108-20-3	c / ht	hs
1,4-Dioxane	123-91-1	pp	nd
Ethylbenzene	100-41-4	c	hvs
Ethylene oxide	75-21-8	pp	nd
Ethyl methacrylate	97-63-2	c	ms
Ethyl *tert*-butyl ether (ETBE)	637-92-3	c / ht	hs
Hexachlorobutadiene	87-68-3	c	ms
2-Hexanone	591-78-6	pp	hvs
Iodomethane	74-88-4	c	nd
Isobutyl alcohol	78-83-1	ht / pp	nd
Isopropylbenzene	98-82-8	c	ms
Malononitrile	109-77-3	pp	nd
Methacrylonitrile	126-98-7	pp	hs
Methylene chloride	75-09-2	c	hs
Methyl methacrylate	80-62-6	c	ms
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	ms
Methyl *tert*-butyl ether (MTBE)	1634-04-4	c / ht	hs
Naphthalene	91-20-3	c	ms
Nitrobenzene	98-95-3	c	nd

(continued)

Compound	CAS No.[a]	Response	Stability
2-Nitropropane	79-46-9	c	nd
N-Nitroso-di-*n*-butylamine	924-16-3	pp	nd
Paraldehyde	123-63-7	pp	nd
2-Pentanone	107-87-9	pp	nd
2-Picoline	109-06-8	pp	nd
1-Propanol	71-23-8	ht / pp	nd
2-Propanol	67-63-0	ht / pp	nd
â-Propiolactone	57-57-8	pp	nd
Propionitrile (ethyl cyanide)	107-12-0	ht	nd
n-Propylamine	107-10-8	c	nd
Styrene	100-42-5	c	hvs
1,1,1,2-Tetrachloroethane	630-20-6	c	hs
1,1,2,2-Tetrachloroethane	79-34-5	c	nd
Tetrachloroethene	127-18-4	c	ms
Toluene	108-88-3	c	hs
o-Toluidine	95-53-4	pp	nd
1,2,4-Trichlorobenzene	120-82-1	c	hs
1,1,1-Trichloroethane	71-55-6	c	ms
1,1,2-Trichloroethane	79-00-5	c	hs
Trichloroethene	79-01-6	c	ms
Trichlorofluoromethane	75-69-4	c	ls
1,2,3-Trichloropropane	96-18-4	c	ls
Vinyl acetate	108-05-4	c	ls
Vinyl chloride	75-01-4	c	hvs
o-Xylene	95-47-6	c	hvs
m-Xylene	108-38-3	c	hvs
p-Xylene	106-42-3	c	hvs

[a] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
nd = Not determined
hs = High stability in preserved water samples (> 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ms = Medium stability in preserved water samples (15 - 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ls = Low stability in preserved water samples (< 14 days), analyses should be performed as soon as possible.
hvs = Highly variable stability in preserved water samples. Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are minimized. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030.

1.5 This method can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are significantly higher because of poor purging efficiency. The purging efficiency can be improved for water soluble analytes, e.g. ketones and alcohols, when purging at an elevated temperature of 80°C as compared to 20° or 40°C.

1.6 This method, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use this method and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 8.2.2).

1.9 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.10 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained laboratory analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg (refer to Appendix A for additional information).

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. To ensure minimal loss of volatile constituents prior to analysis the entire sample vial is placed, unopened with an unpierced septum, into the instrument auto sampler device. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg (refer to Appendix A for additional information).

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/ELCD, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect an appropriate sample volume in a pre-weighed vial with a septum-sealed screw-cap (see Sec 6) that contains a water-miscible organic solvent (e.g., methanol). At the time of analysis, an aliquot of the solvent is removed from the vial and diluted into water along with the internal standards and surrogates, then purged using Method 5030 and analyzed by an appropriate determinative method.

2.2.2 The second option is to collect a bulk sample in a VOA vial without the use of a chemical preservative. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, a significant amount of volatile constituents may be lost during handling. (See Appendix A, Sec. 5.1 for additional details)

<u>NOTE</u>: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

3.0 DEFINITIONS

Refer to Chapter One for a listing of applicable quality assurance/quality control (QA/QC) definitions.

4.0 INTERFERENCES

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which can be concentrated in the trap during the purge operation. These compounds can result in interferences or false positives in the determinative step.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from an appropriate organic-free matrix and sample container, and carried through sampling and handling protocols, serves as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

4.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken when analyzing 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 GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed can also lead to random background levels and the same precautions must be taken.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals included in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 Sample containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 6.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices. Additional information on sample containers can be found in Appendix A, Secs. 1.6, 3.0, 7.0 and 8.0.

6.2 Purge-and-trap system

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

6.2.1 The purging device should be capable of accepting a vial sufficiently large enough to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 6.2.2).

NOTE: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

6.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all desired target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000, Supelco, Inc., Bellefonte, PA), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 (Supelco, Inc., Bellefonte, PA) is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

6.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carbopack/Carbosieve (Supelco, Inc., Bellefonte, PA).

6.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

6.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

6.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

6.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

6.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications as noted above.

6.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

6.3 Syringe and syringe valves

6.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

6.3.2 2-way syringe valves with Luer ends.

6.3.3 25-µL micro syringe with a 2-inch x 0.006-inch ID, 22° bevel needle (Hamilton #702N or equivalent).

6.3.4 Micro syringes - 10-, 100-µL.

6.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

6.4 Miscellaneous

6.4.1 Glass vials

6.4.1.1 60-mL, septum-sealed, to collect samples for screening, moisture determination.

6.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

6.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

6.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

6.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

6.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

6.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

6.4.7 Disposable Pasteur pipettes.

6.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

6.5 Field sampling equipment

6.5.1 Purge-and-trap soil sampler - Model 3780PT (Associated Design and Manufacturing Company, Alexandria, VA), or equivalent.

6.5.2 EnCore™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.3 Terra Core™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.4 EasyDraw™ syringe and PowerStop™ handle - (US Oil Company, Inc., Kimberly, WI), or equivalent.

6.5.5 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

6.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

6.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

6.5.6 Additional types of field sampling equipment and accessories are described in Appendix A, Secs. 1.6 and 7.0.

7.0 REAGENTS AND STANDARDS

7.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

7.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

7.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes.

7.4 Low concentration sample preservative

7.4.1 For determination as to whether sample preservation is necessary and for selection of appropriate preservation options, see Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0.

7.4.2 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

7.4.3 The preservative, if necessary, should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

7.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure. The recommended surrogates are 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and toluene-d_8. Other compounds may be used as surrogates, depending upon the analysis requirements and the specific target analytes. The recommended internal standards are chlorobenzene-d_5, 1,4-dichlorobenzene-d_4, and fluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the target analytes being detected.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, and Appendix A for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process.

As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

8.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in this method.

8.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 6.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

8.1.1.2 Add preservative, if necessary, (See Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0) to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2.

8.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

8.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

8.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

8.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

8.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 6.4). More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030. See the water-miscible solvent dilution effect information in Sec. 11.5 and Method 8000 for guidance on correcting results for data reporting purposes. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3.1 Add 10 mL of methanol to each vial.

8.1.3.2 Seal the vial with the screw-cap and septum seal.

8.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

8.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

8.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 8.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 8.1.2.

8.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler ™, or any other sampling device listed in Sec. 6.5, or equivalent. Always wear gloves whenever handling the tared sample vials. More detailed information and additional sample collection options can be found in Appendix A, Sec. 7.0.

8.2.1 Low concentration soil samples

8.2.1.1 Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution or other preservation options as discussed in Appendix A. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C. Alternatively, samples can be collected into an empty vial or vial containing reagent water (with or without preservative) and stored frozen at < -7°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution option in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials without chemical preservation.

8.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.1.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis, if needed. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

8.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, moisture determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine % moisture. If high concentration samples are collected in

vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the moisture determination in a vial without either methanol or the low concentration aqueous preservative solution.

8.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 8.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

8.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

8.2.1.9 The collection of low concentration soil samples in vials that contain methanol is <u>not</u> appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 8.2.2).

8.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is <u>not</u> appropriate for use with the low concentration soil procedure described in this method.

<u>NOTE</u>: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of three potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 100, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. Secondly, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (see Sec. 11.5 and Method 8000) The final problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, or cause it to become a listed waste, thereby requiring the unused sample volume to be managed as a hazardous waste.

8.2.2.1 When samples are <u>known</u> to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

8.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

8.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

8.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the moisture content, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

8.2.3 High concentration sample <u>not</u> preserved in the field

The collection of high concentration bulk samples, i.e., wastes containing percent level concentrations, that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 8.2.1 and 8.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for moisture determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

8.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

8.2.4.1 When an oily waste is <u>known</u> to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 8.1.4), using procedures similar to those described in Sec. 8.2.2.

8.2.4.2 When the solubility of the oily waste is <u>not</u> known, the sample should either be collected in a vial without a preservative, as described in Sec. 8.2.3, or the

solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 8.2.2. Otherwise, collect an unpreserved sample as described in Sec. 8.2.3.

8.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample handling options.

8.4 Sample storage

8.4.1 Once in the laboratory, store samples at the recommended temperature until analysis (refer to Appendix A, Secs. 3.0 and 7.4 for additional sample storage information). The sample storage area should be free of organic solvent vapors.

8.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

8.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at <-7°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 8.2.1.2 for additional information.

8.4.4 See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample storage options.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols and Method 5000 for sample preparation QC procedures. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One.

9.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

9.3 Initial demonstration of proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See the Quality Control Section of Methods 5000 and 8000 for information on how to accomplish this demonstration.

9.4 Sample quality control for preparation and analysis - See the Quality Control Section of Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.6 The laboratory should have quality control procedures to make sure that sample integrity is not compromised during the sample collection and sample handling process, e.g., making sure that septa and vial caps do not leak, etc. (See Appendix A, Secs. 1.6 and 7.1.1) In addition, it would be advisable for the laboratory to monitor the internal standard's (IS) area counts for the low concentration samples, since leaks attributed to a poor seal with the vial caps and septa will be evident by low IS area counts. Sample containers and data results for instances where low IS area counts are observed and leaks are suspected, should be discarded.

10.0 CALIBRATION AND STANDARDIZATION

Refer to the appropriate determinative method for calibration and standardization procedures.

11.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

11.1 Sample screening

11.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 11.2), the high concentration (methanol extraction) method (Sec. 11.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 11.4).

11.1.2 The analyst may employ any appropriate screening technique. Three suggested screening techniques employing SW-846 methods are:

11.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with an appropriate detector,

11.1.2.2 Screening with a portable photoionization detector (PID) (Method 3815) or,

11.1.2.3 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

11.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

11.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 11.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 11.3), or the oily waste method (Sec. 11.4).

11.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

11.2.1 Initial set-up

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

11.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 6.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

11.2.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by baking out with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned by baking for 10 minutes at 245°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.3 If the standard trap in Sec. 6.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by baking out with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be

conditioned by baking for 10 min at 180°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

11.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). When the sodium bisulfate preservation technique is used, the calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

11.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 11.2.3. to 11.2.5.

11.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

11.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

11.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

11.2.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

11.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

11.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem. Extra rinsing of the purge vessel after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bake-out of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

11.2.2 Calibration verification (see appropriate determinative method)

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate if the samples are also preserved in this manner.

11.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

11.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

11.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

11.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in the Reagents Section of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in the Quality Control Section of Method 8000.

11.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for the appropriate purge time (usually 11 minutes) while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

11.2.4 Sample desorption

11.2.4.1 Non-cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

11.2.4.2 Cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Method 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

11.2.5 Trap reconditioning

After desorbing the sample, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

11.2.6 Data interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 8.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be corrected for moisture content, proceed to Sec. 11.5.

11.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 11.3.8).

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below,

beginning at Sec. 11.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 11.3.4.

11.3.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

11.3.2 If the sample is from an unknown source, perform a solubility test preferably using a sample container reserved for the % moisture determination before proceeding. Remove several grams of material from the sample container. If the sample material is obtained from a vial dedicated for analysis, quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 11.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 11.3.8.

11.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution, or 10.0 mL of methanol without surrogates to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. See Appendix A, Sec. 6.2.1 for methanol contact time information. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 or 10.0 mL of PEG in place of the methanol. Proceed with Sec. 11.3.5.

NOTE: The steps in Secs. 11.3.1, 11.3.2, and 11.3.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

11.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 8.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum, and proceed with Sec. 11.3.5. See Appendix A, Sec. A.6.2.1 for methanol contact time information.

11.3.5 Pipet approximately 1 mL of the extract from either Sec. 11.3.3 or 11.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

11.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (based on the approximate sample concentration as noted in the table below) to 5.0 mL of organic-free reagent water containing if applicable, surrogates, internal standards, and matrix spike compounds, and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to the Procedure Section in Method 5030 and follow the procedure for purging high concentration samples.

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500 - 10,000 µg/kg	100 µL
1,000 - 20,000 µg/kg	50 µL
5,000 - 100,000 µg/kg	10 µL
25,000 - 500,000 µg/kg	100 µL of 1/50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding those in this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

11.3.7 If results are to be reported using a correction factor for moisture content, determine the moisture content of a separate aliquot of the sample, using the procedure in Sec. 11.5, after the sample extract has been transferred to a GC vial and the vial sealed.

11.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in the Procedure Section of Method 3585.

11.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in the Procedure Section of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 11.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 11.4.3.

11.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

11.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

11.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

11.4.2 Quickly add 1.0 mL of surrogate spiking solution, if desired, to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and proceed with Sec. 11.4.4. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

11.4.5 Add 10 - 50 µL of the methanol extract to 5 mL of organic-free reagent water containing if applicable, surrogates and internal standards, followed by purge-and-trap analysis, using Method 5030.

11.4.6 If necessary, prepare a matrix spike sample by adding 10 - 50 µL of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 11.4.2 - 11.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in the Procedure Section of Method 3585.

11.5 Determination of % moisture

If results are to be reported using a correction factor for moisture content, it is necessary to determine the moisture content of the sample. Also note that solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) In order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

NOTE: It is highly recommended that the moisture content determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the moisture content determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

11.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

11.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % moisture as follows:

$$\%\ \text{moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

12.0 DATA ANALYSIS AND CALCULATIONS

There are no calculations explicitly associated with this extraction procedure. See the appropriate determinative method and Method 8000 for calculation of final sample results.

13.0 METHOD PERFORMANCE

13.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 µg/kg. These data are listed in tables found in Method 8260.

13.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste*

Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 12.2.

16.0 REFERENCES

1. Bellar, T., "Measurement of Volatile Organic Compounds in Soils Using Modified Purge-and-Trap and Capillary Gas Chromatography/Mass Spectrometry" U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH, November 1991.

2. Siegrist, R. L., Jenssen, P. D., “Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils”, Envir Sci Technol, 1990; 24; 1387-92.

3. Hewitt, A. D., Jenkins, T. F., Grant, C. L., “Collection, Handling and Storage: Keys to Improved Data Quality for Volatile Organic Compounds in Soil”, Am Environ Lab, 1995; 7(1); 25-8.

4. Liikala, T. L., Olsen, K. B., Teel, S. S., Lanigan, D. C., “Volatile Organic Compounds: Comparison of Two Sample Collection and Preservation Methods”, Envir Sci Technol, 1996; 30; 3441-7.

5. Lewis, T. E., Crockett, A. B., Siegrist, R. L., Zarrabi, K., “Soil Sampling and Analysis for Volatile Organic Compounds”, Envir Monitoring & Assessment, 1994; 30; 213-46.

6. Hewitt, A. D., “Enhanced Preservation of Volatile Organic Compounds in Soil with Sodium Bisulfate”, SR95-26, U. S. Army Cold Regions Research and Engineering Laboratory, Hanover, NH.

7. Hewitt, A. D., Lukash, N. J. E., “Sampling for In-Vial Analysis of Volatile Organic Compounds in Soil”, Am Environ Lab, 1996; Aug; 15-9.

8. Hewitt, A. D., Miyares, P. H., Sletten, R. S., “Determination of Two Chlorinated Volatile Organic Compounds in Soil by Headspace Gas Chromatography and Purge-and-Trap Gas Chromatography/Mass Spectrometry”, Hydrocarbon Contaminated Soils, 1993, 3; 135-45, Chelsea, MI, Lewis Publishers.

9. Hewitt, A. D., “Methods of Preparing Soil Samples for Headspace Analysis of Volatile Organic Compounds: Emphasis on Salting Out”, 12th Annual Waste Testing and Quality Assurance Symposium, Washington, DC, 1996, 322-9.

10. Hewitt, A. D., Miyares, P. H., Leggett, D. C., Jenkins, T. F., “Comparison of Analytical Methods for Determination of Volatile Organic Compounds”, Envir Sci Tech, 1992; 26; 1932-8.

APPENDIX A

THE COLLECTION AND PRESERVATION OF AQUEOUS AND SOLID SAMPLES FOR VOLATILE ORGANIC COMPOUND (VOC) ANALYSIS

FOREWORD

The information provided in this Appendix is based on EPA's evaluation of currently available data and technology as applied to the most appropriate sample handling and preservation procedures in order to minimize the loss of volatile organic compounds (VOCs) during the collection and analysis of aqueous and solid materials, such as groundwater, wastewater, soils, solid waste, or sediments. These procedures are designed to minimize the losses of VOCs through the two most common mechanisms, volatilization and biodegradation. The intended users of this Appendix guidance are those individuals and organizations involved in the collection and preparation of samples for VOC analyses during the characterization of solid materials under the Resource Conservation and Recovery Act (RCRA). The procedures and techniques described in this Appendix are not presented in any preferential order nor do they represent EPA requirements, but rather they are intended solely as guidance and should be selected and utilized based on the stated project-specific data quality objectives.

This Method 5035 Appendix was developed under the direction of Mr. Barry Lesnik, U.S. EPA, Office of Solid Waste (OSW), Methods Team in collaboration with Mr. David Payne, U.S. EPA, Region 5, Mr. Alan Hewitt, U.S. ACE CRREL, and the SW-846 Organic Methods Workgroup Members. The Methods Team is the focal point within OSW for expertise in analytical chemistry and characteristic testing methodologies, environmental sampling and monitoring, and quality assurance. The Methods Team provides technical support to other OSW Divisions, EPA Program Offices and Regions, state regulatory agencies, and the regulated community.

DISCLAIMER

The U.S. Environmental Protection Agency's Office of Solid Waste (EPA or the Agency) has prepared this Method 5035 Appendix to provide guidance to those individuals involved in the collection and preparation of samples for volatile organic compounds (VOCs) analysis during the characterization of aqueous and solid materials under the Resource Conservation and Recovery Act (RCRA). This Appendix provides guidance for selecting an appropriate sample collection and preservation technique that may be suitable for VOC analyses in order to meet the data quality requirements or objectives for the intended use of the results.

EPA does not make any warranty or representation, expressed or implied with respect to the accuracy, completeness or usefulness of the information contained in this report. EPA does not assume any liability with respect to the use of, or for damages resulting from the use of, any information, apparatus, method or process disclosed in this report. Reference to trade names or specific commercial products, commodities, or services in this report does not represent or constitute an endorsement, recommendation, or favoring by EPA of the specific commercial product, commodity, or service. In addition, the policies set out in this Appendix are not final Agency action, but are intended solely as guidance. They are not intended, nor can they be relied upon, to create any rights enforceable by any party in litigation with the United States. EPA officials may decide to follow the guidance provided in this Appendix, or to act at variance with the guidance, based on an analysis of specific site or facility circumstances. The Agency also reserves the right to change this guidance at any time without public notice.

CONTENTS

A.1.0 PURPOSE AND OVERVIEW 33
A.1.1 What are VOCs? 33
A.1.2 What is sample preservation? 33
A.1.3 Do all VOA samples need to be chemically preserved? 34
A.1.4 Who is the intended audience for this Appendix? 35
A.1.5 What does this guidance not cover? 35
A.1.6 What equipment is needed? 35
A.1.7 How is the guidance organized? 35

A.2.0 PROJECT PLANNING 37

A.3.0 AQUEOUS SAMPLE MATRICES AND VOLATILE ORGANIC COMPOUNDS 38
A.3.1 Alternative Considerations for Sample Holding Time Criteria 39

A.4.0 SOLID MATERIALS/COHESIVE SOILS AND VOLATILE ORGANIC COMPOUNDS 44

A.5.0 HISTORY OF PRACTICES IN THE SAMPLING AND PREPARATION OF SOLID MATERIALS FOR VOC ANALYSIS 46
A.5.1 Traditional Practices 46
A.5.2 Improvement of Sample Collection Techniques 46
A.5.3 New Developments 47

A.6.0 OVERVIEW OF VAPOR PARTITIONING AND METHANOL EXTRACTION TECHNOLOGIES 48
A.6.1 Vapor Partitioning 48
A.6.2 Methanol Extraction 49
A.6.2.1 Contact Time Effect 49
A.6.2.2 Safety and Hazardous Waste Generation Concerns 49

A.7.0 SAMPLE COLLECTION 50
A.7.1 Collection of Samples for Analysis 50
A.7.1.1 Subsampling of Cohesive Granular Uncemented Materials Using Devices Designed to Obtain a Sample Appropriate for Analysis 50
A.7.1.2 Devices that can be Used for Subsampling a Cemented Material . 51
A.7.1.3 Devices that can be Used for subsampling a Non-Cohesive Granular Material 51
A.7.2 Use of the EnCore™ Sampler (or Equivalent) for Sample Transport and Storage 51
A.7.3 Concerns Regarding Use of Core Barrel Liners 52
A.7.4 After Collection – Sample Handling and Storage 52
A.7.4.1 Holding Times 52
A.7.5 Quality Control 53
A.7.6 Interferences / Artifacts of Analysis 54

A.8.0 APPROACHES TO SAMPLE PREPARATION 55
A.8.1 Overview of Empty Vial Technique 56
A.8.2 Preservation Approaches Using Empty VOA Vials 57

A.8.2.1 Preservation by freezing (< -7°C) 57
A.8.2.2 Refrigerate vials at 4 ± 2°C for 48 hours or less, then preserve by freezing at < -7°C upon laboratory receipt 58
A.8.2.3 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less, then preserve with methanol upon laboratory receipt 58
A.8.2.4 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less and complete VOC analysis (Method 5021/5035) within 48 hours 59
A.8.2.5 Refrigerate/freeze coring tool used as transport device for 48 hours or less (Refs. 15,26) 59
A.8.3 Preservation Approaches Using Prepared VOA Vials 59
A.8.3.1 Collection with reagent water, preservation by freezing (< -7°C) and analysis by vapor partitioning 60
A.8.3.2 Collection with reagent water, preservation by refrigeration at 4 ± 2°C for 48 hours or less and immediate laboratory analysis or freezing storage at -7°C for subsequent vapor partitioning . . . 60
A.8.3.3 Collection with 5 mLs of water and 1 g of $NaHSO_4$ and analysis by vapor partitioning 60
A.8.3.4 Collection and preservation with methanol at 4 ± 2°C 61

A.9.0 SUMMARY OF FINDINGS 62

A.10.0 REFERENCES 65

Tables

Table A.1 Recommended VOC Sample Preservation Techniques and Holding Times 41

A.1.0 PURPOSE AND OVERVIEW

This Appendix provides guidance in sample collection and preservation procedures that may be suitable for use during the characterization of volatile organic compounds (VOCs) in solid materials, such as soils, solid wastes, or sediments and aqueous samples or leachates from solid matrices.

A.1.1 What are VOCs?

VOCs are a class of organic compounds that includes low molecular weight aromatics, hydrocarbons, halogenated hydrocarbons, ketones, acetates, nitriles, acrylates, ethers, and sulfides with sufficiently low boiling points to give them appreciable vapor pressures at 1 atmosphere of pressure. Although EPA has never defined a strict boiling point cut-off for this compound class, most VOCs of concern to EPA have boiling points below 150°C, while some members of this class may have boiling points as high as 200°C.

The solubilities of the individual VOCs in water vary widely, from insoluble to soluble, with many of the oxygenated compounds (ketones and ethers) at the soluble end of the range and the hydrocarbons and substituted hydrocarbons at the insoluble end of the range.

Given that water may be present to varying degrees in such solid materials of environmental significance as soils, solid wastes, and sediments, the water solubility of an individual VOC may in fact control its "solubility" in solid samples.

A.1.2 What is sample preservation?

The sample collection procedures described in EPA analytical methods are designed to ensure that at the time of analysis, the chemical composition of the small volume of material collected from the parent bulk material is representative of the chemical composition of the original material. Considerations regarding sample support and sampling design (discussed in Chapter Nine of the SW-846 manual) ensure that the physical aspects of sample collection (e.g., sample volume and orientation, numbers and distribution of samples) produce data estimates that are representative of the bulk material subject to regulatory decision-making, perhaps millions of gallons a day of discharged wastewater, or thousands of kilograms of solid material. Once collected, a sample should be maintained in a manner that preserves the relationship between it and the bulk material, e.g., the chemical composition of the sample should not change by virtue of being collected. Maintaining that relationship between the sample and the bulk material is referred to as sample preservation.

Several types of sample preservation are employed in EPA methods. The most common method of preservation is to cool the sample to 4 ± 2°C. Cooling may be applied to many types of sample matrices, including water, soil, sediments, and solid wastes. The temperature of 4 ± 2°C is used because it represents the temperature at which pure water exhibits its maximum density, hence its minimum volume. However, if aqueous samples are cooled below 0°C, the water expands significantly as it freezes and may crack the sample container.

By lowering the temperature of the sample, many of the physical, chemical, and biological processes that may cause environmental contaminants to leave the sample (e.g., loss of volatiles to the air) or be transformed into other compounds (e.g., chemical breakdown or biodegradation) are greatly slowed. However, even if the rates of biodegradation are reduced by physical preservation, many environmental matrices of interest contain large numbers of microorganisms that may break down contaminants. Examples include wastewaters from sewage

treatment, surface waters, and surface soil. In these types of matrices, simply reducing the rate at which biodegradation occurs may not be enough to maintain the condition of the original sample.

The most practical way in which to reduce this biological activity in aqueous samples is through the use of chemical preservatives that act as biocides. Historically, this has included preservatives such as sodium bisulfate or hydrochloric acid to adjust the pH for aqueous samples to less than pH 2, at which point, virtually all biological activity ceases.

Adjusting the pH of a solid sample such as a soil, sediment, or solid waste presents a number of other difficulties. In particular, samples containing carbonates should not be acidified due to the potential for effervescence which may result in loss of volatile compounds. Precautions should also be taken when preserving by acidification since certain compounds within the olefins, ketones, esters, ethers, and sulfides classes may react under low pH conditions and possibly not be representative of the material as sampled. Additionally, acidification of solid wastes may evolve toxic gases that may be harmful to field and laboratory personnel. **It is therefore recommended that when collecting wastes of unknown composition, preliminary screening and characterization of potential sample contents should be performed prior to use of acidification as a means to chemically preserve samples designated for determinative analyses.**

Sample collection and preservation procedures should be carefully selected in order to minimize VOC losses prior to sample preparation and determination in the laboratory. Although this guidance discusses some traditional approaches to VOC sample collection and preservation, its main purpose is to provide guidance regarding newer approaches, such as freezing the samples, which may particularly decrease VOC loss in some materials. For additional information regarding the challenges associated with collecting and handling VOC samples, recommended reading includes the "Standard Guide for Sampling Waste and Solids for Volatile Organic Compounds" (ASTM D 4547-98), published by the American Society for Testing and Materials (ASTM). (Ref. 15)

Currently, **it is recommended that VOC solid samples are to be collected, while maintaining a closed-system approach to prevent constituent losses, using an appropriate coring device and immediately transferred to the VOA vial to be used for analysis and should be stored for no longer than 48 hours at 4 ± 2°C prior to analysis or preservation.** Longer storage times at 4 ± 2°C may be appropriate if it can be demonstrated that the VOC concentrations are not adversely affected or that the data generated at the time of sample analysis meets the project-specific data quality objectives. Extended sample storage, up to 14 days from sample collection, may be obtained by either physical or chemical preservation techniques as noted in this Appendix guidance. These preservation techniques can be initiated at the time of sample collection or after arrival in a laboratory. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A.1.3 <u>Do all VOA samples need to be chemically preserved?</u>

No. Only samples that contain analytes that are subject to biological degradation prior to analysis need to be preserved. Samples where aromatic hydrocarbons are target analytes, which are most subject to biological degradation, need to be preserved, unless they are to be analyzed immediately on-site, even if other VOA compound classes are present. Preservation may be inappropriate for highly reactive compounds, e.g., styrene, vinyl chloride, since it may accelerate loss by polymerization or other rapid chemical reaction. Samples for which chlorinated aliphatic hydrocarbons are the only target analytes generally do not need to be preserved. However, all aqueous samples containing free chlorine must be preserved with a dechlorinating agent in order to prevent formation of trihalomethanes and other possible chemical reactions.

A.1.4 Who is the intended audience for this Appendix?

VOCs are frequently Resource Conservation and Recovery Act (RCRA) Program analytes of concern, and thus waste management decisions are often based on characterization of the VOC levels. The intended users of this Appendix guidance are those individuals involved in any way in the collection and preparation of samples for VOC analysis during the characterization of solid materials under RCRA. This may include:

- field sampling personnel
- laboratory analysts
- environmental project managers, whether at a facility regulated under RCRA, or working for a regulatory agency
- Federal, state, and local regulators with oversight responsibilities for sample collection activities
- quality assurance personnel
- data quality assessors.

A.1.5 What does this guidance *not* cover?

This Appendix does *not* provide detailed guidance regarding sampling design or the actual steps in sample preparation and VOC determination in the laboratory. For such guidance, users of this manual should refer to Chapter Nine of SW-846 and the preparation and determinative methods that are selected for analysis as part of the planning process in order to meet the intended data quality objectives.

A.1.6 What equipment is needed?

The site-specific Sampling and Analysis Plan should clearly list the required sample collection equipment necessary to ensure that the loss of volatile constituents will be minimized during the sample collection process. As with all environmental sampling applications, the analytical data usability and representativeness will be affected by improper sample collection techniques. Sampling personnel will be responsible for ensuring that VOA vials are sealed properly using a septum of sufficient thickness without any punctures. The improper vial sealing (i.e., due to excess sample retained on the vial threads) and tightening of caps are the primary factors in the loss of volatiles due to sample collection activities. Care should also be exercised in the selection of approved pre-cleaned and certified VOA vials absent of burrs on the glass. Procedures should be in place for the selection and appropriate use of sample collection devices (i.e., bailer, coring tool, etc.) along with the required decontamination measures. It is also recommended to store one trip blank per cooler when collecting volatile samples in order to assess possible field induced contamination.

A.1.7 How is the guidance organized?

This Appendix is organized as follows:

Section A.2.0 - Project Planning -- Provides an overview of the data quality objectives (DQOs) process as related to the suggested project planning activities prior to sample collection.

Section A.3.0 - Aqueous Sample Matrices and Volatile Organic Compounds – Outlines the appropriate sampling and preservation strategy for aqueous sample matrices.

Section A.4.0 - Solid Materials/Cohesive Soils and Volatile Organic Compounds -- Describes the two most common mechanisms (volatilization and biodegradation) for potential VOC losses during the sample collection process.

Section A.5.0 - History of Practices in the Sampling and Preparation of Solid Materials for VOC Analysis – Provides a summary of the common historical VOC loss mechanisms and discusses the improvements and new developments in sample collection techniques.

Section A.6.0 - Overview of Vapor Partitioning and Methanol Extraction Technologies – Discusses the two most commonly used methods for the laboratory preparation of soils for VOC analysis.

Section A.7.0 - Sample Collection – Describes the sample collection and storage process for various solid matrices.

Section A.8.0 - Approaches to Sample Preparation -- Provides examples of several sample preparation techniques that may be appropriate based on the intended use of the data.

Section A.9.0 - Summary of Findings – Lists the key highlights as discussed in Sections A.2.0 through A.8.0.

Section A.10.0 - References

A.2.0 PROJECT PLANNING

The EPA requires that a systematic planning process such as, but not limited to, the Data Quality Objectives (DQOs) Process be used for all EPA environmental data collection activities. Systematic Planning is necessary to define the type, quantity, and quality of data a decision maker needs before collecting or generating environmental data. As part of the DQO process, questions such as "what are the possible sample matrices?," "why is the sample being collected?," and "what are the appropriate analytical methods?" can be answered based on the intended use of the data. The Systematic Planning process should also include the preparation of a Quality Assurance Project Plan (QAPP) along with a site-specific Sampling and Analysis Plan (SAP) prior to any sample collection activities. Refer to *Guidance for the Data Quality Objectives Process* (G-4) (August 2000, EPA/600/R-96/055), *Guidance for Quality Assurance Project Plans* (G-5) (February 1998, EPA/600/R-98/018) and Chapter Nine of SW-846 for guidance on how to perform the DQO process and planning guidance associated with RCRA waste sampling and analysis.

During the project planning period it is important to stress to all interested parties that any samples identified as a result of the planning process must be representative of the material subject to investigation, and that each sample handling activity can affect sample integrity and representativeness up through analysis (e.g., VOCs can be lost if samples are not appropriately collected and preserved [See Sec. A.1.3]).

The EPA encourages the use of a performance-based measurement system (PBMS) during selection of sample collection and preparation approaches. The EPA defines PBMS as "a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified, and serve as criteria for selecting appropriate methods to meet those needs in a cost effective manner." The PBMS process permits the use of any appropriate method that demonstrates the ability to meet established criteria while complying with specified data quality needs. In addition, analysts must generate initial and continuing method performance data that demonstrate that the selected approaches were appropriate. Implementation of PBMS does not negate the need for or use of standard or consensus methods. It only eliminates the mandate that they be used exclusively. The following are typical items that should be considered during selection of approaches to VOC sample collection and preservation:

1. VOC concentration range.

2. VOC constituents of interest.

3. Physical characteristics of material, i.e., water content and particle size distribution.

4. Chemical and biological characteristics of material, i.e., acid/base properties, chlorine residual, carbonate content, and microbial activity.

5. Compatibility with selected preparation method.

6. Holding time constraints.

7. Data quality requirements.

A.3.0 AQUEOUS SAMPLE MATRICES AND VOLATILE ORGANIC COMPOUNDS

All environmental aqueous samples are physically preserved at 4 ± 2°C immediately after collection in order to improve the overall VOC stability prior to analysis. This preservation process alone has been shown to be effective in preventing the degradation of most constituents for up to seven days from the sample collection date. Depending on the project required VOC constituents, an aqueous sample stability or holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid. The chemical preservatives act as acidifying agents to lower the sample pH and thereby inhibit microbial activity which may cause biological degradation of aromatic hydrocarbons. However, since reactive compounds such as 2-chloroethyl vinyl ether are unstable at low pHs, if these analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. In addition, aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs 48,49) (NOTE: if the aromatic constituents such as benzene, toluene, ethylbenzene, and xylenes (BTEX) are among the analytes of interest, acidification is required for biologically active samples because it has been demonstrated that losses can occur within four hours of sample collection).

The presence of free chlorine in aqueous samples must be monitored and controlled in order to prevent the possible formation of trihalomethanes and reaction with certain compounds such as styrene after sample collection. Therefore, samples containing residual chlorine should be treated with a 10% sodium thiosulfate solution or ascorbic acid prior to acidification in order to reduce the chlorine to unreactive chloride.

Details of procedures and protocols for sample collection must be identified in an approved sampling plan. Aqueous samples for volatile constituents should be collected in vials or containers specifically designed to prevent loss of analytes. In most cases, containers should be provided by the laboratory conducting the analysis. If chemical preservation is required and the laboratory has not pre-preserved the containers, add the appropriate preservative prior to sample collection. Store empty VOC containers on ice in order to reduce potential volatilization while they are being filled. During the sample collection process do not rinse the container before filling and take care to minimize sample overflow that may dilute the preservative. The container should be filled until the water sample forms a positive meniscus at the brim. At this point the container is capped immediately to prevent bubbles and headspace. After the sample has been collected and the container capped, the formation of bubbles can be verified by inverting and lightly tapping the side of the container. Sometimes it is not possible to collect a sample without air bubbles, particularly if the water is aerated. In these cases, the field personnel should record the problem and account for the probable cause. (NOTE: dechlorinating agents should not be mixed with the acid preservative prior to sample collection).

During transport and prior to analysis, samples should be stored in a cooler or refrigerator maintained at 4 ± 2°C and care should be taken to prevent freezing of the sample and possible container breakage. The sampling plan should indicate how sample shipment will occur along with method of packaging, shipping, and the time schedule relative to sample collection and analytical holding times. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A large number of water VOC sample holding time and stability studies have been performed to determine the degree of degradation which may occur at a variety of concentrations, preservation, and storage conditions. Data from these studies have been reviewed by the Oak Ridge National Laboratory (ORNL) in order to develop an approach for assessing the data

confidence from analyses completed beyond the regulatory holding time of 14 days. This approach is based on methodology, referred to as "Practical Reporting Times," that were developed by ORNL in the early 1990's, and described in a summary report listed in Ref. 47. Users may find the data provided in Tables 2 and 3 of this referenced report to be helpful in estimating the post-holding time degradation of VOCs in water and for determining the potential data impact from analyses completed beyond the required holding time. However, the user is cautioned that the holding times provided in this report are estimations based on actual analytical data, and the true values are relative to the on-site sample matrix conditions. See the footnote following Table A.1 regarding holding time extensions.

A.3.1 Alternative Considerations for Sample Holding Time Criteria

Recognition that holding times for environmental contaminants are analyte-specific and highly variable is not new. (Refs. 52,53,54). Environmental contaminants may be short-lived, destroyed by preservation, or highly resistant to degradation. Understanding and applying historical knowledge (Table A.1) can be important and valuable. (Ref. 55) Therefore, we encourage consideration of alternative holding times for several reasons:

1. Project planning,
2. Performance based data review processes,
3. Analytical method selection,
4. Streamlined verification of unexpected or suspect analytical results, and
5. Design of alternative quality control procedures.

Specific examples of how to implement the information incorporated in Table A.1 include the following: During project/systematic planning, field measurement or quick-turn-around analyses must be identified as critical if particular contaminants of concern for a project are easily lost or destroyed. Currently, data review guidelines suggest samples analyzed within 2 weeks of collection be accepted as uniformly reliable, and analyses completed >2 weeks after sample collection are uniformly assessed as unacceptably uncertain. This review judgement is not technically defensible. Many of the most common contaminant decision drivers listed in Table A.1 are important, because they are stable over time, e.g., chlorinated solvents. For these contaminants, cooperative Inter-Agency research has demonstrated no significant change in results from analyses performed at 30 days, often as long as 96 days, after collection and preservation. *NOTE: this extension assumes preservation of samples as identified in Table A.1.* In addition, longer holding times than those specified in Table A.1 may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

The resistance to degradation of these frequent environmental drivers offers additional process improvement opportunities. Utilization of a second VOA sample analyzed beyond the recommended holding time is a mechanism to verify or independently determine unexpected results or correct laboratory errors that cannot be addressed within the current 2-week window. With no significant loss of confidence in the results, this approach eliminates the schedule delays and expense of sampling crew mobilization.

In addition, the use of site-specific performance evaluation material is recognized as a high confidence mechanism to ensure reliability of project data. However, the historical perception of short shelf-life for volatile organics in water eliminates implementation of this approach as a viable

quality control/quality assurance system component for water monitoring programs. Table1 and the associated references contain documentation of appropriate analytes and procedures to develop and implement these alternatives.

Table A.1
Recommended VOC Sample Preservation Techniques and Holding Times

Sample Matrix	Preservative	Holding Time	Comment
Aqueous Samples With No Residual Chlorine Present	Cool to 4 ± 2°C.	7 days	If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples With No Residual Chlorine Present	Cool to 4 ± 2°C and adjust pH to less than 2 with HCl or solid $NaHSO_4$.	14 days[1]	Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
Aqueous Samples With Residual Chlorine Present	Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to 4 ± 2°C.	7 days	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples With Residual Chlorine Present	Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to 4 ± 2°C and adjust pH to less than 2 with HCl or solid $NaHSO_4$	14 days[1]	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. Caution: never add acid preservative directly to a dechlorinating agent prior to sample collection.
Solid Samples[2]	Sample is extruded into an empty sealed vial and frozen on-site to < -7°C.	14 days[1]	Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Table A.1 (Continued)

Sample Matrix	Preservative	Holding Time[1]	Comment
Solid Samples[2]	Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C for no more than 48 hours then frozen to < -7°C upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C for no more than 48 hours then preserved with methanol upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not preserved with methanol prior to the expiration of the 48 hour period.
	Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C.	48 hours	
	Cool to 4 ± 2°C the coring tool used as a transport device	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7°C or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing.
	Freeze to < -7°C the coring tool used as a transport device	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7°C or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing.
	Sample is extruded into a vial containing reagent water and frozen on-site to < -7°C.	14 days[1]	Sample vials should not be frozen below - 20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into a vial containing reagent water and cooled to 4 ± 2°C for 48 hours or less then frozen to < -7°C upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Table A.1 (Continued)

Sample Matrix	Preservative	Holding Time[1]	Comment
Solid Samples[2]	Sample is extruded into a vial containing reagent water and 1 g $NaHSO_4$ and cooled to 4 ± 2°C.	14 days[1]	Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
	Sample is extruded into a vial containing methanol and cooled to 4 ± 2°C.	14 days[1]	Additional methanol extract storage time beyond 14 days may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

[1] A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

[2] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

A.4.0 SOLID MATERIALS/COHESIVE SOILS AND VOLATILE ORGANIC COMPOUNDS

During the selection of VOC sample collection and preservation approaches, it is important to understand the mechanisms of VOC loss inherent to solid materials and VOCs. In general, uncontrolled losses occur through both volatilization and biodegradation. However, for some compounds, e.g., vinyl chloride, acrylonitrile, 2-chloroethylvinyl ether, and styrene, rapid losses can occur through chemical reaction, as well. (Ref. 46)

In most solid materials, VOCs coexist in gaseous and liquid phases, as well as sorbed to the solid particles. The molecular diffusion coefficients of VOCs in the gaseous phase are high enough to allow for the immediate volatilization of those VOCs from a freshly exposed sample surface, resulting in a loss to the surrounding atmosphere. If the sample matrix is porous, these losses will continue as VOCs below the surface diffuse outward. Furthermore, once the gaseous phase is lost, the dynamic equilibrium between the gaseous phase and the liquid and sorbed VOC phases will result in rapid transformations of the liquid and sorbed VOCs to the gaseous phase, where they can continue to be lost to the atmosphere. (Ref. 4) Thus, the primary goal of preservation is to minimize or eliminate the loss of the compounds of concern through direct volatilization to the atmosphere.

The biodegradation of VOCs usually involves compound loss by biological processes mediated by naturally-occurring micro- and macro-organisms found in solid environmental samples such as soils and sediments. Aerobic processes are often of greatest concern, but anaerobic organisms in some sediments and soils can also result in significant losses of VOCs. Biodegradation may be of concern in waste samples, particularly those that may have been stored outdoors.

Most soil sample collection procedures involve intrusive sampling operations that can create or enhance aerobic conditions within a sample. Aerobic conditions can occur by disaggregation of the particles in the solid, or by simple exposure of the sample to air (e.g., collection of a sediment sample from under standing water). Soil samples should be collected immediately or as soon as practical after exposure of the soil during such activities as tank removal or excavation in order to minimize VOC losses from uncontrolled aerobic processes. Unless precautions as noted in this Appendix are employed, aerobic conditions will then persist during handling and storage of the sample.

The rate of biodegradation is dependent on several factors, including the indigenous microbes, the chemical properties of the individual VOC, the total VOC concentration, the chemical properties of the solid matrix, and temperature. In general, the biodegradation mechanism for soil VOCs is not as large a source of determinate error as volatilization. Volatilization losses of an order of magnitude can occur in minutes to hours, whereas losses of a similar magnitude due to biodegradation usually take days to weeks.

Biodegradation is compound selective whereby, under aerobic conditions, the biological mechanisms favor the degradation of aromatic hydrocarbons over the loss of halogenated (chlorinated) hydrocarbons. (Refs. 1,2,4) Aromatic hydrocarbons such as benzene, toluene, ethyl benzene, and xylenes (collectively referred to as BTEX) can be lost in days from samples stored at $4 \pm 2^\circ$ C, while losses of chlorinated hydrocarbons by biodegradation over the same period can be relatively insignificant. Major benzene and toluene biodegradation losses (50% or more) have been observed when soils are stored at room temperature (22°C) for five (5) days and near complete concentration reduction when stored for fourteen (14) days at 4°C. (Refs. 1,2,4,6,11,17) For extremely biologically active soils this can occur in less than five days. (Ref. 11)

Due to the above mechanisms, attempts are made from the beginning to maintain sample integrity and representativeness. In doing so, approaches often use various combinations of chemical (e.g., methanol) and physical (e.g., freezing) preservation procedures and collection (e.g., single transfer to air-tight vial) and storage practices (e.g., holding times) to minimize VOC loss. Some of these approaches are presented within this guidance.

A.5.0 HISTORY OF PRACTICES IN THE SAMPLING AND PREPARATION OF SOLID MATERIALS FOR VOC ANALYSIS

A.5.1 Traditional Practices

Over the past 20 years, solid samples obtained for VOC analysis were collected using a spatula -type device to completely fill a container for transfer off-site before the introduction of certain preparation steps and analysis within a 14-day holding time. VOC sampling procedures recommended the use of clean stainless steel utensils to completely fill either 40-mL to 250-mL glass containers. The containers were then closed with polytetrafluoroethylene (PTFE)-lined caps. Sample containers were stored in coolers at 4 ± 2° C and shipped to field or off-site support laboratories for subsampling (usually with 1 to 5 g aliquots) and subsequent analysis. The common holding time for these bulk soil samples, held at 4 ± 2°C, was 14 days.

During the 1990s, research efforts demonstrated that the above VOC bulk sampling procedure is inaccurate and produces VOC results that are biased low. (Refs. 3,8,10,16,30,31,32,33,34) The studies showed that bulk samples can lose 90% or more of their VOC content prior to analytical measurement. (Refs. 3,8,10,16,29,31,32,33) Reasons identified for these losses include:

1. Volatilization from exposure of the solid surface near the time of collection. (Refs. 3,8)

2. Volatilization from intermediate storage containers (e.g., core barrel liners, plastic bags, etc.). (Refs. 4,10,13,17,30)

3. Volatilization from disaggregation of the solid during collection. (Refs. 3,8)

4. Volatilization from failed seals on the PTFE-lined caps of the bottles or volatile organic analyte (VOA) vials (can be caused by soiling of cap and bottle ring closures during filling of containers). (Refs. 3,8)

5. Volatilization during laboratory subsampling of the bulk samples. (Refs. 3,8)

6. Biodegradation (principally of aromatic hydrocarbons, especially benzene and toluene) during storage (probably hastened by disaggregation of soils during sampling). (Refs. 3,8,11)

7. Reaction of chemically reactive compounds during sample storage.

8. Pressure changes during sample collection and transport.

A.5.2 Improvement of Sample Collection Techniques

Due to concerns regarding the loss of VOCs, particularly in samples containing low concentrations of VOCs (<200 µg/kg) during traditional sampling practices, the scientific community investigated other approaches to VOC sample collection and preparation. A closed-system purge-and-trap technique was developed and tested for the analysis of low-level concentrations of VOCs in solids. The methanol extraction option for high concentrations (>200 µg/kg) and oily wastes remained unchanged. The Office of Solid Waste promulgated Method 5035 as part of Update III to the Third Edition of SW-846 on June 13, 1997. (Ref. 38) As an active participant in these studies in conjunction with OSW, the American Society for Testing and Materials (ASTM) published the

"Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds" (ASTM D 4547-98). (Ref. 15)

These documents include the immediate in-field transfer of the sample (by a coring tool of 2 to 5 g capacity) into a tared VOA vial (of 22 to 40 mL capacity) that contains acidified reagent water (most often acidified by 1g $NaHSO_4$ per 5g of soil) so that a vapor partitioning preparation procedure (see Sec. A.6.0 of this Appendix) can be performed by the laboratory on the sample without reopening the vial. A second in-field transfer to a tared VOA vial containing 5 to 10 mLs of methanol is used for VOC soil concentrations larger than 200 µg/kg.

Another technique described is the immediate in-field collection and maximum 48 hour storage in an air-tight coring device/container (such as the EnCore™ sampler) so that the laboratory preservation and preparation procedures described for the closed-system purge-and-trap (Method 5035) or headspace (Method 5021) can be performed. (Ref. 39) (An EnCore™ sampler is a device that can be used for both sample collection and as the sample transport and storage device. See Sec. A.8.0)

Both documents recommend similar approaches to sample preservation and preparation in order to minimize VOC loss and address the collection of cohesive solids whereby a coring tool collects a relatively undisturbed sample by compression, and then extrudes the sample into an appropriate VOA vial. The documents also provide guidance for the collection of cemented materials and non-cohesive materials (e.g., dry sand, mixtures of gravel and fines) and collectively address factors that must be considered when selecting the most appropriate approach for VOC sample preservation and preparation, including expected concentrations of VOCs (high versus low). A screening method for determining whether a sample contains high or low concentrations of VOCs (Method 3815) is available for making these determinations on-site. (Ref. 40)

A.5.3 New Developments

Since the publication of the new VOA sampling techniques for solids, the scientific community has continued to investigate additional techniques to further improve sample collection and preservation to minimize VOC loss. For example, studies were conducted regarding the freezing of samples without the use of chemical preservatives (see Sec. A.8.0), use of "empty VOA vials," and more information was gained regarding acidification of samples, as discussed below. (Refs. 4,19)

Current practice recommends the use of $NaHSO_4$ to acidify reagent water in VOA vials prior to addition of the sample when preservation is necessary. (Ref.1) This acidification is one means used to minimize loss of VOC due to biodegradation. However, acidification is not recommended for solids or aqueous samples with significant levels of carbonates, because the acidification can cause effervescence and the loss of VOCs. In 1998 and 1999, other adverse effects of acid preservation of soils were discovered i.e., chemical breakdown of certain classes of compounds. Additionally, certain VOC components such as 2-chloroethylvinyl ether are lost by the acidification. An artifact is sometimes observed for acetone in that acidification of certain soils may cause the formation of acetone. (Refs. 4,37)

The approaches recommended in Sec. A.8.0 of this guidance incorporate the new developments in solid sample preparation for VOC analysis.

A.6.0 OVERVIEW OF VAPOR PARTITIONING AND METHANOL EXTRACTION TECHNOLOGIES

Vapor partitioning and methanol extraction are the two most commonly used methods for the laboratory preparation of soils for VOC analysis. This section briefly discusses these two procedures, and their relative advantages and disadvantages. For further information, ASTM D 4547-98 (Ref. 15) discusses the merits of vapor partitioning relative to the use of methanol extraction; and Method 5035 relates concerns regarding the use of methanol.

Selection of the preparation technology should be made during the systematic planning process prior to sample collection given that the selection will dictate subsequent sample collection and preservation practices. One technology may be preferred based on the project data quality objectives and target analytes, and the sample collection and handling approaches need to be compatible with the chosen technology.

Each preparation technology involves use of a VOA vial for sample collection and transport. Approaches for preparation of the vials (with and without preservatives), often based on the technology to be used, will be discussed in a section to follow.

A.6.1 Vapor Partitioning

One means of vapor partitioning involves the direct analysis of a sample by purge-and-trap (Method 5035). This technique is routinely used for the analysis of volatiles in environmental samples and is considered more sensitive than the headspace technique. By purging samples at higher temperatures, higher molecular weight compounds can be detected. However, the purge-and-trap technique requires more time for sample preparation.

Another means of vapor partitioning involves the direct analysis of a sample by equilibrium headspace (Method 5021). This technique is most suited for the analysis of very light molecular weight volatiles in samples that can be efficiently partitioned into the headspace gas volume from the liquid or solid matrix sample. Higher boiling point volatiles are not detected with this technique due to their low partition rate in the gas headspace volume. In addition, the technique is generally less sensitive than purge-and-trap, however, it is preferred for the analysis of gases, highly water-soluble compounds, and very light molecular weight volatiles which may not be analyzed using the purge-and-trap technique.

For both vapor partitioning techniques, the vapor is removed for analysis without opening the container. Heat and water are usually used to assist in the direct partitioning of VOCs from the solid matrix. Vapor partitioning is applicable to VOC soil concentrations of 2 to 200 ppb. Methods 5021 or 5035 commonly require 2- to 5-g soil aliquots collected in individual 20- to 40-mL VOA vials, depending on the specific instrumentation used in the selected purge-and-trap or headspace method. Only one analysis per VOA vial can be done using purge-and-trap or headspace (Methods 5035 or 5021).

Vapor partitioning can offer lower detection limits than methanol extraction because no dilution is involved. In addition, there are no organic solvent interferences and no use of regulated organic solvents (e.g., methanol), which requires special handling and disposal practices. Use of methanol may generate a flammable waste that is hazardous based on the ignitability characteristic (40 CFR § 261.21) or a listed waste (40 CFR § 261, Appendix VII).

A.6.2 Methanol Extraction

Methanol extraction involves the extraction of VOCs from a sample with methanol, and the subsequent transfer of an aliquot of the extract to water (dilution) for either purge-and-trap or headspace analysis. After extraction with methanol (anywhere from 1:1 methanol to soil to a 10:1 methanol to soil ratio); the extract typically receives a 50-fold dilution. Methanol extracts must be diluted to minimize adverse effects of methanol on analytical instrumentation. However, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report results for samples containing significant moisture contents on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

One advantage of a methanol extract is it may be tested more than once. Methanol extracts of soil are applicable to a wide range of high to low concentrations, e.g., 50 ppb to several ppm. Once a methanol extract is obtained, it can be stored at 4 ± 2 °C for two weeks, and sufficient volume is present for multiple VOC determinations. Additional extract storage time beyond two weeks may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

As noted above, concerns exist regarding the use of methanol extraction. The information to follow provides recent observations regarding the use of methanol for VOC analysis.

A.6.2.1 Contact Time Effect

Methanol extraction can provide more robust, larger or accurate values for VOCs when compared to vapor partitioning results. (Refs. 5,9,16,27,29,30,32,33,41,42) However, methanol extract results tend to increase with time as the sample contact time increases. (Refs 27,33) State agencies implementing methanol extraction for soil VOCs often require either a minimum contact time of one day, or the soil is to be sonicated for 20 to 30 minutes at 40° C with the methanol prior to analytical measurement of VOCs. The actual contact time should be sufficient enough to efficiently extract all VOC constituents of interest and to allow for the complete breakdown of agglomerated solid materials.

Particularly volatile VOCs (e.g., benzene, dichloroethene) in sandy soils are not expected to show this effect of contact time. The less volatile VOCs (e.g., xylenes) in an organic rich soil or clay can be expected to demonstrate higher results with increased contact time. (Refs. 5,9,27,33)

A.6.2.2 Safety and Hazardous Waste Generation Concerns

A primary disadvantage of methanol extraction is that it poses hazards to personnel due to its toxicity and flammability. Finally, the addition of methanol to a sample is likely to cause the sample to fail the ignitability characteristic or to become a listed waste, thereby making the unused sample volume a hazardous waste.

A.7.0 SAMPLE COLLECTION

A.7.1 Collection of Samples for Analysis

After a fresh surface of the solid material is exposed to the atmosphere, the subsample collection process should be completed in the least amount of time in order to minimize the loss of VOCs due to volatilization. Removing a subsample from a material should be done with the least amount of disruption (disaggregation) as possible. Additionally, rough trimming of the sampling location's surface layers should be considered if the material may have already lost VOCs (been exposed for more than a couple of minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Removal of surface layers can be accomplished by scraping the surface using a clean spatula, scoop, knife, or shovel. (Refs. 15,51)

A.7.1.1 Subsampling of Cohesive Granular but Uncemented Materials Using Devices Designed to Obtain a Sample Appropriate for Analysis

Subsamples of the appropriate size for analysis should be collected using a metal or rigid plastic coring tool. For example, coring tools for the purpose of transferring a subsample can be made from disposable plastic syringes by cutting off the tapered front end and removing the rubber cap from the plunger or can be purchased as either plastic or stainless steel coring devices. These smaller coring devices help to maintain the sample structure during collection and transfer to the VOA vial as do their larger counterparts used to retrieve subsurface materials. When inserting a clean coring tool into a fresh surface for sample collection, air should not be trapped behind the sample. If air is trapped, it could either pass through the sampled material causing VOCs to be lost or cause the sample to be pushed prematurely from the coring tool. The commercially available EasyDraw Syringe™ and Powerstop Handle™ and Terra Core™ sampler coring devices are designed to prevent headspace air above the sample contents. For greater ease in pushing into the solid matrix, the front edge of these tools can be sharpened. The optimum diameter of the coring tool depends on the following: size of the opening on the collection vial or bottle (tool should fit inside mouth), dimensions of the original sample, particle size of the solid materials (e.g., gravel-size particles would require larger samplers), and volume of sample required for analysis. For example when a 5-g subsample of soil is specified, only a single 3-cm^3 volume of soil has to be collected (assuming the soil has density of 1.7 g/cm^3). Larger subsample masses or more subsample increments are preferred as the heterogeneity of the material increases. After an undisturbed sample has been obtained by pushing the barrel of the coring tool into a freshly exposed surface and then removing the corer once filled, the exterior of the barrel should be quickly wiped with a clean disposable towel. The next step varies, depending on whether the coring device is used for sample storage and transfer or solely for transfer. If the coring tool is used as a storage container, cap the open end after ensuring that the sealing surfaces are cleaned. If the device is to be solely used for collection and not for storage, immediately extrude the sample into a VOA vial or bottle by gently pushing the plunger. The volume of material collected should not cause excessive stress on the coring tool during intrusion into the material, or be so large that the sample easily falls apart during extrusion. Obtaining and transferring a sample should be done rapidly (<10 seconds) to reduce volatilization losses. If the vial or bottle contains methanol or another liquid, it should be held at an angle when extruding the sample into the container to minimize splashing. Just before capping, a visual inspection of the lip and threads of the sample vessel should be made, and any foreign debris should be removed with a clean towel, allowing an airtight seal to form.

A.7.1.2 Devices that Can be Used for Subsampling a Cemented Material

The material requiring sampling may be so hard that even metal coring tools cannot penetrate it. Subsamples of such materials can be collected by fragmenting a larger portion of the material using a clean chisel to generate aggregate(s) of a size that can be placed into a VOA vial or bottle. When transferring the aggregate(s), precautions must be taken to prevent compromising the sealing surfaces and threads of the container. Losses of VOCs by using this procedure are dependent on the location of the contaminant relative to the surface of the material being sampled. Therefore, caution should be taken in the interpretation of the data obtained from materials that fit this description. As a last resort when this task can not be performed onsite, a large sample can be collected in a vapor-tight container and transported to the laboratory for subsampling. Collecting, fragmenting, and adding the sample to a container should be accomplished as quickly as possible.

A.7.1.3 Devices that Can be Used for Subsampling a Non-cohesive Granular Material

As a last resort, gravel, or a mixture of gravel and fines that can not be easily obtained or transferred using coring tools, can be quickly sampled using a stainless steel spatula or scoop. If the collection vial or bottle contains methanol or an aqueous solution, samples should be transferred with minimal splashing and without the spatula or scoop contacting the liquid contents. For some solids, a wide-bottom funnel or similar channeling device may be necessary to facilitate transfer to the container and prevent compromising of the sealing surfaces of the container. Caution should be taken in the interpretation of the data obtained from materials that fit this description. Losses of VOCs are likely because the nature of the sampling method and the noncohesive nature of the material expose more surface area to the atmosphere than other types of samples. During the sampling process, noncohesive materials also allow for the separation of coarser materials from fines, which can skew the concentration data if the different particle sizes, which have different surface areas, are not properly represented in the sample.

A.7.2 Use of the EnCore™ Sampler (or Equivalent) for Sample Transport and Storage

The EnCore™ sampler is a sampling device that can be used as both a simultaneous coring tool for cohesive soils and a transport device to a support laboratory (field or off-site). The EnCore™ sampler is intended to be a combined sampler-storage device for soils until a receiving laboratory can initiate either immediate VOC analysis, or preserve extruded soil aliquots for later VOC analysis. It is meant to be disposed after use. The commercially available device is constructed of an inert composite polymer. It uses a coring/storage chamber to collect either a 5 g or 25 g sample of cohesive soils. It has a press-on cap with hermetically vapor tight seal and locking arm mechanism. It also has a vapor tight plunger for the nondisruptive extrusion of the sample into an appropriate container for VOC analysis of soil.

An individual disposable EnCore™ sampler (or equivalent) is needed for each soil aliquot collected for vapor partitioning or methanol sample preparation. Upon soil sample collection, the EnCore™ sampler is stored at 4 ± 2°C until laboratory receipt within 48 hours. Upon laboratory receipt, soil aliquots are extruded to appropriate tared and prepared VOA vials.

Validation data have been provided to support use of the EnCore™ sampler for VOC concentrations in soil between 5 and 10 ppm, for two (2) sandy soils, with a 2-day holding time at 4 ± 2°C. Preliminary data (Ref. 25) demonstrate an effective 2-day (48-hour) holding time at 4 ±

2°C for three sandy soil types with VOC concentrations at 100 ppb (benzene and toluene at 300 ppb), as well as an effective 1 or 2 week holding time at -12°C (freezing temperature). Recent published work (Ref. 43) neither definitively supports or shows the EnCore™ device to be ineffective for sample storage at these preservation temperatures. Soils stored in the EnCore™ device for 2 calendar days at 4 ± 2°C are subject to loss of BTEX compounds by biodegradation if the soil is an aerated, biologically active soil (e.g., garden soil) (Ref. 24), but this BTEX loss is eliminated for up to 48 hours under freezing conditions. (Ref. 2)

Further details on the EnCore™ sampler can be found in ASTM D4547-98 (Ref. 15) or other publications.

A.7.3 Concerns Regarding Use of Core Barrel Liners

One geotechnical technique for retrieval of bulk soil from subsurface regions is ring-lined barrel samples. Core barrel liners fit snugly within a corer and can be constructed of steel or brass (which is inert to VOCs). Cylindrical cores of subsurface soil can be obtained anywhere from 1 to 4 inches in diameter of varying lengths in feet.

Core barrel liners have been used as both a sample collection and storage device for VOC soil samples. Upon retrieval with subsurface soil, the core barrel liner (brass) is covered on both ends with a thin sheet of PTFE or with aluminum foil. Plastic caps are pressed over the ends to hold the PTFE/aluminum in place. The core barrel liner sample is maintained at 4 ± 2°C during shipment and storage at a laboratory. Sample preparation for VOC analysis is initiated by opening the core barrel coverings and sub-sampling the soil with a coring tool for analyses by either the vapor partitioning or methanol extraction options.

Experimental work has demonstrated that the core barrel transport and storage procedure is ineffective for a 2-day storage and holding time. (Refs. 4,10,13,16, 36) PTFE coverings (0.02 mm and 0.05 mm thickness) and aluminum foil will not prevent losses of 30-90% for certain volatile compounds (dichloroethene, benzene and trichloroethene). Therefore, the core barrel liners should be used as sample collection and transfer devices only with the least amount of elapsed time as possible prior to sample preparation.

A.7.4 After Collection -- Sample Handling and Storage

A.7.4.1 Holding Times

Published holding times should be followed, unless performance data can be produced to support longer time periods.

This guidance assumes a 48-hour holding time, unpreserved at 4 ± 2°C, between sample collection and analysis or preservation of VOC soil aliquots in VOA vials. Most validation data provided to support or justify an approach listed the holding time as 48 hours. The 48-hour holding time results for VOC in soil can provide average recoveries of 80% or more. However, recoveries from samples stored for 5 days are less successful. Little data exists on the impact of holding times between 48 hours and 5 days.

Implementing a 48-hour holding time can be difficult when transporting VOC soil samples (via overnight air carrier) from the field to an off-site support laboratory. All interested parties i.e., field and laboratory personnel need to be cognizant that the 48 hour holding time begins **from the time of sample collection**. If the VOC analysis cannot be completed prior to the expiration of the initial 48 hour period, other

preservation measures (i.e., freezing, chemical preservation, and methanol extraction) are required in order to extend the analysis holding time to 14 days from the time of sample collection.

A.7.5 Quality Control

Quality control checks to be employed during field sampling activities should include the collection, preparation, and analysis of the various QC samples discussed below:

Note: The exact specifications and need for the following QC samples should be outlined in the project planning documents.

1. Field duplicate: A field duplicate may be prepared at a frequency of one per day per matrix. The field duplicate is an independent sample which is collected as close as possible to the same point in time and space as the primary field sample. Field duplicates are used to estimate the reproducibility (precision) of the sampling process.

2. Trip blank: Trip blanks should be prepared at a frequency of one per day of sampling during which samples will be collected for VOCs. Trip blanks are prepared using reagent water (see Chapter One for definition) prior to the site visit at the time sample containers and kits are transported to the site. The trip blank will accompany the field samples throughout all sample collection and transport operations. This blank will not be opened during sampling activities and will be used to assess sample contamination originating from sample transport, shipping, or site conditions.

3. Field blank: A field blank conversely is prepared on-site during the sample collection activities using the same reagent water source used to prepare the trip blank. The field blank should be collected and preserved in the same manner as the environmental samples. The results from this analysis are used to assess sample contamination originating predominantly from field sampling conditions.

4. Equipment rinsate: An equipment rinsate blank should be collected from sample collection devices used for each distinct sample matrix. The equipment blanks are obtained either prior to or during sample collection activities. The results from these analyses are used to assess possible sample contamination from sampling equipment.

5. Temperature blank: A temperature blank prepared with a water-filled vial or a suitable thermometer, should be included with each cooler of samples designated for transport. Upon sample receipt, the laboratory will use the temperature blank or thermometer to determine the internal temperature of each cooler. Acceptable temperatures are $4 \pm 2\,^{\circ}C$ for refrigerated aqueous and solid samples and $< -7\,^{\circ}C$ for frozen solid samples.

6. Matrix spike and matrix spike duplicate: Additional sample aliquots should be collected when matrix spike and matrix spike duplicate analyses are required. Matrix spikes are aliquots of environmental samples to which known concentrations of certain target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. The matrix spike analysis is used to assess the performance of the method by measuring the effects of interferences caused by

the sample matrix and reflects the accuracy of the method for the particular matrix in question.

7.6 Interferences / Artifacts of Analysis

When aqueous and solid samples are acidified it can lead to losses of highly reactive compounds such as 2-chloroethylvinyl ether through chemical reaction. Additionally, acidification of certain soils with sodium bisulfate may produce a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) Furthermore, *meta*- and *para*-xylene co-elute on most analytical columns and need to be reported as an isomeric pair. Acid preservation of samples to be analyzed for methyl *tert*-butyl ether (MTBE) should be avoided because use of a high temperature sample preparation method (Methods 5021, 5030, or 5032) can cause degradation of the MTBE to *tert*-butyl alcohol (TBA) during the high temperature sample preparation step. (Refs. 49,50)

Since aqueous samples containing residual chlorine must be dechlorinated to prevent the formation of trihalomethanes and other chlorinated compounds, the sample should be added to the dechlorinating agent prior to acid preservation. The addition of sodium thiosulfate to an acidified sample will generate sulfur dioxide which may interfere with the determination of gaseous VOC constituents of interest.

The project chemist should research and review historical data pertaining to the use of VOCs at the site under investigation. If previous data indicates that tetrachloroethylene or trichloroethylene were used at the site and their daughter products dichloroethene and dichloroethane are present, then vinyl chloride may also be present. In this scenario acid preservation would not be appropriate due to the reactive nature of vinyl chloride.

If the sampling location is known to contain polymers that were manufactured from monomers, then both vinyl chloride or styrene could be present. For this situation, due to the potential for reactive compounds present, acid preservation would not be necessary.

Pre-testing of a representative soil sample, prior to collection, with acid or bisulfate may show effervescence if carbonaceous materials are present. If bubbling occurs during chemical preservation, samples should not be collected with acid or bisulfate preservative. If the soil sample is a loamy material or contains a high proportion of decayed matter then acid preservation may generate acetone as a byproduct. The sampling personnel should examine and pre-test the soils to be collected prior to actual collection in order to make the proper determination for the correct preservation technique.

The laboratory should fully document whenever sample matrix interferences are suspected and can be attributed to poor analytical method quality control data. It is also important for the laboratory area where volatile analyses are performed to be completely free of solvents. Special precautions must be taken for the analysis of methylene chloride, since random background levels will result if the analytical and storage areas are not isolated from all sources of atmospheric methylene chloride.

A.8.0 APPROACHES TO SAMPLE PREPARATI0N

This section provides examples of approaches to sample preparation that include prepared vials (e.g., chemical preservation approaches) and use of empty vials (other means such as freezing used for preservation). Complete validation data is not available for all approaches. Analysts are responsible for showing that any given approach is appropriate for the intended use of the data.

Typically, as part of these procedures, a cohesive soil subsample (2 to 5g) from a freshly exposed sampling trench, geotechnical coring device/probe, etc., using a coring tool such as a cut-off syringe or purchased device (e.g., EasyDraw Syringe™ and Powerstop Handle™ or EnCore™), is extruded immediately to either a tared empty VOA vial or to a tared prepared VOA vial. Precautions with handling tared vials i.e., not applying additional labels, markings, and seals are necessary to ensure an accurate sample weight. Once filled with sample, the VOA vials are then capped (with PTFE-lined septa) until VOC sample preparation. Three or more replicate VOA vials (e.g., two for vapor partitioning and additional ones for any matrix spike QC analysis) are utilized by either technique, as well as one more soil aliquot for a percent moisture determination. One coring tool (disposable or reusable) can be used at each soil sampling location by providing co-located cores for the replicate VOA vials. The same coring tool can be used to collect an additional co-located soil for the percent moisture determination typically required by the laboratory preparation procedures. If the coring tool can be properly capped to prevent moisture loss, the coring tool can be used as a storage container for percent moisture. Note: should freezing be used as a means to preserve samples in the field, the aliquot reserved for percent moisture determination should not be frozen.

The preparation of samples for VOC analysis can be initiated either in the field at the time of collection using the prepared VOA vials, or at either an on- or off-site support laboratory using either the empty VOA vials (note the manual puncture of septa to introduce reagent water prior to analysis is not recommended) or a coring tool (e.g., the EnCore™ sampler) that can also serve as a sample transport device. A separate EnCore™ sampler is required for each replicate VOA vial used for VOC analysis.

When determining VOCs over the complete concentration range of ppb to several ppm, four (4) or more VOA vials may be required for each sampling point. For example, at least one VOA vial is necessary for methanol extraction when selected to analyze high VOC concentrations, while at least two vials are necessary for when vapor partitioning is to be used because low VOC concentrations (<200 ppb) are expected. A fourth VOA vial may be necessary for percent moisture determination so that VOC concentrations can be corrected for moisture content and/or methanol dilution factor, if required. A set of replicates for a single investigative soil sample are often composed of the following:

1. Two (2) 40-mL VOA vials for direct vapor partitioning measurement. These are needed for the most sensitive measurements - one is kept in reserve for any necessary repeat analysis. The upper concentration value of the vapor partitioning method's calibration range limits the usability of these direct measurements.

2. One (1) 40-mL VOA vial for methanol extraction of soil aliquot prior to vapor partitioning. Once a methanol extract is obtained, an aliquot of this extract is diluted fifty-fold (50) or more with water and is tested by vapor partitioning as a water matrix. The 50-fold dilution is necessary to minimize interferences in vapor partitioning measurements of water matrices. Methanol extracts have no

upper limit of measured VOC concentration since the extract can be sub-aliquoted for different dilutions.

3. One (1) 60-mL VOA vial for any percent moisture determination to report VOC results on a moisture corrected basis, if necessary. Also note that solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations. The physical preservation (4 ± 2 °C) of this vial is not as critical as for the VOC analytes in soil.

4. VOA vials for any QC audits such as duplicates, matrix spikes, etc.

Please note that a VOA vial should always be collected for methanol extraction unless it is known in advance that VOCs will not exceed the upper usable concentration values for direct vapor partitioning measurements.

Before presenting the different approaches using empty or prepared vials, a discussion is included regarding the study of the preservation of soils by freezing. As noted, this was studied using empty VOA vials. Some of the empty VOA vial approaches that follow in Sec. A.8.2 use freezing as a preservative.

A.8.1 Overview of Empty Vial Technique

Hewitt (Refs. 2, 4, 7,10) and Ricker (Refs. 19, 20, 21) independently developed “empty vial” techniques. Using a coring tool (Hewitt’s cut-off syringe or Ricker’s commercially available syringe and 5- to 13-g sample) a 5-g aliquot of undisturbed soil is transferred to a tared empty VOA vial and capped with a PTFE-lined septa (PTFE of 0.25 mm thickness). The two "empty vial" techniques were evaluated using methanol extraction (Method 5035) measurements.

The sealed vial with the soil aliquot is maintained either frozen (< -7 °C), or at 4 ± 2 °C until laboratory receipt and analysis. Multiple VOA vials can be collected, as necessary based on the sample preparation technique to be used. Sample vials should not be frozen below -20 °C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Upon laboratory receipt of VOA vials maintained at 4 ± 2 °C (within 48 hours of sample collection), one “empty VOA vial” is selected for methanol extraction and the methanol reagent is added through the septum using a glass syringe equipped with a 23-gage Luer Lock needle. The methanol is mixed with the soil and any pressure can be relieved by cracking the VOA vial’s cap once. The methanol extract, stored at 4 ± 2 °C or less, has a shelf life of up to two weeks. Upon laboratory receipt of frozen VOC samples, a vial may be thawed and methanol added through the septa as described above.

To determine VOCs by vapor partitioning, “empty VOA vials” should have 10 mLs of reagent water added, either through the septa liner by a laboratory’s automated sampler at the time of analysis, or be present in the vial prior to sample collection (see Sec. A.8.3) when Method 5035 is used. For VOC samples maintained at 4 ± 2°C this must be done within 48 hours of sample collection. Experimental work by En Novative Technologies, Inc., and Hewitt (Refs. 44, 45) indicates that VOCs are slowly lost through the pierced septa after reagent water is manually added to an

"empty VOA vial," prior to Method 5035 purge and trap measurements. To avoid any clogging of the needle of an automated purge-and-trap system, reagent water or the sodium bisulfate solution can be present in the VOA vial (Sec. A.8.3) prior to sample collection, thereby, allowing the soil/solid to be dispersed prior to the purge-and-trap analysis.

Ricker and Hewitt in their experimental work demonstrated that the empty VOA vial, with a suitable PTFE-lined septa cap, has integrity for several days. Significant VOC losses do not occur at 4 ± 2 °C through the septum of the sealed VOA vial. A 48-hour holding time for soils, at 4 ± 2 °C storage of samples, has been found effective with the "empty VOA vial" for most target VOCs studied, except for aromatic compounds in biologically active, aerated garden soils (Refs. 2, 20). Hewitt studied freezing of soils (< -7 °C) as a preservative for soils, in conjunction with the "empty VOA vial" technique and found it effective for all target VOCs studied, including aromatic compounds, so long as freezing starts at the time of collection.

When soils are maintained at 4 ± 2°C for 48 hours until freezing starts, the same condition or stability is found for the VOCs except for benzene in biologically active soil. Use of freezing at the time of lab receipt of empty VOA vials can therefore simplify sample handling of soil materials. ASTM D 4547-98 (Ref. 15) and Method 5035 briefly mention freezing, but do not endorse it because data were not available at the time of their publication to support preservation by freezing. With this approach, chemical preservatives are not needed. VOA vials, maintained at < -7 °C, need only be thawed on the day of analysis, whether it be by vapor partitioning or by methanol extraction.

A.8.2 Preservation Approaches Using Empty VOA Vials

This section provides five examples of approaches to sample preparation using empty VOA vials -- no preservatives or solutions are added to the vials.

A.8.2.1 Preservation by freezing (< -7 °C)

Upon collection, the soil is added to replicate empty vials and frozen at < -7 °C until thawed for analysis. The design of newer vials makes it possible to freeze the contents in an upright position, however, it may be advisable to place the vials on their side during the freezing process to prevent breakage. Freezing has been found effective to preserve both aromatic and chlorinated hydrocarbon VOCs in soil for two weeks at all VOC concentrations studied. (Refs. 2,4,11) Sample vials should not be frozen below -20 °C due to potential problems with vial seals and the loss of constituents upon sample thawing.

The on- or off-site support laboratory thaws a VOA vial when needed and either adds 5 or 10 mLs of methanol through the PTFE-lined septum using a 23-gage Luer lock syringe for methanol extraction and preservation. (Refs. 4,21,22) Addition of 5-10 mLs of water to the vial through the septum should not be performed, since this technique will create a punctured septum capable of producing VOC losses prior to purge-and-trap analysis.

This technique is unpopular for vapor partitioning because a prepared VOA vial with reagent water fits the operations of Methods 5021/5035 better than the empty VOA vial.

This technique can be undesirable when soil samples are transported to a support laboratory because dry ice, gel packs or salt-ice mixtures can be required to

maintain sub-zero temperature conditions during shipment. This technique has merit when freezers are available at a field site or on a sampling vessel.

A.8.2.2 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less, then preserve by freezing at < -7°C upon laboratory receipt

Upon laboratory receipt, replicate soil VOA vials are frozen (< -7°C) then thawed as needed for preparation by methanol extraction, or if possible by vapor partitioning. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. The 48-hour time period prior to freezing is practical and can be supported by the studies:

1. The chlorinated hydrocarbon volatiles that were studied have been found to be stable for two weeks at 4°C, with dichloroethene isomers not being as stable as other chlorinated compounds studied. (Refs. 1,2,4,7,11,16)

2. For spiked (at 5 ppm) typical soils, aromatic hydrocarbons demonstrate major losses at room temperature (22°C) after 5 days of storage. (Refs. 1,2,4) When these soil types are stored at 4°C, major losses occur between 10 and 14 days for aromatic hydrocarbons (e.g., benzene) spiked at 5 ppm. (Refs. 1,2,4) When these soil types are spiked at 30-40 ppb with aromatic hydrocarbons, major losses for benzene and toluene occur at 3-5 days of storage. (Refs. 2,4)

3. Aromatic hydrocarbons (such as benzene or toluene) when spiked into biologically active soil (aerated garden soil or fertilized soil) and stored at 4°C demonstrate losses of 20-30% within 48 hours. (Refs. 2,16,17,19,20). Limited disruption sampling techniques in conjunction with a maximum holding time of 48 hours can minimize this loss, but not eliminate it. Soils containing manure exhibited a major loss of aromatic hydrocarbons within one day while soil sterilization eliminated this loss. (Ref. 16)

4. Observed losses of aromatic or dichloroethene volatile compounds in soil, stored at 4°C, cease when soil is frozen at < -7°C. (Refs. 2,4).

A.8.2.3 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less, then preserve with methanol upon laboratory receipt

Upon laboratory receipt, the volume of methanol necessary for methanol extraction sample preparation is added to one of the replicate VOA vials through the PTFE-lined septum cap, using a 23-gage needle on a Luer lock syringe. Methanol will preserve VOCs in soil for 2 weeks if stored at 4 ± 2°C. See Sec. A.8.2.2 above for discussion on initial ≤ 48-hour transport at 4 ± 2°C. Certain PTFE-lined septa caps were found to be effective seals for 10 days prior to the addition of methanol. (Refs. 19,20,21)

When methanol is added through the septum cap to a soil aliquot core in an empty VOA vial, the mixture is swirled to provide contact with the soil and methanol, to wet the head space, and dissolve gaseous and sorbed VOC compounds into the methanol. At this point, there can be a pressure build-up within the vial that can be removed by cracking the VOA vial cap and immediately resealing it. (Ref. 4) There is believed not to be significant VOC loss so long as the methanol remains in contact with

the soil material. The methanol extraction efficiency can be improved by sonicating and heating the mixture at 40°C for 30 minutes followed by centrifuging and transferring the supernatant to a disposable, screw-top glass centrifuge tube. (Ref. 33)

A.8.2.4 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less and complete VOC analysis (Method 5021/5035) within 48 hours

VOC sample preparation by vapor partitioning is completed within 48 hours from sample collection. See Secs. A.8.2.2 and A.8.2.3 above for further details.

A.8.2.5 Refrigerate/freeze coring tool used as transport device for 48 hours or less (Refs. 15,26)

Each replicate soil aliquot is collected by a suitable coring device, (e.g., EnCore™) that is used as a transport device to the laboratory. Upon laboratory receipt, soil aliquots from each replicate transport device are extruded into individual empty or prepared tared VOA vials as noted in Secs. A.8.2.2 to A.8.2.4. Upon cap closure, the vial is weighed again and the wet sample weight is determined by difference.

For spiked soils characteristic of a waste site, some VOC losses were observed in 2 days for soils stored at 4 ± 2°C and losses continued further at a 5-day and 12-day storage time period. Losses during the first 2 days for aromatics and dichloroethene, were equivalent to the empty vial techniques as noted in Sec. A.8.2.2. (Ref. 4) Also, sampling of TCE contaminated soil showed reasonable agreement between the EnCore™ and cut-off syringe/empty vial techniques. (Ref. 4) Significant losses after 2 days at 4°C have been observed for the EnCore™ for biologically active soils. (Refs.16,24).

The EnCore™ sampler has been systematically evaluated for three sandy soil types (at high VOC concentrations (5 -10 ppm) and at low VOC concentrations (100 ppb). (Refs. 22,23,24,25). The EnCore™ was effective as a 2-day transport device when stored at 4 ± 2°C, for the above studies, and storage could be extended from 1 week to 12 days further under freezing conditions (< -7°C) during the low VOC concentration study. (Ref. 25) The EnCore™ was ineffective for one soil type using high concentration spikes, because the soil was non-cohesive (dry clumped sand) - any coring device could be ineffective. (Refs. 15,22) The three soils exhibited little biodegradation of aromatic hydrocarbons discussed above.

For the original EnCore™ of stainless steel construction, it was found to be the only sampling/storage device that was as effective as the original single vial technique (Dynatech vial of January 1995 draft Method 5035). (Ref. 16)

A.8.3 Preservation Approaches Using Prepared VOA Vials

This section provides four examples of preservation approaches using prepared VOA vials. During sample collection, a coring tool is used to extrude the collected sample into a VOA vial containing methanol. Co-located soil cores are extruded into replicate VOA vials containing reagent water, or reagent water acidified with 1 g $NaHSO_4$ per 5 mLs water.

Coordination between field and laboratory personnel is required so specific vials and reagents are consistent with laboratory instrumentation and reagents. Vials with reagents, and any magnetic stirring bars (e.g., for Method 5035) need be tared prior to field use. If prepared VOA vials contain methanol or water they must be tared with the septum caps and the added reagent. Once

methanol or water reagent is added, a meniscus level of the liquid in the VOA vial can be marked. This allows field personnel to note any apparent liquid loss (especially methanol) during shipment to the field. If field personnel are concerned with reagent weight loss during shipment to the field and return, individual vials can be periodically weighed after initial tare or after addition of cored soil aliquot.

A.8.3.1 Collection with reagent water, preservation by freezing (< -7°C) and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs water (Method 5035), turn vial on its side and freeze contents. It may be problematical to freeze 10 mLs of water in the 22 ml vial used for Method 5021. Maintain at < -7°C until thawed for analysis. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. Few published data exist to validate this preservation technique, but its effectiveness is inferred from Sec. A.8.2.1, and should be demonstrated by appropriate performance data results. (Ref. 28)

A.8.3.2 Collection with reagent water, preservation by refrigeration at 4 ± 2°C for 48 hours or less and immediate laboratory analysis or freezing storage at < -7°C for subsequent vapor partitioning

Sample is collected as in Sec. A.8.3.1 but transported to the laboratory within 48 hours at 4 ± 2 °C for:

1. Immediate analysis by vapor partitioning within 48 hours of sample collection.

2. Freezing at < -7°C upon laboratory receipt for vapor partitioning analysis within 2 weeks from sample collection. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

One investigator has found that a spiked hazardous waste site soil provided the same results one week after freezing with water as the initial spiked soil results. (Ref 28). Another investigator used headspace techniques with soil added to 10 mLs of reagent water to develop justification for certain variables discussed in Sec. A.8.3.2 for the initial 48-hour holding time. (Refs. 6,12) Sec. A.8.2.2 should be consulted for biodegradation effects for aromatic hydrocarbons. This technique allows the laboratory to observe the dispersion of soils in water and take any corrective action prior to purge-and-trap analysis. This technique is also most consistent with automated purge-and-trap samplers where stirring occurs prior to the purge cycle.

A.8.3.3 Collection with 5 mLs of water and 1 g of $NaHSO_4$ and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs of reagent water and 1 g of $NaHSO_4$ for vapor partitioning by Method 5035. For a spiked soil, $NaHSO_4$ was found to preserve the aromatic hydrocarbons at room temperature for more than 2 weeks. (Refs.1,11) The same soil showed major losses of aromatic hydrocarbons (5-10 ppm) when stored at room temperature for 5 days or at 4 ± 2°C after 10 days when no $NaHSO_4$ was present. (Ref. 1) The studied chlorinated hydrocarbons demonstrated insignificant losses during these storage conditions. The

use of $NaHSO_4$ with sample acidification to pH 2 or less eliminates the biodegradation of the important aromatic hydrocarbon volatile compounds.

1 g of $NaHSO_4$ will acidify 5 g of soil with an alkaline content (as $CaCO_3$) of 5%. It is insufficient to neutralize a soil with an alkaline content of 10%. This technique has been found to be somewhat problematic since publication of Method 5035. Carbonaceous soils cause effervescence of the acidic soil slurry with loss of volatiles and even cause failure of the septa VOA vial cap or even the VOA vial itself. Upon acidification, certain soils exhibit a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) The $NaHSO_4$ corrosive vapors may cause increased purge-and-trap maintenance by laboratories due to creation of active sites on the trapping material. A very few target compounds such as styrene, vinyl chloride, and 2-chloroethylvinyl ether react under acidic conditions and are not detected. Note that the sodium chloride matrix modifying reagent of Method 5021 was found to be as effective as $NaHSO_4$ for inhibiting biodegradation of aromatic hydrocarbons in soil and may be more advantageous to use with calcareous soils, since the inhibitory agent is not dependent on the concentration of hydrogen ion present. (Ref. 1)

A.8.3.4 Collection and preservation with methanol at 4 ± 2° C

Extrude collected soil from a coring device into a VOA vial containing 5 -10 mLs of methanol. Larger volumes of methanol may be used if compositing of soils is required. Methanol preservation is effective for 2 weeks if stored at 4 ± 2 °C. Also, one investigator has found methanol preservation of a sand spiked with gasoline to be effective when traditional techniques were ineffective. (Ref. 36)

A.9.0 SUMMARY OF FINDINGS

1. An aqueous sample holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid, however, since reactive compounds such as 2-chloroethylvinyl ether are unstable at low pHs, if these types of analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. Aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs. 49,50) (Sec. A.3.0)

2. The solid material to be characterized should be sampled with limited disruption (e.g., by a coring device for cohesive soils) and single transfer to an air tight VOA vial (PTFE-lined septa cap) that will be used for storage and preparation for VOC analysis. (Sec. A.7.1)

3. Data have been published or presented to validate different storage devices, procedures, preservative reagents and techniques for the VOC analysis of aqueous and solid samples. A wide range of recovery results have been observed. Acceptable devices, procedures, preservatives, and techniques should provide an average recovery of greater than 80% for important volatile contaminants such as benzene, dichloro- and trichloroethanes/ethenes. A recovery of 80% may be difficult for gaseous VOC contaminants such as vinyl chloride and chloroethane; however, the acceptability of a procedure should not be solely based on the less volatile VOCs such as chlorobenzene, xylenes, and trimethyl benzene. (Secs. A.2.0, A.6.0, A.7.0 and A.8.0)

4. VOCs in solids can be successfully sampled using coring tools (usually 5-g aliquots but can be 2 to 25 g) if the material is cohesive. Sampling procedures are not available to prevent VOC loss during sampling of non-cohesive soil material (dry sand, gravel, liquid sediment) or cemented material. (Secs. A.4.0 and A.7.0)

5. The following two techniques have been found accurate (minimal VOC loss) for preparation of soils for VOC analysis; however, they are not without problems:

   a. Soil is added to empty VOA vials at time of collection and is frozen at < -7°C until thawed for analysis. Validation data have not been provided yet, but it is believed that a prepared VOA vial with reagent water only is also acceptable for low concentration VOC in soil (<200 ppb) if frozen at < -7°C at time of collection.

   b. Soil is added to a prepared VOA vial, with methanol reagent, at time of collection and stored at 4 ± 2°C until time of analysis. This is applicable only to VOC in soil concentrations greater than 50 ppb. (Sec. A.6.2) (See comments below regarding use of methanol.)

6. The following techniques have been found to be the most practical, currently available alternatives for preparation of soil for VOC analysis. Validation data are not available to fully support their use for all types of soil or to fully differentiate them in accuracy relative to each other. The techniques rely on transport of sealed VOA vials or coring tools, at 4 ± 2°C, to a support laboratory within 48

hours where they are preserved/stored appropriately or immediately tested for VOCs. As more validation data and experience occur with time, their relative worth will become more apparent. The techniques listed below are superior to the traditional procedures of ten years ago.

a. Soil is added to tared replicate "empty VOA vials" at time of collection, preserved, refrigerated at 4 ± 2°C until laboratory receipt within 48 hours, and then preserved by freezing (< -7°C). Individual vials are thawed prior to sample preparation within 14 days of collection. A thawed vial must be processed within 24 hours by either screening using methanol extraction or analysis by vapor partitioning. At time of laboratory receipt, laboratories have the option of immediately testing a soil by vapor partitioning where the required reagent water is added through the PTFE-lined septa cap using the automated instrument sampling devices after weighing an "empty VOA vial" and obtaining wet sample weight by difference. In addition at time of laboratory receipt, laboratories have the option of immediately preparing a soil for methanol extraction by weighing an "empty VOA vial," obtaining the wet sample weight by difference, then adding methanol reagent through the PTFE-lined septa cap using a 23-gage needle on a Luer lock syringe. The sample-methanol mixture is shaken for 15 seconds to wet the vial's head space. The vial cap is opened once to vent pressure and then closed for the extraction process. (Sec. A.8.0)

b. For carbonate-containing soils (or soils suspected as such), ASTM D4547-98 (Ref. 15) provides for adding 2 to 5 g of soil (using coring tool) to tared, replicate prepared VOA vial containing 5 mLs of reagent water. Prepared VOA vials are maintained at 4 ± 2°C until laboratory receipt within 48 hours, and immediately tested for VOCs by vapor partitioning. This approach offers the advantage of mixing and dispersing the soil into the water and to observe any problematic samples prior to vapor partitioning analysis. Alternatively, the reagent water prepared VOA vials may be preserved by freezing (< -7°C) by placing vials in horizontal position. This technique is an alternative or fall-back from the prepared VOA vial with acidified reagent water; however, little or no data are available to validate its use. (Sec. A.5.3)

c. Soil is collected in replicate "Coring Tool Used as Transport Device" (e.g., the EnCore™ sampler), maintained at 4 ± 2 °C until laboratory receipt within 48 hours, then extruded into individual "Empty VOA Vials" for preservation by freezing (< -7°C) or into prepared VOA vials for immediate analysis by vapor partitioning or for sample preparation by methanol extraction. (Sec. A.7.2)

d. For known non-carbonate soils, a coring tool soil aliquot for BTEX type VOC analysis is added to a tared prepared VOA vial containing 5 mLs reagent water acidified with 1g $NaHSO_4$. The prepared VOA vial is maintained at 4 ± 2°C for BTEX testing by vapor partitioning within 14 days of sample collection. Acidified reagent water has been problematic when applied to a wide range of soil types for a large analyte list; however, it is effective for the volatile BTEX compounds in known non-carbonate soils. It is a specialized, preservation technique that minimizes aromatic VOC losses from biodegradation at 4 ± 2 °C.

Acetone artifacts are sometimes observed in soil samples preserved with $NaHSO_4$.

7. Use of a prepared VOA vial with acidified ($NaHSO_4$) reagent water is not recommended as a primary preservation technique for all soil types and a broad VOC analyte list. This technique is applicable to volatile aromatic hydrocarbons in soils known not to contain carbonates as discussed above.

8. A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from storage and analyses performed outside the recommended holding times.

A.10.0 REFERENCES

1. Hewitt, A.D., "Chemical Preservation of Volatile Organic Compounds in Soil," *Environmental Science & Technology*, 31, pp. 67-70 (1997).

2. Hewitt, A.D., "Frozen Storage of Soil Samples for VOC Analysis,"*Environmental Testing & Analysis*, 8 (5), pp. 18-25, 46 (1999).

3. Hewitt, A.D., T.F. Jenkins, and C.L. Grant, "Collection, Handling and Storage: Keys to Improved Data Quality for Volatile Organic Compounds in Soil,"*American Environmental Laboratory*, (February 1995).

4. Hewitt, A.D., "Storage and Preservation of Soil Samples for Volatile Compound Analysis; Special Report 99-5," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1999.

5. Hewitt, A.D., "Comparison of Sample Preparation Methods for the Analysis of Volatile Organic Compounds in Soil Samples: Solvent Extraction vs Vapor Partitioning," *Environmental Science & Technology*, 32, pp.143-149 (1998).

6. Hewitt, A.D., "Concentration Stability of Four Volatile Organic Compounds in Soil Samples; Special Report 94-6," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1994.

7. Hewitt, A.D., "Freezer Storage of Soil Samples Containing Volatile Organic Compounds," *Proceedings of the 15th Annual Waste Testing and QA Symposium,* U.S. EPA, Washington, D.C. pp. 125-128, 1999.

8. Hewitt, A.D., "Losses of Trichloroethylene from Soil During Sample Collection, Storage and Laboratory Handling, Special Report 94-8," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1994.

9. Hewitt, A.D., "Preparing Soil Samples for Volatile Organic Compound Analysis; Special Report 97-11," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1997.

10. Hewitt, A.D., and N.J.E. Lukash, "Obtaining and Transferring Soils for In-Vial Analysis of Volatile Organic Compounds; Special Report 96-5," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1996.

11. Hewitt, A.D., "Determining Volatile Organic Compound Concentration Stability in Soil," *Proceedings of the 11th Annual Waste Testing and QA Symposium,* U.S. EPA, Washington, D.C. pp. 173-183, 1995.

12. Hewitt, A.D., "Evaluation of Methanol and $NaHSO_4$ for Preservation of Volatile Organic Compounds in Soil Subsamples," *American Environmental Laboratory*, pp. 16-18 (1995).

13. Hewitt, A.D., and N.J.E. Lukash, "Sampling for In-Vial Analysis of Volatile Organic Compounds in Soil," *American Environmental Laboratory*, pp. 15-19, (1996).

14. Hewitt, A.D., "A Tool for the Collection and Storage of Soil Samples for Volatile Organic Compound Analysis," *American Environmental Laboratory*, 9, (10) pp. 14-16, 1997.

15. ASTM D 4547-98; "Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds."

16. Turriff, D. and C. Klopp, "Studies of Sampling, Storage and Analysis of Soils Contaminated with Gasoline and Diesel," PUBL-SW-513-95, Wisconsin Department of Natural Resources, 1995.

17. Turriff, D., "Comparison of Alternatives for Sampling and Storage of VOCs in Soil," *Proceedings of the 11th Annual Waste Testing and QA Symposium*, U.S. EPA, Washington, D.C. pp. 51-58, 1995.

18. Turriff, D., "A New Soil Sampling and Soil Storage System for Volatile Organic Compound Analysis," *Proceedings of the 11th Annual Waste Testing and QA Symposium*, U.S. EPA, Washington, D.C. pp. 95-96, 1995.

19. Ricker, M.J., "Determining Volatiles in Soils: A New Sampling Procedure," *Environmental Testing & Analysis*, July/August, pp. 13-16, 1999.

20. Ricker, M.J., "An Easy, Cost-Effective Solution for Sampling Volatile Organic Compounds in Soil," *Proceedings of the 15th Annual Waste Testing and QA Symposium*, U.S. EPA, Washington, D.C. pp. 134-162, 1999.

21. Ricker, M.J., "Validation of a New Sampling Procedure for Determining Volatiles in Soils," Report to the Wisconsin Department of Natural Resources, U.S. Oil Company, Kimberly, WI, 1998.

22. Soroni, S.S. and J.F. Schabron, "Validation of a New Soil VOC Sampler: Development of a Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis," Topical Report WRI-99-R003; Laramie, WY; Western Research Institute, 1999.

23. Soroni, S.S. and J.F. Schaborn, "Development of a Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis," Laramie, WY; Western Research Institute, 1999.

24. Soroni, S.S. and J.F. Schaborn, "Performance of the Disposable En Core Sampler for Storing Soil for Volatile Organic Analysis," *Proceedings of the 15th Annual Waste Testing and QA Symposium,* U.S. EPA, Washington, D.C. pp. 129-134, 1999.

25. Soroni, S.S., J.F. Schaborn and J.F. Rovani, "Validation of a New Soil VOC Sampler: Performance of the En Core Sampler for Storage of Low VOC Concentrations and EPA Method 1311 Volatile Organic Analytes," Topical Report WRI-01-R005; Laramie, WY; Western Research Institute, 2001.

26. ASTM D6418-99; "Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis."

27. Phillips, J.H., A.D. Hewitt and J.P. Glaser, "Recovery of VOCs from Soils With and Without Methanol Preservation," *Proceedings of the 15th Annual Waste Testing and QA Symposium,* U.S. EPA, Washington, D.C. pp. 163-169, 1999.

28. Severn Trent Services, "Method 5035 EnCore Holding Time Study," Denver, CO, April 2000.

29. Minnich, M.M., B.A. Schumacher, and J.H. Zimmerman, "Comparison of Soil VOCs Measured by Soil Gas, Heated Headspace, and Methanol Extraction Techniques," *Journal of Soil Contamination*, 6 (2), pp. 187-203, (1997).

30. Siegrist, R.L. and P.D. Jenssen, "Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils," *Environmental Science & Technology*, 24, pp. 1387-1392, (1990).

31. Urban, M.J., J.S. Smith & R.K. Dickinson, "Volatile Organic Analysis for a Soil, Sediment or Waste Sample," *Proceedings of the 5th Annual Waste Testing and QA Symposium*, U.S. EPA, Washington, D.C. pp. II-87-II-101, 1989.

32. Liikala, T.L., K.B. Olsen, S.S. Teel and D.C. Lanigan, "Volatile Organic Compounds: Comparison of Two Sample Collection and Preservation Methods," *Environmental Science & Technology*, 30, pp. 3441-3447, (1996).

33. Askari, M.D.F., M.P. Maskarinec, S.M. Smith, P.M. Beam and C.C. Travis, "Effectiveness of Purge-and-Trap for Measurement of Volatile Organic Compounds in Aged Soils," *Analytical Chemistry*, 68, pp. 3431-3433, (1996).

34. Voice, T.C. and B. Kolb, "Static and Dynamic Headspace Analysis of Volatile Organic Compounds in Soils," *Environmental Science & Technology*, 27, pp. 709-713, (1993).

35. Vitale, R.T., "Report: Comparison of Volatile Organic Compound Results Between Method 5030 and Method 5035 on a Large Multi-State Hydrocarbon Investigation; Poster Session - WTQA Symposium 1998 or 1999.

36. King, P.H., "Evaluation of Sample Holding Times and Preservation Methods for Gasoline in Fine-Grained Sand," Report to - National Symposium on Measuring and Interpreting VOCs in Soils: State of the Art and Research Needs, 1993.

37. Uhlfelder, M.M., "Study of Acetone Production in SW-846 Method 5035 (Low Level) Associated with Various Preservation Techniques and Storage Conditions,"*Proceedings of the 16th Annual Waste Testing and QA Symposium*, U.S. EPA, Washington, D.C. p. 13, 2000.

38. USEPA Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*," EPA Publication SW-846, Third Edition, as amended by Updates I, II, IIB, and III, finalized in the Federal Register on June 13, 1997.

39. USEPA Method 5021, "Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*," EPA Publication SW-846, Third Edition, as amended by Updates I, II, IIB, and III, finalized in the Federal Register on June 13, 1997.

40. USEPA Method 3815, "Screening Solid Samples for Volatile Organics," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*," EPA Publication SW-846, Third Edition, Draft Update IVB, listed in the Federal Register on November 27, 2000.

41. Minnich, M.M., J.H. Zimmerman and B.A. Schumacher, "Extraction Methods for Recovery of volatile Organic Compounds from Fortified Dry Soils," *Journal of AOAC International*, Vol. 79, No. 5, 1996.

42. Sawhney, B.L., J.J. Pignatello and S.M. Steinberg, "Determination of 1,2-Dibromoethane (EDB) in Field Soils: Implication for Volatile Organic Compounds," *Journal of Environmental Quality*, Vol. 17, No. 1, pp. 149-152, 1988.

43. USEPA OERR, "Multi-Laboratory Study on the Effects of Temperature and Sample Container on Volatile Organic Compounds from Time of Sampling to Time of Analysis - Final Report," April 24, 2001.

44. En Novative Technologies, Inc., Personal Communication to D. Payne, USEPA Region 5, October 5, 2000.

45. Hewitt, A. D., Personal Communication to D. Payne, USEPA Region 5, August 9, 2001.

46. Solomons, Graham, T.W., *Fundamentals of Organic Chemistry*, 3rd ed., pp 403, 505, 1990.

47. Oak Ridge National Laboratory, "Summary Report - Evaluation of the Impact of post-Holding Time Analyte Degradation on Regulatory Decision-Making -VOCs in Water," May 2001.

48. Oak Ridge National Laboratory, "Summary Report - Method for Preparing Performance Evaluation Water Samples for Volatile Organic Compounds," May 2001.

49. USEPA OSW, "Development and Evaluation of Methods for the Analysis of MTBE," September 17, 2001.

50. USEPA OUST, *Environmental Fact Sheet: Analytical Methods for Fuel Oxygenates,* EPA 510-F-02-001, May, 2002, www.epa.gov/OUST/mtbe/.

51. Hewitt, A.D., and K.F. Myers, "Sampling and On-Site Analytical Methods for Volatiles in Soil and Groundwater - Field Guidance Manual; Special Report 99-16," Hanover, NH; U.S. Army Cold Regions Research and Engineering Laboratory, 1999.

52. Maskarinec, M.P., L.H. Johnson, S.K. Holladay, R.L. Moody, C.K. Bayne, and R.A. Jenkins, “Stability of Volatile Organic Compounds in Environmental Water Samples during Transport and Storage,” *Environmental Science & Technology*, 24(11) pp1665-1670, 1990.

53. Bottrell, D.W., J.F. Fisk, G.L. Robertson, J.D. Dempsey, C.H. Petty, and M.L Bartling, “Holding Times of Volatile Organics in Water,” *Waste Testing and Quality Assurance: Third Volume, ASTM STP 1075*, C.E. Tatsch, Ed., American Society for Testing and Materials, Philadephia, PA, 1991.

54. Bottrell, D.W., “Suggested Modification of Pre-Analytical Holding Times - Volatile Organics in Water Samples,” *Proceddings of the 11th Annual Waste Testing and Quality Assurance Symposium*, U.S. EPA, Washington D.C. pp. 507-516, 1995.

55. Bayne, C.K., D.D. Schmoyer, and R.A. Jenkins, ”Practical Reporting Times for Environmental Samples,” *Environmental Science & Technology*, 28 (8) pp1430-1436, 1994.

USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 1225
SOIL SAMPLING FOR VOLATILE ORGANIC COMPOUNDS (VOCs)

Revision 0
Effective Date: February 10, 2016

Reviewed by: _____ 2/5/2016
Peter Husby — Date
Biology Team Leader/Technical Director

Reviewed by: _____ 2/5/2016
Lucrina Jones, Laboratory QA Officer — Date

Approved by: _____ 2/8/2016
Duane James, Acting Laboratory Director — Date

Periodic Review:

Signature	Title	Date

TABLE OF CONTENTS

1 SCOPE AND APPLICABILITY 3
2 METHOD SUMMARY 3
3 DEFINITIONS 4
4 RESPONSIBILITIES 4
5 SAFETY & HEALTH 4
6 SAMPLE HANDLING AND PRESERVATION 6
7 INTERFERENCES 7
8 APPARATUS AND MATERIALS 7
9 SAMPLING PROCEDURES 8
10 QUALITY CONTROL 15
11 DOCUMENTATION 16
12 REFERENCES 17

APPENDIX A. EPA, DOT, IATA REGULATIONS
APPENDIX B. CHAIN OF CUSTODY FORM
APPENDIX C. COLLECTED SAMPLE FORM – SOIL/SEDIMENT
APPENDIX D. REVISION HISTORY

1 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes field collection, handling, and preparation of soil samples for analyses of volatile organic compounds (VOCs) in solid material (e.g., soils, sediments and solid waste). This SOP includes guidance published by EPA Office of Solid Waste (1998) and complements EPA Laboratory Methods 5035 (purge and trap) and EPA 8015, 8021, 8260 (gas chromatography). Specialized equipment and containers are designed to maintain sample integrity for soil or solid materials which may contain contaminants with boiling points less than 200°C.

Research shows that traditional sample collection techniques may result in substantial losses of volatiles. These losses (an order of magnitude or more) are the culmination of volatilization and biodegradation that occur during the sampling, storage, and subsequent sub-sampling in the laboratory. To address significant problems with soil VOC analyses, Method 5035 as revised in Update III of SW-846 (June 13, 1997 Federal Register) and further amended under Method 5035A, July 2002. Samples are handled differently from the onset of sample collection, depending upon the action levels for the project and the anticipated concentrations of VOCs at the site. Field personnel may need to collect samples with both low (<200 ug/kg) and medium (>200 ug/kg) level VOC concentrations. Field preservation and laboratory preservation options are described in order to address sampling for soils at various concentrations.

2 METHOD SUMMARY

Collect approximately 5-25 grams from freshly exposed soil into a specially-designed hand-held coring device, or a pre-weighed/pre-preserved vial and ship to the laboratory for overnight delivery for further preservation and/or analysis. In addition to the samples collected for VOC analysis, collect an additional co-located sample for moisture content determination for results on a dry-weight basis. A brief summary of each method option is presented below.

2.1 Thermal Preservation Low Level (<200 ug/kg)

This method requires either a specially designed coring devise tested to withstand freezing temperatures (i.e., Encore ™) and or further chemical/thermal preservation upon receipt at the laboratory. However, no field chemicals are required. Thermal (freezing) or chemical ($NaHSO_4$) preservation within 24 hours of collection at the laboratory may extend the analysis holding times to two weeks. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 0.5 to 5 ug/kg. Bias may exist for some soil types (e.g., high clay or organic carbon). Additional volume is often required for laboratory QC and percent moisture determinations. Further dilution/analysis requires a sample collected under the medium level methanol preservation procedure summarized below.

2.2 Chemical Preservation Sodium bisulfate ($NaHSO_4$) – Low Level (<200 ug/kg)

This method requires that $NaHSO_4$ solution be added to a pre-weighed and tared vial prior to sample collection. This method is limited to consolidated soils which can be collected in a coring device and cannot be used on carbonaceous soils. The sample container (glass vial) also serves as the purge vessel. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 0.5 to 5 ug/kg. Since the vial is also used as sparge vessel, samples cannot be diluted, and VOA vials cannot be reanalyzed. Bias may exist for some soil types (e.g., high clay or organic carbon). Further dilution/analysis requires a sample collected under the methanol preservation procedure below. Additional volume is often required for laboratory QC and percent moisture determinations. Shipments must comply with special DOT labeling, limited quantities and special packaging requirements.

2.3 Methanol Preservation Medium Level (>200 ug/kg)

This method requires that purge and trap grade methanol be added to a pre-weighed and tared vial prior to sample collection. This method is often referred to as preservation/extraction procedure and holding time is two week from date and time of collection. This method may be used for aggregate and cemented materials by increasing sample size and volume of methanol. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 5 to 50 ug/kg. Additional volume is often required for laboratory QC and percent moisture determinations. Shipments must comply with special DOT labeling, limited quantities and special packaging requirements.

3 DEFINITIONS

There are no terms or definitions specific to this procedure. For terms and acronyms in general use at the EPA Region 9 Laboratory refer to Appendix A of the Laboratory Quality Assurance Plan.

4 RESPONSIBILITIES

Field Team Leader – It is the responsibility of the Field Team Leader to assure that all members of the field team have the proper training and are aware of and follow the guidelines outlined in this procedure and in other applicable documents.

Field Team Members – It is the responsibility of each Field Team Member to be aware of the requirements and follow the guidelines outlined in this procedure.

5 SAFETY & HEALTH

When working with potentially hazardous materials, follow U.S. EPA, OSHA and specific health and safety procedures. More specifically, depending upon the site-specific contaminants, various protective programs must be implemented to ensure the samplers

safety prior to collecting the first sample. The EPA Region 9 Field and Biology (FAB) Team member collecting the sample should not climb into trenches where bank failure may cause him or her to lose their balance. To prevent this, the person performing the sampling should be completed via augers with extensions or from directly from the backhoe immediately after removal from the ambient ground area.

5.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a well-vented laboratory fume hood.

5.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

The site health and safety plan should be reviewed with specific emphasis placed on the protection program planned for the sampling tasks. Standard safe operating practices should be followed such as minimizing contact with potential contaminants using respirators and disposable clothing as deemed necessary. At a minimum, skin protection will be afforded by disposable protective clothing. For example

Volatile organic compounds:

- Avoid breathing constituents venting from a borehole drill cuttings or trenching
- Pre-survey the borehole drill cuttings and trenches with an FID/PID prior to sampling.
  - If pre-survey results indicate parts-per billion (ppb) levels of organic constituents, sampling activities may be conducted in Level D protection.
  - If pre-survey results indicate parts-per million (ppm) levels or higher of organic constituents, sampling activities should be conducted in Level C protection.

Physical hazards associated with soil sampling are:

- Lifting injuries associated with drill moving equipment
- Pinch/crush injuries associated with drilling and hammering
- Heat/cold stress because of exposure to extreme temperatures
- Slip, trip, fall conditions because of discharge, and
- Restricted mobility due to the wearing of protective clothing.

Methanol is a toxic and flammable liquid and must be handled with all safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided and vials opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area wearing protective gloves

and safety glasses. Methanol should be stored away from open flames, areas of extreme heat, and other ignition sources.

Aqueous sodium bisulfate is a strong mineral acid. Therefore, solutions must be handled with all safety precautions related to mineral acids. Protective clothing (gloves, safety glasses, etc.) should be worn when vials containing sodium bisulfate are handled.

5.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management.

5.4 Waste Management

The EPA Region 9 Laboratory FAB Team complies with all applicable rules and regulations in the management of investigation derived waste (IDW). All team members must collect and manage IDW in a manner consistent with applicable site specific work plans. Solid and hazardous wastes are disposed of in compliance with site standards, hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

6 SAMPLE HANDLING AND PRESERVATION

Decontaminate all sampling devices and wrap in aluminum foil until needed. Each sampler should be used for only one sample. Dedicated samplers for sediment samples may be impractical when collecting numerous samples. In this case, decontaminate samplers in the field using SOP 1230 - *Sampling Equipment Decontamination.*

Samples for volatile organic analysis must be collected directly from the devise, before mixing the sample, to minimize loss due to volatilization of contaminants. Samples should be transferred to a VOA vial with a chemical preservative within 24-hours of collection. If this is done, a 14-day holding time will apply. If this action cannot be performed, either due to carbonaceous soils scheduled for low level analysis, or due to laboratory logistical issues, the samples should be stored in a freezer (-12°C). Such samples can be held for up to 7-days after sample collection.

Samples should be shipped the day of collection for overnight delivery to the laboratory. If

overnight transit is not feasible due to site logistics, samples should be held at 4°C until shipping. Samples collected in the Encore™ sampler should be received at the laboratory within 4 days of sampling. *Note: DOT regulations associated with the use of preservatives in the field may be avoided by using Encore ™ samplers*

Chemically preserved samples should be stored at 4°C until analysis. A 14-day holding time is applicable. Depending on the quantity and method of packaging, sodium bisulfate and methanol may be DOT Hazardous Materials and may be subject to the DOT and International Air Transportation Association (IATA) hazardous materials regulations.

Field personnel involved in the shipment of samples prepared in the field for laboratory analysis by Method 5035, should be aware of the pertinent EPA, Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations so that regulatory compliance can be maintained. Three levels of regulations apply depending on type and quantity of preservative and method of packaging. These regulations are summarized in Appendix A:

7 INTERFERENCES

Substrate particle size and organic content are directly related to water velocity and flow characteristics of a body of water. Contaminants are more likely to be concentrated in soils typified by fine particle size and a high organic content. This type of soil is most likely to be collected from depositional zones. In contrast, coarse soils with low organic content do not typically concentrate pollutants and are found in erosion zones. The selection of a sampling location can, therefore, greatly influence the analytical results.

Contamination of preservatives could result in a high bias of data. Personnel should optimize handling preservatives and sample vials in laboratory with controlled conditions. When samples are preserved in the field, it is especially critical to avoid the introduction of contamination from external sources. Consequently, personnel should work upwind of any possible source of VOCs (emissions from engines and backhoes, tobacco smoke, etc.) while adding preservatives to soil samples.

Free methanol or acidic solutions can solubilize contaminants in ambient air. Forethought while sampling or handling vials is crucial to avoid possible contamination. <u>Do not leave vials open and exposed to ambient air</u>. Equipment blank and field blanks should be specified in the sampling plan.

8 APPARATUS AND MATERIALS

8.1 Equipment needed for collection of soil samples includes:

- maps/plot plan
- Global Positioning Receiver/Recorder
- safety equipment--photoionization detector, OVM

- compass
- tape measure
- survey stakes, flags, or buoys and anchors
- camera and film
- stainless-steel, plastic, or other appropriate composition bucket
- 4-oz, 8-oz, and one-quart, wide-mouth jars w/Teflon-lined lids
- Ziploc plastic bags
- Logbook
- purge and trap grade methanol
- sample jar labels
- disposable plastic syringes
- glass vials, 40 mL, screw cap, TFE lined, septum sealed
- TFE coated magnetic stir bars
- portable top-loading balance ±0.01g
- balance weights for reference and calibration once per day
- chain-of-custody forms
- custody seals
- field data sheets
- cooler(s)
- ice
- decontamination supplies/equipment
- spade or shovel
- scoop
- bucket auger
- hand auger
- extension rods
- T-handle
- power auger
- backhoe
- drill rig

Reagents are not used for preservation of soil samples. Decontamination solutions are specified in SOP 1230 - *Sampling Equipment Decontamination.*

9 SAMPLING PROCEDURES

9.1 Preparation

Project preplanning is essential to successful sample collection. The following are prerequisites that should be addressed in advance of a full-scale field mobilization.

- Determine the extent of the sampling effort, the sampling method(s) employed, and required equipment and supplies according to the sampling QA plans for the site.
- Obtain necessary sampling and monitoring equipment.

- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.
- Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.
- Use stakes, flags, or buoys to identify and mark all sampling locations. Specific site characteristics, including flow regime, basin morphometry, soil characteristics, and depth of overlying aqueous layer, and extent and nature of contaminant should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

Whether sampling from the surface or from depth using such devices as a split spoon, collection of the sample will be the same. Samples should be collected as quickly as possible (< 10-15 seconds). *Temporary storage of soil in split spoons, jars, or ziplock bags is not permitted*. Field screening may still be used to decide which samples will be submitted for analysis but all potential samples must be immediately chemically preserved or placed in a coring device. All protocols have been written assuming that both medium and low level sample will need to be collected.

In order to help maintain the physical structure of samples, for cohesive granular material, a hand-operated coring device must be used to collect samples of appropriate size for laboratory analysis (e.g., cylindrical soil plugs are extruded into vials using disposable plastic syringes with the tapered front ends removed). Field personnel transfer samples into pre-weighed vials containing liquid preservatives (e.g., sodium bisulfate solution or methanol). The vials are weighed in the laboratory before use and are subsequently reweighed at the laboratory after the sample aliquots are added to obtain the net sample weights.

Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed and shipped to the field location. Gloves should be worn during the preparation steps.

9.2 Low level samples field-preserved with sodium bisulfate solution

9.2.1 Add a clean magnetic stirring bar to each clean vial. If the purge and trap device employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

9.2.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If sample volume is markedly smaller or larger than 5 g, adjust the amount of preservative at a ratio of 0.2 g preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of <2.

9.2.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority

of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

9.2.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.

9.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures the tare weight of the vial includes the label. (The weight of any added markings is negligible.)

9.2.6 Weigh the prepared vial ± 0.01 g, record the tare weight, and write it on the label.

9.2.7 As VOCs will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards must be introduced in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis

9.3 Medium level samples field preserved/extracted with methanol

9.3.1 Add 10 mL of methanol to each vial.

9.3.2 Seal the vial with the screw-cap and septum seal.

9.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures the tare weight of the vial includes the label. (The weight of any added markings is negligible.)

9.3.4 Weigh the prepared vial ± 0.01 g, record the tare weight, and write it on the label.

9.3.5 Surrogates, internal standards and matrix spikes (if applicable) must be added to the sample in the laboratory and prior to analysis.

9.4 Field collection with coring type samplers followed by laboratory preservation

Expose a fresh surface using a clean spatula or other suitable tool. Collect a sample using a coring device (e.g., the hand-held Encore™) and immediately cap following manufacturer's directions. Collect two five gram cores for the low level (if needed) and one five gram core for the medium level. Label cores and transfer to laboratory on ice as soon as possible.

9.4.1 Unconsolidated Materials

Certain soil types may not be sufficiently consolidated to collect a core sample. Two examples would be dry sand or sludges/sediments with a very high moisture content. In such cases, the plunger of the Encore™ should be pulled

back and locked. The Encore™ should be held with the opening facing upward and the sample transferred by spatula or pouring until the Encore™ is filled. The Encore™ is then capped and handled as previously outlined. *Note: Samples which are unconsolidated should be labeled as such on the chain of custody so that the laboratory can handle these samples with additional caution.*

9.4.2 Aggregate or Cemented Material

The Encore™ sampler should not be used for these materials. It can only be used for soil types that can be collected using a small diameter coring device. For other materials, the only collection technique which will maintain the integrity of the sample is field collection with methanol–protocol.

9.5 Low level soil samples field preserved with Sodium Bisulfate

The sample vials for the low-level method are designed to be placed directly in the laboratory's instrument so that they remain hermetically sealed until the VOCs are withdrawn during analysis. The entire content of each vial is processed during analysis. Hence, when low-level VOC analyses are required, it is necessary to collect at least two co-located samples. This gives the laboratory an opportunity to perform an additional analysis should the first analysis be unacceptable. Since the vials remain sealed, dilutions cannot be performed. When low-level VOC analyses are required, an extra co-located sample for the medium-level method must be collected with each set of low-level samples. Also aqueous acidic solutions are used to preserve samples for the low-level analyses therefore, low-level samples must be initially tested for carbonate interferences in the field before samples are collected.

9.5.1 Laboratory Preparation

Add 1 gram sodium bisulfate, clean magnetic stir bar, and 5 milliliters (mL) of de-ionized water to a 40 mL VOA vial. Label vial and record weight ± 0.01 grams.

Note: VOA vials with special low bleed septa must be used to prevent false positives due to siloxane peaks from standard septa. Teflon coated stir bars absorb VOCs. This is a potential loss of VOCs. Disposable stir bars should be used or if stir bars are to be re-used, the stir bars should be cleaned and the cleanliness verified.

9.5.2 Field Sampling

9.5.2.1 Samples should be collected using a coring device (modified plastic syringe) as a transfer tool. A simple coring device can be made by cutting off the front part (with tip) of a disposable non-lubricated syringe, removing the rubber plunger tip and (with repeated

experimentation) marking the length of core (2--3 cm) that corresponds to 5.0 ± 0.5 g. *Note: Use disposable syringes are NOT lubricated since so as to avoid contaminating the VOC sample.*

9.5.2.2 If the test sample passes the initial test for both effervescence and pH (section below), use the plastic syringe to collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most.

9.5.2.3 Transfer the 5 g soil sample ("or plug") into the prepared and pre-labeled sample vial by placing the syringe tip inside the vial and squeezing the syringe plunger. Cap immediately and carefully wipe the exterior of the sample collection device with a clean cloth towel.

9.5.2.4 For each sampling point, use a new plastic syringe to collect soil. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap.

9.5.2.5 An initial test sample should be collected to evaluate effervescence and chemical preservation (pH ≤ 2). A five gram core should be placed in a VOA vial which contains the acid solution. If effervescence occurs, the sample should be collected in a VOA vial with no sodium bisulfate. (Note: if effervescence does occur, immediately unscrew the cap to release built up pressure.) The unpreserved sample should be analyzed within 48 hours, the holding time. Results from the analysis may be biased low and should be flagged as estimated.

9.5.2.6 The test sample must also have the pH evaluated either by a pH meter or test strip to ensure that the pH has been reduced to <2 to limit biodegradation. If the sample has not been properly acidified, there are two options:

- Vials can be used which contain a higher amount of sodium bisulfate. This additional sodium bisulfate should be added in the lab when the vial is prepared since addition in the field would affect tare weight. The exact amount of sodium bisulfate will be determined by the buffering capacity of the soil. Sodium bisulfate can be added in the field if the field personnel record the weights of the additional preservative and sample.
- Analyze the sample within the 48 hour holding time and flag the data as estimated.

9.5.2.7 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 g ± 0.5 g of sample were added. The balance should be calibrated in the field using an

appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

9.5.2.8 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of soil in the syringe that corresponds to 5.0 + 0.5 g. Discard each trial sample.

9.5.2.9 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

9.5.2.10 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1- 2 grams instead of 5 grams as described above.

NOTE: When the low level samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Additional steps may be required to preserve the samples. Such steps include: addition of larger amount of the sodium bisulfate preservative to non-calcareous samples, storage of low level sample as -12°C, or significantly reduce the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel.

9.5.2.11 Record weight and transfer to ice.

9.5.2.12 A duplicate low level sample should be collected for the laboratory since low level samples cannot be reanalyzed. Ship to the laboratory per DOT regulations. (Corrosive.) *Note: Additional samples need to be collected for matrix spikes or other QC objectives.*

9.5.3 Aggregate or Cemented Material

Coring tools should not be used for these materials. For other materials, the only collection technique which will maintain the integrity of the sample is field collection with methanol–protocol below.

9.6 Medium level samples field preserved with Methanol

This particular sampling protocol has been suggested by some as a combined

preservation and extraction procedure. Carbonates are not problematic for methanol preservation and methanol sample extracts may be diluted in the laboratory when concentrations exceed the calibration range of the instrument. In addition, when samples are preserved with methanol, field personnel are not limited to single grab samples (as in the low-level method) but may composite subsamples from several locations.

9.6.1 Laboratory Preparation

Add 5 mL of methanol to a 40 mL VOA vial. Label vial and record weight ± 0.01 grams.

9.6.2 Field Sampling

9.6.2.1 Collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 g ± 0.5 g. Discard each trial sample.

9.6.2.2 Using the appropriately sized sample collection device, quickly (within 1-2 minutes exposure to the atmosphere) collect approximately 5 g of sample from the surface of the soil or sediment. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

9.6.2.3 Using the collection device, add about 5 g (2--3 cm) of soil to the vial containing 10 mL of methanol. Brush any soil off the vial threads and seal the vial with the septum and screw-cap. Total sub-sampling time should not to exceed 5 minutes. Store the samples on ice at 4°C± 2°C.

9.6.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 g ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

9.6.2.5 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate the sensitivity of the overall analytical procedure is appropriate for the intended application.

9.6.2.6 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in above. Samples collected in methanol should be shipped as described in Attachment A and must be clearly labeled as containing methanol, so the that the samples are not analyzed using the closed-system purge and trap equipment described in this procedure.

NOTE: Collection of medium concentration soil samples that are NOT preserved in the field generally follow similar procedures as for the other types of samples described above, with the exception that the

sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

9.6.3 Large Aggregate and Cemented Materials

Sample will need to be placed in a larger, wide mouth, 4 0z. glass jar and preserved with a proportionately larger volume of methanol (to maintain the 1:1 ratio). In this event, the weight or volume of methanol must be recorded.

9.7 No preservation

Under limited circumstances, Region 9 will permit the collection of unpreserved samples, such as hard or cementitious materials, debris, or large aggregates which cannot be easily collected using the options above. Field methanol preservation is the preferred approach for these types of materials. Losses of VOCs are likely and all results should be considered as estimated values.

9.8 Moisture Content Sample

In addition to the samples collected as described above, a separate container must be collected to determine moisture content. This sample can be any conveniently sized container, of glass or plastic. Ordinary soil sampling procedure are used to collect samples to measure moisture content. If samples are being collected for other analytes (e.g. metals, semivolatiles) that sample container can serve as the container for moisture content.

10 QUALITY CONTROL

The EPA Region 9 Laboratory operates a formal quality control program and tracks compliance using the Lab QC Database.

10.1 Quality Control Samples

10.1.1 Field duplicate samples should be collected at a frequency of 10 percent, or as otherwise specified in project specific work plans to assess sampling precision.

10.1.2 Equipment or rinsate blanks should be collected if equipment is field cleaned and re-used or when necessary, to document that low-level contaminants were not introduced by sampling tools.

10.1.3 Ambient or field blanks should be collected at the discretion of the field team leader based on extreme site conditions (e.g., strong odors, vapors, dust storms) or as otherwise specified in project specific work plans to document that low-level contaminants were not introduced from the airborne sources.

11 DOCUMENTATION

Sample documentation includes chain-of-custody forms and custody seals.

11.1 Chain-of-Custody

11.1.1 EPA chain-of-custody forms (Appendix B) will be used to document sample collection and transportation to the laboratory for analysis. The chain-of-custody record will identify the contents of each sample cooler and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are transported to the laboratory, the custody of the samples will be the responsibility of the sampler.

11.1.2 The sample location on the chain-of-custody form should be sufficiently descriptive so that another person could resample the same location. Other identifiers such as permanent sample station numbers, or global coordinates should also be recorded.

11.1.3 Use the “Remarks” column to record additional information and any problems encountered sampling.

11.1.4 When finished sampling be sure that all samples are recorded on the chain-of-custody form and that the sample locations and sample times on the form match those on the sample bottles. The sampler’s name, address and phone number will also be written on the form. The sampler will sign the chain-of custody record and the "relinquished by" box and note date and time when the samples are delivered to the courier service or the Regional Laboratory Sample Custodian.

11.2 Sample Collection Information.

All sample collection information pertinent to the Project will be recorded in a bound Collected Sample Logbook. Each Collected Sample Form (Appendix C) will be consecutively numbered, dated, and signed. All entries will be made in indelible ink and all corrections will consist of line-out deletions that are initialed and dated. The person making the correction will provide a brief explanation for the change. There should be no blank spaces on the form. A single line or “Not Applicable” or “NA” should be written in spaces where no information was collected. Entries may include some or all of the following information:

- Date (yyyymmdd)
- Time (military format)
- Depth start of sample
- Depth end of sample
- Units
- Sample type
- Sample method
- Analytical Method
- Sample Preservation
- Sample matrix
- QC Information:
- Rinse blank ID,
- Trip blank ID,
- Duplicate sample ID,
- Split Sample ID
- Requested
- Sample container/no. of containers
- color, texture,
- FID, PID, RAD
- Weather
- Temperature
- Comments and
- Signature of Sampler and Date

11.3 SOP Distribution and Acknowledgement

After approval, distribute an electronic copy of the final SOP to all field and laboratory staff expected to perform the SOP or review data generated by the SOP. (The Lab QC Database contains a list of assigned analysts for each SOP). All approved EPA Region 9 Laboratory SOPs are maintained in the Lab QA Sharepoint site in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

11.4 SOP Revisions

Revisions to this SOP are summarized in Appendix D.

12 REFERENCES

EPA Region 9 Laboratory documents (SOPs, the Laboratory Quality Assurance Plan, etc.) are not included in this list. Analysts are referred to the SOP database on the Lab QA Sharepoint site or the local area network (G:\USER\SHARE\QA PROGRAM\LAB SOPS PDF) for these documents; laboratory users should contact the Chemistry Team Leader or Laboratory QAO for copies of any supporting documents.

Askari, M.D.F., M.P. Maskarinec, S.M. Smith, P.M. Beam and C.C. Travis, "Effectiveness of Purge-and-Trap for Measurement of Volatile Organic Compounds in Aged Soils," Analytical Chemistry, 68, pp. 3431-3433, (1996).

ASTM D 4547-98 "Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds."

ASTM D6418-99 "Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis."

Bayne, C.K., D.D. Schmoyer, and R.A. Jenkins, ”Practical Reporting Times for Environmental Samples,” Environmental Science & Technology, 28 (8) pp1430-1436, 1994.

Bottrell, D.W., “Suggested Modification of Pre-Analytical Holding Times – Volatile Organics in Water Samples,” Proceedings of the 11th Annual Waste Testing and Quality Assurance Symposium, U.S. EPA, Washington D.C. pp. 507-516, 1995.

Bottrell, D.W., J.F. Fisk, G.L. Robertson, J.D. Dempsey, C.H. Petty, and M.L Bartling, “Holding Times of Volatile Organics in Water,” Waste Testing and Quality Assurance: Third Volume, ASTM STP 1075, C.E. Tatsch, Ed., American Society for Testing and Materials, Philadelphia, PA, 1991.

En Novative Technologies, Inc., Personal Communication to D. Payne, USEPA Region 5, October 5, 2000.

Hewitt, A.D., "Chemical Preservation of Volatile Organic Compounds in Soil," Environmental Science & Technology, 31, pp. 67-70 (1997).

Hewitt, A.D., "Frozen Storage of Soil Samples for VOC Analysis," Environmental Testing & Analysis, 8 (5), pp. 18-25, 46 (1999).

Hewitt, A.D., T.F. Jenkins, and C.L. Grant, "Collection, Handling and Storage: Keys to Improved Data Quality for Volatile Organic Compounds in Soil," American Environmental Laboratory, (February 1995).

Hewitt, A.D., "Storage and Preservation of Soil Samples for Volatile Compound Analysis Special Report 99-5," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1999.

Hewitt, A.D., "Comparison of Sample Preparation Methods for the Analysis of Volatile Organic Compounds in Soil Samples: Solvent Extraction vs Vapor Partitioning," Environmental Science & Technology, 32, pp.143-149 (1998).

Hewitt, A.D., "Concentration Stability of Four Volatile Organic Compounds in Soil Samples Special Report 94-6," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1994.

Hewitt, A.D., "Freezer Storage of Soil Samples Containing Volatile Organic Compounds," Proceedings of the 15th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 125-128, 1999.

Hewitt, A.D., "Losses of Trichloroethylene from Soil During Sample Collection, Storage and Laboratory Handling, Special Report 94-8," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1994.

Hewitt, A.D., "Preparing Soil Samples for Volatile Organic Compound Analysis Special Report 97-11," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1997.

Hewitt, A.D., and N.J.E. Lukash, "Obtaining and Transferring Soils for In-Vial Analysis of Volatile Organic Compounds Special Report 96-5," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1996.

Hewitt, A.D., "Determining Volatile Organic Compound Concentration Stability in Soil," Proceedings of the 11th Annual Waste Testing and QA Symposium, U.S. EPA, Washington,

D.C. pp. 173-183, 1995.

Hewitt, A.D., "Evaluation of Methanol and NaHSO4 for Preservation of Volatile Organic Compounds in Soil Subsamples," American Environmental Laboratory, pp. 16-18 (1995).

Hewitt, A.D., and N.J.E. Lukash, "Sampling for In-Vial Analysis of Volatile Organic Compounds in Soil," American Environmental Laboratory, pp. 15-19, (1996).

Hewitt, A.D., "A Tool for the Collection and Storage of Soil Samples for Volatile Organic Compound Analysis," American Environmental Laboratory, 9, (10) pp. 14-16, 1997. 5035A - 66 Draft Revision 1 July 2002

Hewitt, A. D., Personal Communication to D. Payne, USEPA Region 5, August 9, 2001. 46. Solomons, Graham, T.W., Fundamentals of Organic Chemistry, 3rd ed., pp 403, 505, 1990.

Hewitt, A.D., and K.F. Myers, "Sampling and On-Site Analytical Methods for Volatiles in Soil and Groundwater - Field Guidance Manual Special Report 99-16," Hanover, NH U.S. Army Cold Regions Research and Engineering Laboratory, 1999.

King, P.H., "Evaluation of Sample Holding Times and Preservation Methods for Gasoline in Fine-Grained Sand," Report to - National Symposium on Measuring and Interpreting VOCs in Soils: State of the Art and Research Needs, 1993.

Liikala, T.L., K.B. Olsen, S.S. Teel and D.C. Lanigan, "Volatile Organic Compounds: Comparison of Two Sample Collection and Preservation Methods," Environmental Science & Technology, 30, pp. 3441-3447, (1996).

Maskarinec, M.P., L.H. Johnson, S.K. Holladay, R.L. Moody, C.K. Bayne, and R.A. Jenkins, “Stability of Volatile Organic Compounds in Environmental Water Samples during Transport and Storage,” Environmental Science & Technology, 24(11) pp1665-1670, 1990.

Minnich, M.M., J.H. Zimmerman and B.A. Schumacher, "Extraction Methods for Recovery of volatile Organic Compounds from Fortified Dry Soils," Journal of AOAC International, Vol. 79, No. 5, 1996.

Minnich, M.M., B.A. Schumacher, and J.H. Zimmerman, "Comparison of Soil VOCs Measured by Soil Gas, Heated Headspace, and Methanol Extraction Techniques," Journal of Soil Contamination, 6 (2), pp. 187-203, (1997).

National Environmental Laboratory Accreditation Conference, 2001. *National Environmental Laboratory Accreditation Conference Standards, May 21, 2001*.

Oak Ridge National Laboratory, "Summary Report - Evaluation of the Impact of post-Holding Time Analyte Degradation on Regulatory Decision-Making -VOCs in Water," May 2001.

Oak Ridge National Laboratory, "Summary Report - Method for Preparing Performance Evaluation Water Samples for Volatile Organic Compounds," May 2001.

Phillips, J.H., A.D. Hewitt and J.P. Glaser, "Recovery of VOCs from Soils With and Without Methanol Preservation," Proceedings of the 15th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 163-169, 1999.

Ricker, M.J., "Determining Volatiles in Soils: A New Sampling Procedure," Environmental Testing & Analysis, July/August, pp. 13-16, 1999.

Ricker, M.J., "An Easy, Cost-Effective Solution for Sampling Volatile Organic Compounds in Soil," Proceedings of the 15th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 134-162, 1999.

Ricker, M.J., "Validation of a New Sampling Procedure for Determining Volatiles in Soils," Report to the Wisconsin Department of Natural Resources, U.S. Oil Company, Kimberly, WI, 1998.

Sawhney, B.L., J.J. Pignatello and S.M. Steinberg, "Determination of 1,2- Dibromoethane (EDB) in Field Soils: Implication for Volatile Organic Compounds," Journal of Environmental Quality, Vol. 17, No. 1, pp. 149-152, 1988.

Siegrist, R.L. and P.D. Jenssen, "Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils," Environmental Science & Technology, 24, pp. 1387-1392, (1990).

Soroni, S.S. and J.F. Schaborn, "Validation of a New Soil VOC Sampler: Development of a Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis," Topical Report WRI-99-R003 Laramie, WY Western Research Institute, 1999.

Soroni, S.S. and J.F. Schaborn, "Development of a Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis," Laramie, WY Western Research Institute, 1999.

Soroni, S.S. and J.F. Schaborn, "Performance of the Disposable En Core Sampler for Storing Soil for Volatile Organic Analysis," Proceedings of the 15th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 129-134, 1999.

Soroni, S.S., J.F. Schaborn and J.F. Rovani, "Validation of a New Soil VOC Sampler: Performance of the En Core Sampler for Storage of Low VOC Concentrations and EPA Method 1311 Volatile Organic Analytes," Topical Report WRI-01-R005 Laramie, WY Western Research Institute, 2001.

Severn Trent Services, "Method 5035 EnCore Holding Time Study," Denver, CO, April 2000.

Turriff, D. and C. Klopp, "Studies of Sampling, Storage and Analysis of Soils Contaminated with Gasoline and Diesel," PUBL-SW-513-95, Wisconsin Department of Natural Resources, 1995.

Turriff, D., "Comparison of Alternatives for Sampling and Storage of VOCs in Soil," Proceedings of the 11th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 51-58, 1995.

Turriff, D., "A New Soil Sampling and Soil Storage System for Volatile Organic Compound Analysis," Proceedings of the 11th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. 95-96, 1995.

Uhlfelder, M.M., "Study of Acetone Production in SW-846 Method 5035 (Low Level) Associated with Various Preservation Techniques and Storage Conditions," Proceedings of the 16th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. p.13, 2000.

Urban, M.J., J.S. Smith & R.K. Dickinson, "Volatile Organic Analysis for a Soil, Sediment or Waste Sample," Proceedings of the 5th Annual Waste Testing and QA Symposium, U.S. EPA, Washington, D.C. pp. II-87-II-101, 1989.

USEPA Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, Third Edition, as amended by Updates I, II, IIB, and III, finalized in the Federal Register on June 13, 1997.

USEPA Method 5021, "Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, Third Edition, as amended by Updates I, II, IIB, and III, finalized in the Federal Register on June 13, 1997. 5035A - 68 Draft Revision 1 July 2002

USEPA Method 3815, "Screening Solid Samples for Volatile Organics," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, Third Edition, Draft Update IVB, listed in the Federal Register on November 27, 2000.

USEPA OUST, Environmental Fact Sheet: Analytical Methods for Fuel Oxygenates, EPA 510-F-02-001, May, 2002, www.epa.gov/OUST/mtbe/.

USEPA OERR, "Multi-Laboratory Study on the Effects of Temperature and Sample Container on Volatile Organic Compounds from Time of Sampling to Time of Analysis - Final Report," April 24, 2001.

USEPA. 1997. Test Methods for Evaluating Solid Waste, Update III. (SW-846). Office of Solid Waste, U.S. Environmental Protection Agency, Washington, D.C.

U.S. Environmental Protection Agency, 2001. *Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents (QA/G-6).*

Voice, T.C. and B. Kolb, "Static and Dynamic Headspace Analysis of Volatile Organic Compounds in Soils," Environmental Science & Technology, 27, pp. 709-713, (1993).

Vitale, R.T., "Report: Comparison of Volatile Organic Compound Results Between Method 5030 and Method 5035 on a Large Multi-State Hydrocarbon Investigation Poster Session - WTQA Symposium 1998 or 1999.

APPENDIX A.
EPA, DOT, IATA REGULATIONS

Pertinent EPA, Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations

- Small quantity exception--(< 30 mL inner containers), not subject to Hazardous Material Regulations (HMR) provided package is in accordance with 49 CFR 173.4.

- Limited quantity DOT hazardous material--must meet regulatory requirements minus UN specification containers (49 CFR 172.700 training applies)

- Fully regulated DOT hazardous material--Ltd Qty exception not taken, package must be in full compliance with HMRs (49 CFR 172.700 training applies)

1. Shipment as a Small Quantity Exception (49 CFR 173.4)

The DOT small quantity exception described in 49 CFR 173.4(a)(1)(i) states that the maximum quantity of material per inner container is limited to thirty (30) mL for authorized liquids, other than Division 6.1, Packing Group I materials (i.e., poisons). As applied to the preservatives of Method 5035, if there is less than or equal to 30 mL of methanol or aqueous sodium bisulfate solution per inner container (VOC vial), this material is not subject to any other requirements of the hazardous materials regulations except those presented in 49 CFR 173.4. However, aside from the 30 mL receptacle limit, there are additional restrictions:

- Each inner receptacle with a removable closure, has its closure held securely in place with wire, tape or other positive means.
- Unless equivalent cushioning and absorbent material surrounds the inside packaging, each inner receptacle is securely packed in an inside packaging with cushioning and absorbent material that (i) will not chemically react with other material and (ii) is capable of absorbing the entire contents (if liquid) of the receptacle.
- The inside packaging is securely packed in a strong outside packaging.
- The completed package, as demonstrated by prototype testing, is capable of sustaining each of the following free drops made from a height of 1.8 m (5.9 feet) directly onto a solid unyielding surface without breakage or leakage from any inner receptacle and without a substantial reduction in the effectiveness of the package:
- One drop flat on bottom
- One drop flat on top
- One drop flat on the long side
- One drop flat on the short side
- One drop on a corner at the junction of three intersecting edges
- A compressive load as specified in 49 CFR 178.606(c)

The gross mass of the completed package must not exceed 29 kg (64 pounds). The package must not be opened or otherwise altered until it is no longer in commerce. The shipper must certify conformance with this section by marking the outside of the package with the statement: This package conforms to 49 CFR 173.4," or, until 1 October 2001, with the statement: This package conforms to the conditions and limitations specified in 49 CFR 173.4. Furthermore, the shipper must indicate on the air waybill under nature and quantity of goods: Dangerous Goods in

Excepted Quantities.

IATA also requires the application of an excepted quantities label. This label contains the certification language identified above. Label entries include shipper signature, title, date, address and indication of the hazard class and associated UN number.

While 49 CFR 173.4 does not have a total net quantity limitation, IATA Dangerous Goods Regulations (DGR Section 2.7.4.2) does. For packing group II materials (e.g., methanol and sodium bisulfate), the total net quantity limit is 500 mL. This equates to 60 inner containers (VOC vials) containing approximately 8 mL of material (sample plus preservative) per outer package (i.e., sample cooler).

When discussing the shipment of DOT hazardous materials in the air mode, shippers have additional restrictions that are identified in Columns 9A/9B of the 49 CFR 172.101 hazardous materials table. Net quantity limits for methanol for passenger and cargo aircraft are one (1) liter and sixty (60) liters, respectively. The net quantity limits for sodium bisulfate solutions are one (1) liter and thirty (30) liters, respectively. Shippers should note that these quantities exceed the IATA small quantity exception. Therefore, if preservative volume (methanol or sodium bisulfate solution) is less than 30 mL per VOC vial (inner container) and the total net quantity per cooler (outer package) is limited to 500 mL, DOT HMRs or IATA DGR's quantity limits are never an issue provided packaging conforms with 49 CFR 173.4.

If more than 30 mL of methanol is used per VOC vial, shippers must address regulations for DOT-regulated hazardous material.

2. DOT Regulated Hazardous Materials Shipments, Limit Quantity

Personnel offering chemically preserved environmental samples for shipment in commerce in inner packaging (containers) containing more than 30 mL of methanol are Hazmat employees and are subject to the DOT training requirements in 49 CFR 172.700. If these individuals do not possess DOT training and do not have an employer certification, it is a violation of DOT regulations to offer these materials for transportation in commerce! Also, some generally used air shipping couriers may not ship hazardous materials or limited quantity hazardous materials. It is recommended that the proposed carrier be consulted in advance to determine if there are any company-specific requirements or limitations.

Methanol-preserved samples in greater than 49 CFR 173.4 inner-container quantities will void the 49 CFR 173.4 small quantity exception. These materials meet the definition of a DOT flammable liquid. On the shipping paper, these samples must be described using any of the following text:

- Methanol solution, 3, UN1230, PGII
- Methanol solution, 3, UN1230, PGII Limited Quantity
- Methanol solution, 3, UN1230, PGII Ltd Qty.

(Note that methyl alcohol may be substituted for Amethanol.)

It is emphasized that DOT allows for a limited quantity exception. Under the limited quantity exceptions, packages need not be UN specification. Labels are not required unless the shipment

is by air. Additionally, limited quantity shipments are not subject to placarding requirements. There are restrictions on the type of combination packaging that is acceptable for use. Since methanol is a PGII flammable liquid, 49 CFR 173.150 states the inner packaging limitation is one (1.0) liter. The outer packaging is described in the regulations as a strong outer package (i.e., a box, can, or cooler). Marking requirements must be met. An outline of the limited quantity exceptions and requirements for methanol is as follows:

Packaging:
Inner packaging: Plastic or glass < 1.0 liters
Outer Packaging: Strong outer package
Gross Weight: 66 lb (30 kg)

Labeling: Not required unless shipped by air
Primary Hazard: Flammable Liquid
Secondary Hazard: Poison

Marking: The outer package must be marked with the following items:

1. Proper shipping name: Methanol Solution
2. UN Number: Not required for Ltd Qty shipment/Otherwise required
3. DOT specification orientation arrows (See 49 CFR 172.312 for exception)
4. Shipper or receiving facility name and address
5. Cargo Aircraft only may be required depending on quantity shipped.

Shipping Paper:
1. Complete DOT shipping description
2. Number of containers (i.e., complete package)
3. Weight
4. Emergency Response information (ERG #)
5. Emergency Contact information

3. DOT Regulated Hazardous Materials Shipments, Fully Regulated

If shippers do not take a limited quantity exception and their materials are regulated in commerce, they must have DOT specification packages and would probably have to consider the cooler a DOT over pack (49 CFR 173.25). All inner packaging must be marked and labeled. Also, since the inner markings and labels will not be visible, the over pack must be marked and labeled on the outside and be marked with the following statement:

Inside (inner) packages comply with prescribed specifications

This means that the inner receptacles (glass jars or vials) must be in an authorized (UN specification) outer package. These combination packages would then be placed in the cooler (DOT over pack). For this case, all DOT shipping paper, labeling, marking (including UN numbers), and placarding requirements in 49 CFR 171 - 177 apply.

4. RCRA Regulations

1. The RCRA hazardous waste regulations are also be applicable to shipping of chemically preserved samples. 40 CFR 261.4 discusses the RCRA exemption for shipping samples. These regulations provide an exemption from the hazardous waste regulations for "samples" but not for materials which are not analyzed. Materials preserved with aqueous sodium bisulfate or methanol, which are not considered "samples," would be classified as hazardous wastes due to characteristics (corrosivity and ignitability) and would need to meet the RCRA manifesting and shipping requirements in 40 CFR 262.

APPENDIX B.
CHAIN OF CUSTODY FORM

ENVIRONMENTAL PROTECTION AGENCY
Region 9 Laboratory

CHAIN OF CUSTODY RECORD

1337 S. 46th St., Bldg. 201
Richmond, CA 94804-4698

PROJ. NO.	PROJECT NAME					NO. OF CON-TAINERS							REMARKS
SAMPLERS: *(Signature)*													
DATE	TIME	MATRIX	COMP.	GRAB	SAMPLE IDENTIFICATION								

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*		Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*		Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Received for Laboratory by: *(Signature)*	Date / Time	Temp.	Seals Intact (Y/N)	Conditions / Remarks		

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

9-3986

APPENDIX C.
COLLECTED SAMPLE FORM – SOIL/SEDIMENT

Installation/Site:_____ Area/OU:_____

Date:_____ Time:_____

Location ID:_____ Field Sample ID:_____

Begin Depth:_____ ☐ feet ☐ meters End Depth::_____ ☐ feet ☐ meters

NCoord:_____ ECoord:_____

Coord Type: ☐ UTM ☐ STP ☐ GEO Survey Type: ☐ Optical ☐ GPS ☐ Estimate

Datum: ☐ NAD83 ☐ WGS84 ☐ Other: _____ Coord Precision (±) :_____ ☐ feet ☐ meters

Elevation: _____ ☐ feet ☐ meters Elev. Type: ☐ Optical ☐ GPS ☐ Estimate

Photo Reference:_____ Elevation Precision (±) :_____

Sample Information

Sample Type	**Sample Method**	**Sample Container**
☐ Normal (N)	☐ Trowel	☐ Stainless Steel Sleeve
☐ Field Duplicate (FD)	☐ Scoop	☐ Acetate Sleeve
☐ Split Sample (SP)	☐ Hand Auger	☐ 8 oz. WM Glass Jar
☐ Trip Blank (TB)	☐ Power Auger	☐ 4 oz. WM Glass Jar
☐ Rinse Blank (RB)	☐ Cone Penetrometer	☐ Ziplock Bag (metals only)
☐ Performance Eval. (PE)	☐ Split Spoon	☐ Encore (VOCs only)
	☐ Other	☐ 40 mL vial w/ (CH_3OH)
		☐ 40 mL vial w/ ($Na_2H_2SO_3$)
		☐ Other: _____

Weather Notes:

Start: ☐ Clear ☐ Overcast ☐ Rain Temperature Start:_____ ☐ (°F) ☐ (°C)

Stop: ☐ Clear ☐ Overcast ☐ Rain Temperature Stop:_____ ☐ (°F) ☐ (°C)

Comments:_____

_____

_____

_____ _____ _____

Sampler (Print): Sampler (Sign) (Date)

APPENDIX D.
REVISION HISTORY

STANDARD OPERATING PROCEDURE: 1225
Revision: 0, Effective: 2/10/2016

SOIL SAMPLING FOR VOLATILE ORGANIC COMPOUNDS (VOCs)

Revision	Effective Date	Description
0	2/10/16	1. Initial Revision. Prepared in accordance with SOP 850 *Preparation of Standard Operating Procedures.*

APPENDIX C

Quality Assurance Project Plan (QAPP)

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN (QAPP)

277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE

MOUNTAIN VIEW, CALIFORNIA

TABLE OF CONTENTS

Page

1.0 INTRODUCTION 1

2.0 PROJECT TEAM AND RESPONSIBILITIES 1

2.1 ENGEO 1
2.1.1 Project Manager—Shawn Munger, CHG 1
2.1.2 Project Field Manager—Divya Bhargava, PE 1
2.1.3 Quality Assurance Manager—Jeff Adams, PE 1
2.1.4 Site Health and Safety Manager—Divya Bhargava, PE 2
2.2 LABORATORY SUBCONTRACTORS 2
2.3 TRAINING 2

3.0 DATA GENERATION AND ACQUISITION 2

3.1 SAMPLE HANDLING AND DOCUMENTATION 2
3.1.1 Sample Containers 2
3.1.2 Sample Labeling 2
3.1.3 Chain of Custody Record 3
3.1.4 Hold Time Compliance 3
3.1.5 Equipment Decontamination 3
3.2 ANALYTICAL METHODS 3
3.3 QUALITY CONTROL 4
3.3.1 Field QC Samples 4
3.3.2 Laboratory QC Samples 4
3.4 PRECISION, ACCURACY, AND COMPLETENESS 5
3.4.1 Precision 5
3.4.2 Accuracy 5
3.4.3 Completeness 6

4.0 INSTRUMENT CALIBRATION AND MAINTENANCE PROCEDURES 6

5.0 DATA MANAGEMENT 6

5.1 DATA VALIDATION 7
5.2 CORRECTIVE ACTION 7

1.0 INTRODUCTION

ENGEO prepared this Quality Assurance Project Plan (QAPP) to provide Quality Assurance (QA) and Quality Control (QC) procedures for the data collected in association with the SMP. This QAPP, along with the SMP, provide the sampling and analysis protocols consistent with U.S. Environmental Protection Agency (USEPA) guidance The QAPP outlines the project organization and responsibilities with respect to the SMP and provides procedures and methodologies to ensure that the proposed field and laboratory activities generate reliable data.

2.0 PROJECT TEAM AND RESPONSIBILITIES

The following section provides a description of the organizational structure and responsibilities of the individual positions for this project.

2.1 ENGEO

ENGEO is the consulting firm for Warmington Residential and is responsible for implementing the SMP. The project management team is comprised of the Project Manager (PM), Project Field Manager, QA Manager, and Site Safety Manager.

Roles and responsibilities of the ENGEO management team are presented as follows.

2.1.1 Project Manager—Shawn Munger, CHG

The Project Manger will hold overall responsibility for technical and quality-related project matters. The Project Manager will be responsible for committing the necessary resources of ENGEO for the timely completion of the project tasks. Final decisions on recommendations, personnel assignments, and the submittal of final reports to the City of Mountain View and the USEPA will be made by the Project Manager. All of the documents prepared by ENGEO will be reviewed and signed by the Project Manager.

2.1.2 Project Field Manager—Divya Bhargava, PE

The Project Field Manager will be responsible for managing the field personnel and ensuring that field activities are completed pursuant to the workplan. The Project Field Manager will be responsible for day-to-day coordination of field activities, including the coordination of subcontractors, as necessary. The Project Field Manager will schedule pick up or delivery of samples to the appropriate analytical laboratory.

2.1.3 Quality Assurance Manager—Jeff Adams, PE

The Quality Assurance Manager will work with the Project Field Manager to ensure that field activities are carried out in compliance with the QAPP. The Quality Assurance Manager shall approve QA and technical procedures and deliverables from the field and the laboratory.

2.1.4 Site Health and Safety Manager—Divya Bhargava, PE

The Site Health and Safety Manager will ensure that requirements specified in the Health and Safety Plan are being met (Appendix D).

2.2 LABORATORY SUBCONTRACTORS

An offsite laboratory will be utilized for chemical analysis of media and waste materials. The laboratory will be responsible for providing data subject to their own QAPP as well as this QAPP and will designate a Laboratory Quality Assurance Manager. The Laboratory Quality Assurance Manager will review laboratory QA/QC reports, identify problems, and ensure the proper use maintenance, and storage of analytical equipment. Pursuant to California Health and Safety Code Section 25198, all subcontractor laboratories will be certified by the State of California Department of Health Services under the Environmental Laboratory Accreditation Program (NELAP) for the requested analyses. The Laboratory Quality Assurance Manager will report to the Project Field Manager.

2.3 TRAINING

All personnel will be trained and qualified to perform the assigned tasks and will have completed the necessary safety training pursuant to the Health and Safety Plan.

3.0 DATA GENERATION AND ACQUISITION

A description of the sampling scope, including types and numbers of samples required, locations of samples, rationale for design, and sampling methods, is detailed in the SMP and associated Sampling and Analysis Plan (SAP – Appendix B). This section presents the activities associated with data generation and acquisition to ensure that appropriate methods for sampling, measurement and analysis, and QC activities are employed throughout the project.

3.1 SAMPLE HANDLING AND DOCUMENTATION

The types of samples to be collected under the SMP include soil samples and air samples.

3.1.1 Sample Containers

All containers used to hold collected samples will be new containers supplied by the laboratory or an environmental equipment supply company. Appropriate measures shall be made to ensure that the samples are stored at the appropriate temperature while they are held at the site, during transport to the laboratory, and during storage at the laboratory.

3.1.2 Sample Labeling

Each sample taken will be labeled appropriately prior to submittal to the laboratory. All samples subject to analysis will require a sample ID that indicates where the sample was taken and the

matrix associated with the sample (i.e., soil). The sample label will include the following information:

- Sample identification
- Sampling date and time
- Name of the person who collected the sample
- The type of analysis to be performed on the sample
- Site name
- Type of preservative

3.1.3 Chain of Custody Record

All samples will be collected, transported, and received under chain-of-custody (COC) protocols. The COC will contain the following information:

- Project name
- Sample identifications
- Date and time of sample collection
- Analyses to be performed
- Signatures of personnel relinquishing and receiving the samples

The COC forms shall accompany the samples at all times. When transferring possession of the samples, the transferor shall sign the form and enter the date and time the samples were relinquished. The receiver shall also sign and date the form upon receiving the samples.

3.1.4 Hold Time Compliance

For each sample collected, the time a sample is held prior to analysis will be verified with the laboratory prior to submittal. It is the responsibility of the laboratory to analyze the submitted samples within the time indicated on the chain-of-custody form as the hold time, or turnaround time.

3.1.5 Equipment Decontamination

Equipment used in the field will be decontaminated prior to collection of each sample. The equipment will be subjected to a rinse with a biodegradable cleanser, a subsequent rinse with potable water, and a final rinse with de-ionized water. A more detailed description of the decontamination process is provided in the SAP.

3.2 ANALYTICAL METHODS

Analytical methods for the collected samples are discussed in the SMP. The analytical methodologies, required equipment, and laboratory procedures shall meet the standards for the associated USEPA methodology.

3.3 QUALITY CONTROL

QC procedures will be carried out during field sampling and laboratory analysis activities. QC procedures in the field will consist of strict protocols for field sampling and decontamination procedures (described in the SAP) and the collection of field duplicates and equipment rinsate samples for laboratory analysis. Laboratory QC procedures will include the analysis of matrix spike and matrix spike duplicates. A description of the QC procedures is presented in the following sections.

3.3.1 Field QC Samples

QC samples ensure that the sampling and field measurements activities are in control and generate quality data. QC samples for this project will consist of equipment blanks and duplicates/split samples.

- As necessary, equipment blanks will be collected at a frequency of one per day per matrix, or a minimum of 10 percent of the samples collected. An equipment blank will collected by pouring ASTM Type II reagent grade water through or over the purging or sampling device, transferring the water to a sample bottle, and transporting it to the laboratory for analysis.

- As necessary, field duplicates/splits will be collected for every 10 samples collected. Field duplicates will be collected from one sampling location during a single act of sampling. The precision goal for field duplicate analyses will be plus or minus 30 relative percent difference (RPD).

3.3.2 Laboratory QC Samples

Laboratory QC samples will consist of method blanks, laboratory control samples, matrix spikes, matrix spike duplicates, and surrogate spikes.

- Method blanks will be analyzed at a minimum frequency of one per batch, and the concentration of target compounds in the blank must be less than the practical quantitation limit (PQL).

- Laboratory control samples (LCSs) will be analyzed at a minimum frequency of one per batch. Laboratory control samples consist of blank spikes, which are used to determine the accuracy of the analytical procedure by measuring a known concentration of an analyte of interest.

- Surrogate spikes will be performed for all organic standards, samples, and blanks. Each organic standard sample matrix spike, matrix spike duplicate, LCS, and blank is spiked with surrogate compounds prior to purging or extraction. Surrogate spike recoveries must fall within the limits established by the analytical method and if a surrogate spike recovery is outside of acceptable ranges, then a corrective action will be taken.

- Matrix spike/matrix spike duplicates (MS/MSD) are conducted to evaluate the matrix effect of the sample on the analytical method. The MS/MSD analyses will be performed at a minimum frequency of one per each group of 20 samples of the sample matrix. As a result, it is necessary to collect triplicate sample volumes in the field for one sample out of every 20. The RPD goal will be 20 percent.

3.4 PRECISION, ACCURACY, AND COMPLETENESS

The procedures used to assess data are precision, accuracy, and completeness. In the absence of laboratory-specific precision and accuracy limits, the QC limits presented in this section must be met.

3.4.1 Precision

Precision refers to the reproducibility of data under a specified set of conditions. Precision will be evaluated in conjunction with accuracy for the LCS samples. Precision will be determined using the MS/MSD samples and will be expressed as **RPD. RPD** is defined as:

$$RPD = X — X' \frac{\quad}{(X_i + X_2)I2}^{2} x100$$

Here, X, and X2 refer to the larger and smaller of the two observed values, respectively.

Acceptable levels of precision vary with the sample matrix, analytical method, and sample concentration. EPA precision data will be used as a basis for developing acceptance criteria for assessing precision.

3.4.2 Accuracy

Accuracy refers to the degree of agreement between a measurement or set of measurements to an accepted reference value. Accuracy is assessed by means of reference samples and percent recoveries. The calculation of percent recovery (AR) or accuracy is given by the equation:

$$\%R\ 100x_x$$
$$IL_s — LI$$

Here, L_s refers to the measured value of the spiked sample, L refers to the measured value of the unspiked sample, and X refers to the known value of the spike solution. Acceptable levels of accuracy vary with the sample matrix, analytical method, and sample concentration. EPA accuracy data will be used as a basis for developing acceptance criteria for assessing precision.

Field accuracy will be assessed through the analysis of field equipment blanks. The goal for field equipment blanks is that all values are less than the reporting limit for each target constituent.

3.4.3 Completeness

Completeness is the amount of valid data collected as compared to the amount of data that was expected to be collected under normal operating conditions. Two completeness values will be calculated: total number of samples collected, and total number of samples reaching the laboratory intact. The objective for completeness is to recover at least 90 percent of the planned data. The formula for completeness is:

$$\% \bullet completeness = 100 \times \frac{number\ of\ valid\ _results}{number\ _of\ _expected\ _results}$$

4.0 INSTRUMENT CALIBRATION AND MAINTENANCE PROCEDURES

Laboratory analytical instruments will be calibrated in accordance with the procedures specified in the applicable method. All analytes that are reported shall be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in the reference method. Records of standard preparation and instrument calibration will be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration records will be traceable to standard materials.

At the onset of analysis, instrument calibrations will be checked using all of the analytes of interest. At a minimum, calibration criteria will satisfy method requirements. Analyte concentrations can be determined with either calibration curves or response factors, as defined in the method. Guidance provided in USEPA SW-846, or applicable method, should be considered to determine appropriate evaluation procedures.

All equipment and instruments used in the field will be maintained and calibrated to operate within the manufacturer's specifications to ensure the required sensitivity and QA/QC parameters.

Use of all field instruments will be documented by maintaining a log for each individual instrument. Calibration and maintenance requirements of the field instruments follow the manufacturer's specifications. Any required modifications, adjustments, repairs, or replacement of parts for any piece of equipment are recorded in the instrument's logbook. The effects of the modifications will be tested with appropriate standards and the related procedures recalibrated, if required, prior to continued use of the equipment.

5.0 DATA MANAGEMENT

Data measured by field instruments will be recorded in field notebooks or logsheets. The field data will be reviewed by the Project Field Manager to evaluate completeness of the records,

problems encountered in the field, and compliance with workplan-specified field methods. All field records will be retained in the project files.

Laboratory reports will be consistent with Level II documentation and include the following data:

- Narrative, chain-of-custody, and method references
- Analytical results
- Laboratory QC results including surrogate recoveries and LCS recoveries
- Sample spike recoveries
- Acceptance criteria for applicable QC samples

The laboratory QA/QC manager will determine if the data quality objectives (DQOs) for the analytical data have been met. Results will be documented and summarized in a data validation memorandum, which is reported along with the associated data.

All data generated will be accessible to downloading into USEPA's system as necessary.

5.1 DATA VALIDATION

Data validation is the process of screening and accepting or rejecting data to use for project evaluation and decision-making. Whether or not data is acceptable is based on compliance with specified criteria, including meeting holding times, verifying calibration results, and meeting goals for laboratory QC sample results (i.e., blank and duplicate sample results).

Data validation activities include the following:

- Examination of the raw data to verify adequacy of documentation and check the accuracy of spreadsheet calculations and the data entered into tables and figures.

- Confirmation that the calibration standards are within the expected values.

- Review of all associated blank, duplicate, spike, standard, and QC data to ensure that results meet analytical objectives.

- Flagging laboratory results that may be suspect. Suspect data will be assessed to determine whether the information is usable or not.

5.2 CORRECTIVE ACTION

Corrective action will be initiated when deficiencies are encountered during the project or when QAPP guidelines/objectives are not met. Corrective action will be performed prior to analysis of the next batch of data to help prevent recurrent erroneous data. Corrective actions may consist of one or more of the following:

- Datum is annotated
- Documentation is reviewed for adherence to QA/QC procedures
- Measurement is repeated to check the error
- Duplicate sample is reanalyzed
- Calibrations are checked and/or repeated
- Measuring device is repaired or replaced
- Sample is re-collected

The Quality Assurance Manager will be responsible for initiating and approving corrective actions. All corrective actions will be handled individually.

APPENDIX D

Standard Operating Procedures (SOPs): Field Monitoring of VOCs by Modified Method TO-14

APPENDIX D

Standard Operating Procedures (SOP)
Field Monitoring of VOCs by Modified Method TO-14

277 Fairchild Drive, 228/236 Evandale Avenue
Mountain View, California

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATIONS 1
2.0 METHOD SUMMARY 1
3.0 INSTRUMENTATION AND EQUIPMENT 1
3.1 GAS CHROMATOGRAPH AND PERIPHERALS 1
4.0 REAGENTS AND STANDARDS 2
4.1 HIGH PURITY NITROGEN COMPRESSED GAS 2
4.2 PRIMARY (STOCK) STANDARDS 2
4.3 SECONDARY (WORKING) STANDARDS 2
5.0 DETECTION AND REPORTING LIMITS 3
5.1 METHOD DETECTION LIMIT 3
5.2 REPORTING LIMITS 4
6.0 INTERFERENCES 5
7.0 PRECISION, BIAS AND WORKING RANGE 5
8.0 SAMPLE COLLECTION AND HOLDING TIMES 5
9.0 PROCEDURES, CALIBRATION, QA/QC ANALYSIS AND SAMPLE ANALYSIS 6
9.1 SYSTEM SET-UP 6
9.2 SYSTEM START-UP – INSTRUMENT 6
9.3 SYSTEM CALIBRATION 6
9.4 DAILY CONTINUING CALIBRATION VERIFICATION (CCV) 6
9.5 BLANKS 7
9.6 SAMPLES 7
10.0 QUALITY ASSURANCE AND QUALITY CONTROL 7
10.1 INITIAL CALIBRATION (ICAL) 7
10.2 CONTINUING CALIBRATION (CCAL) 8
10.3 BLANKS 8
10.4 SAMPLE DUPLICATES AND REPLICATES 8
11.0 FIXED-BASED LABORATORY VERIFICATION SAMPLING 9
12.0 DATA/RECORDS MANAGEMENT AND REPORTS 9

13.0 TROUBLESHOOTING AND MAINTENANCE.....10

13.1 PREVENTIVE MAINTENANCE.....10

13.1.1 Preparation PM 10

13.1.2 Instrument PM 10

13.2 TROUBLESHOOTING 11

13.2.1 Low sensitivity.....11

13.2.2 Poor peak shapes and tailing.....11

13.2.3 Noise in one or both detectors 11

1.0 SCOPE AND APPLICATIONS

This Standard Operating Procedures (SOP) document has been prepared to help ensure consistent analytical protocol for field monitoring of volatile organic compounds (VOCs) using modified method TO-14. The scope of topics discussed in this SOP includes the following:

- Method Summary
- Instrumentation and Equipment
- Reagents & Standards
- Detection and Reporting Limits
- Interferences
- Precision, Bias and Working Range (Method Performance)
- Sample Collection and Holding Times
- Procedures, Calibration, QA/QC and sample Analysis
- QA/QC Requirements
- Fixed-Based Laboratory Verification Sampling
- Data and Records Management and Reporting
- Troubleshooting Problems and Preventative Maintenance

2.0 METHOD SUMMARY

The monitoring system measures a select group of VOCs in a gas matrix (indoor air, outdoor air or soil gas). The primary VOCs are chlorinated compounds, including tetrachloroethene (PCE) and trichloroethene (TCE), but it can also analyze for hydrocarbons such as benzene and ethylbenzene. The system may be controlled remotely and data downloaded via the internet in real-time.

3.0 INSTRUMENTATION AND EQUIPMENT

3.1 GAS CHROMATOGRAPH AND PERIPHERALS

The system consists of the following elements:

- Gas chromatograph (GC) (SRI 8610) with an electron capture detector (ECD) and optional photoionization detector (PID).

- Column: 30M MTX-1 Megabore.

- Sample injection valve with 1 milliliter (ml) sample loop.

- TCE and PCE MDL: H&P Mobile Geochemistry Inc. (a California, NELAC and DOD certified environmental laboratory) conducted a method detection limit (MDL) study on November 5, 2015, which established an MDL of 0.38 micrograms per cubic meter ($\mu g/m^3$) for TCE and 0.69 $\mu g/m^3$ for PCE.

- TCE and PCE Quantitation Limit: For the MDL study conducted in November 2015 a 0.5 parts per billion by volume (ppbv) standard was used, resulting in 0.54 □g/m^3 for TCE and 0.69 □g/m^3 for PCE. A lower quantitation limit may be achieved by using a lower concentration standard.

- Performance Data: Performance Data: Inter lab comparison data from EPA-ORD[1] indicates accuracy expressed as %RPD of 16.6%, -16.5% and -24%, all within accuracy of the method.

- Computerized data acquisition system (*Peaksimple* by SRI Instruments).

- Remote connection via Wireless connection (ethernet cable, cell or wifi).

A low-flow vacuum pump draws the air sample through the tubing and through the sample loop from the selected sample location. When purging is complete (approximately 30 seconds), the sample is injected into the GC for analysis. Analysis time is approximately 8 to 10 minutes.

The above-mentioned equipment requires 115 VAC as a power source to operate. This power can be applied by external power sources available at the site, or by an internal, gasoline-operated generator located on the site itself.

The data acquisition software (*Peaksimple*) acquires the chromatographic data and also controls the sample injection, GC analysis, and stores the data to a summary file on a laptop. Remote access to the laptop and the data is enabled by a wireless connection.

4.0 REAGENTS AND STANDARDS

4.1 HIGH PURITY NITROGEN COMPRESSED GAS

4.2 PRIMARY (STOCK) STANDARDS

Vapor standards (TCE) are purchased from a certified supplier at 1,000 ppbv. Certificates and preparations of all secondary standards are recorded on a log sheet.

4.3 SECONDARY (WORKING) STANDARDS

Made by diluting primary standard with ultra-pure air or nitrogen. Typical concentrations range from 1 ppbv to 100 ppbv. It is preferable to prepare these standards in SUMMA canisters, as they are stable for a longer time period. Tedlar® bags may be used in lieu of SUMMA canisters if prepared fresh daily.

[1] http://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=308904

- Using the Entech can cleaner, clean five, 1-liter SUMMA cans. Refer to the Entech literature for guidance. In general, 10 cycles of evacuation down to 1,000 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.

- Introduce about 20 microliters (□) of reagent grade water into the quick connects on each of the three SUMMA canisters to be used.

- Using a gas-tight 100 milliliter (ml) syringe, introduce the following amounts of 1 parts per million by volume (ppmv) primary stock vapor standard into each of the 6 humidified SUMMA canisters.

Target Concentration (ppbv)	Volume of Stock (ml)
5	10
10	20
25	50
100	200
500	1000
1000	Fill Can

- Then attach SUMMA canister to nitrogen and pressurize to a stable 14.7 pounds per square inch (psi) (2 ATM) gage pressure. Record preparation in the standard logbook. Standards may be prepared at other concentrations if a different analytical range is possible and desired. (See section 5.2 for more information).

5.0 DETECTION AND REPORTING LIMITS

5.1 METHOD DETECTION LIMIT

The 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. MDLs for each target analyte are established prior to a project. The analyst shall maintain proof of the MDL demonstrations (i.e., before project samples are analyzed) and upon request in the format specified.

MDLs will be demonstrated using the following instructions:

1. Estimate the MDL using one of the following methods:

   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 at least 7 replicate measurements of the target analyte, 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 5 to 10 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:

$$MDL = 3.14(s)$$ [2]

6. If the spike level used in step 2 is more than 10 times the calculated MDL, repeat the process using a smaller spike level. If the calculated MDL is more than the spike level concentration, repeat the process using a higher spike level. If the calculated MDL is less than 10 percent of the spike level concentration, repeat the process using a lower spike level.

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

5.2 REPORTING LIMITS

The reporting limits (RLs) will be dependent upon the sample matrix, indoor air or soil vapor, and the calibration range. The ECD detector has a lower sensitivity than the PID and therefore can be used to achieve lower RLs than the PID. At mid-range concentrations, the results from the PID and

[2] 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

ECD can be compared as verifications. At higher concentrations, the ECD will be over-range and will not provide accurate results.

The normal RLs are nominally from 0.5 ppbv to 100 ppbv of each compound. These limits should be compared annually to the MDLs to make certain that they are appropriate. Depending upon client data quality objectives (DQOs), the RLs may change. The RLs; however, are still dependent upon the calibration curve. The RLs can be as low as 0.2 ppbv. However, the lowest point in the calibration curve cannot be greater than 5 times the RL. Some projects may require that the lowest point on the calibration curve be set at the RLs and these will be project specific.

When samples are diluted, the RLs are raised proportionately. All multipliers must also be applied to the RLs as well. Dilutions are recorded on the daily extraction/run logs and are entered into the *Peaksimple* software for the particular analysis.

6.0 INTERFERENCES

When analyzing for VOCs, samples can contain high concentrations of target and non-target analytes. These analytes may interfere with the ECD and PID detectors. 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 QA/QC requirements. In addition, the sample loops may be prone to carry over. The sample loop should be flushed well after a high concentration sample is analyzed (greater than 50 times the RL). In addition, a blank should be analyzed after any high level sample (greater than 50 times the RL) to ensure that carry over is eliminated.
Specifically for TCE, potential interferences may be due to the presence of chloroform and bromodichloromethane. This may be resolved by slowing the analytical run. There are no common interferences for PCE.

7.0 PRECISION, BIAS AND WORKING RANGE

The working range of the instrument is between the MDL for the analyte and the concentration of the high standard used for system calibration. In the event that sample results are greater than the amount used for the high standard, then sample dilution is necessary. Results reported that are between the MDL and RL should be flagged with the “J” flag as a quantitative estimate.

The required precision of this method is 30 percent. Precision is determined prior to each sampling program by performing replicate analysis of a mid-range standard.

The required bias of this method is +/- 30 percent, which may be determined by analyzing a mid-point standard, mid-point second source standard, or a performance test sample.

8.0 SAMPLE COLLECTION AND HOLDING TIMES

The sample is pulled through the tubing and flushed through the sample loop by a low-flow vacuum pump in real-time; therefore, holding times are not applicable.

9.0 PROCEDURES, CALIBRATION, QA/QC ANALYSIS AND SAMPLE ANALYSIS

9.1 SYSTEM SET-UP

A flat surface approximately 3 feet by 3 feet is necessary to set up the gas chromatograph and supporting equipment. The nitrogen cylinder should be secured in an upright or horizontal position in a mobile tank rack. Connect the nitrogen to the instrument using clean copper tubing.

9.2 SYSTEM START-UP – INSTRUMENT

Open the valve on the nitrogen carrier gas and ensure that no leaks are detected. Let the nitrogen run for approximately 5 minutes to flush all air out of the columns and detector bodies. Load the *Peaksimple* program into the computer. Turn on the GC and check to make sure the carrier flow is within acceptable range.

The recommended GC conditions are:

- Initial temp: 60 to 90°C
- Program rate: Isothermal
- Injection temp: 125°C
- ECD Detector temp: 250°C
- PID Detector temp: 150°C

Turn on the PID lamp and allow the PID and ECD to stabilize. Determine when stable by monitoring the baseline detector voltages on the *Peaksimple* software. PID voltage should be less than 20 mV and stable. ECD voltage should be less than 500 mV and stable. This may take up to 1 hour. The system is now ready for operation.

9.3 SYSTEM CALIBRATION

Prior to performing analyses, the GC must be calibrated to ensure system accuracy. To calibrate the GC, standards that were prepared following the procedures outlined in Section 5.1 are injected into the GC and analyzed. A minimum of 3 concentration levels are to be used to generate the calibration curve for each analyte. The end result is a calibration curve for each analyte on each detector. The linearity of the calibration curve is to be evaluated per Section 10.1. For the real-time TCE air monitoring, a 1 ppbv TCE sample, or lower, will be analyzed daily.

9.4 DAILY CONTINUING CALIBRATION VERIFICATION (CCV)

Once an acceptable calibration curve is generated for all analytes to be reported, this curve may be used to analyze client samples as long as it is still valid. To determine if a curve is still valid, a

mid-point standard is analyzed at a regular interval, and the percent difference (drift) of each analyte is calculated using the following equation:

$$\% D = C_E - C_C / C_E * 100$$

Where: C_E = Expected analyte concentration of CCV
C_C = Determined analyte concentration of CCV

See Section 10.2 for CCV criteria and corrective actions.

9.5 BLANKS

Once the calibration is verified as valid, a method blank is to be analyzed. This is performed by injecting clean air into the sample loop and analyzing per the method. The blank must meet criteria set in Section 10.3.

9.6 SAMPLES

Once an acceptable blank is analyzed, sample analysis can proceed. All samples are to be analyzed under the same analytical conditions as the standards and blanks. Air samples will be recovered using 50 cc glass syringes (KDS 78-0877, or equivalent).Samples are to be evaluated according to criteria set forth in Section 10.4.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

Quality Assurance (QA) and Quality Control (QC) are a set of procedures and conditions implemented to assure data produced are of known and proven quality. 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 insure proper QA/QC.

10.1 INITIAL CALIBRATION (ICAL)

The computer will construct the calibration "curve" according to one of several methods. Among these are (a) straight line, (b) straight line through origin, (c) point to point, (d) quadratic. For the PID detector, the recommended curve is either method (a) or (b). For the ECD, method (c) is used due to the small linear range of the ECD detector.

Area counts from each calibration standard are entered into an Excel spreadsheet template (svfixed.xls), which is in the laboratory. The spreadsheet computes the response factor (RF) for each standard, the average response factor for all the standards, the standard deviation (SD) of the response factors, and the %RSD as:

$$\%RSD = SD/ave\ RF*100$$

If the %RSD is less than or equal to 30 percent, the values are entered into the *Peaksimple* software and used for quantitation. If the %RSD is greater than 30 percent, a new ICAL is performed.

All calibrations are to be reviewed and approved by the laboratory director or QAO before use on client samples.

Hardcopy outputs of the chromatograms are to 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.

10.2 CONTINUING CALIBRATION (CCAL)

The calibration of the instrument is checked prior to running samples. The continuing calibration (CCAL) or continuing calibration verification (CCV) checks the validity of the ICAL. Normally a standard corresponding to the midpoint of the calibration curve is chosen. Response of the compounds of interest must be within 30 percent of the calibrated amount for the curve to be valid.

Corrective Action: Re-prepare and/or re-analyze the CCV standard. If failure is confirmed, perform a new ICAL.

10.3 BLANKS

Method blanks are performed at least daily, and typically every sequence, by drawing clean air through the sampling equipment and analyzing. These blanks verify all components of the sampling and analytical system are free of contamination. Additional blanks are recommended immediately after any high concentration samples. The results of all blank analyses are recorded in the data tables. If a contaminant is found, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the blank if above the RL.

10.4 SAMPLE DUPLICATES AND REPLICATES

A sample replicate is a sample that is collected as soon as possible after the original sample was collected from that same location. A sample duplicate is a repeat analysis of the same sample. Sample replicates may be performed with the system by repeating the analysis of the same sample location. Sample duplicates cannot be performed with the system. Replicate results are evaluated against the original sample results by calculating the Relative Percent Difference (RPD). The RPD may be calculated using the following general equation:

$$RPD = \frac{CS - CD}{(CS + CD)/2} \times 100$$

Where: CS = Concentration of analyte in sample
CD = Concentration of analyte in replicate or duplicate

This RPD criterion is 30 percent or less.

Corrective Action: Re-collect and re-analyze one more replicate.

11.0 FIXED-BASED LABORATORY VERIFICATION SAMPLING

Initially on a daily basis during real-time monitoring activities, one split sample will be recovered. The split samples will be recovered as follows:

A Teflon® "T" and tubing will be attached to 1-liter SUMMA canister and a 50 cc glass syringe (KDS 78-0877, or equivalent). The sample will be extracted with the syringe, simultaneously opening the summa canister valve. The SUMMA canister(s) will be submitted to an ELAP-certified laboratory for TO-15(SIM) analysis on 72-hour turnaround. For TCE, 1,2-dichloroethene (1.2-DCE), and vinyl chloride, the respective reporting and detection limits will be maintained at 0.2 □g/m^3 and 0.1 □g/m^3, respectively.

12.0 DATA/RECORDS MANAGEMENT AND REPORTS

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

<u>The Data Package</u>

For the continuous monitoring system, the data package will consist of the following in the order listed or as near as possible to that order:

a. The log files created by *Peaksimple* summarizing all the results for each location;
b. An Excel file containing all of the data for all of the probes;
c. Summaries of the ICAL for each detector;
d. Chromatograms from the daily ICAL and the CCAL (electronic);
e. Chromatograms for all sample analyses (electronic);
f. Analyses report.

<u>Final Report</u>

The following information should be included in the analyses report:

- Locations of the samples;

- Summary of the results;
- Description of the system as configured;
- Any deviations from the QA/QC requirements listed in this SOP.

The analyst shall verify that the test results meet all the requirements of this SOP or shall provide a reasonable explanation as to why they do not.

13.0 TROUBLESHOOTING AND MAINTENANCE

13.1 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 described PM in two sections: preparation and instrument PM.

13.1.1 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 the glass syringes before use.

If samples are suspected to be elevated, the sample is to be diluted before it is to be analyzed to prevent contamination of the GC system.

13.1.2 Instrument PM

There are 5 pieces of instrumentation involved in the analysis of VOCs by GC/ECD/PID. They are the following:

- The gas chromatograph and 2 detectors;
- Sample loop;
- The computer system.

The analyst follows the manufacturer's recommendations on the gas chromatograph. The 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 computer system PM consists of keeping dust and dirt out of the components and backing up the data and methods as often as possible.

13.2 TROUBLESHOOTING

There are potential issues that may occur during analyses. The following are the most frequent issues, along with the suggested investigative steps:

13.2.1 Low sensitivity

Possible causes: Leaks or dirty PID lamp.

Leaks can occur at the septa and at the analytical column connections. These are to be inspected and the septa and/or ferrules replaced if necessary. Remove and clean PID lamp. Replace and re-test.

13.2.2 Poor peak shapes and tailing

Possible causes: Poor column installation or poor carrier flow.

Reinstall column, check and adjust carrier flow, and re-test.

13.2.3 Noise in one or both detectors

Noise in the PID detector could suggest a leak or a loose wire to the PID detector. A leak detector may be used to find the source so that it can be corrected. If the noise is in both detectors, the ECD and the PID, the leak will be outside the gas chromatograph.

APPENDIX E

Health and Safety Plan

APPENDIX E

HEALTH AND SAFETY PLAN

277 FAIRCHILD DRIVE, 228/236 EVANDALE AVENUE

MOUNTAIN VIEW, CALIFORNIA

TABLE OF CONTENTS

Page

1.0 INTRODUCTION 1

2.0 SITE SAFETY REGULATORY REQUIREMENTS 1

3.0 STAFF ORGANIZATION, QUALIFICATIONS, AND RESPONSIBILITIES 1

3.1 GENERAL 1
3.2 PROJECT MANAGER 1
3.3 HEALTH AND SAFETY MANAGER (HSM) 2
3.4 SITE SAFETY OFFICER (SSO) 2
3.5 PERSONS CERTIFIED IN FIRST AID AND CPR 3

4.0 HAZARD/RISK ANALYSIS 3

4.1 IDENTIFIED TASKS 3
4.2 POTENTIAL HAZARDS 3
4.2.1 General Safety Hazards 3
4.2.1.1 Noise 4
4.2.1.2 Heat Stress 4
4.2.1.3 Cold Stress 4
4.2.1.4 Sunburn 5
4.2.1.5 Heavy Equipment Operation 6
4.2.1.6 Slip/Trip/Fall Hazards 6
4.2.1.7 Lifting Hazards 6
4.2.1.8 Tool and Equipment Hazards 6
4.2.1.9 Fire Hazard Control 6
4.3 BIOLOGICAL HAZARDS 7
4.3.1 Ants, Bees, Wasps, Hornets and Yellow Jackets 7
4.3.2 Spiders and Fleas 7
4.3.3 Rattlesnakes and Scorpions 7
4.3.4 Poisonous Plants 8
4.3.5 Hantavirus 8
4.3.6 Bird Excrement and Amplified Fungal Growth 8
4.4 CHEMICAL HAZARDS 8

5.0 SAFETY AND HEALTH TRAINING 8

5.1 CERTIFICATION OF TRAINING 8
5.2 TAILGATE SAFETY MEETINGS 9

6.0 PERSONAL PROTECTIVE EQUIPMENT 9

6.1 LEVELS OF PPE 9
6.1.1 Level D 9
6.1.2 Level D Modified 9

6.1.3 Level C 10
6.1.4 Level B 10
6.1.5 Level A 10
6.2 INSPECTION OF PPE 11

7.0 SAFETY PROCEDURES, ENGINEERING CONTROLS, AND WORK PRACTICES 11

7.1 GENERAL SITE RULES/PROHIBITIONS 11
7.1.1 Engineering Controls and Work Practices 12
7.1.2 Employee Rotation 12
7.1.3 Work Practices and Procedures 12
7.2 MATERIALS HANDLING 12
7.2.1 Spill and Discharge Control 12
7.2.2 Notification of Spills and Discharges 13
7.2.3 Material Storage, and Disposal 13
7.3 HAZARD COMMUNICATION 14
7.4 SANITATION 15
7.5 MACHINERY AND MECHANIZED EQUIPMENT 15
7.6 CONFINED SPACE OPERATIONS 17
7.7 SITE ILLUMINATION 17

8.0 SITE CONTROL MEASURES 17

8.1 SITE CONTROL LOG 17
8.2 DECONTAMINATION 18
8.2.1 Personnel 18
8.2.2 Equipment 18

9.0 EMERGENCY EQUIPMENT 18

10.0 EMERGENCY RESPONSE AND CONTINGENCY PROCEDURES 19

10.1 PRE-EMERGENCY PLANNING 19
10.2 LINES OF AUTHORITY 19
10.3 EMERGENCY RECOGNITION AND PREVENTION 19
10.4 PROCEDURES FOR SITE EVACUATION 19
10.4.1 Emergency Equipment 19
10.4.2 Adverse Weather Conditions 19
10.4.3 Earthquakes 20
10.4.4 Evacuation Routes and Places of Refuge 20
10.4.5 Site Security and Control 20
10.5 NEAREST HOSPITAL 21
10.6 EMERGENCY ALERTING AND RESPONSE PROCEDURES 21
10.6.1 Emergency Alerting Procedures 21
10.6.2 Emergency Telephone Numbers 22
10.7 COMMUNITY ALERT PROGRAM 22
10.8 PROCEDURES FOR INCIDENT REPORTING 22

11.0 CERTIFICATE OF WORKER/VISITOR ACKNOWLEDGMENT.....23

12.0 REPORTING 23

12.1 LOGS, REPORTS, AND RECORDKEEPING 23

ATTACHMENT A – Accident/Injury/Illness Investigation
ATTACHMENT B – Project Manager Daily Log
ATTACHMENT C – Worker/Visitor Entry and Exit Control Log
ATTACHMENT D – Acknowledgement
ATTACHMENT E – Job Site Emergency Procedures
ATTACHMENT F – Hospital Map

GLOSSARY

APR	Air Purifying Respirator
ACGIH	American Conference Governmental Industrial Hygienists
AIHA	American Industrial Hygiene Association
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
CCR	California Code of Regulations
CFR	Code of Federal Regulations
COPC	Contaminant of Potential Concern
CPR	Cardiopulmonary resuscitation
CRZ	Contaminant Reduction Zone
dBA	Decibels on the A scale
DOT	Department of Transportation
DTSC	California Department of Toxic Substance Control
EPA	U.S. Environmental Protection Agency
^{0}F	Degrees Fahrenheit
eV	Electron Volt
EZ	Exclusion Zone
FEV	Forced expiratory volume
FVC	Forced vital capacity
GISO	General Industry Safety Order
GPS	Global Positioning System
HSM	Health and Safety Manager
mg/m^3	Milligrams per cubic meter
MSDS	Material Safety Data Sheets
OSHA	Occupational Safety and Health Administration
PAH	Polyaromatic Hydrocarbons
PEL	Permissible Exposure Limit
PID	Photoionization Detector
PM	Project Manager
ppb	Parts per Billion
PPE	Personal Protective Equipment
ppm	Parts per million
PSHM	Program Safety and Health Manager
ROPS	Roll over protection structure
RV	Reserve volume
HASP	Health and Safety Plan
SSO	Site Safety Officer
SVOC	Semi-volatile organic compound
TLV	Threshold Limit Value
TPH	Total Petroleum Hydrocarbons
TWA	Time Weighted Average
µg/L	Micrograms per liter
VCP	Voluntary Cleanup Program
VOC	Volatile Organic Compound

DISCLAIMER

This Health and Safety Plan (HASP) was prepared for use at the 277 Fairchild Drive, 228/236 Evandale Avenue in Mountain View, California (Site). The Plan was prepared based on the best available information regarding the physical and chemical hazards known or suspected to be present at the Site and for the execution of the proposed scope of work. It is not possible in advance to discover, evaluate, and protect against all possible hazards, which may be encountered during the duration of this project. Therefore, this HASP may not be appropriate if the work is not performed by or using the methods presently anticipated. In addition, as the work is performed, conditions different from that anticipated may be encountered and this HASP may have to be modified.

Adherence to the requirements of this HASP will significantly reduce, but not eliminate, the potential for occupational injury and illness at the Site. The guidelines contained in this HASP were developed specifically for the soil removal project at the Site described herein and should not be used at any other site without the review and approval of a qualified health and safety professional.

1.0 INTRODUCTION

This Health and Safety Plan (HASP) sets forth the minimum health, safety, and emergency response requirements for activities involving, or potentially involving, employee exposure to physical or chemical health hazards associated with the implementation of the Site Management Plan (SMP) prepared for the Site located at 277 Fairchild Drive, 228/236 Evandale Avenue in Mountain View, California.

The proposed development consists of 22 row homes and four single-family homes with appurtenant utilities, parking, and landscaping. Proposed residential development of the Property will include the demolition/removal of existing structures, paving, and utilities. Minimal site grading will occur to achieve residential pad grades. Utility trenching, up to 6 feet in depth, will also be conducted as part of site development.

2.0 SITE SAFETY REGULATORY REQUIREMENTS

Work performed under this HASP will comply with applicable Federal, State of California, and local safety and occupational health laws and regulations. Applicable regulations include, but are not limited to, Occupational Safety and Health Administration (OSHA) Standards 29 CFR, Part 1910.120, "Hazardous Waste Site Operations and Emergency Response"; 29 CFR 1910.1025, General Industry Standards; 8 CCR 5216. General Industry Standard 29 CFR 1926.62, Lead in the Construction Industry; and 8 CCR 1532.1, Lead in the Construction Industry. Where the requirements of these specifications, applicable laws, criteria, ordinances, regulations and referenced documents vary, the most stringent will apply.

3.0 STAFF ORGANIZATION, QUALIFICATIONS, AND RESPONSIBILITIES

3.1 GENERAL

This section of the HASP outlines the organizational structure and Site personnel responsible for the safety and health of personnel during the proposed work. The replacement of any member of the Health and Safety Staff requires the acceptance of the Project Manager. Replacement requests will include the names, qualifications, duties, and responsibilities of each proposed replacement.

3.2 PROJECT MANAGER

The Project Manager (PM) for ENGEO is Shawn Munger, CHG. Mr. Munger is responsible for:

- Oversight of Site activities required to implement this HASP.
- Directing work performed under this contract.
- Verifying that work is completed in accordance with the project workplan.

3.3 HEALTH AND SAFETY MANAGER (HSM)

The Site Health and Safety Manager (HSM) is Divya Bhargava, PE. Ms. Bhargava is responsible for:

- Implementing and enforcing of the HASP.
- Providing the initial and periodic site-specific training.
- Monitoring of remediation activities.
- Coordinating activities in the event of an onsite emergency.
- Evaluating air monitoring data and changes to engineering controls, work practices, and personal protection equipment (PPE) that may be warranted.
- Receiving onsite accident reports.

3.4 SITE SAFETY OFFICER (SSO)

The Site Safety Officer is Divya Bhargava, PE. Ms. Bhargava is responsible for:

- Conduct onsite training and the day to day onsite implementation and enforcement of the HASP.

- Be assigned to the site on a full-time basis for the duration of field activities.

- Have authority to ensure Site compliance with specified safety and health requirements, Federal and State of California OSHA regulations and all aspects of the HASP including, but not limited to, activity hazard analyses, air monitoring, use of PPE, decontamination of personnel and equipment, site control, standard operating procedures used to minimize hazards, safe use of engineering controls, the emergency response plan, confined space entry procedures, spill containment-program, and preparation of records by performing a daily safety and health inspection and documenting results on the Daily Safety Inspection Log.

- Have the authority to stop work if unacceptable health or safety conditions exist, and take necessary action to re-establish and maintain safe working conditions.

- Consult with proper authorities and coordinate any modifications to the HASP with the Project Manager.

- Serve as a member of ENGEO's quality control staff on matters relating to safety and health.

- Conduct accident investigations and prepare accident reports (Attachment A).

- Review results of daily quality control inspections and document safety and health findings into the Project Manager's Daily Log (Attachment B).

- In coordination with site management recommend corrective actions for identified deficiencies and oversee the corrective actions.

3.5 PERSONS CERTIFIED IN FIRST AID AND CPR

CPR, if needed, will be conducted by trained personnel, or offsite emergency responders (i.e. paramedics, fire fighters). Many personnel with 40-hour Hazardous Waste Operations and Emergency Response training will have completed CPR and first aid courses as part of their training. The consultant/contractor is responsible for identifying and informing workers of designated first aid trained personnel. These persons may perform other duties but will be immediately available to render first aid when needed. The identity of these persons will be posted and made known to all personnel involved in this project.

4.0 HAZARD/RISK ANALYSIS

4.1 IDENTIFIED TASKS

The tasks identified for the conduct of this soil exploration project include the following:

- Mobilization/demobilization.
- Subsurface exploration (rotary drilling and cone penetration testing).
- Containerization of soil cuttings.
- Backfilling of soil borings.

4.2 POTENTIAL HAZARDS

4.2.1 General Safety Hazards

Potential safety hazards will include, but are not limited to, general construction hazards, such as:

- Physical contact with heavy equipment.
- Physical contact with motor vehicles.
- Slips/trips/falls due to unstable surfaces, or uneven terrain.
- Exposure to site contaminants including dust.
- Equipment noise.
- Buried utility lines and energized overhead and underground power lines.
- Heat stress and cold stress.
- Lifting heavy objects.
- Sunburn.
- Biological hazards.

These hazards are described below.

4.2.1.1 Noise

Noise exposures will be controlled to levels below the permissible noise exposure levels, which are equivalent to an 8-hour time weighted average (TWA) level of 85 decibels (dBA). Reduction of exposures may be by engineering controls or adequate hearing protection. Engineering controls will include isolation of the noise source by their enclosure and reduction of noise transmission by application of noise absorbing materials.

Most work site noise will originate from heavy equipment. As a result, equipment operators and observers will be required to use hearing protection when exposed at or above 85 decibels. A copy of the OSHA Occupational Noise Standard, 29 CFR 1910.95 will be available and copies will be made available to employees upon request.

4.2.1.2 Heat Stress

A worker's risk for developing heat stress is greatly increased when wearing impermeable clothing or respirators. This type of clothing interferes with the body's normal cooling mechanisms by preventing the evaporation of perspiration. For workers who wear permeable clothing, work/rest schedules recommended in the current ACGIH Threshold Limit Values (TLV) for Heat Stress will be followed. For workers who wear semi-permeable or impermeable clothing, technical guidelines in "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities" will be followed. Monitoring of personnel wearing impermeable clothing will commence when the ambient temperature is above 70 degrees Fahrenheit. Monitoring frequency will increase as the ambient temperature increases or as slow recovery rates are observed. A shady rest area and an adequate supply of cool drinking water will be provided for the workers.

4.2.1.3 Cold Stress

Cold stress may be an exposure hazard during the project based on the current work schedule and anticipated weather conditions. Exposure to cold weather can lead to frost bite and/or hypothermia. The signs and symptoms of excessive exposure to cold are listed in Table 4.2.1.3-1.

TABLE 4.2.1.3-1
Different Levels of Cold Exposure and Associated Symptoms

Condition	Signs and symptoms
Hypothermia - A condition when a person's body loses heat faster than it can be produced.	Vague, slow, slurred speech, impaired judgment, forgetfulness, memory lapses, drowsiness, inability to use the hands.
Frostbite - A condition where a part of the body is frozen	Loss of the sensation of touch, pressure and pain in the affected part of the body. This may occur without awareness of any numbness. Just before freezing, the skin becomes bright red and at freezing, small patches of white appear on the skin.

When weather conditions are cold, wet and windy, the following precautions will be instituted:

- Field personnel should wear layered clothing. Mittens, heavy socks, hats, jackets/vests, long underwear, glove liners or other suitable clothing should be worn when air temperatures fall below 40°F. Chemical protective clothing will be worn over the warm garments when protective clothing is required by the field operations.

- At temperatures below 30°F, temperature insulating suits and gloves should be considered.

- Protective outerwear should be used to prevent wetting of work shoes and feet, when appropriate.

- Additional clothing worn in layers allows gradual removal as work activities generate metabolic heat.

- At temperatures below 35°F, raingear should be worn if an employee could become wet on the job.

- At temperatures below 35°F, employees shall be provided with warm (65°F or above) break areas. If appropriate, space heaters will be provided to warm hand and feet.

- Hot liquids such as soups and warm drinks should be consumed during break periods. Caffeine beverages should be limited due to attendant diuretic and circulatory effects.

- A buddy system shall be practiced at all times. An employee that is observed shivering or showing signs of frostbite shall leave the cold area immediately.

- Work should be arranged to avoid sitting or standing for long periods.

- All employees who work in cold areas should be trained in the following subjects:
  - Proper first aid treatment for cold stress
  - Proper clothing practices
  - Proper eating and drinking habits
  - Recognition of impending adverse health effects due to cold
  - Safe work practices

4.2.1.4 Sunburn

Sunburn is caused by overexposure to ultraviolet light (sunshine). The symptoms of exposure are not usually apparent until two to four hours after the exposure ceases. Depending upon the severity of the exposure, the symptoms can range from reddening of the skin, accompanied by mild discomfort, to painful deep burns and blisters. Although light-haired, fair-skinned, blue-eyed personnel are at the greatest risk of sunburn, all complexion types can develop sunburn.

The physical hazard of sunburn can be controlled by: (1) providing a shady rest area; (2) wearing appropriate clothing (long pants and tee shirts, i.e. no tank tops); (3) wearing sunscreen with an appropriate protection factor, as appropriate; and (4) working in shifts.

4.2.1.5 Heavy Equipment Operation

The contractor is responsible for all personnel associated with heavy equipment operation. Equipment operators should maintain a constant awareness of their surroundings and associated hazards. Constant visual or verbal contact between the equipment operators and laborers will facilitate such awareness. When operating heavy equipment near an embankment, a spotter shall be present at all times to observe the soil behavior on which the unit is situated. All heavy equipment shall be equipped with a roll over protection structure (ROPS) and seat belts. Operators shall use seat belts at all times when in the cab of operating equipment. All personnel will wear high visibility safety vests and hearing protection if appropriate.

4.2.1.6 Slip/Trip/Fall Hazards

Prevention of slips/trips and fall hazards can be reduced to a minimum if employees use caution when working on slick, uneven or unsteady surfaces. The risk of injury will be minimized by implementing proper site control measures such as daily safety meetings, proper footwear and by keeping the work area free of obstructions.

4.2.1.7 Lifting Hazards

Field operations often require that heavy physical labor tasks be performed. All employees will be instructed by the SSO and contractor in proper lifting techniques through safety meetings and demonstration. Additionally, employees will be instructed to not attempt to lift objects heavier than 60 pounds without mechanical assistance or the assistance of a fellow worker.

4.2.1.8 Tool and Equipment Hazards

Improper tool handling and inadequate tool maintenance will increase risk of injury during their use. Management of these hazards requires rigorous maintenance of tools and equipment. The contractor is responsible for effective training of employees in the proper use of the tools. Hand tools that are damaged shall be tagged and removed from the work area. Equipment in need of maintenance or repair shall be tagged and removed from operation until repairs or replacement is accomplished. Only tools with immediate use will be present onsite. Unused tools shall be assembled at a collection point and removed from underfoot and immediate use.

4.2.1.9 Fire Hazard Control

Caution will be used to prevent sparks or open flames within the vicinity of vegetation. When welding or cutting, be sure hot sparks or slag does not come in contact with flammables. An approved A or B fire extinguisher, sufficient in size, will be immediately available (usually 25 feet) when welding or cutting. All heavy equipment (drill rigs, loaders, backhoes, dozers, etc.) shall have a minimum of one 5-pound AB fire extinguisher mounted on it. A minimum of one

AB fire extinguisher shall be at each remediation site. Only approved containers will be used for storing flammable liquids. Oily rags and waste will be placed in appropriate containers. Fire protection equipment will be used for firefighting only. The proper use and location of fire extinguishers will be known by all employees. Gasoline or other flammable liquids will not be used for cleaning. All fire hazards will be reported to the site superintendent immediately. Fire and emergency access lanes will be kept clear at all times in order to facilitate equipment entry and exit.

4.3 BIOLOGICAL HAZARDS

Biological Hazards have not been identified but the following discussions may be relevant to activities. Potential biological hazards may consist of bees, wasps, snakes, spiders, ticks, fleas, poisonous plants such as poison oak and poison ivy, Hantavirus, and bird excrement.

4.3.1 Ants, Bees, Wasps, Hornets and Yellow Jackets

Nests and hives for ants, bees, wasps, hornets and yellow jackets often occur in ground, trees, brush and overhangs on buildings. The area will be checked for obvious nests and hives before it is cleared. If a nest or hive is detected, the PM or site SSO will be contacted before the nest is disturbed. If necessary a pest management consultant will be brought onsite to recommend procedures for by passing or moving the nest. Workers with identified insect allergies will not be allowed to work in the area of a nest or hive. If simple first aid measures do not alleviate the symptoms of a sting, the victim will be taken to the nearest medical center for consultation with a physician. An attempt will be made to kill the offending insect and take it to the emergency room with the victim if this can be done quickly and without endangering personnel.

4.3.2 Spiders and Fleas

These insects exist in cool dark moist areas. The potential for encounters exist when reaching into dark covered places. Suggestions for control include using a long stick to break apart webs or loosen soil from certain areas. A flashlight should also be used before reaching into a dark area. Field personnel shall be aware of their surrounding and avoid contact with all insects.

4.3.3 Rattlesnakes and Scorpions

These creatures are indigenous to many parts of the United States, although are not expected to be encountered at the Site. The SSO will inform field team members at the daily tailgate safety meetings to be on the lookout for rattlesnakes and scorpions. It should be noted that the American Red Cross does not advocate the use of snakebite kits for snakebite injuries. Rather, experience has shown that the victim has a better chance of recovery without permanent damage when the site of the wound is immobilized and the victim rushed to the closest emergency medical facility (preferably within thirty (30) minutes).

4.3.4 Poisonous Plants

Plants such as poison ivy and poison oak grow wild in shady, moist area and at the base of surrounding seedling or adult trees. Many individuals are prone to break out in dermal (skin) rashes upon contact with the plant oil. A visual site inspection and identification of the plants should be completed prior to each work shift so that all individuals are aware of the potential exposure.

4.3.5 Hantavirus

Hazards associated with Hantavirus are not expected.

4.3.6 Bird Excrement and Amplified Fungal Growth

Hazards associated with bird excrement and/or amplified fungal growth are not expected.

4.4 CHEMICAL HAZARDS

Previous sampling and analysis of soil gas and groundwater on the Site has shown elevated levels of volatile organic compounds (VOCs), specifically trichloroethylene (TCE). During construction activities, site workers may be exposed to fugitive soil gas in near surface soils, particularly within trench excavations. Section 3.0 of the SMP provides detailed protocols for required air monitoring at the Site.

5.0 SAFETY AND HEALTH TRAINING

5.1 CERTIFICATION OF TRAINING

Certification of 40- or 24-hour OSHA initial training by the consultant/contractor(s) must be provided to ENGEO before work on the site. Certification records must indicate the type and time period of training. Certification of supervised field experience must also be provided for previous work. If not available, supervised field experience may be obtained at the Site. In addition, workers must demonstrate the completion of annual 8-hour refresher training, as necessary. Requirements for initial training in hazardous substances and supervised field experience contained in 29 CFR 1910.120 and 8 CCR 5192 vary with the degree of anticipated exposure to hazardous substances. The initial training requirements for workers involved in the investigation and remediation activities that may involve exposure to contaminated soils are summarized in the following table:

TABLE 5.1-1
OSHA Initial Training and Field Experience Requirements

Activity	Function	Initial Training (hours)	Supervised Field Experience (days)
Excavation	Equipment operator	24	3
Site Safety Officer	Consultant—onsite during operations	24	3
Health and Safety Manager Project Manager	Consultant— onsite part time	24	3

5.2 TAILGATE SAFETY MEETINGS

At a minimum, daily tailgate health and safety meetings will be held and documented at the site for all field personnel. The SSO will be responsible for scheduling and conducting this safety meeting. All personnel will be required to attend. Hands-on refresher training on PPE, decontamination procedures, work practices, changes in work-tasks, schedule changes, results of air monitoring, and review of safety discrepancies noted may be discussed. Should an operation change affect the onsite fieldwork, a meeting prior to implementation of the change will be convened to explain the changes to all concerned (Attachment D).

6.0 PERSONAL PROTECTIVE EQUIPMENT

6.1 LEVELS OF PPE

All personnel working on the project site will wear the appropriate level of protection as described herein. It is anticipated that EPA level D modified will be required as the initial level of protection. The SSO, in consultation with the HSM may upgrade levels of protection, as necessary. In general, all onsite work will be conducted in Modified Level D PPE. Level A, B or C work is not anticipated for the project. A description of the PPE ensembles is presented below.

6.1.1 Level D

- Hearing Protection - custom fitted or disposable ear plugs/ear muffs (85dBA or above).
- Hard hat (meets ANSI requirements).
- Safety glasses with side shields (meets ANSI requirements).
- Safety shoes or boots.
- Coveralls or long pants and orange shirts or high visibility safety vests.
- Leather work gloves (as appropriate).

6.1.2 Level D Modified

- Hearing protection as described above.
- Work clothing, as dictated by the weather.
- Safety shoes or boots.

- Hard hat.
- Tyvek (or equivalent) coveralls (optional).
- Nitrile gloves (when handling or contact may occur with contaminated soils or materials).
- Safety glasses with side shields.
- High Visibility Safety vest.

6.1.3 Level C

- Full-face or half-mask, air purifying respirators (NIOSH approved).
- Hooded chemical-resistant clothing (overalls; two-piece chemical-splash suit; disposable chemical-resistant overalls).
- Coveralls.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots (outer), chemical-resistant steel toe and shank.
- Boot-covers, outer, chemical-resistant (disposable).
- Hard hat.
- Face shield.

Depending on air monitoring during construction, it is conceivable the SSO, in consultation with the HSM could upgrade to Level C, particularly for trench workers.

6.1.4 Level B

- Positive pressure, full-facepiece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA (NIOSH approved).
- Hooded chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; one or two-piece chemical-splash suit; disposable chemical-resistant overalls).
- Coveralls.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots, outer, chemical-resistant steel toe and shank.
- Boot-covers, outer, chemical-resistant (disposable).
- Hard hat.
- Face shield.

<u>This level of protection is not anticipated for the Scope of Work assigned to this project.</u>

6.1.5 Level A

- Positive pressure, full face-piece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA, approved by the National Institute for Occupational Safety and Health (NIOSH).

- Totally-encapsulating chemical-protective suit.
- Coveralls.
- Long underwear.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots, chemical-resistant, steel toe and shank.
- Hard hat (under suit).
- Disposable protective suit, gloves and boots (depending on suit construction, may be worn over totally-encapsulating suit).

This level of protection is not anticipated for the Scope of Work assigned to this project.

6.2 INSPECTION OF PPE

Specific procedures recommended by equipment manufacturers should be followed for inspection of PPE. A general inspection checklist for PPE before use includes:

- Determining that the clothing material is correct for the specified task at hand.
- Visually inspect for imperfect seams, non-uniform coatings, tears, closure malfunctions, hold up to light and check for pinholes.
- Hard Hats - Head harness is intact and installed properly. Check for cracks.
- Safety Glasses/Goggles - Lenses are clear and free of scratches. Side shields are present.
- Safety Shoes/Boots - Free of holes, damage, soles have ample thread, and laces are adequate.

7.0 SAFETY PROCEDURES, ENGINEERING CONTROLS, AND WORK PRACTICES

7.1 GENERAL SITE RULES/PROHIBITIONS

During soil removal work, all employees, subcontractors or persons entering the work site shall sign in with the SSO and shall sign out upon departing. Employees, subcontractors or persons who will be engaged in hazardous materials or waste operations or have the potential to be exposed to hazardous materials will be informed of the nature, and level of exposure. Each person engaged in such operations will be required to indicate they have been informed of the associated hazards and requirements by signing the Project Manager's notification form.

7.1.1 Engineering Controls and Work Practices

Engineering controls are not anticipated for the proposed activity. Work practices to minimize exposure to nuisance dust will include the wetting down of dusty operations and relocating employees upwind of dusty areas, if necessary.

7.1.2 Employee Rotation

A schedule of employee rotation will not be implemented as a means of compliance with permissible exposure.

7.1.3 Work Practices and Procedures

The following health precautions will be implemented:

- Avoid skin contact and ingestion of stockpiled soil.
- Avoid excessively dusty areas
- Keep work areas clean and well ventilated
- Clean up spills promptly

7.2 MATERIALS HANDLING

7.2.1 Spill and Discharge Control

Should a spill or discharge of petroleum products or contaminated soil occur, the following measures will be taken:

- Take immediate measures to control and contain the spill to the smallest area possible.
- Keep unnecessary people away, isolate the hazardous area, deny entry to unauthorized people, do not allow unauthorized people to touch spilled material.
- Stay upwind.
- Keep out of low areas.
- Keep combustibles away from the spilled material.
- Use a water spray to reduce vapor or dust generation being cautious not to cause the migration of water outside the set boundaries.
- If necessary, take samples for analysis to determine adequate clean up was performed.

- Remove or retrieve any discharged liquids or slugs. Absorb discharged materials with absorbents such as commercial pillows, kitty litter, sand, clean fill, or other noncombustible absorbent material. Place the absorbent/spill mixture into leak proof containers and dispose per EPA and DOT requirements.

7.2.2 Notification of Spills and Discharges

If the spill or discharge is reportable, and/or human health or the environment is threatened, notify the National Response Center, Santa Clara County Environmental Health Department, Mountain View Police Department (non-emergency line), Mountain View Fire Department (non-emergency line), and the Project Manager. Spills or leaks, regardless of their quantity will be reported to the Project Manager immediately following discovery. A follow-up written report will be submitted to the Project Manager within seven (7) days after the initial report. The written report will be in narrative form and as a minimum include the following:

- A description of the material spilled including identity and quantity. Photographs showing the location and extent of the spill.

- A statement as to whether the amount spilled is EPA/State reportable and when and to whom it was reported.

- Exact time and location of the spill, including a description of the area involved.

- Containment procedures initiated and a full description of the cleanup measures taken, or to be taken, including disposal location of the spill residue.

7.2.3 Material Storage, and Disposal

Employees will be trained in and will use proper lifting techniques. Material handling devices will be available for the material handling needs of an activity. Whenever heavy or bulky material is to be moved, the material handling needs will be evaluated in terms of weight, size, and distance and path of move. The following hierarchy will be followed in selecting a means for material handling:

- Elimination of material handling need by engineering controls
- Movement by mechanical device (e.g. lift truck, backhoe, loader, etc.)
- Movement by manual means with handling aid (e.g. dolly or cart)
- Movement by manual means with protective equipment (e.g. lifting belt or lifting monitor).

Materials will not be moved over or suspended above personnel unless positive precautions have been taken to protect the personnel from falling objects. Where the movement of materials may be hazardous to personnel, taglines or other devices will be used to control the loads being handled by hoisting equipment. These devices will be nonconductive when used near energized lines.

Non-compatible materials will be segregated in storage.

Work areas and means of access will be maintained safe and orderly. Sufficient personnel and equipment will be provided to insure compliance with all housekeeping requirements. Work areas will be inspected daily for adequate housekeeping and findings recorded on daily inspection reports. Work will not be allowed in those areas that do not comply with the requirements of this section.

Waste material and rubbish, if generated, will be placed in suitable containers. Waste material and rubbish will not be stored in areas that are away from the general work areas. Separate covered, non-flammable/non-reactive containers will be provided for the collection of garbage, oily, flammable, and dangerous wastes. The containers will be labeled with a description of their contents. The contents will be properly disposed of on a scheduled basis.

Hazardous material waste (i.e. vehicle and equipment oils and lubricants, containers and drums for solvents, adhesives, etc.) will be collected, stored, and disposed of in accordance with Federal, state, and local agencies.

7.3 HAZARD COMMUNICATION

All personnel must follow established work practices to safely handle hazardous materials and chemicals. A hazardous chemical is broadly defined as a chemical that is a health hazard, a physical hazard or both. A hazard communication program has been developed to limit the risks of personnel exposures, damage to equipment, and the unplanned release of hazardous materials and chemicals to the environment due to normal operations. The written program includes protocols for:

- Assessment of the hazards associated with chemicals onsite.
- Inventory and labeling of chemicals and their containers.
- Communication of hazards to the employee through Material Safety Data Sheets (MSDSs) for chemical products and tailgate meetings to discuss hazards of impacted environmental media, such as impacted soil or water.
- Training on the safe handling of chemicals.
- Acquisition, transportation and handling of chemicals.
- Emergency response to releases of chemicals.

The requirements of this program will apply to consultant/contractor in the event that they need to store hazardous materials and/or chemicals such as equipment, fuel, caustic compounds for sample preservation, or solvents for equipment decontamination on the site. The consultant/contractor will be responsible for coordinating the inventory of hazardous materials

and chemicals used or stored at the site. The inventory will be utilized for reporting and emergency response purposes. Data contained in the inventory will include the name, quantity, and location of the chemical. Material Safety Data Sheets shall be readily available onsite for reference.

7.4 SANITATION

Given the expected duration of work, sanitation facilities should be provided on-site. Personnel will be encouraged to use safe sanitation practices with respect to washing and consumption of potable water.

7.5 MACHINERY AND MECHANIZED EQUIPMENT

Before any machinery or mechanized equipment is placed in use, the contractor is responsible for the inspection and testing by a competent person and certified to be in safe operating condition. Inspections and tests will be in accordance with manufacturer's recommendations and will be documented in the daily logs. Records of tests and inspections will be maintained at the site, and will be made available upon request of the designated authority.

Daily/shift inspections and tests:

- All machinery and equipment will be inspected daily (when in use) to ensure safe operating conditions. The Site Superintendent will designate competent persons to conduct the inspections. These inspections will be documented and incorporated into the field logs.

- Tests will be made at the beginning of each shift during which the equipment is to be used to determine that the brakes and operating systems are in proper working condition and that all required safety devices are in place and functional.

Whenever any machinery or equipment is found to be unsafe, or whenever a deficiency, which affects the safe operation of equipment, is observed, the equipment will be tagged and immediately taken out of service until the unsafe condition(s) have been corrected. The tag will indicate the equipment will not be operated. The tag will not be removed and will be placed in a conspicuous location on the equipment. The tag will remain in its attached location until it is demonstrated to the individual dead lining the equipment that it is safe to operate. When corrections are complete, the machinery or equipment will be re-tested and re-inspected prior to being returned to service.

Machinery and mechanized equipment will be operated only by designated qualified personnel. Machinery or equipment will not be operated in a manner that will endanger persons or property nor will the safe operating speeds or loads be exceeded. Getting off or on any equipment where it is in motion is prohibited. Machinery and equipment will be operated in accordance with the manufacturer's instructions and recommendations. Inspections or determinations of road

conditions and structures will be made in advance to assure that clearances and load capacities are safe for the passage or placing of any machinery or equipment.

Mobile equipment, operating within an off-highway job site not open to public traffic, will have a service brake system and a parking brake system capable of stopping and holding the equipment while fully loaded on the grade of operation. In addition, it is recommended that heavy-duty hauling equipment have an emergency brake system, which will automatically stop the equipment upon failure of the service brake system. This emergency brake system should be manually operable from the driver's position.

Preventive maintenance procedures recommended by the manufacturer will be followed. All machinery or equipment will be shut down and positive means taken to prevent its operation while repairs or maintenance is being done. Equipment designed to be serviced while running are exempt from this requirement. All repairs on machinery or equipment will be made at a location, which will protect repair personnel from traffic. Heavy machinery, equipment, or parts thereof which are suspended or held apart by slings, hoist, or jacks also will be substantially blocked or cribbed before personnel are permitted to work underneath or between them. Only, authorized factory trained personnel shall do repairs to heavy equipment. Routine daily lubrication, fueling, etc. shall be conducted by the operator.

All vehicles which will be parked or moving slower than normal traffic on haul roads will have a yellow flashing light or four-way flashers visible from all directions.

All industrial trucks will meet the requirements of design, construction, stability, inspection, testing, maintenance, and operation, defined in ANSI/ASME B56.1, Safety Standards for Low Lift and High Lift Trucks.

Self-propelled construction equipment, whether moving alone or in combination, will be equipped with a reverse signal alarm. Equipment designed and operated so that the operator is always facing the direction of motion does not require a reverse signal alarm. Reverse signal alarms will be audible and sufficiently distinct to be heard under prevailing conditions. Alarms will operate automatically upon commencement of backward motion. Alarms may be continuous or intermittent (not to exceed 3-second intervals) and will operate during the entire backward movement. Reverse signal alarms will be in addition to requirements for signal persons. A warning device or signal-person will be provided where there is danger to persons from moving equipment, swinging loads, buckets, booms, etc.

All belts, gears, shafts, pulleys, sprockets, spindles, drums, flywheels, chains, or other reciprocating, rotating or moving parts of equipment will be guarded when exposed to contact by persons or when they otherwise create a hazard. All hot surfaces of equipment, including exhaust pipes or other lines, will be guarded or insulated to prevent injury and fire. All equipment having a charging skip will be provided with guards on both sides and open end of the skip area to prevent persons from walking under the skip while it is elevated. Platforms, foot walks, steps, handholds, guardrails, and toe boards will be designed, constructed, and installed on machinery and equipment to provide safe footing and access. Equipment will be provided with suitable

working surfaces of platforms, guard rails, and hand grabs when attendants or other employees are required to ride for operating purposes outside the operator's cab or compartment. Platforms and steps will be of nonskid material. Substantial overhead protection will be provided for the operators of forklifts and similar material handling equipment.

Fuel tanks, if any, will be located in a manner, which will not allow spills or overflows to run onto engine, exhaust, or electrical equipment. Exhaust or discharges from equipment will be so directed that they do not endanger persons or obstruct view of operator.

All points requiring lubrication during operation will have fittings so located or guarded to be accessible without hazardous exposure.

7.6 CONFINED SPACE OPERATIONS

All work will be performed at or above current grades, with the exception of some utility trenching at the Site. In the event worker entry into trenches greater than 5 feet in depth is necessary, all required OSHA regulations for trench safety will be followed.

7.7 SITE ILLUMINATION

All work will be performed during daylight hours. No special illumination devices will be necessary to perform the proposed scope of work. Vehicular headlights will be required in the event that vehicular transport operations are required outside of daylight hours or during times of limited visibility (i.e. fog, rain). Additionally, site workers will be required to use vehicular headlights in accordance with State of California motor vehicle laws during onsite or offsite transport.

8.0 SITE CONTROL MEASURES

All employees and personnel entering the site during soil removal work will be required to report to the Site Safety Officer and sign in and out on the site control log. In addition, all workers will be required to complete the worker/visitor acknowledgment form informing them of the potential hazards onsite. Copies of both the site control log and the worker/visitor acknowledgment form are presented in Attachments B and D. The purpose of the site control measures is to prevent the spread of contamination, control the flow of personnel, vehicles, and materials into and out of work areas. Procedures for preventing the spread of contamination include maintaining a site control log, developing a communications program, and implementing site security measures are presented below.

8.1 SITE CONTROL LOG

ENGEO will maintain documentation of sign-in/out forms, employee training records, PPE use and applicable medical surveillance records. In addition, any unsafe conditions present or work practices that have been identified and action taken to correct the identified unsafe conditions

and work practices will be identified by the SSO and documented on the site control log. Record keeping will be performed in accordance with the following.

ENGEO will maintain logs and reports covering the implementation of the HASP. If necessary the format will include training logs and a report. The training log will include the following information for both initial training and refresher training sessions:

- Date and place.
- Area (specific zone) checked.
- Employees in a particular area.
- Equipment being utilized by employees named.
- Protective clothing being worn by employees named.
- Protective devices being used by employees named and area assignment.

Should this project be extended, the bi-weekly reports will include the following information:

- Summary sheet covering the range of work being done.

- Any incidents of nonuse of protective devices in an area where required, nonuse of protective clothing, disregard of buddy system, violation of eating, smoking, and chewing in prohibited areas, instances of job-related injuries and illness, and monitoring results.

- Copies of medical certificates for employees and the waivers of visitors.

8.2 DECONTAMINATION

8.2.1 Personnel

Decontamination will consist of the removal of disposal of protective coverings (i.e. gloves, coveralls) and washing of skin surfaces that may have been exposed or soiled during operations. Additionally, all onsite personnel will be required to wash hands or any other potentially exposed or soiled skin surface prior to breaks, leaving the Site, and at the end of daily operations.

8.2.2 Equipment

Equipment and automobiles will have visible soil deposits removed prior to equipment being transported offsite.

9.0 EMERGENCY EQUIPMENT

The following items, at a minimum, will be maintained onsite and available for immediate use:

- First aid equipment and supplies.

- Fire extinguishers with a minimum rating of 5-A, B, or C will be carried in all vehicles and heavy equipment. Fire extinguishers will also be available at any site where flammables or combustible materials present a fire risk.

10.0 EMERGENCY RESPONSE AND CONTINGENCY PROCEDURES

10.1 PRE-EMERGENCY PLANNING

Emergency response agencies will be contacted, and notified of upcoming site activities and potential emergency situations. The capabilities and commitment of the local agencies will be ascertained and obtained. ENGEO will verify that this Emergency Response Plan is compatible and integrated with disaster, fire, and emergency response plans of the local, state, and federal agencies.

10.2 LINES OF AUTHORITY

The HSM and SSO are responsible for overall Site safety. In the case of a Site safety concern, emergency or accident, the SSO should be contacted immediately. Upon notification, the SSO will make appropriate decision regarding the incident.

10.3 EMERGENCY RECOGNITION AND PREVENTION

The recognition and prevention of hazards and potential emergencies are discussed in detail elsewhere in this plan. In general, emergency situations occur when personnel are seriously injured and require first aid or hazardous or potentially hazardous materials are spilled or released to the environment. Attachment E provides a checklist for critical information and procedures in cases of emergency.

10.4 PROCEDURES FOR SITE EVACUATION

10.4.1 Emergency Equipment

The following items, as a minimum will be immediately available for onsite use:

- First aid equipment and supplies
- Spill control materials and equipment
- Fire extinguishers
- Telephone

10.4.2 Adverse Weather Conditions

In the event of adverse weather conditions, the SSO will assess if work can continue without sacrificing the health and safety of any field workers. Items to be considered prior to assessing if work should continue include:

- Potential for heat stress and heat-related injuries
- Limited visibility
- Potential for electrical storms
- Potential for high winds resulting in contaminant transport

10.4.3 Earthquakes

This guidance assumes that personnel will be outdoors. In the event of a major earthquake:

- Field personnel should immediately evacuate any trenches, excavations or elevated positions in machinery, heavy equipment or structures.

- Field personnel should move away from structures or overhead electrical transmission poles and wires or any other objects or structures that might topple over or collapse.

- Personnel should move to an area where there is the least chance of something falling from above.

- Personnel should assume a position of low center of gravity to avoid being thrown or falling to the ground. A position on “all fours” can minimize shaking.

- Personnel should remain alert for rolling or traveling objects to avoid injury.

- The buddy system shall be maintained in the event of earthquake.

10.4.4 Evacuation Routes and Places of Refuge

Prior to access into the work areas and during soil removal work, workers will be instructed as to designated evacuation routes and procedures. A route map detailing directions to the emergency medical facility will be posted conspicuously at the job site. Additionally, each support vehicle should be equipped with copies of this map and each driver should be familiar with the route and travel time to that facility. A copy of the hospital route map is included as Attachment F.

Workers will be instructed during the preliminary and subsequent tailgate meetings to proceed away from the hazard in a direction of 90° to the prevailing wind for at least 50 feet prior to heading up wind of the hazard should an emergency evacuation occur. A place of refuge will be identified. The purpose of the place of refuge is to provide an offsite meeting place in the event that site evacuation is required. The actual place of refuge will be determined during the weekly onsite safety meetings.

10.4.5 Site Security and Control

Areas onsite where hazardous substances are known to exist will be secured through demarcation tape and warning signs. Access to the work area will be restricted, and all personnel (regardless

of status) requesting entry the work area will be required to report to the Site safety officer and sign in on the Site control log. All visitors will be briefed onsite-specific safety and health issues. The gated Site will be kept closed and locked during hours of non-operation.

10.5 NEAREST HOSPITAL

The nearest hospital is located in the City of Brentwood, approximately 2.5 miles from the project site. Attachment F provides a map and directions to the nearest hospital. The address and telephone numbers are as follows:

El Camino Hospital
2500 Grant Road
Mountain View, CA 94040

10.6 EMERGENCY ALERTING AND RESPONSE PROCEDURES

10.6.1 Emergency Alerting Procedures

If physical injury or illness due to accidental exposure to hazardous materials or waste occurs, uninjured/unaffected personnel should do the following:

- Evacuate all non-essential personnel.
- Remove injured/exposed person(s) from the work zone.
- Remove protective gear from injured/exposed person(s).
- Decontaminate exposed person(s).
- Render first aid if necessary.
- **Call 911.**
- *If medical assistance is urgent, decontamination of the victim may not be practical or required.*
- Evacuate other onsite personnel to a safe place until the SSO determines that it is safe to resume work
- The senior person present will notify the SSO and superintendent and advise them of the incident and the steps taken to prevent recurrence.
- Submit a written report on the incident to the contracting officer or representative within 24 hours. The report will be made part of the final closure file.

- Accident reporting records and investigative reports will be maintained at the site office and ENGEO's corporate office as part of the Department of Labor record keeping requirements.

Following any emergency response, an evaluation of procedures will be performed. The evaluation should include cause and proposed remedy for subsequent incident prevention. Should an emergency situation develop the site superintendent will notify work site personnel by hand held radio. Work activities shall be stopped if necessary.

10.6.2 Emergency Telephone Numbers

The universal emergency response number is 911. When 911 is dialed, a public safety answering service will ascertain the type of assistance needed and quickly summon the appropriate emergency service (Fire Department, Police Department, emergency medical or paramedics, ambulance, etc.) to the site. A complete listing of emergency telephone numbers for project personnel is provided in Table 10.6.2-1 below.

TABLE 10.6.2-1
Emergency Contact Telephone Numbers

Name	Telephone number	Alternate Number
National Emergency Response Center	1-800-424-8802	N/A
Santa Clara County Environmental Health Department	(408) 918-3400	N/A
National Poison Control	1-800-222-1222	N/A
Mountain View Police Department	(650) 903-6344 (Non-emergency)	911
Mountain View Fire Department	(650) 903-6365 (Non-emergency)	911
Project Manager, Shawn Munger	916-580-6535	916-416-9000
Health and Safety Manager, Divya Bhargava	(925) 395-2559	(650) 804-2402
Site Safety Officer , Divya Bhargava	(925) 395-2559	(650) 804-2402

10.7 COMMUNITY ALERT PROGRAM

The universal emergency response number is 911. When 911 is dialed, a public safety answering service will ascertain the type of assistance needed and quickly summon the appropriate local and/or municipal emergency service (Fire Department, Policy Department, emergency medical or paramedics, ambulance, etc.) to the site.

10.8 PROCEDURES FOR INCIDENT REPORTING

In the event that an incident such as an explosion or fire, or a spill or release of toxic material occurs during the course of the project, the appropriate government agencies will immediately notified. ENGEO will notify Cal/OSHA, EPA, and the contractor/subcontractor supervisor(s). A

written notification shall be forwarded to the contracting officer within 24 hours. The report should include the following items:

- Name, organization, telephone number, and location.
- Name and title of the person(s) reporting.
- Date and time of the incident.
- Location of the incident, i.e. site location, facility name.
- Brief summary of the incident giving pertinent details including type of operation ongoing at the time of the incident.
- Cause of the incident, if known.
- Casualties (fatalities, disabling injuries).
- Details of any existing chemical hazard or contamination.
- Estimated property damage, if applicable.
- Nature of damage, effect on contract schedule.

11.0 CERTIFICATE OF WORKER/VISITOR ACKNOWLEDGMENT

A copy of a certificate of worker/visitor acknowledgment (Attachment D) will be completed and submitted for each visitor allowed to enter the work site during soil removal work.

12.0 REPORTING

12.1 LOGS, REPORTS, AND RECORDKEEPING

The following logs, reports, and records will be developed, retained, and submitted to the contracting officer when requested:

- Training logs (site specific and visitor)
- Daily inspection logs
- Equipment Safety and Maintenance Logs
- Employee/visitor register (Site Control Log)
- Environmental and personal exposure monitoring/sampling results

ATTACHMENT A

Accident/Injury/Illness Investigation

Job Site: _____

Please Print - complete all items - submit immediately

PART I – SUPERVISOR		
Employee	Employee #	Phone # ( )
Address	City	State Zip
Date of Birth / / Age Sex	Social Security #	
Shift ☐ Day ☐ Evening ☐ Night	Date of Hire / /	Occupation
Date of Injury / /	Time of Injury : AM : PM	
Location of Incident		
Date Reported / /	Time Reported	Reported to Whom?

PART II - SUPERVISOR

(1) Was employee given First-Aid? Yes ☐ No ☐
(2) Sent to: Emergency Room Yes ☐ No ☐
Preferred Provider Yes ☐ No ☐
Personal Physician Yes ☐ No ☐
Company Nurse Yes ☐ No ☐
Other Yes ☐ No ☐

(3) Was Employee Placed on Transitional Duty? Yes ☐ No ☐
(4) Will Employee lose time/work? Yes ☐ No ☐
(5) If lost time, approx. days_____
(6) Was treatment refused? Yes ☐ No ☐

Name & Address of Clinic Phone number of clinic

Attach statement of all witnesses

PART III – SUPERVISOR

Name of Witness	Address	Phone
(1)		
(2)		

Describe in detail what employee was doing at the time of injury (what, how why)

Did employee wear protective equipment? Yes ☐ No ☐ If Yes, specify

Part of body (check) indicate right or left when applicable

1 ☐ Head	5 ☐ Mouth	9 ☐ Arm	13 ☐ Knee	17 ☐ Toe	21 ☐ Groin
2 ☐ Face	6 ☐ Heart	10 ☐ Wrist	14 ☐ Leg	18 ☐ Hip	22 ☐ None
3 ☐ Eye	7 ☐ Back	11 ☐ Hand	15 ☐ Ankle	19 ☐ Neck	23-☐ other_____
4 ☐ Ear	8 ☐ Trunk	12 ☐ Finger	16 ☐ Foot	20 ☐ Shoulder	

Type of injury (check)

1 ☐ Reaction to foreign substances/objects
2 ☐ Puncture
3 ☐ Laceration
4 ☐ Contusion
5 ☐ Burn
6 ☐ Fracture
7 ☐ Amputation
8 ☐ Sprain/Strain
9 ☐ Other

What type of training has been conducted to prevent recurrence?

Describe what acts or conditions may have contributed to the incident. (Analyze all the facts concerned. If either the injured person, a machine or other physical condition was involved, find out How. Use the Possible Worker's Compensation Accident Causes on the back of this form to complete this section.)

Corrective Action(s) taken:

Investigated by: _____Date:_____

PART IV - MANAGEMENT REVIEW
Are you satisfied with your review of Part I-III that the accident has been thoroughly investigated? ☐ Yes ☐ No If NO, return for a more detailed report.
As a result of your review, have you identified any additional reasons why the accident occurred: ☐ Yes ☐ No If YES, list the reasons:
Corrective action(s) you are taking?
Who have you made responsible for corrections?
Signature of Superintendent Date:
Manager Comments

As a result of the Foreman's investigations and my comments above, I am satisfied that the accident has been thoroughly investigated. Corrective actions will be personally followed up by me until complete.

Signature of Manager_____Date:_____

POSSIBLE WORKER'S COMPENSATION ACCIDENT CAUSES

UNSAFE ACT - PERSONAL FACTORS	UNSAFE CONDITION
Making safety devices inoperable	Inadequate guards or protection
Failure to use guards provided	Defective tools or equipment
Using defective equipment	Unsafe condition of machine
Servicing equipment in motion	Congested work area
Failure to use proper tools or equipment	Poor housekeeping
Operating machinery or equipment at unsafe speed	Unsafe floors, ramps, stairways, platforms
Failure to use personal protective equipment	Improper material storage
Operating without authority	Inadequate warning system
Lack of skill or knowledge	Fire or explosion hazards
Unsafe loading or placing	Hazardous atmosphere: gases, dust, fumes, vapors
Improper lifting, lowering or carrying	Hazardous substances
Taking unsafe position	Inadequate ventilation
Unnecessary haste	Radiation exposures
Influence of alcohol or drugs	Excessive noise
Physical limitation or mental attitude	Inadequate lighting
Unaware of hazards	
Unsafe act or other	

THE PURPOSE OF THIS INVESTIGATION FORM IS NOT TO PLACE FAULT OR BLAME. ITS PURPOSE IS TO INVESTIGATE ALL POSSIBLE CAUSES OF THE ACCIDENT TO TAKE NECESSARY CORRECTIVE ACTIONS AND CONTINUALLY IMPROVE PROJECT SAFETY.

ATTACHMENT B

Project Manager Daily Log

Date: _____ Project Name: _____

Log Information:_____

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

ATTACHMENT C

Worker/Visitor Entry and Exit Control Log

Name	Date	Time	In	Out

SSO Signature_____ Date_____

ATTACHMENT D

Acknowledgement

This is to acknowledge my participation in a geotechnical exploration project. I accept the responsibility to protect myself with the appropriate personal protective equipment. In the event I have any safety questions I will not hesitate to ask the Head Site Safety Officer.

_____
(Signature)

_____
(Date)

ATTACHMENT E

Job Site Emergency Procedures

Job Site:_____Date:_____

EMERGENCY TELEPHONE NUMBERS:
FIRE _____
POLICE _____
AMBULANCE _____
HOSPITAL _____

IN CASE OF FIRE:
- exit the site using the evacuation route
- call the fire department
- go immediately to the assembly point

EVACUATION ROUTE:
_____
_____
_____

ASSEMBLY POINT:_____

IN CASE OF SERIOUS INJURY:
- immediately contact first aid trained personnel
- call for medical assistance

Job site first aid trained personnel:
_____
_____
_____

Trained personnel will take immediate charge of the emergency situation. (Supervision to perform accident investigation)

In case of natural disaster: (check)

☐ Tornado: Seek inside shelter, preferably underground. Stay away from windows. If outside, move away from the tornado's path at a right angle, or lie flat in a ditch or ravine

☐ Earthquake: Evacuate the building and go directly to the designated assembly point for instructions.

☐ Other: _____

ATTACHMENT F

Hospital Map

bing Maps

A **277 Fairchild Dr, Mountain View, CA 94043**

B **2500 Grant Rd, Mountain View, CA 94040**
El Camino Hospital Emergency Services (650) 940-7055

My Notes

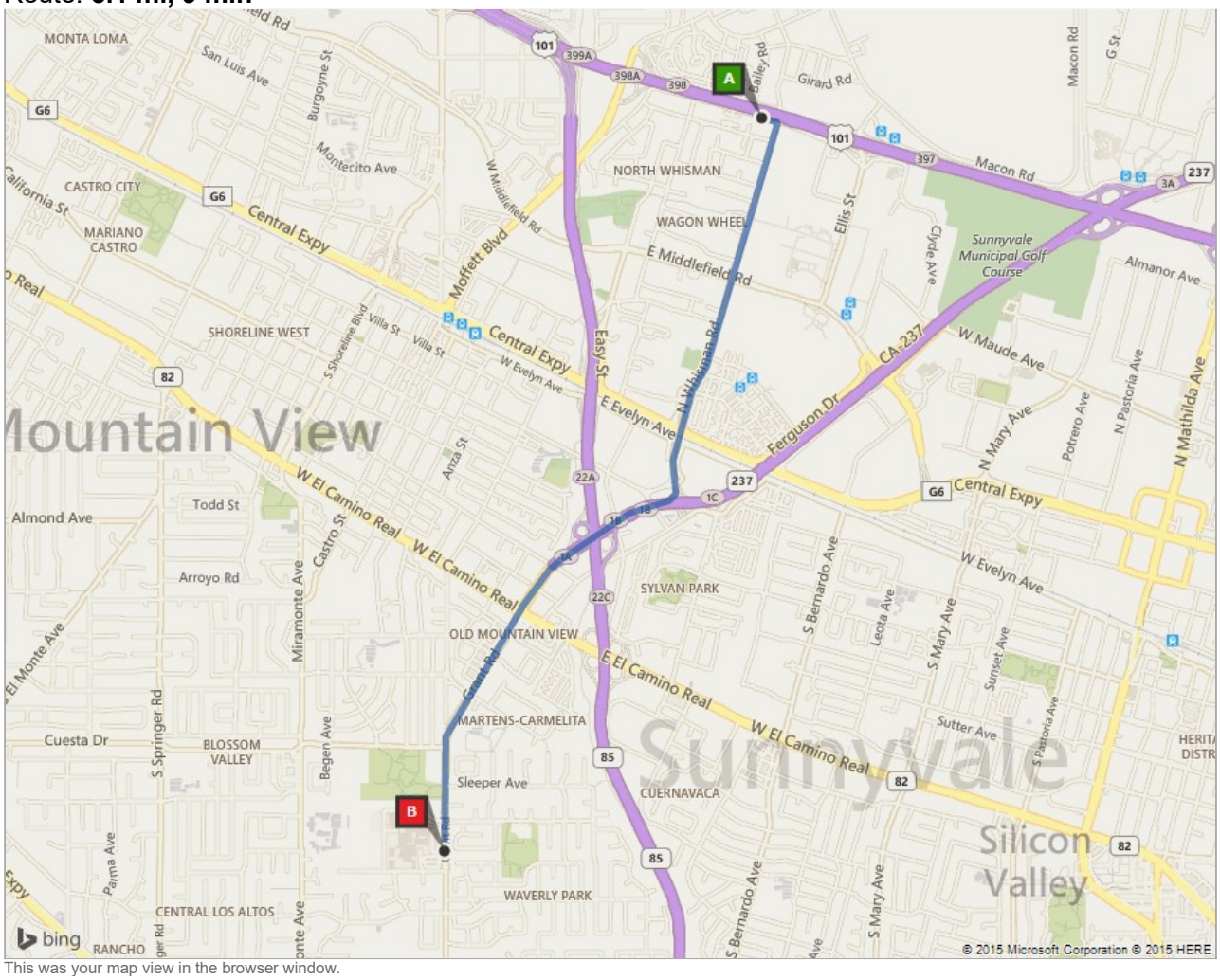
On the go? Use **m.bing.com** to find maps, directions, businesses, and more

Route: **3.1 mi, 9 min**

A	**277 Fairchild Dr, Mountain View, CA 94043**	**A–B: 3.1 mi** 9 min
	1. Depart **Fairchild Dr** toward N Whisman Rd	318 ft
	2. Turn **right** onto **N Whisman Rd**	1.1 mi
	3. Road name changes to **S Whisman Rd**	0.1 mi
237	4. Take ramp **right** for **CA-237 W**	0.8 mi
	5. Keep **straight** onto **Grant Rd**	0.9 mi
B	6. Arrive at **2500 Grant Rd, Mountain View, CA 94040** *The last intersection is North Dr* *If you reach South Dr, you've gone too far*	

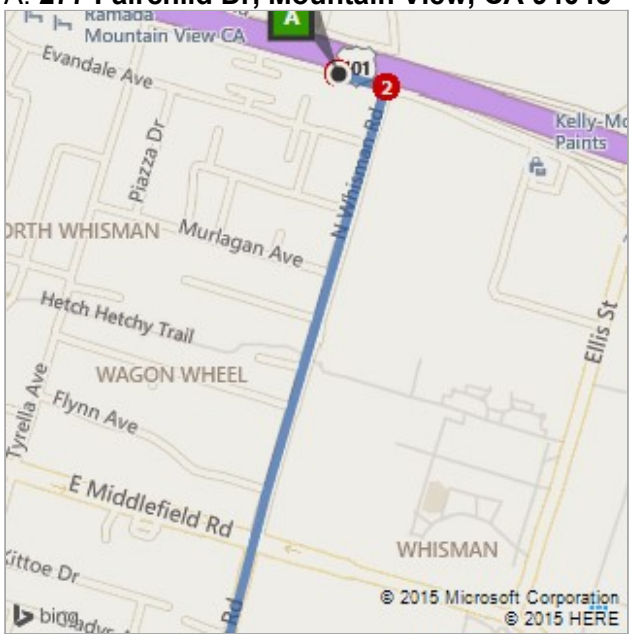
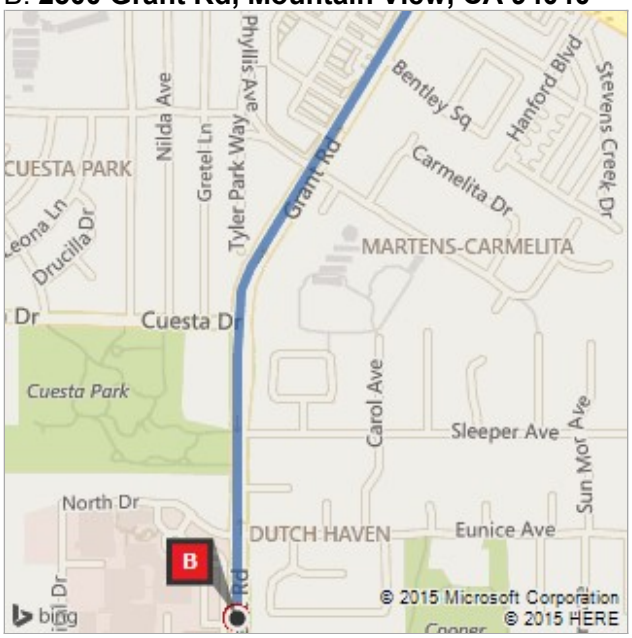
These directions are subject to the Microsoft® Service Agreement and for informational purposes only. No guarantee is made regarding their completeness or accuracy. Construction projects, traffic, or other events may cause actual conditions to differ from these results. Map and traffic data © 2015 NAVTEQ™.

Route: **3.1 mi, 9 min**



This was your map view in the browser window.

A: **277 Fairchild Dr, Mountain View, CA 94043**



B: **2500 Grant Rd, Mountain View, CA 94040**