WORK PLAN
FOR SOIL INVESTIGATION
AT SITE SCREENING AREA 12
BETWEEN SITE 2 AND SITE 5

NAS JRB Willow Grove
Horsham, Pennsylvania

Naval Facilities Engineering Command
Mid-Atlantic

Contract No. N62472-03-D-0057
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WORK PLAN FOR SOIL INVESTIGATION
AT SITE SCREENING AREA 12
BETWEEN SITE 2 AND SITE 5
NAVAL AIR STATION JOINT RESERVE BASE
WILLOW GROVE, PENNSYLVANIA
COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:
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1.0 INTRODUCTION

Tetra Tech NUS (TtNUS) has prepared this Work Plan for investigation of surface and shallow subsurface soils at the former Drum and Debris Removal Area between Site 2 and Site 5 also known as Site Screening Area 12 (SSA 12) at the Naval Air Station Joint Reserve Base (NAS JRB) Willow Grove, Pennsylvania. This soil investigation supersedes a previous investigation by the Navy contractor Resource Management Concepts (RMC, 2003) that was performed to obtain soil quality data in the area of discarded surficial drums (since removed) and selected EPIC features that were discovered after the completion of the Phase II RI (TtNUS, 1997). These areas and features are not considered to be part of Site 2. Data quality issues (high detection limits) limited the usability of the analytical data obtained during the RMC investigation. The Navy has directed TtNUS to perform this resampling and analysis effort under Contract Task Order No. 003 of the Contract N62472-03-D-0057, Comprehensive Long-Term Environmental Action-Navy (CLEAN). The new analytical data will be compared to the appropriate U.S. Environmental Protection Agency (EPA) and Pennsylvania Department of Environmental Protection (PADEP) risk-based criteria and medium-specific screening criteria for soils.

1.1 SITE DESCRIPTION AND SETTING

NAS JRB Willow Grove, Pennsylvania is located in Horsham Township, Montgomery County in southeastern Pennsylvania, approximately 20 miles north of the city of Philadelphia (Figure 1). NAS JRB Willow Grove occupies approximately 1,000 acres of 1,200 acres the Department of Defense (DoD) maintains at the Air Station. The Willow Grove Air Reserve Station (ARS) occupies approximately 200 acres of land in the northeastern section of the Air Station and shares common facilities with the NAS JRB. Figure 1 shows the location of NAS JRB Willow Grove and ARS. The Air Station is comprised of flat to slightly rolling terrain and is generally bounded by State Route 611 to the east, State Route 463 to the southwest, and Keith Valley Road to the north.

The primary mission of NAS JRB Willow Grove is to provide support for operations involving aviation training activities and to train Navy reservists. NAS JRB Willow Grove supports DoD tenants such as the Marine Reserve, Pennsylvania National Guard and the Army Reserve, and shares facilities/services with the Air Force Reserve. The Base provides facilities, services, materials, and training in direct support of all assigned units. These units include anti-submarine warfare squadrons, a helicopter squadron, a fleet logistic support squadron, and other DoD units.

SSA 12 is located north of the Antenna Field Landfill in the southern portion of NAS JRB Willow Grove, southwest of Runway 10/28 (Figure 2). SSA 12 is located in a relatively undeveloped section of the
station that is covered by vegetation, including grass, brush, and short trees. The drum and Environmental Photographic Interpretation Center (EPIC) soil sampling locations are located north of the former landfill (between Sites 2 and Site 5), generally either along a road leading northwest from the Antenna Field Landfill site through the undeveloped area, or north west of the drainage swale that bisects the former landfill.

1.2 BACKGROUND

Subsequent to completion of the field work for the Phase II RI for Site 2, EPA requested that the Navy investigate various EPIC features that had been identified adjacent to Site 2. During their field reconnaissance of these features, the Navy discovered several drums abandoned on the surface in a limited area. As a result, in 2003 the Navy tasked RMC to obtain surface and shallow subsurface soil samples at EPIC feature and drum locations, and analyze the soil for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, and metals. The report of the soil investigation (RMC, 2003) is included as Appendix A.

The results of the RMC investigation indicated that SVOCs, pesticides, and metals were present at some locations. Subsequent review of the analytical results, however, indicated that data quality issues (high detection limits) compromised the usability of the data, resulting in inconclusive results, precluding further decision making relative to the regulatory disposition of the site. As a result, the Navy has directed TtNUS to resample the soils at each location for VOCs, SVOCs, pesticides, PCBs and metals. This resampling effort is the subject of this work plan.
2.0 SUMMARY OF PLANNED FIELD INVESTIGATIONS

The objective of this study is to collect surface and shallow subsurface soil samples from 29 locations in the vicinity of SSA 12 (see Figure 2). All 29 locations will be sampled from the interval 0 to 6 inches, and ten of the locations will also be sampled at a depth of 24 to 30 inches. These are the same locations and features sampled by RMC, when the drums and debris were removed in 2003. The TtNUS SOPs for soil sampling, field documentation, utility locating and excavation clearance, and decontamination of field equipment are included in Appendix B. Soil samples for VOC analysis will be collected and analyzed according to the EPA Region III Fact Sheet: Field Samplers Guide to Collection and Handling of Soil Samples for Volatile Organic Analysis using SW 846 Method 5035A (Appendix C).

All work will be performed in accordance with the site specific Health and Safety Plan prepared for this investigation following procedures outlined in the Work Plan for Phase II Remedial Investigation, NAS JRB Willow Grove, Pennsylvania submitted to the Northern Division, Naval Facilities Engineering Command (Brown & Root Environmental, May 1997) and updated in the Health and Safety Plan for Soil Investigation and Well Maintenance for Site 1 (OU 1), Site 3 (OU 10) and Site 5 (OU 4) (TtNUS September 2005).

2.1 SAMPLE LOCATION

TtNUS will subcontract with a Pennsylvania-licensed surveyor to relocate and stake the historical sample locations before the new samples are obtained. It is anticipated that many of the locations may still have the pin flag placed by the RMC sampling team. The Universal Trans Mercator (UTM) survey coordinates for the RMC sample locations, which were determined in the field by using a hand-held geographical positioning system (GPS) device, are included on page 16 of Appendix A. Because the expected accuracy of the measurements ranged from plus or minus 6 to 40 feet (with a reported “average” accuracy of plus or minus 9 to 12 feet), each location may be adjusted in the field by referring to RMC’s hand-drawn maps from the field notes. The surveyor will determine the location of the new sample to an accuracy of plus or minus 6 feet or less.

TtNUS will inform Pennsylvania One Call of the planned investigation. Utility clearance will be the responsibility of the NAS JRB Willow Grove Civil Works Division. Actual intrusive activities will be limited to a maximum of approximately 30 inches deep.

2.2 SAMPLING PROCEDURE

In accordance with the previous investigation, one soil sample will be obtained from a depth interval of 0 to 6 inches at each EPIC feature location (19 samples). Two soil samples will be obtained (from the interval 0 to 6
inches and at 24 to 30 inches) at each former drum location (20 samples). For each sample, a field technician will obtain the sample material by digging with a stainless-steel hand-auger, and placing the soil into a clean stainless steel bowl. The sample material will be homogenized, transferred into the laboratory supplied sample jars, and placed in iced coolers for temporary storage. In the case of soil samples for VOC analysis, the sample will be collected directly from the hand auger bowl using an EnCore sampler following the procedures described in Appendix C. At the end of each sampling day, the samples will be packaged in iced coolers and shipped to a subcontracted, Navy-certified analytical laboratory via overnight courier.

2.3 SAMPLE NOMENCLATURE

All samples will identified by the prefix “SSA12” for Site Screening Area 12. The EPIC features will be identified by the letter “E”. The drum locations will be identified by the letter “D”.

The surface samples (obtained from the interval 0 to 6 inches) will be designated by the prefix SS, followed by their location number, followed by either a “D” or an “E”, followed by “000.5” to identify the sample depth in feet. For example, the surface sample obtained at the EPIC 2 location will be designated SSA12-SS02E-000.5.

The subsurface samples (obtained at the depth of 24 to 30 inches) will be designated by the prefix SB, followed by their location number, followed by a “D”, followed by “2.02.5” to identify the sample depth in feet. For example, the subsurface sample obtained at the Drum 4 location will be designated SSA12-SB04D-2.02.5.

Field quality assurance/quality control (QA/QC) samples will be taken and designated with the following nomenclature:

- Field duplicate samples will be collected at the rate of 1 duplicate per 20 field samples. The sample nomenclature will be similar to that for the field samples, but the duplicate samples will be assigned fictitious sample designations (location numbers and sample collection times) on the sample bottle ware and the chain-of-custody paperwork. The cross-reference between the actual and fictitious sample designations will be kept in the site logbook and on the sample collection field log sheet.

- Rinsate blanks will be collected at the rate of 1 per day per sampling device. The rinsate blank will be labeled with an alphanumeric code identifying the sample as a rinsate blank, the date of collection, and a sequential number specifically designating that sample (assuming that more than one rinsate blank may be collected in a day). For example, the sample RB-20070515-02 would be the second rinsate blank collected on May 15, 2007.
2.4 FIELD DECONTAMINATION

The sampling equipment used for collecting samples will be decontaminated before field sampling, between samples and after sampling activities. It is anticipated that the hand auger and stainless-steel mixing bowl will require decontamination. Dedicated, disposable trowels will be used to obtain the sample from the auger, or from the stainless-steel mixing bowl. The following decontamination steps will be followed:

- Remove all soils to the extent possible.
- Alconox or liquinox detergent solution wash.
- Potable water rinse.
- Rinse with pesticide-grade isopropanol.
- Deionized water rinse.
- Air dry.
- Wrap in aluminum foil (if not immediately used).

2.5 INVESTIGATION DERIVED WASTE

Surplus soil generated during sampling will be placed back at the point of generation. Decontamination fluids will be containerized and disposed in the sanitary sewer for ultimate treatment at the onsite waste water treatment plant (WWTP). Used personal protection equipment (PPE), including Tyvek suits and latex and nitrile gloves, will be bagged and disposed as municipal-type waste.

2.6 LABORATORY METHODS AND DETECTION LIMITS

All analyses will be performed using EPA SW-846 methodologies at a Navy-certified laboratory. The required laboratory detection limits for VOCs, SVOCs, PAHs, PCB/pesticides and metals in soil samples are presented in Appendix D.

2.7 DATA VALIDATION

TiNUS will begin the data review process by initially examining and accepting the data submitted by the laboratory. The data package will be reviewed for accuracy, precision, and completeness (all pertinent information is included, all appropriate forms are signed and dated, calculations are correct, and holding times and QC sample acceptance criteria have been met). The laboratory project manager will also review the data package to ensure that the submittal meets the TiNUS specifications. The data package will include a narrative, copies of the chains-of-custody, method summaries and references, summary of the laboratory
identification numbers, receipt logs, extraction and analysis logs, analytical results, QC results, and raw data. The laboratory will deliver the analytical data as a hard copy and electronic disk. The EDD format requirements are included as Appendix E.

All (100%) of the laboratory data will be validated by TtNUS according to EPA Region III guidelines for M 3 data validation. The results of the data validation effort will include a data summary, narrative, Form 1s, and support documentation. TtNUS SOPs DV-02 and DV-04 for data validation are included in Appendix B. TtNUS will provide a complete data package to the Navy as an appendix to the investigation's report of results. This data package will include a data summary in tabular format, the complete data package delivered by the laboratory, and validation forms and documentation of the data validation performed by TtNUS.
FIGURES
APPENDIX A

RMC REPORT
ANTENNA LANDFILL SITE SOIL CHARACTERIZATION
AND DRUM REMOVAL

WILLOW GROVE, PENNSYLVANIA

PROJECT 710-005-005

Contract No.: N62472-99-D-0826, EM0024

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31 JULY 2003
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ANTENNA LANDFILL SOIL CHARACTERIZATION
AND DRUM REMOVAL

SUMMARY

This report details the soil characterization and the drum removal efforts at the
antenna landfill and the area that drains through the drum removal area. Since
not all the metallic debris was actually drums, this report uses the terms “drum
removal” and “debris removal” synonymously.

Organic soil contamination is irregular. Certain chemicals associated with
incomplete fuels combustion are present in the parts per million range in the
EPIC feature sites. The maximum is fluoroanthene at 9,200 ppb (9.2 ppm) at
feature site 16. Organic contamination at the drum sites was even more
irregular, with the maximum contamination at drum site 8.

Metals presence was ubiquitous across the area. This area is or has been an
area of considerable hunting activity, as evidenced by the many shell casings,
camping residues, and beer cans in the area. It is likely that much of the soil
“contamination” is a result of hunting.

The full set of laboratory data accompany this report electronically as PDF files.

All visible heavy metal debris was removed from the wooded area, including
several metallic pieces not discovered in the original site walk-through. A few
dozens aluminum cans were also removed, and some larger pieces of exposed
sharp glass shards were removed.

SOIL SAMPLING

Sampling Locations

According to the work order, there were 19 selected locations indicated by
number on an aerial photograph and defined by a list of universal transverse
mercator (UTM) coordinates. These locations are known as EPIC locations, and
they required a single soil sample. There were also ten “drum” sites to be
sampled at two depths: at six inches and 24 inches. The drum sites were
marked in the field, and a supplemental map was hand drawn by EFANE to show
the directions to each drum site.

The EPIC feature sites were located in the field using a Garmon GPS Map 76
(hand-held GPS device). Depending on the overhead cover, the device
displayed a computed accuracy of ± 40 feet to ± 6 feet. A computed accuracy of
± 12 feet to ± 9 feet seemed to be the average range. The device was also used
to derive the UTM coordinates of the drum sites.

The aerial photograph has been edited by RMC to show the approximate
locations of the drums areas, and also to show the approximate line of the
existing fence. The lay of the land has changed slightly in the decade since the aerial photograph was taken. The hand drawn maps have been edited to show the sample number of each drum site, and the field derived drum site UTM coordinates have been entered on the maps.

The edited aerial photograph and the edited hand drawn maps are in Appendixes A and B, and the list of UTM Epic coordinates is in Appendix C. An orange construction pin flag was labeled according to the sample number and placed at the site of each sample.

Sampling Dates
Soil characterization sampling was performed on two dates. The first sampling date was April 8, 2003. Due to severe and inclement weather it was impossible to complete sampling on this date, and a second sampling trip was scheduled for June 19, 2003. Based on the data from the first sampling trip, it was determined that the drums and other debris would be removed and transported to a metals recycler as ordinary metallic waste.

Sample Numbers
The EPIC feature samples were labeled as sample number 1-19. The drum samples were labeled as D#-6 or D#-24. For instance D1-6 would indicate that the sample was collected at drum site D1 at the six-inch level, and D8-24 would indicate that the sample was collected at drum site D8 at the 24 inch level.

Sampling Protocol
From each sampling location the surface detritus was brushed away. A disturbed-soil sampling augur was used to collect samples to a depth of six inches for the first drum sample. Using the same hole, for each drum sample site the soil was augured to a depth of 24 inches. A single sample was collected from each EPIC feature. The sampling augur was cleaned between each sample collection by brushing with a clean wipe, and wiping with USP isopropyl alcohol.

Each collected sample was displaced from the augur into a sealable plastic bag, and samples were withdrawn from the plastic bag and placed in labeled jars for transport to the laboratory. The samples were placed in an ice cooler immediately upon collection. A temperature blank was placed in the cooler, and the cooler was refreshed with fresh ice as required until the cooler could be shipped to the laboratory (generally the next day). A sketch of the disturbed soil sampling augur may be found in Appendix D.

SITE DESCRIPTION
Referring to the aerial photograph, the area north of the fence is a prairie with growths of berries and wild shrubs. During the April sampling event it was possible to generally walk through the area, although in certain locations such as points 6, 7 and 9 the residual growth was dense.
The EPIC feature sites south of the fence, points 10-19, is thickly wooded with growth of berries and shrubs in areas where the sun penetrates. Except for some difficulty in establishing the field UTM coordinates of sample points 13, 14, 15 and 16 there were no particular problems. The drum sites D1-D8 and D-10 are in mature woodland with animal trails, occasional stands of thick honeysuckle, and occasional stands of berries and shrubs where the sun penetrates. Drum site D9 was an isolated drum under a stand of honeysuckle near the fence, as indicated on the edited aerial photograph.

**SAMPLING PARAMETERS**

**Parameters for Analysis**

The sampling parameters were as follows:

- GC/MS Volatiles  Method SW846 8260B
- GC/MS Semivolatiles Method SW846 8270C
- GC Semivolatiles Method SW846 8081A (Aroclors)
- Total Metals Method SW846 6010B
- Mercury Method SW846 7471A

**Analysis Summary**

The tables on the following pages indicate the maximum level of selected parameters. The full results may be compared against the action levels for soil cleanup in Pennsylvania for further determinations. For all GC/MS volatile compounds the results were either non detectable or below the detection limit (ND or <RL) for all samples. For GC/MS semivolatile compounds there were detectable levels of dieldrin and similar compounds. For GC Semivolatiles (Aroclors) the results were either non detectable or below the detection limit for all samples.

Metals were ubiquitous. The maximum level and the location of TCLP metals is indicated in the following tables. Note that the analysis was for total metals and not for TCLP metals. As a rule of thumb, the total metals analysis may be divided by 1/3 to estimate the TCLP results.
## GC/MS Volatiles

### Method SW846 8260B

<table>
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<tr>
<th>Parameter</th>
<th>Epic Feature</th>
<th>D-6&quot;</th>
<th>D-24&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>ND or &lt;RL</td>
<td>ND or &lt;RL</td>
<td>ND or &lt;RL</td>
</tr>
<tr>
<td>Benzene</td>
<td>ND or &lt;RL</td>
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<td>ND or &lt;RL</td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>ND or &lt;RL</td>
<td>ND or &lt;RL</td>
<td>ND or &lt;RL</td>
</tr>
<tr>
<td>Bromoform</td>
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<tr>
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</tr>
<tr>
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</tr>
<tr>
<td>Carbon disulfide</td>
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<tr>
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<td>cis-1,3-Dichloropropene</td>
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<td>methylene chloride</td>
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<td>Vinyl chloride</td>
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<td>xylenes (total)</td>
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### GC/MS Semivolatiles*

#### Method SW846 8270C

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<th>D-24&quot;</th>
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*Values not reported are ND or <RL
# GC Semivolatiles*

## Method SW846 8081A

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*Values not reported are ND or <RL*
**GC Semivolatiles**

**Method SW846 8082**

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# Total Metals For Selected Parameters*

## Method SW846 6010B

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## Method SW846 7471A

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*Full laboratory data include:
  - Calcium
  - Iron
  - Magnesium
  - Manganese
  - Potassium
  - Selenium
  - Sodium
  - Thallium
  - Vanadium
  - Zinc
DEBRIS REMOVAL AND DISPOSAL
All drums and other metal debris were taken to Mayer Pollack Steel Corporation, Pottstown, PA, for metals recycling. A copy of the delivery ticket accompanies this report as APPENDIX E.

In addition to the marked drum sites other metallic debris in the area was also removed, including signs, electrical components, miscellaneous pieces of steel, and over 100 beer cans.

SITE INSPECTION
As mentioned in the sampling section, each site has been marked with an orange construction pin flag. In addition, the area of the drums removal has been cleared with a weed eater and other tools to facilitate return to the sites. The path has been marked with day-glo lime green or orange tape.
# APPENDIXES

<table>
<thead>
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<tr>
<td>APPENDIX A</td>
<td>Edited Aerial Photograph</td>
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<td>APPENDIX B</td>
<td>Edited Hand Drawn Maps</td>
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<tr>
<td>APPENDIX C</td>
<td>UTM Coordinates for Epic Features and Drum Sites</td>
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<tr>
<td>APPENDIX D</td>
<td>Sketch of Disturbed Soil Core Sampler</td>
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<td>APPENDIX E</td>
<td>Metals Recycling Delivery Ticket</td>
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APPENDIX A: Edited Aerial Photograph
Sheet 3 of 3

LARGE SUGAR MAPLE

CRUSHED METAL OBJ. RIVETED LIKE A PRESSURE VESSEL

Cleaned

100 FT 15° N

CRUSHED METAL WIRE WORKS

LARGE SCAB

0487677 4448980

From Sheet 2
## APPENDIX C: UTM Coordinates of EPIC Feature Sites and UTM Coordinates of Drum Sites

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</table>

DATE: 6/17/03

NAME: D. OOP/ROD

MAYER POLLOCK STEEL CORPORATION

70568

DRIVER ON: WILLOP GROVE

CLASSIFICATION

NONFERROUS
APPENDIX B

TtNUS SOPs
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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.
## FIGURE 1
BORING LOG (EXAMPLE)

### BORING LOG

**PROJECT NAME:**

**PROJECT NUMBER:**

**DRILLING COMPANY:**

**DRILLING RIG:**

**BORING NUMBER:**

**DATE:**

**GEOLOGIST:**

**DRILLER:**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Depth (ft) or Run No.</th>
<th>Blows / 8&quot; or RQD (%)</th>
<th>Sample Recovery / Sample Length</th>
<th>Lithology Change (Depth or Screened Interval)</th>
<th>Soil Density / Consistency or Rock Hardness</th>
<th>Color</th>
<th>Material Classification</th>
<th>USCS</th>
<th>Remarks</th>
<th>Pd/Rd Reading (ppm)</th>
</tr>
</thead>
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<tr>
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</tr>
</tbody>
</table>

* When rock coring, enter rock brokenness.

** Include monitor reading in 8 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks:

Drilling Area

Background (ppm):

Converted to Well: Yes ______ No ______ Well I.D. #: ______

019611/P

Tetra Tech NUS, Inc.
5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued). This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silt and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:
Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

### 5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

<table>
<thead>
<tr>
<th>Terms of Identifying Proportion of the Component</th>
<th>Defining Range of Percentages by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trace</td>
<td>0 - 10 percent</td>
</tr>
<tr>
<td>Some</td>
<td>11 - 30 percent</td>
</tr>
<tr>
<td>Adjective form of the soil type (e.g., &quot;sandy&quot;)</td>
<td>31 - 50 percent</td>
</tr>
</tbody>
</table>
## FIGURE 2

**CONSISTENCY FOR COHESIVE SOILS**

<table>
<thead>
<tr>
<th>Consistency</th>
<th>Standard Penetration Resistance (Blows per Foot)</th>
<th>Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)</th>
<th>Field Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very soft</td>
<td>0 to 2</td>
<td>Less than 0.25</td>
<td>Easily penetrated several inches by fist</td>
</tr>
<tr>
<td>Soft</td>
<td>2 to 4</td>
<td>0.25 to 0.50</td>
<td>Easily penetrated several inches by thumb</td>
</tr>
<tr>
<td>Medium stiff</td>
<td>4 to 8</td>
<td>0.50 to 1.0</td>
<td>Can be penetrated several inches by thumb with moderate effort</td>
</tr>
<tr>
<td>Stiff</td>
<td>8 to 15</td>
<td>1.0 to 2.0</td>
<td>Readily indented by thumb but penetrated only with great effort</td>
</tr>
<tr>
<td>Very stiff</td>
<td>15 to 30</td>
<td>2.0 to 4.0</td>
<td>Readily indented by thumbnail</td>
</tr>
<tr>
<td>Hard</td>
<td>Over 30</td>
<td>More than 4.0</td>
<td>Indented with difficulty by thumbnail</td>
</tr>
</tbody>
</table>
Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual’s judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features
### FIGURE 3

**BEDDING THICKNESS CLASSIFICATION**

<table>
<thead>
<tr>
<th>Thickness (metric)</th>
<th>Thickness (Approximate English Equivalent)</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 1.0 meter</td>
<td>&gt; 3.3'</td>
<td>Massive</td>
</tr>
<tr>
<td>30 cm - 1 meter</td>
<td>1.0' - 3.3'</td>
<td>Thick Bedded</td>
</tr>
<tr>
<td>10 cm - 30 cm</td>
<td>4&quot; - 1.0'</td>
<td>Medium Bedded</td>
</tr>
<tr>
<td>3 cm - 10 cm</td>
<td>1&quot; - 4&quot;</td>
<td>Thin Bedded</td>
</tr>
<tr>
<td>1 cm - 3 cm</td>
<td>2/5&quot; - 1&quot;</td>
<td>Very Thin Bedded</td>
</tr>
<tr>
<td>3 mm - 1 cm</td>
<td>1/8&quot; - 2/5&quot;</td>
<td>Laminated</td>
</tr>
<tr>
<td>1 mm - 3 mm</td>
<td>1/32&quot; - 1/8&quot;</td>
<td>Thinly Laminated</td>
</tr>
<tr>
<td>&lt; 1 mm</td>
<td>&lt;1/32&quot;</td>
<td>Micro Laminated</td>
</tr>
</tbody>
</table>

(Weir, 1973 and Ingram, 1954)
5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth’s surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite (CaCO₃). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.
FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

<table>
<thead>
<tr>
<th>Particle Name</th>
<th>Grain Size Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cobbles</td>
<td>&gt; 64 mm</td>
</tr>
<tr>
<td>Pebbles</td>
<td>4 - 64 mm</td>
</tr>
<tr>
<td>Granules</td>
<td>2 - 4 mm</td>
</tr>
<tr>
<td>Very Coarse Sand</td>
<td>1 - 2 mm</td>
</tr>
<tr>
<td>Coarse Sand</td>
<td>0.5 - 1 mm</td>
</tr>
<tr>
<td>Medium Sand</td>
<td>0.25 - 0.5 mm</td>
</tr>
<tr>
<td>Fine Sand</td>
<td>0.125 - 0.25 mm</td>
</tr>
<tr>
<td>Very Fine Sand</td>
<td>0.0625 - 0.125 mm</td>
</tr>
<tr>
<td>Silt</td>
<td>0.0039 - 0.0625 mm</td>
</tr>
</tbody>
</table>

After Wentworth, 1922
5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures
The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

\[
\text{RQD \%} = \frac{r}{l} \times 100
\]

\[r = \text{Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.}\]

\[l = \text{Total length of the coring run.}\]

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.
5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- **Seam** - Thin (12 inches or less), probably continuous layer.
- **Some** - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- **Few** - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- **Interbedded** - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- **Interlayered** - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- **Basalt** - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- **Rhyolite** - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- **Granite** - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- **Diorite** - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- **Gabbro** - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- **Slate** - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- **Phyllite** - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- **Schist** - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- **Gneiss** - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- **Quartzite** - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.
5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

<table>
<thead>
<tr>
<th>C</th>
<th>Coarse</th>
<th>Lt</th>
<th>Light</th>
</tr>
</thead>
<tbody>
<tr>
<td>Med</td>
<td>Medium</td>
<td>BR</td>
<td>Broken</td>
</tr>
<tr>
<td>F</td>
<td>Fine</td>
<td>BL</td>
<td>Blocky</td>
</tr>
<tr>
<td>V</td>
<td>Very</td>
<td>M</td>
<td>Massive</td>
</tr>
<tr>
<td>SI</td>
<td>Slight</td>
<td>Br</td>
<td>Brown</td>
</tr>
<tr>
<td>Occ</td>
<td>Occasional</td>
<td>BI</td>
<td>Black</td>
</tr>
<tr>
<td>Tr</td>
<td>Trace</td>
<td></td>
<td></td>
</tr>
<tr>
<td>YI</td>
<td>Yellow</td>
<td>Or</td>
<td>Orange</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SS</td>
<td>Sandstone</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sh</td>
<td>Shale</td>
</tr>
<tr>
<td></td>
<td></td>
<td>LS</td>
<td>Limestone</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Fgr</td>
<td>Fine-grained</td>
</tr>
</tbody>
</table>

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.
### FIGURE 5
**COMPLETED BORING LOG (EXAMPLE)**

**BORING LOG**

<table>
<thead>
<tr>
<th>Sample No. and Type or RQD</th>
<th>Depth (ft) or Run No.</th>
<th>Blow / 4&quot; or RQD %</th>
<th>Sample Recovery / Sample Length</th>
<th>Lithology Change (Depth/Flx) or Screened Interval</th>
<th>Color</th>
<th>Soil Density/Consistency or Rock Hardness</th>
<th>Material Classification</th>
<th>Remarks</th>
<th>USCS</th>
<th>Remarks</th>
<th>PID/VID Reading (ppm)</th>
</tr>
</thead>
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<tr>
<td>5-1</td>
<td>0.0</td>
<td>7</td>
<td>13/2.0</td>
<td>Dense</td>
<td>Silty Sand - Some</td>
<td>Moist Silt Clay</td>
<td>Drill to 4 ft</td>
<td>5</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.00</td>
<td>2.0</td>
<td>9</td>
<td>10</td>
<td>Rock Fr - TR Bricks</td>
<td>Fill to 4 ft</td>
<td>Siltstone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td>Wet</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5-2</td>
<td>0.0</td>
<td>6</td>
<td>2.0</td>
<td>Dense</td>
<td>Silty Sand - TR Fine</td>
<td>Moist - W Odor</td>
<td></td>
<td>10</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.10</td>
<td>6.0</td>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td>Siltstone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>9.0</td>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td>Clay - SW</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5-3</td>
<td>0.0</td>
<td>10</td>
<td>12.0</td>
<td>Dense</td>
<td>Fine to Coarse Sand</td>
<td>Wet</td>
<td>Hit Water 17 ft</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.20</td>
<td>12.0</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td>TrF. Gravel</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>12.0</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td>Siltstone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>14.0</td>
<td>15</td>
<td>15.0</td>
<td>Hard</td>
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<td></td>
<td>15.0</td>
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<tr>
<td></td>
<td>19.0</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td>Lost Core</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td>drill H2O 17 ft</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>25.0</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
<td>Siltstone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>28.0</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
<td>Sand 14 - 25</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*When rock coring, enter rock brokenness.

**Remarks:**
- Drilling Area: 1-20' [D] 80' [B] 1-80' [D] Background (ppm): 0

**Converting to Well:** Yes [X] No [ ] Well I.D. #: MW-1

019611/P Tetra Tech NUS, Inc.
Enter color of the material in the appropriate column.

Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

- Trace: 0 - 10 percent
- Some: 11 - 30 percent
- And/Or: 31 - 50 percent

Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

- Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

- Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

- Particle shape - flat, elongated, or flat and elongated.

- Maximum particle size or dimension.

- Water level observations.

- Reaction with HCl - none, weak, or strong.

Additional comments:

- Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

- Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

- Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

- At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).
- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:
  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:
  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:
  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.
  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.
5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.

- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.

- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.

- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.

- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).


7.0 RECORDS

Originals of the boring logs shall be retained in the project files.
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**ATTACHMENTS**

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<th>NAME</th>
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<td>A</td>
<td>TYPICAL SITE LOGBOOK ENTRY</td>
</tr>
<tr>
<td>B</td>
<td>SAMPLE LABEL</td>
</tr>
<tr>
<td>C</td>
<td>CHAIN-OF-CUSTODY RECORD FORM</td>
</tr>
<tr>
<td>D</td>
<td>CHAIN-OF-CUSTODY SEAL</td>
</tr>
</tbody>
</table>
1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day
that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project’s central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialled and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project’s central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.
5.3 **Field Forms**

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 **Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results**

5.3.1.1 **Sample Log Sheet**

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 **Sample Label**

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 **Chain-of-Custody Record Form**

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 **Chain-of-Custody Seal**

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.
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<td>Effective Date</td>
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5.3.1.5 **Geochemical Parameters Log Sheets**

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 **Hydrogeological and Geotechnical Forms**

5.3.2.1 **Groundwater Level Measurement Sheet**

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 **Data Sheet for Pumping Test**

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 **Packer Test Report Form**

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 **Boring Log**

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The “Remarks” column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 **Monitoring Well Construction Details Form**

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 **Test Pit Log**

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.
5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is collected, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.
5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day’s field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record
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Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist
TYPICAL SITE LOGBOOK ENTRY

START TIME: ________________ DATE: ________________

SITE LEADER: ______________________ PERSONNEL: ______________________

TNUS DRILLER SITE VISITORS

___________________________ ____________________________ __________________________

___________________________ ____________________________ __________________________

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well _____ resumes. Rig geologist was ________________. See Geologist’s Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist’s Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well ______.
4. Well _____ drilled. Rig geologist was ________________. See Geologist’s Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manager arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _______.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was ________________. See Geologist’s Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ____ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

___________________________
Field Operations Leader
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<tr>
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<td>Time:</td>
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<tr>
<td>Analysis:</td>
<td></td>
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<tr>
<td>Sampled by:</td>
<td>Laboratory:</td>
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Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, PA 15220
(412)921-7090
<table>
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<tr>
<th>DATE</th>
<th>YEAR</th>
<th>TIME</th>
<th>SAMPLE ID</th>
<th>LOCATION</th>
<th>Q (QUANTITY)</th>
<th>TOP DEPTH (FT)</th>
<th>BOTTOM DEPTH (FT)</th>
<th>MATRIX (OW, SW, SRF, QC, ETC.)</th>
<th>COLLECTION METHOD</th>
<th>CONTAINER TYPE</th>
<th>PLASTIC (P) or GLASS (G)</th>
<th>PRESERVATIVE</th>
<th>COMPLIANCE</th>
<th>COMMENTS</th>
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   DATE: [ ] TIME: [ ]

2. RELINQUISHED BY
   DATE: [ ] TIME: [ ] 2. RECEIVED BY
   DATE: [ ] TIME: [ ]

3. RELINQUISHED BY
   DATE: [ ] TIME: [ ] 3. RECEIVED BY
   DATE: [ ] TIME: [ ]

COMMENTS

DISTRIBUTION:
- WHITE (ACCOMPANIES SAMPLE)
- YELLOW (FIELD COPY)
- PINK (FILE COPY)

FORM NO. 7NUS-001
ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

Signature

Date

CUSTODY SEAL

Date

Signature
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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

**Acid** - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

**Alconox/Liquinox** - A brand of phosphate-free laboratory-grade detergent.

**Decontamination Solution** - Is a solution selected/identified within the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

**Deionized Water (DI)** - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

**Potable Water** - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

**Pressure Washing** - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

**Solvent** - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

**Steam Pressure Washing** - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.
4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Failure to meet these objectives are sufficient to restrict equipment from entering the site/exitng the site/ or moving to a new location on the site until the objectives are successfully completed.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations.
Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

- Pedestrian/Vehicle thoroughfares
- Areas where control/custody cannot be maintained
- Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
- Areas potentially contaminated.

Pad – The pad should be constructed to provide the following characteristics

- Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

- Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

- Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

- Liner – Depending on the types of equipment and the decontamination method the liner should be of sufficient thickness to provide a puncture resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. Achieving the desired thickness maybe achieved through layering lighter constructed materials. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner a light coating of sand maybe applied to provide traction as necessary.

- Wash/drying Racks – Auger flights, drive/hold rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

- Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.
Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.
2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.
3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.
4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).
5) Rinse equipment with pesticide grade isopropanol
6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable.
7) Drain residual deionized water to the extent possible, allow components to air dry.
8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

- The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.
1) Wash with soap and water
2) Rinse with tap water
3) Rinse with deionized water

**Note:** In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

### 5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer’s instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- **Structural integrity** – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.
- **Cleanliness** – As per protocol only volatile organic samples are accompanied by a trip blank. If a cooler’s cleanliness is in question (visibly dirty/stained) or associated with noticeable odors it should be decontaminated prior to use.

1) Wash with soap and water
2) Rinse with tap water
3) Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

### 5.2.2 Down-Hole Drilling Equipment

This includes any portion of the drill rig that is over the borehole including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

### 5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.
1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

During the course of field investigations disposable/single use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.
- Rinsate Blanks – It is recommended that Rinsate samples be collected to
  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.
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## ATTACHMENTS

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4. OSHA Letter of Interpretation ...................................................................................... 13
1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.
4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TinUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2. A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scars and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility...
locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State “one-call” services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a “ticket” number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, “one call” systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

- white excavation/subsurface investigation location
- red electrical
- yellow gas, oil, steam
- orange telephone, communications
- blue water, irrigation, slurry
- green sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TTNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.
5.2 **Overhead Power Lines**

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

<table>
<thead>
<tr>
<th>Nominal Voltage</th>
<th>Minimum Clearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 -50 kV</td>
<td>10 feet, or one mast length; whichever is greater</td>
</tr>
<tr>
<td>50+ kV</td>
<td>10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater</td>
</tr>
</tbody>
</table>

6.0 **UNDERGROUND LOCATING TECHNIQUES**

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 **Geophysical Methods**

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

**Electromagnetic Induction**

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.
Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting
debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

**Hand Excavation**

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

**Tile Probe Surveys**

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

**7.0 INTRUSIVE ACTIVITIES SUMMARY**

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.
5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys
ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

ONE-CALL SYSTEMS INTERNATIONAL
CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5346

Arkansas
Arkansas One Call System, Inc.
1-800-492-1987

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-492-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4960
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8589

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-227-2600

Louisiana
Louisiana One Call System, Inc.
1-800-227-2600

Maine
Dig Safe System, Inc.
1-800-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-800-344-7233

Michigan
Miss Dig System, Inc.
1-800-432-7171

Minnesota
Gopher State One Call
1-800-252-1266

Mississippi
Mississippi One-Call System, Inc.
1-800-227-2477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-8555
Montana One Call Center
1-800-551-8344

Nebraska
Digger's Hotline of Nebraska
1-800-331-0555

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-800-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537

New York
Dig Safely New York
1-800-992-7992
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-832-4849

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-269-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0888

Oklahoma
Dig Safe System, Inc.
1-800-344-7233

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-800-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One Call System, Inc.
1-800-351-1111

Tetra Tech NUS, Inc.
ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-0377
Lone Star Notification Center
1-800-669-6344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-663-9228

Quebec
Info-Excavation
1-800-663-9228
ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION

FROST PENETRATION

Average Depth In Inches

Courtesy U.S. Department Of Commerce
## ATTACHMENT 3
### UTILITY CLEARANCE FORM

**Client:**

**Project No.:**

**Completed By:**

**Location Name:**

**Work Date:**

### Excavation Method/Overhead Equipment:

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<thead>
<tr>
<th>Circle One</th>
<th>yes</th>
<th>no</th>
<th>N/A</th>
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1. **Underground Utilities**

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<td>a)</td>
<td>Review of existing maps?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>b)</td>
<td>Interview local personnel?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>c)</td>
<td>Site visit and inspection?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>d)</td>
<td>Excavation areas marked in the field?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>e)</td>
<td>Utilities located in the field?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>f)</td>
<td>Located utilities marked/added to site maps?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>g)</td>
<td>Client contact notified</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td></td>
<td>Name:</td>
<td></td>
<td>Telephone:</td>
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<tr>
<td>g)</td>
<td>State One-Call agency called?</td>
<td>yes</td>
<td>no</td>
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<tr>
<td></td>
<td>Caller:</td>
<td></td>
<td>Ticket Number:</td>
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<tr>
<td>h)</td>
<td>Geophysical survey performed?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td></td>
<td>Survey performed by:</td>
<td></td>
<td>Method:</td>
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<tbody>
<tr>
<td>i)</td>
<td>Hand excavation performed (with concurrent use of utility detection device)?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td></td>
<td>Completed by:</td>
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<td>Total depth:</td>
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<tbody>
<tr>
<td>j)</td>
<td>Trench/excavation probed?</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td></td>
<td>Probing completed by:</td>
<td></td>
<td>Depth/frequency:</td>
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2. **Overhead Utilities**

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<tr>
<th></th>
<th>Present</th>
<th>Absent</th>
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<tr>
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<th>yes</th>
<th>no</th>
<th>N/A</th>
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<tr>
<td>a)</td>
<td>Determination of nominal voltage</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>b)</td>
<td>Marked on site maps</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>c)</td>
<td>Necessary to lockout/insulate/re-route</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>d)</td>
<td>Document procedures used to lockout/insulate/re-route</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>e)</td>
<td>Minimum acceptable clearance (SOP Section 5.2):</td>
<td></td>
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3. **Notes:**

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**Approval:**

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--- c: PM/Project File Program File
ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651 (Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours ** or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:
When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by \textit{safe and acceptable means}. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, \textit{equally effective means} of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used \textit{where the utilities have not been marked and detection equipment is not being used} -- "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

\begin{quote}
OSHA received two comments ** and input from ACCSH \{OSHA's Advisory Committee on Construction Safety and Health\} ** on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.
\end{quote}

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commenters and ultimately not including any examples of "acceptable means" in the final provision.

\textbf{Non-conductive hand tools are permitted}

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, \textit{when used with appropriate caution}, is an "acceptable means" for locating underground utilities.
Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a “acceptable means” of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be “acceptable means” under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA=s website at http://www.osha.gov.
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1.0 SW-846 ORGANICS BY GC/MS

1.1 Volatiles (Method 8260B)

1.1.1 Applicability

Method 8260B is used to determine volatile organic compounds in most waste matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 8260B analyte list includes of the volatile CLP 3/90 Target Compound List (TCL) (Section 1.1.1) plus the following compounds*:

- Acetonitrile
- Acrolein
- Acrylonitrile
- Allyl chloride
- Chloropropene
- trans-1,2-Dibromo-3-chloropropane
- 1,2-Dibromoethane
- Dibromomethane
- trans-1,4-Dichloro-2-butene
- Dichlorodifluoromethane

- Ethyl methacrylate
- Iodomethane
- Methacrylonitrile
- Methyl methacrylate
- 2-Picoline
- Pyridine
- Trichlorofluoromethane
- 1,2,3-Trichloropropane
- Vinyl acetate

* Appendix IX target compounds

Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared by Method 5030.

1.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

1.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.
1.1.4 Sample Preparation

Method 5030 is a purge-and-trap procedure performed to prepare and extract volatile compounds from samples and introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

1.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

1.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.
Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

1.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIIs. Check the initial calibration date(s) noted in the headings of the Form VIIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form V. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >25%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds whose RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J), when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 25% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).
The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply, and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

1.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance, nondetects are not qualified based on high surrogate recovery.

1.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for appropriately evaluating MS/MSD analyses.

1.1.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate data validation protocol.

1.1.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

1.1.6.8 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than
the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.1.6.9 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

1.2 Semivolatiles (Method SW8250A, 8270C)

1.2.1 Applicability

Methods are applicable to most types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

These methods can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of elution without derivatization as sharp peaks from a gas chromatographic column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.
The above methods specifically analyze for the semivolatile Target Compound List (TCL) (Section 1.1.2) plus the following compounds*:

- Acetophenone
- Aniline
- Benzyl alcohol
- Bis(2-chloroisopropyl)ether
- Chlorobenzilate
- Diallate
- 2,6-Dichlorophenol
- Dimethoate
- p-Dimethylaminoazobenzene
- 7,12-Dimethylbenz(a)anthracene
- 3,3′-Dimethylbenzidine
- a,a-Dimethylphenylamine
- 1,3-Dinitrobenzene
- Diphenylamine
- Ethyl methanesulfonate
- Famphur

- Hexachlorophene
- Hexachloropropene
- Isodrin
- Isosafrole
- Kepone
- Methapyrilene
- 3-Methylcholanthrene
- Methyl methanesulfonate
- 3-Methylphenol
- N-nitrosodimethylamine
- N-nitrosodiethylamine
- N-nitrosodimethylethylamine
- N-nitroso-di-n-butylamine
- N-nitrosomorpholine
- N-nitrosopiperidine
- Pentachlorobenzene
- Pentachloronitrobenzene
- Phenacetin
- p-Phenylenediamine
- Phorate
- 2-Picoline
- Pronamide
- Safrole
- 1,2,4,5-Tetrachlorobenzene
- Thionazin
- o,o,o-Triethylphosphorothioate
- 1,3,5-Trinitrobenzene

* Appendix IX target compounds

The preceding methods are based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures, Method 8270C uses GC/MS capillary column technique.

1.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source, depending upon the diversity of the industrial complex or waste being sampled.

1.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or sonication (Method 3550) procedures.
1.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.

- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

1.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days

- Analysis: 40 days from date of extraction

Affected positive results are generally qualified as estimated (J), nondetects (UJ). Alternately, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance. If the sample was extracted beyond 14 days from collection (28 days for solid samples), the holding time exceedance is considered to be gross and positive results are qualified as estimated (J) or (L); nondetects are rejected (R). Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.
1.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form VVs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs, and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >30%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 30%, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.2.6.3 Blank Contamination

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The action levels for qualification are 10X the maximum amount of phthalates found in the blanks (phthalates are common contaminants) and 5X the maximum amount of other contaminants found in the blanks. The actual action level applied is sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and moisture content. The type and manner in which the qualifiers are applied vary with protocol [i.e., use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate data validation protocol for specific guidance.
1.2.6.4 **Surrogates**

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The quality control ranges are given on the laboratory data package Form IIIs; circle any noncompliances on your working copies of these Forms.

Semivolatile compounds are divided into two classes, base-neutral compounds and acid-extractable compounds. Each class of compounds has its own associated surrogates. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and nondetects are rejected, (R). These results are biased low.

No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.

If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); nondetects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; nondetects are not qualified based on high surrogate recoveries.

1.2.6.5 **Matrix Spike/Matrix Spike Duplicates**

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

1.2.6.6 **Internal Standards**

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

1.2.6.7 **Tentatively Identified Compounds (TICs)**

TICs are evaluated using the laboratory data package Form I BNA-TIC reports and the laboratory raw data. The guidance given in the 3/90 National Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

1.2.6.8 **Other Considerations**

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results. Consider nondetects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be less than 35%; for soil matrix results, less than 50%. Qualification of sample data is limited to that specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); and nondetects (UJ). Bias for these results cannot be determined.
In some USEPA regions a "Percent Solids" rule applies. For example, if a sediment contains less than 50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.2.6.9  Quantitation

Verify and record quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.2.7  Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.0  SW846 NON-CLP ORGANICS BY GAS CHROMATOGRAPHY

2.1  Volatiles (SW 5030/SW 8011/8015B/8021A/8031)

2.1.1  Applicability

Method 8011 is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

1,2-Dibromoethane (EDB)
1,2-Dibromo-3-chloropropane (DBCP)

Method 8021A is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Allyl chloride
Benzyl chloride
Bis (2-chloroethoxy)methane
Bis (2-chloroisopropyl)ether
Bromoacetone
Bromobenzene
Bromodichloromethane
Bromoform
Bromomethane
Carbon tetrachloride
Chlorobenzene
Chloroethane
2-Chloroethanol
Chloroform
1-Chlorohexane
Method 8015B is used to determine the concentration of the following nonhalogenated volatile organic compounds in groundwater, liquid, and solid matrices:

- Diethyl ether
- Acrolein
- n-butyl Alcohol
- Ethanol
- Acetonitrile
- t-butyl Alcohol
- Methyl ethyl ketone (MEK)
- Acetone
- Methanol
- Methyl isobutyl ketone (MIBK)
- Allyl Alcohol
- 1,4-Dioxane

Method 8031 is used to determine the concentration of the following volatile organic compound in groundwater, liquid, and solid matrices:

- Acrylonitrile
All of the above Methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). Method 8021A analyzes for halogenated and aromatic volatile organics via GC/HECP and GC/PID (Electro Conductivity Detector and Photoionization detector), Method 8015B analyzes for nonhalogenated volatile organics via GC/FID (Flame Ionization Detector), and Method 8031 analyzes for the compounds acrylonitrile using GC/FID. Samples can be analyzed by these methods using direct injection, the headspace method (Method 5021) or the purge-and-trap method (Method 5030B and 5035). Groundwater samples should be determined using Method 5030B.

2.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

2.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.1.4 Sample Preparation

Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. Because of the large variability and complicated matrices of waste samples detection limits for this method may vary widely among samples.

Method 5030 is a purge-and-trap method applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, groundwater, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 5035 is a purge-and-trap method applicable to nearly all types of soil samples, regardless of water content, including oily wastes, soils, and sediments.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.
2.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.

- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

2.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

2.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.
In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!). Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.
All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recoveries.

2.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.1.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.1.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.
2.2 **Semivolatiles (SW8041/8061A/8091/8310)**

2.2.1 **Applicability**

Method 8041 is used to determine the concentration of the following phenolic compounds in groundwater, liquid, and solid matrices:

- Phenol
- 2-Chlorophenol
- 2,4-Dichlorophenol
- 2,6-Dichlorophenol
- Trichlorophenols
- Tetrachlorophenols
- Pentachlorophenol
- Cresols (methyl phenols)
- 4-Chloro-3-methylphenol
- 2,4-Dimethylphenol
- 2-Nitrophenol
- 4-Nitrophenol
- 2,4-Dinitrophenol
- 2-sec-Butyl-4,6-dinitrophenol (DNBP)
- 2-Cyclohexyl-4,6-dinitrophenol
- 2-Methyl-4,6-dinitrophenol

Method 8061A is used to determine the concentration of the following phthalate esters in groundwater, liquid, and solid sample matrices:

- Benzyl butyl phthalate
- Bis(2-ethylhexyl)phthalate
- Di-n-butyl phthalate
- Di-n-octyl phthalate
- Diethyl phthalate
- Dimethyl phthalate

Method 8091 is used to determine the concentration of the following nitroaromatic and cyclic ketone compounds in groundwater, liquid, and solid sample matrices:

- Nitrobenzene
- Dinitrobenzene
- 2,4-Dinitrotoluene
- 2,6-Dinitrotoluene
- Isophorone
- Naphthoquinone

Method 8310 is used to determine the concentration of the following polynuclear aromatic hydrocarbons (PAHs) in liquid and solid sample matrices:

- Acenaphthene
- Acenaphthylene
- Anthracene
- Benzo(a)anthracene
- Benzo(a)pyrene
- Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Naphthalene
Phenanthrene
Pyrene

All of the above methods are gas chromatographic (GC), with the exception of Method 8310 which is a High Performance Liquid Chromatography (HPLC) technique, only (i.e., no mass spectrometer detector is employed). These methods use either an electron capture detector (ECD), a flame ionization detector (FID), a ultraviolet detector (UV), or a fluorescence detector.

### 2.2.2 Interferences

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source depending upon the waste being sampled. While general cleanup techniques such as Method 3530 are provided as part of these methods, unique samples may require additional cleanup.

If sample or matrix interferences occur, a secondary column may be employed in addition to the primary column so as to resolve any questionable compound results.

### 2.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

### 2.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using Method 3510 (separatory funnel extraction) or Method 3520 (continuous liquid-liquid extraction). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or Sonication (Method 3550) procedures.

### 2.2.5 Data Overview Prior to Validation

Before commencing validation the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:
• If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.

• The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

2.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

• Extraction:
  Water samples: 7 days
  Solid samples: 14 days
• Analysis: 40 days from date of extraction

Generally, positive results affected by noncompliances are qualified as estimated (J); nondetects (UJ). These results are considered to be biased low. Alternately, the bias qualifiers L and UL may be used. Nondetects may be rejected (R) when the sample was extracted after 14 days (28 days for solid samples). If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Refer to the appropriate data validation protocol for specific guidance.

2.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to
qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the \%RSD is used, determine which compounds have Percent Relative Standard Deviations (\%RSDs) >40\% and between 20\%-40\%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the \%RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the \%Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (\%Ds) >30\%, and between 15\%-30\%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which \%RSD or \%D exceeds 40\% or 30\%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the \%RSD or \%D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which \%RSD is between 20\%-40\% or whose \%D is between 15\%-30\% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.2.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10\%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10\%, positive results are
generally qualified as estimated (J); nondetects (U). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recovery.

2.2.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.2.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (U). Bias for these results cannot be determined.

In some USEPA Regions, a “Percent Solids” rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated, and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.
2.3 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides (SW 8081A/8082/8141A/8151A)

2.3.1 Applicability

Methods 8081A/8082 are used to determine the concentration of the following organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices:

- Aldrin
- alpha-BHC
- beta-BHC
- delta-BHC
- gamma-BHC (Lindane)
- Chlordane
- 4,4'-DDD
- 4,4'-DDE
- 4,4'-DDT
- Dieldrin
- Endosulfan I
- Endosulfan II
- Endosulfan sulfate
- Endrin
- Endrin aldehyde
- Heptachlor
- Heptachlor epoxide
- Methoxychlor
- Toxaphene
- Aroclor-1016
- Aroclor-1221
- Aroclor-1232
- Aroclor-1242
- Aroclor-1248
- Aroclor-1254
- Aroclor-1260

Similarly, Method 8141A is used to determine the following pesticides in groundwater and waste samples:

- Azinphos methyl
- Bolstar (Sulprofos)
- Chlorpyrifos
- Coumaphos
- Demeton-O
- Demeton-S
- Diazinon
- Dichlorvos
- Disulfoton
- Ethoprop
- Fensulfothion
- Fenthion
- Merphos
- Mevinphos
- Naled
Parathion methyl
Phorate
Ronneel
Stirophos (Tetrachlorvinphos)
Tokuthion (Prothiofos)
Trichloronate

Note that when Method 8141A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique if mass spectroscopy is not employed.

Method 8151A is used to determine the following chlorinated acid herbicides in groundwater and waste samples:

2,4-D
2,4-DB
2,4,5-T
2,4,5-TP (Silvex)
Dalapon
Dicamba
Dichloroprop
Dinoseb
MCPA
MCPP
4-Nitrophenol
Pentachlorophenol

Since these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A includes a hydrolysis step to convert the herbicide to the acid form prior to analysis. When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column; alternately, the compounds of interest can be confirmed by detection via a mass spectrometer.

All of the above Methods are Gas Chromatographic (GC) in which sample extracts are analyzed by direct injection. Methods 8081A and 8082 analyze for organochlorine pesticide compounds and PCBs via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used). Method 8141A analyzes for organophosphorous pesticide compounds via GC/FID (Flame Ionization Detector), and Method 8151A analyzes for chlorinated herbicide compounds via GC/ECD (alternately, a Microcoulometric Detector or Hall Electrolytic Conductivity Detector may be used).

### 2.3.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably, and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.
2.3.3 General Laboratory Practices

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicate and laboratory duplicates should also be employed.

Note that herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, when performing Method 8151A, glassware and glass wool must be acid-rinsed and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

2.3.4 Sample Preparation

Prior to the use of Methods 8081, 8082, and 8141A, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Solid samples are extracted with hexane:acetone (1:1) using either the Soxhlet extraction (Method 3540) or sonication (Method 3550) procedures.

Method 8151A provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetone and diethyl ether followed by esterification using diazomethane as a derivatizing agent.

2.3.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.3.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.
2.3.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- **Extraction:**
  - Water samples: 7 days
  - Solid samples: 14 days
- **Analysis:** 40 days from date of extraction

When the holding time criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected (R). These results are biased very low.

2.3.6.2 Calibration

Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.
Method 8081A requires analysis of a DDT/Endrin breakdown check standard. The DDT/Endrin Breakdown should not exceed 20%. Generally, if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE and DDD following the in-last control standard until the next in-control standard (see analytical sequence). If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) nondetects for DDT in associated samples. Generally, if Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable standard. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) nondetect Endrin results.

2.3.6.3 Blank Contamination

When using the information provided below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific, and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; guidelines provided in the appropriate data validation protocol should be followed.

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

2.3.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs; circle any recoveries outside these limits on your working copies of these Forms.

No qualifications are made for surrogates which show zero recoveries because they were “diluted out.” Generally, positive results affected by low surrogate recovery are qualified as estimated (J) or the (L) bias qualifier is used when applicable; nondetects are qualified (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; nondetects are not qualified based on high surrogate recovery. Because the surrogate recovery limits for these fractions are advisory, generally no results are rejected.

The pesticide/PCB surrogates decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII or equivalent must be 0.10 for DCB and 0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peak. The validator’s professional judgment for qualifications should be used.

2.3.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate data validation guidelines for specific procedures for evaluating MS/MSD analyses.

2.3.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.
Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.3.6.7  Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10%.

2.3.7  Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.4  Explosives/Nitroaromatics/Nitroamines(SW 8330)

2.4.1  Applicability

Method 8330 is used to determine the concentration of the following explosives, nitroaromatics, and nitroamines in water, soil, or sediment matrices:

- Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
- Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
- 1,3,5-Trinitrobenzene (1,3,5-TNB)
- 1,3-Dinitrobenzene (1,2-DNB)
- Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
- Nitrobenzene (NB)
- 2,4,6-Trinitrotoluene (2,4,6-TNT)
- 4-Amino-2,6-dinitrotoluene (4-Am-DNT)
- 2-Amino-4,6-dinitrotoluene (2-Am-DNT)
- 2,4-Dinitrotoluene (2,4-DNT)
- 2,6-Dinitrotoluene (2,6-DNT)
- 2-Nitrotoluene (2-NT)
- 3-Nitrotoluene (3-NT)
- 4-Nitrotoluene (4-NT)
- Nitroguanidine
- Nitroglycerin
- Pentaerythritol Tetranitrate (PETN)
The analysis of the compounds listed above is conducted by High Performance Liquid Chromatography equipped with a 254 nm Ultra Violet (UV) detector. This method is capable of determining part per billion (ppb) detection levels in water and soil matrices.

The method requires the use of both a primary (C-18 reverse phase) and a confirmation (CN reverse phase) column.

2.4.2 Interferences

The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

2,4-Dinitrotoluene and 2,6-dinitrotoluene may co-elute. High concentrations of one of the two isomers may cause interference of the other isomer. In instances where this is applicable, both isomers should be reported as one. Baseline resolution should be present for all compounds.

Decomposition of Tetryl occurs rapidly and when exposed to heat. Samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

2.4.3 General Laboratory Practices

Method blanks and instrumentation blanks should be conducted to access laboratory contamination.

Matrix spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field and laboratory duplicates may also be employed.

2.4.4 Sample Preparation

Method 8330 provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetonitrile and a salting-out procedure for aqueous samples. Soil samples are air dried prior to preparation, thus percent moisture is not a consideration when calculating compound concentrations.

2.4.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package.
- If each sample was correctly analyzed and identified for the specified parameters.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the best data quality.
Unless specifically directed by the client, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.4.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols, method requirements, and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this SOP is only intended as a general procedure for the data validation task.

Deficiencies, omissions, and/or other anomalies noted during the review require the data validator to contact the laboratory.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.4.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from the date of collection to the date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- **Extraction:****
  - Water Samples: 7 days
  - Solid Samples: 14 days
- **Analysis:** 40 days from date of extraction

When the holding times criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetected results, (UJ). These results are considered biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If holding times are exceeded by a factor of two or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected, (R). These results are considered to be biased very low.

2.4.6.2 Calibrations

Data pertaining to the initial calibration (i.e. evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels. The initial calibration should consist of a minimum of five concentration levels for each compound of interest.

In general, either the correlation coefficient (r) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is < 0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have %RSDs greater than 20%. Generally, associated data are qualified as estimated (J/UJ) if the calibration %RSD is >20%. Circle these noncompliances on working copies of calibration forms.
Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with each initial calibration by instrument. Write the affected samples on working copies of the appropriate continuing calibration forms. Spot-check (i.e. recalculate) a few of the %Ds to verify the laboratory's computation.

A continuing calibration or daily calibration must be performed at the beginning, midpoint and end of the analytical sequence. The continuing calibration response factor for each analyte must be compared to the response factor of the initial calibration. The continuing calibration response factor must agree within 15% of the initial response factor. Generally, positive and nondetected results are qualified as estimated (J/UJ) if the Percent Difference (%D) is >15%.

2.4.6.3 Blank Contamination

A review of all method and instrument blanks (if provided) is conducted to evaluate laboratory contaminants. An additional review of all relevant field quality control blanks is also conducted. Contaminants, if present, are summarized and the maximum concentration of each contaminant is selected and used to establish blank action levels.

An action level of 5X the maximum amount of each contaminant is used to evaluate sample data. Blank action levels must consider the aliquot used for analysis and sample dilution. Positive results less than the action level are qualified as false positives. The manner in which the qualifiers are applied varies [i.e., use of (U) or (B); replacement by the Reporting Limit]. General regional guidance procedures dictate the most appropriate validation action qualification.

2.4.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II or equivalent and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIIs. Circle any recoveries outside these limits on working copies.

Generally, positive results affected by low surrogate recoveries are estimated, (J) or (L), indicating low bias; nondetected results are qualified, (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated, (J), or the bias qualifier (K), is used when applicable. Nondetected results are not qualified based upon high surrogate recoveries. It should be noted that consideration of interferences may affect surrogate recoveries. If a trend of noncompliance is noted, an evaluation of sample chromatograms should be conducted when surrogate recoveries are noncompliant and a matrix effect is suspected.

No qualifications are made for surrogates which have been diluted out.

Generally, positive results associated with surrogate recoveries <10% are qualified as estimated, (J) or biased low (L). Nondetected results associated with surrogate recoveries <10 are considered unreliable and are qualified rejected (R).

2.4.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the unspiked sample. Typically, Percent Recoveries (%Rs) and the Relative Percent Difference (RPD) are evaluated based upon the laboratory provided control limits.
2.4.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked samples results with MS/MSD analyses result for unspiked compounds. Consider nondetected results and results reported at concentrations less than the reporting limit to be in agreement. Use professional judgment in determining whether to qualify sample results based upon the comparison. The comparison may be presented in terms of a %RSD or an RPD.

Likewise, compare positive compound results for field duplicate samples. Generally, an RPD between field duplicate results for the aqueous matrix should be <35%; for soil matrix results <50%. Qualification of the sample data is limited to specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetected results (UJ).

2.4.6.7 Quantitation

Verify and record the quantitation of at least one compound per fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. The validator and laboratory quantitations must agree within 10%. If quantitation differences are significant, the laboratory must be contacted to investigate and resolve the discrepancy.

2.2.4.7 Deliverable Guidance

In addition to any specific USEPA Regional requirements (i.e., data validation memorandum, data summary spreadsheets, USEPA Regional Worksheets), all laboratory data package quality summary forms, sample Form I reports method blank results and the Chain of Custody records must be included in the validation report.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the narrative is free of transcription and typographical errors before submitting all requested items for quality assurance review.
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1.0 INORGANICS (SW-846 6010B/7470A/7471A/9010A&B/7470/9010)

Inductively Coupled Plasma Emission Spectroscopy (ICP) - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium.

Cold Vapor Methodology - Mercury is commonly analyzed using cold vapor methodology.

Automated Colorimetric Technique - Cyanide is commonly analyzed using automated colorimetric methodology.

1.1 Applicability

These methods are applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

Detection limits for analytes are established on a quarterly basis and are both laboratory and instrument specific.

1.2 Data Overview Prior to Validation Process

1.2.1 Data Completeness

The data reviewer must initially verify that all forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required forms will include:

Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Calibration Summary (Form 2A or equivalent).

Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the blank summary (Form 3 or equivalent) (note, filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).

Verify that all ICP analytes are present in both ICSA and ICSAB solutions. Also, verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report “zeros” or simply leave blank the appropriate solution A columns.

Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels.

Verify that laboratory duplicate analyses were performed for each matrix. **NOTE:** Field quality control blanks are never to be designated for quality control analyses.
Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis.

The Method of Standard Additions (MSA) (Form 8 or equivalent) may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 4.1.3.11 for further details.

Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE:** Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12 (or equivalent). Verify that no GFAA analytical results exceed the highest standard in the associated GFAA calibration.

Verify that the Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillation as applicable.

Examine the Form 14s (or equivalent) to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

### 1.3 Technical Evaluation Summary

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

#### 1.3.1 Holding Times

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

- **Metals**: 6 months; pH <2
- **Mercury**: 28 days; pH <2
- **Cyanide**: 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only. Solid samples do not receive preservative but require maintenance at 4 ºC (2 ºC) during shipment and storage.
The above holding times do not apply to leachate analyses. It is suggested that the data reviewer reference SW-846 Method 1311 for any questions regarding TCLP quality control requirements and analytical procedural requirements; these vary significantly from non-TCLP analyses.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE:** Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

### 1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard

- **GFAA analyses** - must employ a blank and at least three standards. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e. $r = 0.995$ or greater)

- **Mercury analyses** - must employ a blank and at least three standards ($r = 0.995$ or greater).

- **Cyanide analyses** - must employ a blank and at least three standards ($r = 0.995$ or greater). **NOTE:** At least two additional standards (a high or low) must be distilled and compared to similar values on the curve. Values of distilled standards should agree within 10% of undistilled standards.

### 1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

The ICV/CCV %R quality control limits are 90-110% for ICP metals, 80-120% for GFAA metals and mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated, (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects remain unaffected. **NOTE:** Qualify results of only those samples associated with the noncompliant ICV or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

### 1.3.4 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours whichever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in a sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration
present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

1.3.5 ICP Interference Check Sample Results

Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. NOTE: Affected samples include all samples analyzed between the initial and final solutions or within the eight hour working shift whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 or equivalent and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample, while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

\[
\text{Estimated elemental int.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}] \times [\text{Conc. Sample}]}{\text{Interferent Conc. in ICS Soln A}}
\]

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (NOTE: Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).
1.3.6 Matrix Spike Sample Analysis (Pre-digestion)

Verify that at least one matrix spike was performed for each matrix for a given set of samples (maximum of 20 samples) within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Any deviations from the referenced method shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant, no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

1.3.7 Laboratory Duplicate Precision

Verify that one duplicate sample analysis was performed for each group of samples (maximum of 20 samples) of a similar matrix within an SDG. Control criteria used to evaluate the aqueous laboratory duplicates are as follows:

- a control limit of 20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of 35% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

**NOTE:** Review Duplicate Summary (Form 6 or equivalent) carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

1.3.8 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances were field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:
• a control limit of 30% for relative percent difference when sample and duplicate results are >5X CRDL

• a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

• a control limit of 50% for relative percent difference when sample and duplicate results are >5X CRDL

• a control limit of 4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

**NOTE:** The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, field duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

### 1.3.9 Laboratory Control Sample Results

Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R <80%, qualify as estimated (J) positive results and (UJ) nondetects, If aqueous LCS %R >120, qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

### 1.3.10 Method of Standard Additions (MSA)

Review MSA Form 8 or equivalent and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance in the method.

Actions - If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.
1.3.11  ICP Serial Dilution Analysis

Verify that all ICP analytes are included on the Form 9 (or equivalent) with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50 IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of suppressed results exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

1.3.12  Analysis Run Logs Form 14

The Form 14 or equivalent serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify GFAA PDS percent recoveries within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS % R.

1.3.13  Further GFAA Evaluations

It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

1.4  Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.
APPENDIX C

EPA FIELD SAMPLING GUIDE
Summary:

The purpose of this fact sheet is to specify procedures for the collection and handling by field samplers of soil samples for volatile organic analysis (VOA) in Region III. SW-846 Method 5035A is the collection method required for analysis of soil samples for VOA. This method incorporates chemical preservatives and sample storage techniques to limit volatilization and biodegradation of organic compounds. Method 5035A is applicable to both low/medium and high level soil samples.

Collection Procedures:

- Soil samples being analyzed for volatile organic compounds collected via Method 5035A should not be chemically preserved in the field.
- Samples should be collected using the following collection options:

**Option 1:** For most Soil types

Number of samples: 4 EnCore (or similar closed-sampling vessel) samples
4 QC EnCore samplers
1 40-mL vial for moisture analysis

1EnCore samplers (or similar sample collection device, refer to Section 4.5 of Method 5035)

Samples must be cooled to 4°C upon collection and during shipment and bagged individually upon collection.
Samples must be arrive at the laboratory within 24 hours.
Samples must be analyzed or preserved by the lab within 48 hours of collection.

**Option 2:** For Non-Cohesive Granular Material (wet, rocky, sediments, etc.)

Number of samples: 4 40mL vials (sampler may use wide mouth jars if sample not amiable to smaller vials)
2 QC 40 mL vials
1 40 mL vial for moisture analysis

Samples must be cooled to 4°C upon collection and during shipment.
Samples must be arrive at the laboratory within 24 hours.
Samples must be analyzed or preserved by the lab within 48 hours of collection.
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:

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<th>Response</th>
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</tr>
<tr>
<td>Tetrachloroethene</td>
<td>127-18-4</td>
<td>c</td>
<td>ms</td>
</tr>
<tr>
<td>Toluene</td>
<td>108-88-3</td>
<td>c</td>
<td>hs</td>
</tr>
<tr>
<td>(o)-Toluidine</td>
<td>95-53-4</td>
<td>pp</td>
<td>nd</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>120-82-1</td>
<td>c</td>
<td>hs</td>
</tr>
<tr>
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<td>71-55-6</td>
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<tr>
<td>1,1,2-Trichloroethane</td>
<td>79-00-5</td>
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<tr>
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<tr>
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<td>hvs</td>
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<td>hvs</td>
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<tr>
<td>(p)-Xylene</td>
<td>106-42-3</td>
<td>c</td>
<td>hvs</td>
</tr>
</tbody>
</table>

\(^a\) 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°C 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)

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 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, CH₃OH - purge-and-trap quality or equivalent. Store away from other solvents.

7.3 Polyethylene glycol (PEG), H(OCH₂CH₂)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, NaHSO₄ - 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-d₄, and toluene-d₈. Other compounds may be used as surrogates, depending upon the analysis requirements and the specific target analytes. The recommended internal standards are chlorobenzene-d₄, 1,4-dichlorobenzene-d₄, 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 not 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 not appropriate for use with the low concentration soil procedure described in this method.

**NOTE:** 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 known 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 not 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 known 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 not 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

<table>
<thead>
<tr>
<th>Approximate Concentration Range</th>
<th>Volume of Methanol Extract$^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 - 10,000 µg/kg</td>
<td>100 µL</td>
</tr>
<tr>
<td>1,000 - 20,000 µg/kg</td>
<td>50 µL</td>
</tr>
<tr>
<td>5,000 - 100,000 µg/kg</td>
<td>10 µL</td>
</tr>
<tr>
<td>25,000 - 500,000 µg/kg</td>
<td>100 µL of 1/50 dilution$^b$</td>
</tr>
</tbody>
</table>

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
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


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.

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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 Do all VOA samples need to be chemically preserved?

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. Table 1 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

<table>
<thead>
<tr>
<th>Sample Matrix</th>
<th>Preservative</th>
<th>Holding Time</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aqueous Samples With No Residual Chlorine Present</td>
<td>Cool to 4 ± 2°C.</td>
<td>7 days</td>
<td>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.</td>
</tr>
<tr>
<td>Aqueous Samples With No Residual Chlorine Present</td>
<td>Cool to 4 ± 2°C and adjust pH to less than 2 with HCl or solid NaHSO₄.</td>
<td>14 days¹</td>
<td>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.</td>
</tr>
<tr>
<td>Aqueous Samples With Residual Chlorine Present</td>
<td>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.</td>
<td>7 days</td>
<td>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.</td>
</tr>
<tr>
<td>Aqueous Samples With Residual Chlorine Present</td>
<td>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 NaHSO₄.</td>
<td>14 days¹</td>
<td>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.</td>
</tr>
<tr>
<td>Solid Samples²</td>
<td>Sample is extruded into an empty sealed vial and frozen on-site to &lt; -7°C.</td>
<td>14 days¹</td>
<td>Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.</td>
</tr>
<tr>
<td>Sample Matrix</td>
<td>Preservative</td>
<td>Holding Time$^1$</td>
<td>Comment</td>
</tr>
<tr>
<td>-----------------------</td>
<td>------------------------------------------------------------------------------</td>
<td>------------------</td>
<td>-----------------------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Solid Samples$^2$</td>
<td>Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C for no more than 48 hours then frozen to &lt; -7°C upon laboratory receipt.</td>
<td>14 days$^1$</td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>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.</td>
<td>14 days$^1$</td>
<td>Analysis must be completed within 48 hours if samples are not preserved with methanol prior to the expiration of the 48 hour period.</td>
</tr>
<tr>
<td></td>
<td>Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C.</td>
<td>48 hours</td>
<td>The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to &lt; -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.</td>
</tr>
<tr>
<td></td>
<td>Cool to 4 ± 2°C the coring tool used as a transport device</td>
<td>48 hours</td>
<td>The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to &lt; -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.</td>
</tr>
<tr>
<td></td>
<td>Freeze to &lt; -7°C the coring tool used as a transport device</td>
<td>48 hours</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample is extruded into a vial containing reagent water and frozen on-site to &lt; -7°C.</td>
<td>14 days$^1$</td>
<td>Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.</td>
</tr>
<tr>
<td></td>
<td>Sample is extruded into a vial containing reagent water and cooled to 4 ± 2°C for 48 hours or less then frozen to &lt; -7°C upon laboratory receipt.</td>
<td>14 days$^1$</td>
<td>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.</td>
</tr>
</tbody>
</table>
### Table A.1 (Continued)

<table>
<thead>
<tr>
<th>Sample Matrix</th>
<th>Preservative</th>
<th>Holding Time</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solid Samples$^2$</td>
<td>Sample is extruded into a vial containing reagent water and 1 g NaHSO$_4$ and cooled to 4 ± 2°C.</td>
<td>14 days$^1$</td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>Sample is extruded into a vial containing methanol and cooled to 4 ± 2°C.</td>
<td>14 days$^1$</td>
<td>Additional methanol extract storage time beyond 14 days may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.</td>
</tr>
</tbody>
</table>

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.
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 ± 2°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
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-cm³ volume of soil has to be collected (assuming the soil has density of 1.7 g/cm³). 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 available 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 cannot 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 cannot 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 \(4 ± 2\)°C for refrigerated aqueous and solid samples and \(< -7\)°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 PREPARATION

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 $4 \pm 2^\circ C$ for 48 hours or less, then preserve by freezing at $<-7^\circ C$ upon laboratory receipt

Upon laboratory receipt, replicate soil VOA vials are frozen ($<-7^\circ C$) then thawed as needed for preparation by methanol extraction, or if possible by vapor partitioning. Sample vials should not be frozen below $-20^\circ C$ 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 $4^\circ C$, 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 ($22^\circ C$) after 5 days of storage. (Refs. 1,2,4) When these soil types are stored at $4^\circ C$, 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 $4^\circ C$ 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 $4^\circ C$, cease when soil is frozen at $<-7^\circ C$. (Refs. 2,4).

A.8.2.3 Refrigerate VOA vials at $4 \pm 2^\circ C$ 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 $4 \pm 2^\circ C$. See Sec. A.8.2.2 above for discussion on initial 48-hour transport at $4 \pm 2^\circ C$. 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 NaHSO₄ 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 NaHSO₄ 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 NaHSO₄ for vapor partitioning by Method 5035. For a spiked soil, NaHSO₄ 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 NaHSO₄ was present. (Ref. 1) The studied chlorinated hydrocarbons demonstrated insignificant losses during these storage conditions.
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 $4 \pm 2^\circ C$ until laboratory receipt within 48 hours, and then preserved by freezing ($< -7^\circ C$). 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 $4 \pm 2^\circ C$ 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 ($< -7^\circ C$) 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 $4 \pm 2^\circ C$ until laboratory receipt within 48 hours, then extruded into individual “Empty VOA Vials” for preservation by freezing ($< -7^\circ C$) 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 NaHSO₄. The prepared VOA vial is maintained at $4 \pm 2^\circ C$ 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 $4 \pm 2^\circ C$. 

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


APPENDIX D

DETECTION LIMITS REQUIRED
**APPENDIX D**

**REQUIRED COMPOUND LIST AND DETECTION LIMITS**

**NAS JRB WILLOW GROVE, PENNSYLVANIA**

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<thead>
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<th>CHEMICAL</th>
<th>CAS</th>
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### APPENDIX D

**REQUIRED COMPOUND LIST AND DETECTION LIMITS**

**NAS JRB WILLOW GROVE, PENNSYLVANIA**

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<th>CAS</th>
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<th>PADEP MSC Residential Limit</th>
<th>REGION 3 RBC Residential Limit</th>
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### APPENDIX D
REQUURED COMPOUND LIST AND DETECTION LIMITS
NAS JRB WILLOW GROVE, PENNSYLVANIA

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<th>CAS</th>
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<th>PADEP MSC Residential Limit</th>
<th>REGION 3 RBC Residential Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>ENDOSULFAN II</td>
<td>115-29-7</td>
<td>3.3</td>
<td>1.3E+06</td>
<td>4.7E+05</td>
</tr>
<tr>
<td>DDD</td>
<td>72-54-8</td>
<td>3.3</td>
<td>7.5E+04</td>
<td>2.7E+03</td>
</tr>
<tr>
<td>ENDOSULFAN SULFATE</td>
<td>1031-07-8</td>
<td>3.3</td>
<td>1.3E+06</td>
<td>4.7E+05</td>
</tr>
<tr>
<td>DDT</td>
<td>50-29-3</td>
<td>3.3</td>
<td>5.3E+04</td>
<td>1.9E+03</td>
</tr>
<tr>
<td>METHOXYCHLOR</td>
<td>72-43-5</td>
<td>1.7</td>
<td>1.1E+06</td>
<td>3.9E+05</td>
</tr>
<tr>
<td>ENDRIN KETONE</td>
<td>72-20-8</td>
<td>3.3</td>
<td>6.6E+04</td>
<td>2.3E+04</td>
</tr>
<tr>
<td>ENDRIN ALDEHYDE</td>
<td>72-20-8</td>
<td>3.3</td>
<td>6.6E+04</td>
<td>2.3E+04</td>
</tr>
<tr>
<td>ALPHA-CHLORDANE</td>
<td>5103-71-9</td>
<td>1.7</td>
<td>5.1E+04</td>
<td>1.8E+03</td>
</tr>
<tr>
<td>GAMMA-CHLORDANE</td>
<td>5103-74-2</td>
<td>1.7</td>
<td>5.1E+04</td>
<td>1.8E+03</td>
</tr>
<tr>
<td>TOXAPHENE</td>
<td>8001-35-2</td>
<td>33</td>
<td>1.6E+04</td>
<td>5.8E+02</td>
</tr>
</tbody>
</table>

### PCBS

| AROCLOR-1016              | 12674-11-2| 33                        | 1.5E+04                     | 5.5E+03                       |
| AROCLOR-1221              | 11104-28-2| 33                        | 3.6E+04                     | 3.2E+02                       |
| AROCLOR-1232              | 11141-16-5| 33                        | 3.6E+04                     | 3.2E+02                       |
| AROCLOR-1242              | 53469-21-9| 33                        | 3.6E+04                     | 3.2E+02                       |
| AROCLOR-1248              | 12672-29-6| 33                        | 9.9E+03                     | 3.2E+02                       |
| AROCLOR-1254              | 11097-69-1| 33                        | 4.4E+03                     | 3.2E+02                       |
| AROCLOR-1260              | 11096-82-5| 33                        | 3.0E+04                     | 3.2E+02                       |

### Notes:
1. The value for hexavalent chromium was used.
2. The lead value for Region III RBC is the OSWER residential value.
3. The value for methylmercury was used as a surrogate for mercury.
4. The value for 1,3-dichloropropene was used as a surrogate for cis-1,3-dichloropropene and trans-1,3-dichloropropene.
5. The value for cis-1,2-dichloroethene was used as a surrogate for total 1,2-dichloroethene for the PADEP MSC value.
6. The value for acenaphthene was used as a surrogate for acenaphthylene.
7. The value for endosulfan was used as a surrogate.
8. The value for endrin was used as a surrogate.
9. The value for chlordane was used as a surrogate.

Yellow shaded cells indicate the most stringent of the PADEP MSC and Region 3 RBCs for direct contact with soil.
Pink shaded cells indicate the quantitation limit does not meet the most stringent criteria.

APPENDIX E

ELECTRONIC DATA DELIVERABLE FORMAT
ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to provide a compact disk (CD) containing separate text (TXT) files in the format specified in this Attachment. The electronic deliverable includes all environmental samples, sample dilutions, sample reanalyses, and laboratory quality control samples. All entries in the electronic deliverable must agree exactly with the final entries reported on the hardcopy data package sample result summaries. The LAB_RESULT for nondetects should be populated with sample quantitation limits. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Each CD is to be properly labeled with the laboratory name, project name, file name(s), and laboratory point of contact. Electronic files should be delivered in the same fashion, as are the hard copy data packages. A separate .txt file shall be made for each analytical fraction (by method) and each sample delivery group (SDG). The files shall be named with the first character being the analytical fraction designator, followed by an underscore, followed by the SDG name. For example, the file for the volatile fraction for SDG TT001 should be named V_TT001.TXT. Additionally, the laboratory must provide a hardcopy listing all electronic files saved to the CD, indicating what analytical fraction and matrix the file data contained therein pertain to. All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

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I, _______________________, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (______)____________ if there are any questions or problems with the enclosed electronic deliverables.

Signature: ______________________  Title: ______________________  Date: ____________

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The analytical data shall be delivered electronically in an ASCII comma delimited (double quotes around text fields) text file (filename.txt). The exact structure of the database is described in the table below. It shall be the responsibility of the laboratory to ensure that all electronic entries are in strict accordance with the information provided on the Form I.

An example database shall be sent for review prior to the first electronic deliverable in .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable shall be directed to Ricky DePaul at Tetra Tech NUS (412)921-7112.

<table>
<thead>
<tr>
<th>DATA FIELD</th>
<th>DATA TYPE</th>
<th>FIELD WIDTH</th>
<th>DATA FIELD DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE_NO</td>
<td>C</td>
<td>25</td>
<td>Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reanalyses, dilution, and matrix spike results in which a RE, DL, or MS suffix will be added to the sample number respectively.</td>
</tr>
<tr>
<td>MATRIX</td>
<td>C</td>
<td>2</td>
<td>Matrix as indicated on the Chain of Custody.</td>
</tr>
<tr>
<td>COLL_METH</td>
<td>C</td>
<td>2</td>
<td>&quot;G&quot; (Grab) or &quot;CP&quot; (Composite) as indicated on the Chain of Custody.</td>
</tr>
<tr>
<td>LAB_ID</td>
<td>C</td>
<td>15</td>
<td>Laboratory number for the given sample.</td>
</tr>
<tr>
<td>LABORATORY</td>
<td>C</td>
<td>25</td>
<td>Laboratory name.</td>
</tr>
<tr>
<td>BATCH_NO</td>
<td>C</td>
<td>10</td>
<td>Laboratory code for batch of samples included in a given run.</td>
</tr>
<tr>
<td>ASSOC_BLNK</td>
<td>C</td>
<td>15</td>
<td>Laboratory name of the method blank associated with that particular batch of samples.</td>
</tr>
<tr>
<td>QC_TYPE</td>
<td>C</td>
<td>10</td>
<td>Normal Environmental Sample = &quot;NORMAL&quot;, Laboratory Duplicate = &quot;DUPLICATE&quot;, Matrix Spike = &quot;MS&quot;, Matrix Spike Duplicate = &quot;MSD&quot;, Laboratory Control Sample = &quot;LCS&quot;, Laboratory Control Sample Duplicate = &quot;LCSD&quot;, Method Blank = &quot;M_BLANK&quot;, Preparation Blank = &quot;P_BLANK&quot;.</td>
</tr>
<tr>
<td>RUN_TYPE</td>
<td>C</td>
<td>12</td>
<td>Initial, dilution 1, dilution 2, dilution 3, reanalysis 1, reanalysis 2, reanalysis 3</td>
</tr>
<tr>
<td>RES_TYPE</td>
<td>C</td>
<td>5</td>
<td>Surrogate Recoveries = &quot;SUR&quot;, Target Compound = &quot;TRG&quot;, Internal standards = &quot;IS&quot;, Tentatively Identified Compounds = &quot;TIC&quot;</td>
</tr>
<tr>
<td>SAMP_DATE</td>
<td>D</td>
<td>8</td>
<td>Date of sample collection as indicated on the Chain of Custody. Example: 11/07/93</td>
</tr>
</tbody>
</table>

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<table>
<thead>
<tr>
<th>DATA FIELD</th>
<th>DATA TYPE</th>
<th>FIELD WIDTH</th>
<th>DATA FIELD DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMP_TIME</td>
<td>T</td>
<td>5</td>
<td>Time of sample collection as indicated on the Chain of Custody. Reported as five character string.</td>
</tr>
<tr>
<td>REC_DATE</td>
<td>D</td>
<td>8</td>
<td>Date sample was received by the laboratory.</td>
</tr>
<tr>
<td>EXTR_DATE</td>
<td>D</td>
<td>8</td>
<td>Date sample was extracted or prepared by the laboratory.</td>
</tr>
<tr>
<td>ANAL_DATE</td>
<td>D</td>
<td>8</td>
<td>Date sample was analyzed by the laboratory.</td>
</tr>
<tr>
<td>RUN_NUMBER</td>
<td>N</td>
<td>2 (0)</td>
<td>The number of the analytical run for a given sample in sequence. For example, if a sample is diluted and reanalyzed, the original run number would be 1 and the reanalysis would be 2.</td>
</tr>
<tr>
<td>SDG</td>
<td>C</td>
<td>15</td>
<td>Sample delivery group identifier assigned by the laboratory. This number should exactly match the SDG designated on the hardcopy data package.</td>
</tr>
<tr>
<td>PROJECT_NO</td>
<td>C</td>
<td>10</td>
<td>Identification of Project Number or Contract Task Order (CTO) number.</td>
</tr>
<tr>
<td>PROJ_MNGR</td>
<td>C</td>
<td>25</td>
<td>The Tetra Tech NUS Project Manager’s last name, followed by a comma, followed by the first initial of the Project Manager. Example: HUTSON, D.</td>
</tr>
<tr>
<td>PARAMETER</td>
<td>C</td>
<td>45</td>
<td>Chemical or analyte name exactly as reported on Form I.</td>
</tr>
<tr>
<td>CAS_NO</td>
<td>C</td>
<td>10</td>
<td>Chemical Abstract Service number for the parameter listed. The CAS number should be reported exactly as it is listed in publications such as the Merck Index. This field should be left blank for those parameters not having CAS numbers (e.g. Total Organic Carbon).</td>
</tr>
<tr>
<td>FRACTION</td>
<td>C</td>
<td>8</td>
<td>Metals = 'M', Volatiles = 'OV', Semivolatile/BNAs = 'OS', Pesticides = 'PEST', Herbicides = 'HERB', Polychlorinated Biphenyls = 'PCB', Explosives = 'EXP', Any petroleum hydrocarbon or fuel = 'TPH', Radionuclide = 'RAD', Miscellaneous = 'MISC', Dioxin/Furans = 'DIOX'</td>
</tr>
<tr>
<td>SORT</td>
<td>C</td>
<td>5</td>
<td>Leave this field blank. To be filled in by Tetra Tech NUS, Inc.</td>
</tr>
</tbody>
</table>

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LL
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<table>
<thead>
<tr>
<th>DATA FIELD</th>
<th>DATA TYPE</th>
<th>FIELD WIDTH</th>
<th>DATA FIELD DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANAL_METH</td>
<td>C</td>
<td>20</td>
<td>Analytical method used to quantitate parameter concentrations as listed in the laboratory technical specification. Example: 8270C for SW-846 Method 8270C.</td>
</tr>
<tr>
<td>LAB_RESULT</td>
<td>N</td>
<td>20</td>
<td>Reported value in units specified in the UNITS field containing the proper number of significant digits. Non-detects must be reported as sample quantitation limits (i.e. reporting limits adjusted for sample volume, percent moisture, and dilution factors as appropriate). The % Recovery for matrix spikes, laboratory control samples, and surrogates shall ALSO be placed in this field.</td>
</tr>
<tr>
<td>UNITS</td>
<td>C</td>
<td>5</td>
<td>The units of measure as reported on the Form I.</td>
</tr>
<tr>
<td>LAB_QUAL</td>
<td>C</td>
<td>2</td>
<td>The laboratory qualifier as reported on the Form I. For example, a 'U' qualifier should be used for all nondetected results.</td>
</tr>
<tr>
<td>IDL</td>
<td>N</td>
<td>15 (6)</td>
<td>Instrument detection limit in units specified in the UNITS field.</td>
</tr>
<tr>
<td>MDL</td>
<td>N</td>
<td>15 (6)</td>
<td>Method detection limit in units specified in the UNITS field and method specified in the METHOD field.</td>
</tr>
<tr>
<td>DIL_FACTOR</td>
<td>N</td>
<td>6 (1)</td>
<td>Dilution factor.</td>
</tr>
<tr>
<td>PCT_MOIST</td>
<td>N</td>
<td>5 (1)</td>
<td>Percent moisture for soil samples; blank for water samples.</td>
</tr>
<tr>
<td>RET_TIME</td>
<td>T</td>
<td>10</td>
<td>Retention time of analyte. Required for TICs. Format requested as HHHH:MM:SS (e.g. 1 day - 1 hr -10 min as 25:10:00)</td>
</tr>
<tr>
<td>COMMENTS</td>
<td>C</td>
<td>20</td>
<td>Analytical result qualifier or comment other than that listed in the LAB_QUAL field. Example: 'Reanalysis'.</td>
</tr>
</tbody>
</table>

C = Character string (everything shall be reported in capital letters)
N = Numeric string (decimal places are in parentheses in field width column)
D = Date (Ex: 010/07/04)
T = Time HHHH:MM:SS (e.g. 1 day - 1 hr -10 min as 25:10:00)