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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alphanumeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

<table>
<thead>
<tr>
<th>A or N</th>
<th>AAA</th>
<th>A or N</th>
</tr>
</thead>
<tbody>
<tr>
<td>3- or 4-Characters</td>
<td>2- or 3-Characters</td>
<td>3- to 6-Characters</td>
</tr>
<tr>
<td>Site Identifier</td>
<td>Sample Type</td>
<td>Sample Location</td>
</tr>
</tbody>
</table>

Tetra Tech NUS, Inc.
Additional segments may be added as needed. For example:

1. Soil and Sediment Sample ID

<table>
<thead>
<tr>
<th>A or N</th>
<th>AAA</th>
<th>A or N</th>
<th>NNNN</th>
</tr>
</thead>
<tbody>
<tr>
<td>3- or 4-Characters</td>
<td>2- or 3-Characters</td>
<td>3- to 6-Characters</td>
<td>4-Characters</td>
</tr>
<tr>
<td>Site Identifier</td>
<td>Sample Type</td>
<td>Sample Location</td>
<td>Sample Depth</td>
</tr>
</tbody>
</table>

2. Aqueous (groundwater or surface water) Sample ID

<table>
<thead>
<tr>
<th>A or N</th>
<th>AAA</th>
<th>A or N</th>
<th>NN</th>
<th>-A</th>
</tr>
</thead>
<tbody>
<tr>
<td>3- or 4-Characters</td>
<td>2- or 3-Characters</td>
<td>3- to 6-Characters</td>
<td>2-Characters</td>
<td>Filtered Sample only</td>
</tr>
<tr>
<td>Site Identifier</td>
<td>Sample Type</td>
<td>Sample Location</td>
<td>Round Number</td>
<td></td>
</tr>
</tbody>
</table>

3. Biota Sample ID

<table>
<thead>
<tr>
<th>A or N</th>
<th>AAA</th>
<th>A or N</th>
<th>AA</th>
<th>NNN</th>
</tr>
</thead>
<tbody>
<tr>
<td>3- or 4-Characters</td>
<td>2- or 3-Characters</td>
<td>3- to 6-Characters</td>
<td>2-Characters</td>
<td>3-Characters</td>
</tr>
<tr>
<td>Site Identifier</td>
<td>Sample Type</td>
<td>Sample Location</td>
<td>Species Identifier</td>
<td>Sample Group Number</td>
</tr>
</tbody>
</table>

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to
three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an ",-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SAMPLE NOMENCLATURE

| SG | Soil Gas Sample |
| SL | Sludge Sample   |
| SP | Seep Sample     |
| SS | Surface Soil Sample |
| ST | Storm Sewer Water Sample |
| SW | Surface Water Sample |
| TP | Test Pit Sample |
| TW | Temporary Well Sample |
| WC | Well Construction Material Sample |
| WP | Wipe Sample |
| WS | Waste/Solid Sample |
| WW | Wastewater Sample |

Sample Location - Examples of the location field are as follows:

- 001 - Monitoring Well 1
- N32E92 - Grid location 32 North and 92 East
- D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

- BC - Blue Crab
- GB - Blue Gill
- CO - Corn
- SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash ".F".
5.5 **Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature**

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

<table>
<thead>
<tr>
<th>AA</th>
<th>NNNNNN</th>
<th>NN</th>
<th>-F</th>
</tr>
</thead>
<tbody>
<tr>
<td>QC Type</td>
<td>Date</td>
<td>Sequence Number (per day)</td>
<td>Filtered (aqueous only, if needed)</td>
</tr>
</tbody>
</table>

The QC types are identified as:

- TB = Trip Blank
- RB = Rinsate Blank (Equipment Blank)
- FD = Field Duplicate
- AB = Ambient Conditions Blank
- WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 **Examples of Field QA/QC Sample Nomenclature**

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 **DEVIATIONS**

Any deviation from this SOP must be addressed in detail in the site specific planning documents.
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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.
Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request Form included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.
Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.
5.4 **Sample Tracking Forms**

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 **Chain-of-Custody Forms**

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of-Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 **Data Validation Letters**

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 **Historical Data**

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned
editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

- Database Record File
- PROJECT NUMBER: ____
- SITE NAME: ____
- DATE FILED: ___/___/
- SUMMARY OF CONTENTS ENCLOSED
- BOX ___ OF ___

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.
### ATTACHMENT A

**MIS REQUEST FORM**

<table>
<thead>
<tr>
<th>Project Name:</th>
<th>Request Date:</th>
<th>Date Data Available for Production:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Project Manager:</td>
<td>Requestor:</td>
<td>Request in Support of:</td>
</tr>
<tr>
<td>Requester:</td>
<td></td>
<td>Database Lead:</td>
</tr>
<tr>
<td>Program/Client:</td>
<td></td>
<td>GIS Lead:</td>
</tr>
<tr>
<td>State/EPA Region:</td>
<td></td>
<td>Statistics Lead:</td>
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<tr>
<th>Site Name(s) (Area, OU, etc.):</th>
<th>Sampling Date(s):</th>
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<table>
<thead>
<tr>
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<th>GW</th>
<th>SO</th>
<th>SD</th>
<th>SW</th>
<th>Other:</th>
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<table>
<thead>
<tr>
<th>Labels:</th>
<th>Total # of Samples</th>
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<tbody>
<tr>
<td>☐ Labels needed for an upcoming sampling event</td>
<td></td>
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<table>
<thead>
<tr>
<th>Estimated Hours</th>
<th>Additional Instructions:</th>
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</table>

<table>
<thead>
<tr>
<th>Due Date</th>
<th>Complete ETS Charge No.</th>
</tr>
</thead>
</table>

**Data Entry:**

<table>
<thead>
<tr>
<th>☐ Chemical data needs to be entered from hardcopy</th>
<th>Estimated # of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>☐ Chemical data needs to be formatted electronically</td>
<td></td>
</tr>
<tr>
<td>☐ Field analytical data needs to be entered from hardcopy</td>
<td></td>
</tr>
<tr>
<td>☐ Geologic data needs to be entered from hardcopy</td>
<td></td>
</tr>
<tr>
<td>☐ Hydrology data needs to be entered from hardcopy</td>
<td></td>
</tr>
</tbody>
</table>

<table>
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# STANDARD OPERATING PROCEDURES

**Subject**
DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES

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**Effective Date**
08/13/01

**Revision**
0

**Applicability**
Tetra Tech NUS, Inc.

**Prepared**
Risk Assessment Department

**Approved**
D. Senovich

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*019611/P*

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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
- 1,2-Dibromo-3-chloropropane
- 1,2-Dibromoethane
- Dibromomethane
- trans-1,4-Dichloro-2-butene
- Dichlorodifluoromethane
- trans-1,2-Dichloroethene
- 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 Vls 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 Vs. 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) >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
- Methapyriline
- 3-Methylcholanthrene
- Methyl methanesulfonate
- 3-Methylphenol
- 1,4-Naphthoquinone
- 1-Naphthylamine
- 2-Naphthylamine
- 5-Nitro-o-toluidine
- N-methyl-N-nitrosodimethylethylamine
- N-nitroso-di-n-butylamine
- N-nitrosomorpholine
- N-nitrosopiperidine
- Pentachlorobenzene
- Pentachloronitrobenzene
- Phenacatin
- 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 Vs. 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 IIs; 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 (DCP)

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
Bromoaceton
Bromobenzene
Bromodichloromethane
Bromofom
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 (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 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 (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.

2.2.6.7 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
Ronnel
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 IIs. 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°C (2°C) 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 (U), 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 (U) 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 which ever 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 where 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.
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1.0 CLP DFLM 1.1/SW-846 METHODS 8280/8290

1.1 Applicability

Methods 8280, 8290 and CLP SOW DFLM1.1 are applicable for the determination of the tetra-, penta-, hexa-, hepta-, and octachlorinated congeners of dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) (by Gas Chromatography/Mass Spectrometry (GC/MS) via selective ion monitoring) in chemical wastes including fuel oils, sludges, fly ash, still bottoms, reactor residues, soil, and water. Methods 8280 and DFLM1.1 are low resolution GC/MS techniques while Method 8290 is a high resolution GC/MS technique.

1.2 Dioxin Data Package Deliverable Minimum Requirements

The following information must be present in data package prior to the validation effort:

- Appropriate Chain-of-Custody (COC) Form(s)
- Laboratory Case Narrative documenting any particular analytical anomalies encountered and sample description information (i.e., sample cross-reference identifications)
- Calibration Summaries
- Laboratory Control Sample and Duplicate forms
- Single Control Samples and Method Blank Results
- Matrix Spike/Matrix Spike Duplicates
- Retention Time Marker Solutions
- Internal and Recovery Standard Area Summaries

The appropriate laboratory liaison must be contacted immediately if any of the above items have been omitted from the data package.

1.3 Technical Data Evaluation

**NOTE**: Analysis of a fortified standard and blank may be submitted as evidence of compliant Performance Evaluation (PE) analyses as per region-specific requirements. The fortified standard will contain 2,3,7,8-TCDD at a known quantity while the fortified blank will contain 1,2,3,4-TCDD plus other known interferents. The recovery for 2,3,7,8-TCDD recognition must be within the EPA’s 99% confidence interval.

1.4 Quality Control

1.4.1 Holding Times and Sample Preservation

All samples are to be extracted within 30 days of sample collection, and all subsequent analyses are to be conducted within 45 days from the date of collection. **NOTE**: Data qualification based upon holding time noncompliances is rare due to the minor effect of extended storage time on PCDD/PCDF quantitation resulting from the inherent persistence and known stability of these compounds. However, estimation of associated sample data based on holding time shall be subject to the professional judgment of the data validator.

Sample preservation shall be checked by referencing the appropriate Chain-of-Custody (COC) form(s) and verifying that all samples receiving PCDD/PCDF analysis were cooled to and stored at 4 °C.
1.4.2 Initial Calibration Verification

Review the average Relative Response Factors (RRFs) for all dioxin congeners by recalculating approximately 10% of the reported RRFs while also verifying proper use of quantitation ions. The following ions are specified for selective ion monitoring for PCDDs and PCDFs:

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Quantitation Ion</th>
<th>Confirmation Ions</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCDDs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetra</td>
<td>322</td>
<td>320</td>
</tr>
<tr>
<td>Penta</td>
<td>356</td>
<td>354; 358</td>
</tr>
<tr>
<td>Hexa</td>
<td>390</td>
<td>388; 392</td>
</tr>
<tr>
<td>Hepta</td>
<td>424</td>
<td>422; 426</td>
</tr>
<tr>
<td>Octa</td>
<td>460</td>
<td>458</td>
</tr>
<tr>
<td>PCDFs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetra</td>
<td>306</td>
<td>304</td>
</tr>
<tr>
<td>Penta</td>
<td>340</td>
<td>338; 342</td>
</tr>
<tr>
<td>Hexa</td>
<td>374</td>
<td>372; 376</td>
</tr>
<tr>
<td>Hepta</td>
<td>408</td>
<td>406; 410</td>
</tr>
<tr>
<td>Octa</td>
<td>444</td>
<td>442</td>
</tr>
</tbody>
</table>

**Internal Standards**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Quantitation Ion</th>
<th>Confirmation Ion</th>
</tr>
</thead>
<tbody>
<tr>
<td>13C12-2,3,7,8-TCDD</td>
<td>334</td>
<td>332</td>
</tr>
<tr>
<td>13C12-1,2,3,6,7,8-H,CDD</td>
<td>404</td>
<td>402</td>
</tr>
<tr>
<td>13C12-OCDD</td>
<td>472</td>
<td>470</td>
</tr>
<tr>
<td>13C12-2,3,7,8-TCDF</td>
<td>318</td>
<td>316</td>
</tr>
<tr>
<td>13C12-1,2,3,4,6,7,8-H,CDF</td>
<td>420</td>
<td>422</td>
</tr>
</tbody>
</table>

**Recovery Standards**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Quantitation Ion</th>
<th>Confirmation Ion</th>
</tr>
</thead>
<tbody>
<tr>
<td>13C12-1,2,3,4-TCDD</td>
<td>334</td>
<td>332</td>
</tr>
<tr>
<td>13C12-1,2,3,7,8,9-H,CDD</td>
<td>404</td>
<td>402</td>
</tr>
</tbody>
</table>

Next verify the acceptability of isotopic ratios as outlined in the following table:

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Selected Ions</th>
<th>Relative m/z</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCDDs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetra</td>
<td>320/322</td>
<td>0.65-0.89</td>
</tr>
<tr>
<td>Penta</td>
<td>356/358</td>
<td>1.24-1.86</td>
</tr>
<tr>
<td>Hexa</td>
<td>390/392</td>
<td>1.05-1.43</td>
</tr>
<tr>
<td>Hepta</td>
<td>424/426</td>
<td>0.88-1.20</td>
</tr>
<tr>
<td>Octa</td>
<td>458/460</td>
<td>0.76-1.02</td>
</tr>
<tr>
<td>PCDFs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetra</td>
<td>304/306</td>
<td>0.65-0.89</td>
</tr>
<tr>
<td>Penta</td>
<td>340/342</td>
<td>1.24-1.86</td>
</tr>
<tr>
<td>Hexa</td>
<td>374/376</td>
<td>1.05-1.43</td>
</tr>
<tr>
<td>Hepta</td>
<td>408/410</td>
<td>0.88-1.20</td>
</tr>
<tr>
<td>Octa</td>
<td>442/444</td>
<td>0.76-1.02</td>
</tr>
</tbody>
</table>
Internal Standards

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Selected Ions</th>
<th>Relative m/z</th>
</tr>
</thead>
<tbody>
<tr>
<td>13C12-2,3,7,8-TCDD</td>
<td>332/334</td>
<td>0.65-0.89</td>
</tr>
<tr>
<td>13C12-1,2,3,6,7,8-HxCDD</td>
<td>402/404</td>
<td>1.05-1.43</td>
</tr>
<tr>
<td>13C12-OCDD</td>
<td>470/472</td>
<td>0.76-1.02</td>
</tr>
<tr>
<td>13C12-2,3,7,8-TCDF</td>
<td>316/318</td>
<td>0.65-0.89</td>
</tr>
<tr>
<td>13C12-1,2,3,4,6,7,8-HxCDF</td>
<td>420/422</td>
<td>0.88-1.20</td>
</tr>
</tbody>
</table>

Recovery Standards

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Selected Ions</th>
<th>Relative m/z</th>
</tr>
</thead>
<tbody>
<tr>
<td>13C12-1,2,3,4-TCDD</td>
<td>332/334</td>
<td>0.65-0.89</td>
</tr>
<tr>
<td>13C12-1,2,3,7,8,9-HxCDD</td>
<td>402/404</td>
<td>1.05-1.43</td>
</tr>
</tbody>
</table>

Typically, the data reviewer can expect to associate the following congeners with their associated internal standards as follows:

- Internal Standard #1 (13C12-2,3,7,8-TCDD): TCDD, PeCDD
- Internal Standard #2 (13C12-1,2,3,6,7,8-HxCDD): HxCDD, HpCDD
- Internal Standard #3 (13C-OCDD): OCDD, OCDF
- Internal Standard #4 (13C12-TCDF): TCDF, PeCDF
- Internal Standard #5 (13C12-HpCDF): HxCDF, HpCDF

Additionally, verify that the Relative Standard Deviation (%RSD) for all target compounds and internal standards is 15%.

Actions - Qualify as estimated, (J) positive results and (U) nondetects in affected samples if RSD is >15%.

Window Defining Mix

This is a retention time check which must be run prior to the continuing calibration. The composition of the window defining mix may or may not be known. Review the following criteria:

- Peak separation must be 25% valley criterion for TCDD isomers
- Peak separation must be the 50% valley criterion for HxCDD isomers
- Multiple ion detection mass chromatograms and reconstructed ion chromatograms should be present for the window defining mix

Actions - Professional judgment (weighted primarily upon chromatographic expertise) must be employed when assigning data qualifications.

1.4.3 Continuing Calibration Verification

Evaluation of the CCV involves evaluating the Daily Standard (which is a standard that contains the required target compounds plus internal standards), versus the initial standard.

Verify that a Continuing Calibration Verification (CCV) was analyzed prior to sample analysis and at the beginning of each subsequent 12-hour period. A CCV must also be analyzed at the end of the final analysis period.
The Signal-to-Noise ratio (S/N) for all internal standards must be >10:1. No quality control criteria exist to govern internal standard recovery; however, internal standard advisory recovery limits of 40-120% were established in earlier EPA validation protocol.

Verify that the internal standard area count in the sample is -50% to +100% of the internal standard area count in the associated daily standard.

Complete one Percent Recovery (%RRF) calculation for an internal standard as outlined in equation A below:

\[
\text{Equation A: } \%\text{RRF} = \left( \frac{A_{\text{is}}}{A_{\text{rs}}} \right) \left( \frac{Q_{\text{is}}}{Q_{\text{rs}}} \right) \times 100
\]

where:
- \( A_{\text{is}} \) = area of the quantitation ion of the internal standard
- \( A_{\text{rs}} \) = area of the quantitation ion of the recovery standard
- \( Q_{\text{is}} \) = ng of internal standard
- \( Q_{\text{rs}} \) = ng of recovery standard
- \( \text{RRF}_{\text{is}} \) = Relative Response Factor for the internal standard as determined from the associated continuing calibration

An RRF shall be calculated for each congener in the CCV solution. A Percent Difference (%D) of 30% from the average RRF must be accomplished for the CCV. **NOTE**: Recalculate some (approximately 10%) of the continuing calibration RRFs for thoroughness.

Actions - Qualify associated sample data as estimated, i.e., (J) positive results and (UJ) nondetects in affected samples in instances where CCV %D >30%. Qualify as rejected (R) all associated sample data in instances where the internal standard S/N ratio <10:1.

### 1.4.4 Laboratory Method Blank Evaluations

Verify that a laboratory generated method blank was analyzed prior to sample analysis and for each matrix and extraction batch for all samples within an SDG. The laboratory method blanks should be free from contamination and/or interferences stemming from glassware involved in sample preparation and subsequent analytical procedures, associated reagents and solvents, etc. The following criteria shall be employed for evaluation of contaminant levels present in laboratory method blanks:

- The signal of any confirmed analyte present in a method blank must be <2% of the signal of the associated internal standard (based on peak height or peak area). Comparison of contaminants present in the blanks at levels below the calibration range (i.e., contaminants present at levels which constitute <2% of the respective internal standard) shall not require reanalyses as stipulated by the method.
- An action level of 5X the maximum contaminant level shall be used in instances of positive detections.
- The data reviewer should complete a detection limit verification calculation.
- Detection limits are sample-specific dependent upon the concentration of a given analyte to produce a signal with a peak height 2.5 X the background signal.
- The data reviewer shall consider all applicable sample weight, moisture content, and dilution factors prior to application of the aforementioned action level.
The data reviewer shall recalculate at least one Detection Limit (DL) using equation B as follows:

\[
DL = \frac{(2.5) (H_x) (Q_{is})}{(A_{is}) (RRF_A) (W)}
\]

where:
- \(A_{is}\) = area of the quantitation ion of the internal standard
- \(Q_{is}\) = ng of internal standard
- \(H_x\) = peak height of noise for the analyte's quantitation ion
- \(RRF_{is}\) = Relative Response Factor for the analyte as determined from the associated continuing calibration
- \(W\) = dry weight of the sample (g)

Actions - Effects on sample data and subsequent data qualifications shall be upon the professional judgment of the data reviewer, but the following general qualifying guidance shall be employed; Qualify as nondetected (U) any positive result less than the corresponding action level.

### 1.4.5 Duplicate Control Samples

The Duplicate Control Sample (DCS) is a well-characterized matrix which is spiked and analyzed at approximately 10% of the sample load in order to establish method-specific quality control limits. The DCS spike recovery quality control limits of 60-140% shall be employed. Additionally, the RPDs between control sample and duplicate shall be below 50%.

Actions - Qualify as estimated (J) positive results in affected samples when DCS spike recoveries are >140%. Qualify as estimated (J) positive results and (UJ) nondetects in affected samples when DCS spike recoveries are <60%. Qualify as estimated (J) positive results and (UJ) nondetects in affected samples when %RPD between control and duplicate sample exceeds 50%.

### 1.4.6 Matrix Spike/Matrix Spike Duplicate Review

Verify that a matrix spike has been analyzed for each matrix and batch of samples within an SDG.

Verify that the %RSD between matrix spike and duplicate injections is 50%. Additionally, the following recovery limits shall be employed for the respective congeners:

<table>
<thead>
<tr>
<th>Congener</th>
<th>Recovery Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCDD</td>
<td>50-150%</td>
</tr>
<tr>
<td>PCDD</td>
<td>50-150%</td>
</tr>
<tr>
<td>HxCDD</td>
<td>50-150%</td>
</tr>
<tr>
<td>HpCDD</td>
<td>50-150%</td>
</tr>
<tr>
<td>OCDD</td>
<td>50-150%</td>
</tr>
<tr>
<td>TCDF</td>
<td>50-150%</td>
</tr>
<tr>
<td>PeCDF</td>
<td>50-150%</td>
</tr>
<tr>
<td>HxCDF</td>
<td>50-150%</td>
</tr>
<tr>
<td>HpCDF</td>
<td>50-150%</td>
</tr>
<tr>
<td>OCDF</td>
<td>50-150%</td>
</tr>
</tbody>
</table>

Actions - Qualify as estimated (J) only positive results in affected samples when the recovery exceeds the upper quality control limit. Qualify as estimated, (J) positive results and (UJ) nondetects in affected samples when the recovery is below the lower quality control limit.
1.4.7 Chromatographic Performance and Evaluation

Verify that the recovery standard area counts are within -50% to +100% of the area counts in the respective daily check standard.

Examine chromatographic acceptability by checking the chromatographic base-line for fluctuation (i.e., raising or lowering), peak shape and resolution. Proper peak resolution between 13C-2,3,7,8-TCDD and 13C-1,2,3,4-TCDD (or 13C-2,3,7,8-TCDD and its closest eluting isomer), shall be attained at a threshold acceptability level of <25%.

Actions - Data qualification shall be based upon the professional judgment of the data reviewer.

1.4.8 Sample Quantitation

Confirm the quantitation of at least one Estimated Maximum Positive Concentration (EMPC). The laboratory will report an EMPC as opposed to a confirmed, definite positive hit in instances where the S/N 2.5 for both the quantitation ion and confirmation ion for a given target isomer/analyte. The following equation shall be used to verify at least one EMPC calculation:

\[
EMPC = \frac{(A_x)(Q_{is})}{(A_{is})(RRF_A)(W)}
\]

where: \(A_x\) = area of the quantitation or confirmation ion, whichever is lower
\(Q_{is}, A_{is}, RRF_A,\) and \(W\) are defined in the previous equation.

The data reviewer will also confirm at least one positive detection using the following equation:

\[
C_A = \frac{(A_A)(Q_{is})}{(A_{is})(RRF_A)(W)}
\]

where: \(A_{is}, Q_{is}, RRF_A,\) and \(W\) are defined in previous equations
\(C_A = \) analyte concentration (ng/g or ug/kg)
\(A_A = \) analyte quantitation ion area

NOTE: EMPC values are estimates by definition. If these values are used for risk assessment, it must be understood that an EMPC value is "less certain" that positive results which are qualified (J), since the qualified results meet identification criteria while EMPCs do not.

1.5 Deliverables

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided 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, and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.
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<td>3</td>
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<tr>
<td>3.0 GLOSSARY</td>
<td>3</td>
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<td>4.0 RESPONSIBILITIES</td>
<td>4</td>
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<tr>
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1.0 PURPOSE

The purpose of this guideline is to provide a general description of, and technical management guidance on, the use of Resistivity and Electromagnetic Induction (Ground Conductivity) surveys during hazardous waste site investigations.

2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability, and implementability of geophysical methods used during hazardous waste site investigations to determine subsurface resistivity or conductivity. Measurements of subsurface conductivity or resistivity can be used to determine the presence and approximate extent of subsurface contaminants, buried drums, and metal containers. In addition, the depth to the water table, and structural characteristics of the subsurface environment can be determined.

This document is intended to help develop a sufficient understanding of each method to assist in proper work plan development and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation. The highly specialized nature of these methods requires inclusion of project-specific, site-specific, and subcontractorspecific information prior to development of detailed operating procedures. Specialized expertise during both planning and execution of these geophysical methods is also required.

The description focuses on methods and equipment that are readily available and typically applied; it is not intended to provide a complete discussion of the state of the art.

3.0 GLOSSARY

Apparent Conductivity - The quantity measured during an electromagnetic induction survey; proportional to the actual conductivities of subsurface materials.

Apparent Resistivity - The quantity actually deduced during a resistivity survey; proportional to the actual resistivities of subsurface materials.

Conductivity - Intrinsic property of a substance, equal to the reciprocal of resistivity.

Current - The quantity of charge transmitted per unit time.

Electromagnetic (EM) Induction 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.

Potential - Intrinsic property of electrical fields, equating to the ability to do work. A potential field can induce a potential difference (voltage) between two electrodes.

Resistivity - Intrinsic property of a substance, equal to the resistance of a body multiplied by its cross-sectional area and divided by its length.

Resistivity Survey - A geophysical exploration method whereby an electrical current is transmitted into the ground and the resultant potential field is measured to deduce the apparent subsurface resistivity.
4.0 RESPONSIBILITIES

Project Manager - responsible for the scoping of geophysical surveys during development of the Work Plan, with the help of the Field Operations Leader, site geologist, and site geophysicist.

Field Operations Leader (FOL) – Responsible for overall management and coordination of the field work.

Site Geophysicist - as a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of these techniques for providing necessary data. Field work for these surveys is supervised by the site geophysicist.

5.0 PROCEDURES

5.1 Electromagnetics

The electromagnetic induction (EM) method provides a means of measuring the electrical conductivity of subsurface soil, rock, and groundwater. Electrical conductivity is a function of the type of soil and rock, its porosity, its permeability, and the fluid composition and saturation. In most cases the conductivity of the pore fluids will be responsible for the measurement. Accordingly, the EM method applies both to assessment of natural geohydrologic conditions and to mapping of many types of contaminant plumes. In addition, trench boundaries, buried wastes, drums, and utility lines can be located with EM techniques.

5.1.1 Applicability

Although EM is not a definitive technique, it is useful for several reasons. First, an EM survey can be conducted over an entire site very quickly. In addition, EM methods are generally inexpensive, even for coverage of large areas. Often, 100 acres or more may be surveyed in just a few days time (depending on desired detail). More importantly, EM data can be used to focus the more expensive phases of an investigative project, potentially resulting in a large cost savings. For example, rather than drilling several dozen monitoring wells while searching for groundwater contamination, an EM conductivity unit may be used to survey for a conductive (or resistive) plume. Several EM survey lines may be run to provide definition of the plume and an indication of its source area, reducing the number of exploratory wells required and potentially resulting in better well placement providing a potentially significant cost savings. Another reason why EM should be considered is to fill in data gaps and to reduce the risk of missing a facet of the investigation, such as the presence of undetected refuse trenches, buried drums, or changing hydrologic conditions.

Electromagnetic methods may be used in many situations for a variety of purposes. The following list includes major uses related to investigations of hazardous waste sites:

- Defining the location of a contaminant plume [This could lead to the identification of downgradient receptors, source areas, and flow directions if the conductivity of the plume (target) is distinct in comparison to the host (background), hydrogeologic setting.]

- Locating buried metal objects (e.g., drums, tanks, pipelines, cables, monitoring wells).

- Addressing the presence or location of bedrock fault/fracture systems (This is important for identification of groundwater preferential pathways in bedrock.)

- Mapping grain size distributions in unconsolidated sediments.
• Mapping buried trenches and contaminated fill materials.

• Mapping saltwater intrusion.

• Defining lithological (unit) boundaries.

• Determining the rate of plume movements by conducting multiple surveys over time.

The above list is only partial; in fact, EM methods may be used wherever a significant change in conductance can be measured. EM should be considered for use when a suspected target is anticipated to have a conductivity significantly different from background values. Factors such as cost, site-specific conditions, and equipment availability should also be evaluated before deciding to proceed with an EM survey.

5.1.2 General

5.1.2.1 Objectives

The site geophysicist should evaluate the objectives of the site investigation in light of EM capabilities. If the purpose of the site investigation is to confirm the presence of contaminants with minimal effort, EM methods may provide too much detail and no direct evidence. Direct methods, on the other hand, such as installing monitoring wells with limited sampling, may be more suitable. If a site is to be characterized in detail and if assessment of hydrogeologic conditions and identification of all source areas, plumes, and receptors are a priority, then EM (and other geophysical methods) may be a more cost-effective way of selecting strategic locations for monitoring wells, directing test pit operations, efficiently selecting sampling points, and providing information between site sampling points.

5.1.2.2 Existing Data

If EM equipment is identified as a viable alternative for providing the type of information desired, the user should further evaluate the equipment to determine whether it is appropriate for use under the conditions found at a particular site. Evaluation of existing data can identify problems that may be encountered in the field:

• Variations in hydrogeologic conditions (e.g., varied water table conditions or changes in rock or sediment) can result in a conductivity range that envelopes the response of the target (e.g., plume) and effectively masks or blocks out any signals.

• Scattered, near-surface metal may mask buried targets such as drums or trenches.

• Anthropogenic features such as overhead powerlines, buried metallic pipes, etc., may decrease the signal to noise ratio making the technique ineffective.

An analysis of the site history might more closely define a survey area, thereby cutting survey costs by reducing the size of the survey. Deep targets may be out of the penetration range for many EM units, and specialized equipment may be required. It is difficult for EM systems to detect a groundwater contaminant plume through 100 feet of unsaturated overburden. A site reconnaissance should be conducted to identify other site conditions that may affect the data. Drastic topography changes can affect the quality of EM data obtained with some systems, and this possibility should be considered at each site.
5.1.3 Survey Design

Once the EM survey objectives have been defined, existing information has been reviewed, and reconnaissance of the site has been conducted, attention should be given to the design of the geophysical survey. The detail required of an EM survey is a primary factor in designing and planning fieldwork. If the purpose of performing EM work on site is to define a large geologic feature, then a grid using a wide (100- to 1,000-foot) line spacing may be needed. Some instruments are capable of providing a continuous data profile, which makes it less likely to miss small conductors than the typical discrete measurement EM instruments. The importance of designing and implementing a grid system tied into existing "permanent" features (such as roads and buildings) cannot be overstated. This permanent feature will allow the grid to be re-established in the field at a later time in order to place drill holes and monitoring wells. Furthermore, additional surveys may be conducted on the site over time using other geophysical techniques or the same technique to provide an indication of plume movement. These surveys will help in orienting maps and diagrams that are produced later and in defining targets.

5.1.3.1 Background Noise

Background noise can be a significant factor in the success of an EM survey. Evaluation of existing data and a site reconnaissance will help to identify the probable background noise level. A high noise level can make interpretation difficult and may actually cause an anomaly to be overlooked. It is difficult to delineate a conductive contaminant plume contained in overburden that has a wide natural variation in conductivity.

Noise can be divided into two groups: (1) natural, such as changing grain size distributions, steeply dipping strata, undetected mafic dikes, karstic topography, unexpected fault zones; and (2) cultural, such as power lines, houses, railroads, surface metal debris, cars, radio transmission towers, and other metal objects which are not intended to be located by the survey. Some instruments are more sensitive to certain types of noise sources than others. Because there is little published information on this subject, appropriate experience is essential.

5.1.3.2 Limitations

All EM instruments have varying limitations with regard to sensitivity and penetration. Published references, operator’s manuals, and field experience should be used to evaluate instrumentation versus capability. Table 1 lists several commercially available instruments along with operator requirements and productivity estimates.

5.1.3.3 Instrumentation

Table 2 provides guidance for EM equipment selection. These instruments may not be suitable to specific site conditions and investigation objectives. The decision to use a specific instrument is dependent upon site factors.

Electromagnetic techniques have also been adapted for downhole applications. These techniques can be useful in defining the vertical extent of a contaminant zone. Some systems work inside polyvinyl chloride (PVC) or Teflon monitoring well casings (see SOP GH-3.5, Borehole Geophysical Surveys).
TABLE 1

COMMON EM AND RESISTIVITY EQUIPMENT

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Manufacturer</th>
<th>Minimum No. of Operators</th>
<th>Typical Daily Line Miles (50-ft readings)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>EM-16-R</td>
<td>Geonics</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>EM-38</td>
<td>Geonics</td>
<td>1</td>
<td>3 - 4</td>
<td>2</td>
</tr>
<tr>
<td>EM-31-D</td>
<td>Geonics</td>
<td>1</td>
<td>3 - 4</td>
<td>2</td>
</tr>
<tr>
<td>EM-34-3</td>
<td>Geonics</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>PROTEM47P</td>
<td>Geonics</td>
<td>2</td>
<td>0.25</td>
<td>1</td>
</tr>
<tr>
<td>EM-61</td>
<td>Geonics</td>
<td>1</td>
<td>3 - 4</td>
<td>3</td>
</tr>
<tr>
<td>T-VLF</td>
<td>Iris Inst.</td>
<td>1</td>
<td>3 - 4</td>
<td>2</td>
</tr>
<tr>
<td>CEM</td>
<td>Crone</td>
<td>2</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Max Min II</td>
<td>Apex</td>
<td>2</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Syscal Resistivity Meter</td>
<td>Iris Inst.</td>
<td>2</td>
<td>0.5</td>
<td>2</td>
</tr>
</tbody>
</table>

Notes:
1. Primarily useful for geologic features only.
2. Useful for geologic and cultural features.
3. Primarily useful for mapping buried metals.

Designations such as EM-31 or EM-61 are the manufacturer’s model numbers and do not imply equipment complexity or capability.
<table>
<thead>
<tr>
<th>Application</th>
<th>VLF</th>
<th>VLF Resistivity</th>
<th>Frequency Domain EM</th>
<th>Time Domain EM Soundings</th>
<th>Time Domain EM Metal Detection</th>
<th>Resistivity Sounding</th>
<th>Resistivity Profiling</th>
<th>Azimuthal Resistivity Surveying</th>
</tr>
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<tbody>
<tr>
<td>Archeological Studies</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Locate Single Buried Steel Drum</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Locate a Cluster of Buried Steel Drums</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Delineate a Landfill</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Delineate Contaminated Soil and Fill</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Map Groundwater Contamination</td>
<td>X</td>
<td></td>
<td>X</td>
<td></td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Locate Metallic Pipelines and Utilities</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Delineate USTs</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td>X</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Map Stratigraphy</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Map Joints and Faults</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</table>

Notes:  
- X: Sometimes applicable but other techniques may be more cost effective  
- X: Often Applicable and cost effective  
- Not applicable
5.2  Electrical Resistivity

Electrical resistivity surveys provide information about the subsurface distribution of the ground resistivity. The information can be used to infer groundwater quality, lithologic, and geologic information. Both horizontal and vertical changes in ground resistivity can be mapped by resistivity surveys. In practice, resistivity surveys are mostly used to determine the vertical resistivity changes. Lateral resistivity changes are more easily mapped by electromagnetic surveys.

5.2.1  Applicability

Electrical resistivity (ER) data are subject to interpretation; therefore, ER field results should be checked periodically and confirmed by direct methods, such as sampling or drilling.

Although ER is not a definitive technique, the data are useful for several reasons. Typical productivity with conventional resistivity equipment is several thousand line-feet per day. This high productivity rate allows a large amount of useful data to be collected in a relatively short period of time. For example, rather than drilling several dozen monitoring wells or test borings to develop a complete picture of the site stratigraphy and structure, a few wells can be drilled (for control) and information about the rest of the site can be obtained by using resistivity methods. Method integration such as this can reduce the amount of time and the costs required for a project.

Resistivity methods may be used in a wide array of situations and for a variety of purposes. The following is a partial list of major uses related to investigations of hazardous waste sites:

- Definition of a contaminant plume. (This could lead to the identification of downgradient receptors and source areas.)
- Waste pit delineation.
- Definition of bedrock fault/fracture systems.
- Water table mapping (for contour maps).
- Stratigraphic mapping of soil layers (particularly useful in overburden, discriminating clays from sands and establishing their thicknesses).
- Defining bedrock topography (valleys).

Resistivity methods may be used whenever the feature to be mapped has a contrasting resistivity with the background material.

5.2.2  General

Electrodes are typically arranged in one of several patterns, called electrode arrays, depending on the desired information. Electrical resistivity techniques can determine the vertical subsurface resistivity distribution beneath a point. In this type of survey, called vertical electrical soundings, the electrode array is expanded systematically and symmetrically about a point. For each set of electrode spacings, apparent resistivity is determined from measurements of potential and input current. The resultant plot of apparent resistivity versus electrode spacing is interpreted to provide the subsurface resistivity with depth distribution at that one particular point. Examples of three common arrays are given in Figure 1. The Wenner and Schlumberger arrays are somewhat more common than the Dipole-Dipole and other arrays.
These arrays (Wenner, Schlumberger) start with a small electrode spacing that is increased to permit deeper penetration for sounding.

The manner in which the apparent resistivity changes with the electrode separation can be used to determine formation conductivity and layer thickness. To increase accuracy, the user should evaluate the interpretation of resistivity data against the existing subsurface information. With any set of apparent resistivity readings, a number of solutions are possible, so existing data must be used to select the one that fits best. A formation resistivity may be assigned, but without geological control the material is not known. Resistivity electrode arrays can also be used with constant inner-electrode spacing to develop a lateral picture of the site through profiles. Stratigraphic control is even more important when mapping lateral changes with constant electrode spacings, because layer thickness changes alone can cause changes in apparent resistivity. The desired resolution is a major factor in deciding how closely to space measurements for a given survey.

In practical application, a resistivity survey target (such as a plume or clay lens) should have a resistivity contrast (positive or negative) of 20 percent from background. This change in resistivity should be 50 percent or more to provide proper detection and delineation. For example, if a resistivity survey were conducted to delineate a groundwater contaminant plume (in overburden) with a resistivity of 200 ohm-meters, a background, saturated overburden resistivity of over 400 ohm-meters (for a conductive plume) or under 100 ohm-meters (for a resistive plume) would probably allow detection of the plume, providing other factors (such as depth) are not detrimental.

When depth sounding, resolution of individual layers has an accuracy generally around 20 percent; accuracy can be substantially more or less depending on the site conditions and operator expertise. Vertical resistivity sounding is usually less accurate than seismic refraction work, which is often conducted within a 10 percent error tolerance. However, geologic units may be distinguishable (by geophysics) only with the use of resistivity methods at some sites.

5.2.3 Survey Design

Data can be collected at randomly located stations or along survey lines. If vertical electrical soundings are performed to obtain resistivity changes with depth, then the soundings are positioned where the information is most useful. If measurements are made to map lateral resistivity changes, then the survey is best performed on a grid or on survey lines. The station spacing will be determined from the target size.

5.2.3.1 Background Noise

Evaluation of existing data and a site reconnaissance will help to identify the possible background noise level. A background high noise level can make interpretation difficult and may mask an anomaly. It would be difficult to delineate a slightly conductive contaminant plume contained in overburden that has wide natural variations in conductivity. Noise can be divided into two groups: natural, such as discontinuous clay layers, undetected mafic dikes, karstic topography, unexpected fault zones, variable water table, and lightning; and cultural, such as power lines, railroad tracks, and radio transmission towers. Since there is little published information on instrument noise sensitivity, appropriate experience is essential.
FIGURE 1
EXAMPLES OF COMMON ARRAYS

WENNER ARRAY

SCHLUMBERGER ARRAY

DIPOL-DIPOLE ARRAY

Electrode Number
PE Potential Electrode
CE Current Electrode
Voltmeter

5.2.3.2 Depth of Investigation

As a rule of thumb when a lateral resistivity survey is being conducted, the array should be spaced four or five times the distance from the ground surface down to the desired target. For vertical sounding, this suggested spacing should be about ten times the anticipated target depth. These suggestions should be used only as general guidance.

5.2.4 Miscellaneous Considerations

5.2.4.1 Instrumentation

For most shallow work at hazardous waste sites, most resistivity systems will suffice. Generally, equipment capability becomes important only when the desired investigative depth exceeds 70 to 100 feet. Larger power sources are needed to provide a measurable electrical potential with a wider electrode spacing. Some newer resistivity units are capable of electronic data storage, and other features. Often, the peripheral capabilities of an ER system may be the deciding factor when purchase is considered.

Borehole resistivity equipment has been used (in fluid filled unsealed boreholes) to determine relative formation porosity and other factors. For more information on this equipment, the reader should refer to the borehole geophysics subsection of this compendium.

5.2.4.2 Calibration

ER equipment requires calibration, either in the field or in the laboratory; dated records of this calibration should be kept in the equipment management file and in the appropriate project file. Calibration is used to establish the reliability and accuracy of the equipment; calibration typically includes an internal circuit check or actual field trials (e.g., tests over a known target). Equipment that historically exhibits fluctuations in calibration should not be used. The equipment serial number should be recorded on the calibration records. If the manufacturer recalls equipment, this fact should be explained and documented for instrument maintenance in the proper file. The current source and potentiometer must be calibrated on any type of resistivity equipment. The instrument’s current source may be calibrated by placing a reference ammeter in series with the electrode cables. The reading obtained on the reference ammeter is compared with the value read from the instrument’s current source ammeter. The current source ammeter is then adjusted accordingly.

The potentiometer is calibrated by either of two methods. The preferred field method, which is similar to the calibration of the current source, is done by comparing the instrument’s indicated potential to that potential measured with an independent voltmeter. An alternative means of calibration, which can be performed in the laboratory, involves placing a precision resistor of a known value in series with the current load. A potentiometer is then placed across the resistor. The potential measured should be equal to the product of the known resistance and indicated current.

5.2.4.3 Data Reduction

Direct current (DC) and low frequency alternating current (AC) resistivity meters measure two values:

(1) the amount of current injected into the subsurface via the current and sink electrodes; and,
(2) the potential (voltage) between two or more electrodes that are separated a known distance apart (a-spacing).
The resistance (ohms) can be calculated from these known values using Ohm's law.

Ohm's law states the following:

\[ v = ir \text{ or rearranged: } r = v/i \]

where:

\[ v \] voltage (volts)
\[ i \] current (amperes)
\[ r \] resistance (ohms)

Resistivity is a measurement of resistance across a distance and over a cross-sectional area. Resistivity is calculated by the following equation that assumes that the resistor is a rectangular block:

\[ R = ra/L \]

where:

\[ R \] resistivity (commonly expressed in units of ohm-meters or ohm-feet)
\[ r \] resistance (v/i, in units of ohms)
\[ a \] cross-sectional area of the resistor
\[ L \] length of the resistor

When calculating resistivity in the three-dimensional earth, the resistor becomes hemispherical. Ohm's law becomes the following:

\[ R = \left( r * 2\pi d^2 \right)/d \text{ or } R = \left( r * 2\pi d \right) \]

where:

\[ r \] resistance (ohms)
\[ 2\pi d^2 \] surface area of a hemisphere
\[ d \] diameter of sphere

The value for the diameter of the sphere is analogous to the distance between the potential electrodes. This distance is referred to as the "a-spacing" when using the Wenner resistivity array and is measured in units of meters or feet.

Apparent resistivity (\(\rho\)) is determined from a single measurement because the earth is heterogeneous, and horizontal layers of earth act as a circuit with resistors in parallel. True resistivity is found by performing a resistivity sounding. Resistivity soundings are produced by determining the apparent resistivity across successively increasing electrode separation distances. The results of the resistivity soundings are plotted on semi-log paper. Apparent resistivity values are plotted on a linear scale and the potential electrode separation distance is plotted on a log scale for the Wenner array. The resulting curve is then matched to a master set of curves to determine true resistivity. Alternatively, a mathematical model can be used to determine the true resistivity of each layer that is within the depth of investigation of the instrument.
Most modern resistivity meters contain a data logger that records voltage, amperes, and a-spacing values. These resistivity meters often contain processors that calculate apparent resistivity (ρ) and monitor levels of signal noise that are due to anthropogenic features or changes in geology across the measurement area. Apparent resistivity values calculated with these instruments should also be spot checked to ensure the data quality.

Several forward and inverse modeling software packages (e.g., RESIX™) exist that can be used to calculate the true resistivity of the subsurface layers. These computer models require some knowledge of the geology of the site where data collection occurs. Electric log records or estimates of the corresponding geologic layers' electrical resistivity are also required for calibration of the computer model. Typical resistivity values for various types of soil and rock are published in most geophysical textbooks.

6.0 REFERENCES

6.1 Electromagnetic Induction

6.1.1 Electromagnetic (EM) Theory and Interpretation Textbooks


6.1.2 EM General Manuals


6.1.3 Manufacturers

Aerodat Limited
3883 Nashua Drive
Mississauga, Ontario L5V 1R3
416/671-2446 (airborne EM systems)

Phoenix Geophysics Limited
200 Yorkland Boulevard
Willowdale, Ontario M2J 1R5
416/493-6350 (surface EM systems)

Crone Geophysics Limited
3607 Wolfedale Road
Mississauga, Ontario L5C 1V8
416/270-0096 (surface EM systems)

Scintrex
222 Snidercroft Road
Concord, Ontario L4K 1B5
416/669-2280 (surface EM systems)

Geonics Limited
1745 Meyerside Drive
Mississauga, Ontario L5T 1C5
416/676-9580 (borehole and surface EM systems)
6.2 Electrical Resistivity

6.2.1 Electrical Resistivity (ER) Theory and Interpretation Textbooks


6.2.2 Journals


6.2.3 ER General Manuals


Technos, Incorporated. *Application Guidelines for Selected Contemporary Techniques for Subsurface Investigations.* (No publication date given.)

6.2.4 ER Case Histories and Examples Journals


6.2.5 **Manufacturers**

ABEM-Atlas Copco
Distributed by Geotronic Corp.
10317 McKalla Place
Austin, Texas 78758

Phoenix Geophysics Limited
200 Yorkland Boulevard
Willowdale, Ontario M2J 1R5

Bison Instruments, Inc.
570-8 West 36th Street
Minneapolis, Minnesota 55416

Scintrex Limited
222 Snidercroft Road
Concord (Toronto), Ontario L4K 1B5

BRGM-Syscal
Distributed by EDA Instruments
5151 Ward Road
Wheat Ridge, Colorado 80033

7.0 **RECORDS**

The following information will be recorded in the field log book.

- Date
- Equipment operators
- Name and project number of site
- Position and instrument readings
- Position-specific information
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## ATTACHMENTS

1. Listing of Underground Utility Clearance Resources
2. Frost Line Penetration Depths by Geographic Location
3. Utility Clearance Form
4. OSHA Letter of Interpretation
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:

<table>
<thead>
<tr>
<th>Color</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>white</td>
<td>excavation/subsurface investigation location</td>
</tr>
<tr>
<td>red</td>
<td>electrical</td>
</tr>
<tr>
<td>yellow</td>
<td>gas, oil, steam</td>
</tr>
<tr>
<td>orange</td>
<td>telephone, communications</td>
</tr>
<tr>
<td>blue</td>
<td>water, irrigation, slurry</td>
</tr>
<tr>
<td>green</td>
<td>sewer, drain</td>
</tr>
</tbody>
</table>

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 TINUS 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
# Listing of Underground Utility Clearance Resources

## One-Call Systems International Condensed Directory

<table>
<thead>
<tr>
<th>State</th>
<th>Call Center Name</th>
<th>Phone Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alabama</td>
<td>Alabama One-Call</td>
<td>1-800-292-8525</td>
</tr>
<tr>
<td>Alaska</td>
<td>Locate Call Center of Alaska, Inc</td>
<td>1-800-476-3121</td>
</tr>
<tr>
<td>Arizona</td>
<td>Arizona Blue Stake</td>
<td>1-800-782-5348</td>
</tr>
<tr>
<td>Arkansas</td>
<td>Arkansas One Call System, Inc.</td>
<td>1-800-482-8988</td>
</tr>
<tr>
<td>California</td>
<td>Underground Service Alert North</td>
<td>1-800-227-2600</td>
</tr>
<tr>
<td>Colorado</td>
<td>Utility Notification Center of Colorado</td>
<td>1-800-922-1987</td>
</tr>
<tr>
<td>Connecticut</td>
<td>Call Before You Dig</td>
<td>1-800-922-4455</td>
</tr>
<tr>
<td>Delaware</td>
<td>Miss Utility of Delmarva</td>
<td>1-800-262-8555</td>
</tr>
<tr>
<td>Florida</td>
<td>Sunshine State One-Call of Florida</td>
<td>1-800-432-4770</td>
</tr>
<tr>
<td>Georgia</td>
<td>Underground Protection Center, Inc</td>
<td>1-800-282-7411</td>
</tr>
<tr>
<td>Hawaii</td>
<td>Underground Service Alert North</td>
<td>1-800-227-2600</td>
</tr>
<tr>
<td>Idaho</td>
<td>Dig Line Inc.</td>
<td>1-800-342-1585</td>
</tr>
<tr>
<td></td>
<td>Kootenai County One-Call</td>
<td>1-800-425-4950</td>
</tr>
<tr>
<td></td>
<td>Shoshone - Benewah One-Call</td>
<td>1-800-398-3285</td>
</tr>
<tr>
<td>Illinois</td>
<td>JULIE, Inc.</td>
<td>1-800-992-0123</td>
</tr>
<tr>
<td></td>
<td>Digger (Chicago Utility Alert Network)</td>
<td>312-744-7000</td>
</tr>
<tr>
<td>Indiana</td>
<td>Indiana Underground Plant, Protection Service</td>
<td>1-800-382-5544</td>
</tr>
<tr>
<td>Iowa</td>
<td>Iowa One-Call</td>
<td>1-800-292-8989</td>
</tr>
<tr>
<td>Kansas</td>
<td>Kansas One-Call System, Inc.</td>
<td>1-800-344-7233</td>
</tr>
<tr>
<td>Kentucky</td>
<td>Kentucky Underground Protection Inc.</td>
<td>1-800-752-6007</td>
</tr>
<tr>
<td>Louisiana</td>
<td>Louisiana One Call System, Inc.</td>
<td>1-800-272-3520</td>
</tr>
<tr>
<td>Maine</td>
<td>Dig Safe System, Inc.</td>
<td>1-888-344-7233</td>
</tr>
<tr>
<td>Maryland</td>
<td>Miss Utility</td>
<td>1-800-267-7777</td>
</tr>
<tr>
<td>Mississippi</td>
<td>Mississippi One Call System, Inc.</td>
<td>1-800-227-8477</td>
</tr>
<tr>
<td>Massachusetts</td>
<td>Gopher State One Call</td>
<td>1-800-252-1168</td>
</tr>
<tr>
<td>Michigan</td>
<td>Miss Dig System, Inc.</td>
<td>1-800-452-7171</td>
</tr>
<tr>
<td>Minnesota</td>
<td>Mississippian One Call System, Inc.</td>
<td>1-800-515-1168</td>
</tr>
<tr>
<td>Missouri</td>
<td>Missouri One-Call System, Inc.</td>
<td>1-800-344-7483</td>
</tr>
<tr>
<td>Montana</td>
<td>Utilities Underground Protection Center</td>
<td>1-800-624-6555</td>
</tr>
<tr>
<td>Nebraska</td>
<td>Diggers Hotline of Nebraska</td>
<td>1-800-331-5696</td>
</tr>
<tr>
<td>Nevada</td>
<td>Underground Service Alert North</td>
<td>1-800-227-2000</td>
</tr>
<tr>
<td>New Hampshire</td>
<td>Dig Safe System, Inc.</td>
<td>1-888-544-7233</td>
</tr>
<tr>
<td>New Jersey</td>
<td>New Jersey One Call</td>
<td>1-800-272-1000</td>
</tr>
<tr>
<td>New Mexico</td>
<td>New Mexico One Call System, Inc.</td>
<td>1-800-321-2537</td>
</tr>
<tr>
<td>New York</td>
<td>Dig Safety New York</td>
<td>1-800-992-7962</td>
</tr>
<tr>
<td>North Carolina</td>
<td>The North Carolina One-Call Center, Inc.</td>
<td>1-800-832-4949</td>
</tr>
<tr>
<td>North Dakota</td>
<td>North Dakota One-Call</td>
<td>1-800-796-0555</td>
</tr>
<tr>
<td>Ohio</td>
<td>Ohio Utilities Protection Service</td>
<td>1-800-362-2784</td>
</tr>
<tr>
<td>Oklahoma</td>
<td>Call Okie</td>
<td>1-800-922-6543</td>
</tr>
<tr>
<td>Oregon</td>
<td>Oregon Utility Notification Center/One Call Concepts</td>
<td>1-800-332-2344</td>
</tr>
<tr>
<td>Pennsylvania</td>
<td>Pennsylvania One Call System, Inc.</td>
<td>1-800-248-1776</td>
</tr>
<tr>
<td>Rhode Island</td>
<td>Dig Safe System, Inc.</td>
<td>1-800-344-7233</td>
</tr>
<tr>
<td>South Carolina</td>
<td>Palmetto Utility Protection Service Inc.</td>
<td>1-888-721-7877</td>
</tr>
<tr>
<td>South Dakota</td>
<td>South Dakota One Call</td>
<td>1-800-781-7474</td>
</tr>
<tr>
<td>Tennessee</td>
<td>Tennessee One Call System, Inc.</td>
<td>1-800-351-1111</td>
</tr>
</tbody>
</table>

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**ATTACHMENT 1**

LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-Mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY
ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-6377
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
1-800-474-5885

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-863-9223

Tetra Tech NUS, Inc.
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:** [Name]

**Project Name:** [Name]

**Project No.:** [Number]

**Completed By:** [Name]

**Location Name:** [Name]

**Work Date:** [Date]

**Excavation Method/Overhead Equipment:**

<table>
<thead>
<tr>
<th></th>
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<tbody>
<tr>
<td>1.</td>
<td>Underground Utilities</td>
<td></td>
</tr>
<tr>
<td>a)</td>
<td>Review of existing maps?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>b)</td>
<td>Interview local personnel?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>c)</td>
<td>Site visit and inspection?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>d)</td>
<td>Excavation areas marked in the field?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>e)</td>
<td>Utilities located in the field?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>f)</td>
<td>Located utilities marked/added to site maps?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>g)</td>
<td>Client contact notified</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>g)</td>
<td>Name</td>
<td>[Name]</td>
</tr>
<tr>
<td>g)</td>
<td>Telephone</td>
<td>[Number]</td>
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<tr>
<td>g)</td>
<td>Date</td>
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<td>g)</td>
<td>State One-Call agency called?</td>
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</tr>
<tr>
<td>g)</td>
<td>Caller</td>
<td>[Name]</td>
</tr>
<tr>
<td>g)</td>
<td>Ticket Number</td>
<td>[Number]</td>
</tr>
<tr>
<td>g)</td>
<td>Survey performed by</td>
<td>[Name]</td>
</tr>
<tr>
<td>g)</td>
<td>Method</td>
<td>[Method]</td>
</tr>
<tr>
<td>g)</td>
<td>Date</td>
<td>[Date]</td>
</tr>
<tr>
<td>g)</td>
<td>Geophysical survey performed?</td>
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</tr>
<tr>
<td>h)</td>
<td>Survey performed by</td>
<td>[Name]</td>
</tr>
<tr>
<td>h)</td>
<td>Method</td>
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<tr>
<td>h)</td>
<td>Date</td>
<td>[Date]</td>
</tr>
<tr>
<td>i)</td>
<td>Hand excavation performed (with concurrent use of utility detection device)?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>i)</td>
<td>Completed by</td>
<td>[Name]</td>
</tr>
<tr>
<td>i)</td>
<td>Total depth</td>
<td>[Number] feet</td>
</tr>
<tr>
<td>i)</td>
<td>Date</td>
<td>[Date]</td>
</tr>
<tr>
<td>j)</td>
<td>Trench/excavation probed?</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>j)</td>
<td>Probing completed by</td>
<td>[Name]</td>
</tr>
<tr>
<td>j)</td>
<td>Depth/frequency</td>
<td>[Number]</td>
</tr>
<tr>
<td>j)</td>
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<td>[Date]</td>
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2. **Overhead Utilities**

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<tr>
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<tbody>
<tr>
<td>a)</td>
<td>Determination of nominal voltage</td>
<td>yes no N/A</td>
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<tr>
<td>b)</td>
<td>Marked on site maps</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>c)</td>
<td>Necessary to lockout/insulate/re-route</td>
<td>yes no N/A</td>
</tr>
<tr>
<td>d)</td>
<td>Document procedures used to lockout/insulate/re-route</td>
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</tr>
<tr>
<td>e)</td>
<td>Minimum acceptable clearance (SOP Section 5.2):</td>
<td>[Number]</td>
</tr>
</tbody>
</table>

3. **Notes:**

______________________________
______________________________

**Approval:**

______________________________ Date [Name]

**Site Manager/Field Operations Leader**

[PM/Project File Program File] Tetra Tech NUS, Inc.
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:
ATTACHMENT 4 (Continued)

When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by 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, 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 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):

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.

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 commentators and ultimately not including any examples of “acceptable means” in the final provision.

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, 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 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

**Analyte** – Chemical or radiochemical material whose concentration, activity, or mass is measured.

**Composite Sample** – A sample representing a physical average of grab samples.

**Environmental Sample** – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

**Grab Sample** – A portion of material collected to represent material or conditions present at a single unit of space and time.

**Hazardous Waste Sample** – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

**Representativeness** – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

**Project Manager** - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

**Field Operations Leader** - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that
custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

**Site Safety Officer (SSO)** - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

**Project Geologist/Sampler** - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.

- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 **HEALTH AND SAFETY**

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

**Bridge/Boat Sampling** – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.

- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent
Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United States Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

**Entering Water to Collect Samples** – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.
Working in these areas, also recognize the following hazards and means of protection against them:

**Insects** are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.

- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.

- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.

- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

**Safety Reminder**

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

**Poisonous Plants** – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

**Temperature-Related Stress** – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:
Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.

If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.

Where warning systems are not available, follow the 30/30 Rule (if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 procedures

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.
4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.

- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.

- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon’s or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

**Safety Reminder**

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
- Required decontamination equipment.
- Required sample containers.
- Sealable polyethylene bags (e.g., Ziploc® baggies).
- Heavy-duty cooler.
- Ice.
- Paper towels and garbage bags.
- Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.

2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.

3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.

4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.

5. Use a clean paper towel to clean and dry the outside of the container.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.
Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.
3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
4. Raise the sampler and cap the bottle.
5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.
2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.
3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).
4. Use a paper towel to clean and dry the outside of the container.
5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.

- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.

- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.

- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.

- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with
respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicable described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

**SAFETY REMINDER**

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices

- A minimum of three people are required if any of the following conditions are anticipated or observed:
  - Work in a waterway that is turbulent or swift that could sweep a sampler down stream should he or she fall in accidentally.
  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker’s feet, etc.
  - Waterway is tidal, and conditions such as those listed above could rapidly change.
The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon’s or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.
Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.

2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.

2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.

3. Open the sampler to retrieve the sediment.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.
6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

**SAFETY REMINDER**

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

**Coring Samplers**

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.
All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES


# ATTACHMENT A
## SURFACE WATER SAMPLE LOG SHEET

**Tetra Tech NUS, Inc.**

### SURFACE WATER SAMPLE LOG SHEET

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# ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

## Tetra Tech NUS, Inc.

### SOIL & SEDIMENT SAMPLE LOG SHEET

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Signature(s):
APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.
C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 Surface Water Sample Collection

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).
Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overtur" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.
C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- **Mixed Estuary** - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- **Salt Wedge Estuary** - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- **Oceanic Estuary** - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.
# Standard Operating Procedures

## Tetra Tech NUS, Inc.

### Subject
SOIL SAMPLING

### Applicability
Tetra Tech NUS, Inc.

### Prepared
Earth Sciences Department

### Approved
D. Senovich

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## Attachments

- A SOIL & SEDIMENT SAMPLE LOG SHEET
- B SPLIT-SPOON SAMPLER
- C TEST PIT LOG
- D REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING
1.0 PURPOSE

This procedure discusses the methods used to collect surface, near surface, and subsurface soil samples. Additionally, it describes the method for sampling of test pits and trenches to determine subsurface soil and rock conditions, and recover small-volume or bulk samples.

2.0 SCOPE

This procedure is applicable to the collection of surface, near surface and subsurface soils for laboratory testing, which are exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites.

3.0 GLOSSARY

Composite Sample - A composite sample exists as a combination of more than one sample at various locations and/or depths and times, which is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples are not to be collected for volatile organics analysis.

Grab Sample - One sample collected at one location and at one specific time.

Non-Volatile Sample - A non-volatile sample includes all other chemical parameters (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Hand Auqer - A sampling device used to extract soil from the ground in a relatively undisturbed form.

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2-inch OD to 3-1/2 inch OD. The larger sizes are commonly used when a larger volume of sample material is required.

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine the shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher excavator, or bulldozer).

Confined Space - As stipulated in 29 CFR 1910.146, a confined space means a space that: 1) is large enough and so configured that an employee can bodily enter and perform assigned work; 2) has limited or restricted means for entry or exit (for example tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and 3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.
4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for determining sampling objectives, as well as, the field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches, and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring and excavation activities, and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - The FOL is responsible for finalizing the location of surface, near surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits and trenches, and for adherence to OSHA regulations during these operations.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of soil samples and the completion of all required paperwork (i.e., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms).

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions which are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

5.0 PROCEDURES

5.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they have migrated into the water table, and can establish the amount of contamination sorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during the sampling operations, particularly noting the location, depth, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. As a result, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. Often this information on soil properties can be obtained from published soil surveys available through the U.S. Geological Surveys and other government or farm agencies. It is the
intent of this procedure to present the most commonly employed soil sampling methods used at hazardous waste sites.

5.2 **Soil Sample Collection**

5.2.1 **Procedure for Collecting Soil Samples for Volatile Organic Compounds**

The above described traditional sampling techniques, used for the collection of soil samples for volatile organic analysis, have recently been evaluated by the scientific community and determined to be ineffective in producing accurate results (biased low) due to the loss of volatile organics in the sampling stages and microbial degradation of aromatic volatiles. One of the newly adopted sampling procedures for collecting soil samples includes the field preservation of samples with methanol or sodium bisulfate to minimize volatilization and biodegradation. These preservation methods may be performed either in the field or laboratory, depending on the sampling methodology employed.

Soil samples to be preserved by the laboratory are currently being performed using method SW-846, 5035. Laboratories are currently performing low level analyses (sodium bisulfate preservation) and high level analyses (methanol preservation) depending on the end users needs.

It should be noted that a major disadvantage of the methanol preservation method is that the laboratory reporting limits will be higher than conventional testing. The reporting levels using the new method for most analytes are 0.5 µg/g for GC/MS and 0.05 µg/g for GC methods.

The alternative preservation method for collecting soil samples is with sodium bisulfate. This method is more complex to perform in the field and therefore is not preferred for field crews. It should also be noted that currently, not all laboratories have the capabilities to perform this analysis. The advantage to this method is that the reporting limits (0.001 µg/g for GC/PID or GC/ELCD, or 0.010 for GC/MS) are lower than those described above.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

5.2.1.1 **Soil Samples to be Preserved at the Laboratory**

Soil samples collected for volatile organics that are to be preserved at the laboratory will be obtained using a hermetically sealed sample vial such as an Encore™ sampler. Each sample will be obtained using a reusable sampling handle provided with the Encore™ sampler. The sample is collected by pushing the Encore™ sampler directly into the soil, ensuring that the sampler is packed tight with soil, leaving zero headspace. Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler shipment by the manufacturer.

Once the sample is collected, it should be placed on ice immediately and shipped to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

If the lower detection limits are necessary, an option would be to collect several Encore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.
5.2.1.2 **Soil Samples to be Preserved in the Field**

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) method and medium-level (methanol preservation) method.

**Methanol Preservation (Medium Level):**

Soil samples to be preserved in the field with methanol will utilize 40-60 mL glass vials with septum lids. Each sample bottle will be filled with 25 mL of demonstrated analyte-free purge and trap grade methanol. Bottles may be prespiked with methanol in the laboratory or prepared in the field.

Soil will be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol preserved sample bottle. Calibration of the scale should be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected by pulling the plunger back and inserting the syringe into the soil to be sampled. The top several inches of soil should be removed before collecting the sample. Approximately 10 grams ±2g (8-12 grams) of soil should be collected. The sample should be weighed and adjusted until obtaining the required amount of sample. The sample weight should be recorded to the nearest 0.01 gram in the field logbook and/or sample log sheet. The soil should then be extruded into the methanol preserved sample bottle taking care not to contact the sample container with the syringe. The threads of the bottle and cap must be free of soil particles.

After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

**Sodium Bisulfate Preservation (Low Level):**

Samples to be preserved using the sodium bisulfate method are to be prepared as follows:

Add 1 gram of sodium bisulfate to 5 mL of laboratory grade deionized water in a 40-60 mL glass vial with septum lid. Bottles may be prespiked in the laboratory or prepared in the field. The soil sample should be collected in a manner as described above and added to the sample container. The sample should be weighed to the nearest 0.01 gram as described above and recorded in the field logbook or sample log sheet.

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soils containing carbonates (limestone) may cause the sample to effervesc or the vial to possibly explode.

When preparing samples using the sodium bisulfate preservation method, duplicate samples must be collected using the methanol preservation method on a one for one sample basis. The reason for this is because it is necessary for the laboratory to perform both the low level and medium level analyses. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

If the lower detection limits are necessary, an option to field preserving with sodium bisulfate would be to collect 3 EnCore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.
5.2.2 Procedure for Collecting Non-Volatile Soil Samples

Non-volatile soil samples may be collected as either grab or composite samples. The non-volatile soil sample is thoroughly mixed in a stainless steel or disposable, inert plastic tray, using a stainless steel trowel or other approved tool, then transferred into the appropriate sample container(s). Head space is permitted in a non-volatile soil sample container to allow for sample expansion.

5.2.3 Procedure for Collecting Undisturbed Soil Samples (ASTM D1587-83)

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) will be employed. The following method will be used:

1. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and clean out the borehole to the sampling depth, being careful to minimize the chance for disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

2. The use of bottom discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Use of any side-discharge bits is permitted.

3. A stationary piston-type sampler may be required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal and to maintain a suction within the tube to help retain the sample.

4. To minimize chemical reaction between the sample and the sampling tube, brass tubes may be required, especially if the tube is stored for an extended time prior to testing. While steel tubes coated with shellac are less expensive than brass, they're more reactive, and shall only be used when the sample will be tested within a few days after sampling or if chemical reaction is not anticipated. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil by a continuous and rapid motion, without impacting or twisting. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

5. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated. Remove disturbed material in the upper end of the tube and measure the length of sample again. After removing at least an inch of soil from the lower end and after inserting an impervious disk, seal both ends of the tube with at least a 1/2-inch thickness of wax applied in a way that will prevent the wax from entering the sample. Clean filler must be placed in voids at either end of the tube prior to sealing with wax. Place plastic caps on the ends of the sample tube, tape the caps in place, and dip the ends in wax.

6. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label. Mark the "up" direction on the side of the tube with indelible ink, and mark the end of the sample. Complete Chain-of-Custody (see SOP SA-6.3) and other required forms (including Attachment A of this SOP). Do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.
Thin-walled undisturbed tube samplers are restricted in their usage by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soils with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soils. Using these devices normally increases sampling costs, and therefore their use shall be weighed against the need for acquiring an undisturbed sample.

5.3  **Surface Soil Sampling**

The simplest, most direct method of collecting surface soil samples (most commonly collected to a depth of 6 inches) for subsequent analysis is by use of a stainless steel trowel. Surface soils are considered 0-12 inches bgs.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Real-time air monitoring instrument (e.g., PID, FID, etc.).
- Latex gloves.
- Required Personal Protective Equipment (PPE).
- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP).
- Required decontamination equipment.
- Required sample container(s).
- Wooden stakes or pin flags.
- Sealable polyethylene bags (i.e., Ziploc® baggies).
- Heavy duty cooler.
- Ice.
- Chain-of-custody records and custody seals.

When acquiring surface soil samples, the following procedure shall be used:

1. Carefully remove vegetation, roots, twigs, litter, etc., to expose an adequate soil surface area to accommodate sample volume requirements.

2. Using a decontaminated stainless steel trowel, follow the procedure cited in Section 5.2.1 for collecting a volatile soil sample. Surface soil samples for volatile organic analysis should be collected from 6-12 inches bgs only.

3. Thoroughly mix (in-situ) a sufficient amount of soil to fill the remaining sample containers and transfer the sample into those containers utilizing the same stainless steel trowel employed above. Cap and securely tighten all sample containers.

4. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.

5. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

5.4  **Near-Surface Soil Sampling**

Collection of samples from near the surface (depth of 6-18 inches) can be accomplished with tools such as shovels and stainless steel or pre-cleaned disposable trowels.
The following equipment is necessary to collect near surface soil samples:

- Clean shovel.
- The equipment listed under Section 5.3 of this procedure.
- Hand auger.

To obtain near-surface soil samples, the following protocol shall be observed:

1. With a clean shovel, make a series of vertical cuts to the depth required in the soil to form a square approximately 1 foot by 1 foot.

2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 2 through 5 listed under Section 5.3 of this procedure.

5.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e., cylinders 6-1/2" long, and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), and a cross handle. A larger diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then withdrawn. In turn, the larger diameter bit is replaced with a smaller diameter bit, lowered down the hole, and slowly turned into the soil at the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil both from the surface, or to depths in excess of 12 feet. However, the presence of rock layers and the collapse of the borehole normally contribute to its limiting factors.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes).
- Stainless steel mixing bowls.
- The equipment listed under Section 5.3 of this procedure.

To obtain soil samples using a hand auger, the following procedure shall be followed:

1. Attach a properly decontaminated bucket bit to a clean extension rod and further attach the cross handle to the extension rod.

2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).

3. Begin augering (periodically removing accumulated soils from the bucket bit) and add additional rod extensions as necessary. Also, note (in a field notebook, boring log, and/or on standardized data sheets) any changes in the color, texture or odor of the soil.

4. After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.

5. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is commonly smaller in diameter than the bucket bit employed to initiate the borehole.
6. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

7. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

8. Discard the top of the core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the sample material.

9. Fill volatile sample container(s), using a properly decontaminated stainless steel trowel, with sample material directly from the bucket bit. Refer to Section 5.2.1 of this procedure.

10. Utilizing the above trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl and thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

11. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.6 **Subsurface Soil Sampling With a Split-Barrel Sampler (ASTM D1586-84)**

Split-barrel (split-spoon) samplers consist of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-lb. or larger casing driver.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (O.D. 2 inches, I.D. 1-3/8 inches, either 20 inches or 26 inches long); Larger O.D. samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-lb. weight, driving head and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed under Section 5.3 of this procedure.

The following steps shall be followed to obtain split-barrel samples:

1. Remove the drive head and nosepiece, and open the sampler to reveal the soil sample. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.). Carefully separate the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

2. Collect the volatile sample from the center of the core where elevated readings occurred. If no elevated readings were encountered the sample material should still be collected from the core’s...
center (this area represents the least disturbed area with minimal atmospheric contact). Refer to Section 5.2.1 of this procedure.

3. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl. Thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

4. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.7 **Subsurface Soil Sampling Using Direct Push Technology**

Subsurface soil samples can be collected to depths of 40+ feet using direct push technology (DPT). DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

5.8 **Excavation and Sampling of Test Pits and Trenches**

5.8.1 **Applicability**

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise which control the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is still required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden or steel support structures. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments. Any entry may constitute a Confined Space and must be done in conformance with all applicable regulations. In these cases, substantial air monitoring is required before entry, and appropriate respiratory gear and protective clothing is mandatory. There must be at least two persons present at the immediate site before entry by one of the investigators. The reader shall refer to OSHA regulations 29 CFR 1926, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146.

Excavations are generally not practical where a depth of more than about 15 feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If data on soils at depths greater than 15 feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

5.8.2 **Test Pit and Trench Excavation**

These procedures describe the methods for excavating and logging test pits and trenches excavated to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).
Test pits and trenches may be excavated by hand or by power equipment to permit detailed description of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration.
- The space required for efficient excavation.
- The chemicals of concern.
- The economics and efficiency of available equipment.

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table, which is based on equipment efficiencies, gives a rough guide for design consideration:

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<td>Track dozer</td>
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<tr>
<td>Track loader</td>
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<td>Excavator</td>
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<tr>
<td>Scraper</td>
<td>20</td>
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The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous waste materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, field conditions may necessitate revisions to the initial plans. The final depth and construction method shall be determined by the field geologist. The actual layout of each test pit, temporary staging area, and spoils pile will be predicated based on site conditions and wind direction at the time the test pit is made. Prior to excavation, the area can be surveyed by magnetometer or metal detector to identify the presence of underground utilities or drums.

As mentioned previously, no personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is still required, Occupational Safety and Health Administration (OSHA) requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be in the hole at all times, and a temporary guardrail must be placed along the surface of the hole before entry). It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example, samples of leachate, groundwater, or sidewall soils can be taken with telescoping poles, etc.

Dewatering may be required to assure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation dry. This is an important consideration for excavations in cohesionless material below the groundwater table. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.
5.8.3 Sampling in Test Pits and Trenches

5.8.3.1 General

Test pits and trenches are usually logged as they are excavated. Records of each test pit/trench will be made as presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable Health and Safety and OSHA requirements have been met.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information would include soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples, which can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

5.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottleware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps and right angle adapter for conduit (see Attachment D).

5.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 5.8.3.4.

- Excavate trench or pit in several depth increments. After each increment, the operator will wait while the sampler inspects the test pit from grade level to decide if conditions are appropriate for sampling. (Monitoring of volatiles by the SSO will also be used to evaluate the need for sampling.) Practical depth increments range from 2 to 4 feet.
The backhoe operator, who will have the best view of the test pit, will immediately cease digging if:

- Any fluid phase or groundwater seepage is encountered in the test pit.
- Any drums, other potential waste containers, obstructions or utility lines are encountered.
- Distinct changes of material are encountered.

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending upon the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Remove loose material to the greatest extent possible with backhoe.
- Secure walls of pit if necessary. (There is seldom any need to enter a pit or trench which would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)
- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material once it has been deposited on the ground. The sampler or Field Operations Leader directs the backhoe operator to remove material from the selected depth or location within the test pit/trench. The bucket is brought to the surface and moved away from the pit. The sampler and/or SSO then approaches the bucket and monitors its contents with a photoionization or flame ionization detector. The sample is collected from the center of the bucket or pile and placed in sample containers using a decontaminated stainless steel trowel or disposable spatula.
- If a composite sample is desired, several depths or locations within the pit/trench are selected and a bucket is filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.
- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the side wall or bottom of the pit. The face of the pit/trench shall first be scraped (using a long-handled shovel or hoe) to remove the smeared zone that has contacted the backhoe bucket. The sample shall then be collected directly into the sample jar, by scraping with the jar edge, eliminating the need to utilize samplers and minimizing the likelihood of cross-contamination. The sample jar is then capped, removed from the assembly, and packaged for shipment.
- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

### 5.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soils or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:
• There is no practical alternative means of obtaining such data.

• The Site Safety Officer and Competent Person determines that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of volatile organics, explosive gases and available oxygen).

• A Company-designated Competent Person determines that the pit/trench is stable or is made stable (by grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements must be strictly observed.

If these conditions are satisfied, one person will enter the pit/trench. On potentially hazardous waste sites, this individual will be dressed in safety gear as required by the conditions in the pit. He/she will be affixed to a safety rope and continuously monitored while in the pit.

A second individual will be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations. The individual entering the pit will remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon. As an added precaution, it is advisable to keep the backhoe bucket in the test pit when personnel are working below grade. Such personnel can either stand in or near the bucket while performing sample operations. In the event of a cave-in they can either be lifted clear in the bucket, or at least climb up on the backhoe arm to reach safety.

5.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 5.8.3.2, the following equipment is needed for geotechnical sampling:

• Soil sampling equipment, similar to that used in shallow drilled boring (i.e., open tube samplers), which can be pushed or driven into the floor of the test pit.

• Suitable driving (i.e., a sledge hammer) or pushing (i.e., the backhoe bucket) equipment which is used to advance the sampler into the soil.

• Knives, spatulas, and other suitable devices for trimming hand-carved samples.

• Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.

• Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soils in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification, while larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soils using open tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe,
rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the sampler when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 5.8.3.4 of this procedure must be followed. The open tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate, because the sample will not have the correct orientation.

A sledge hammer or the backhoe may be used to drive or push the sampler or tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry of the test pit, the requirements in Section 5.8.3.4 of this procedure must be followed. Prepare, label, pack and transport the sample in the required manner, as described in SOP SA-6.3 and SA-6.1.

5.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew shall photograph all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL.

If a low permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

5.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. In addition, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job.
- Date of boring and excavation.
• Approximate surface elevation.
• Total depth of boring and excavation.
• Dimensions of pit.
• Method of sample acquisition.
• Type and size of samples.
• Soil and rock descriptions.
• Photographs.
• Groundwater levels.
• Organic gas or methane levels.
• Other pertinent information, such as waste material encountered.

6.0 REFERENCES


OSHA, Confined Space Entry 29 CFR 1910.146.
# ATTACHMENT A

## SOIL & SEDIMENT SAMPLE LOG SHEET

<table>
<thead>
<tr>
<th>Project Site Name:</th>
<th>Sample ID No.:</th>
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</thead>
<tbody>
<tr>
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</table>

<table>
<thead>
<tr>
<th>Project No.:</th>
<th>Sample Location:</th>
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<tbody>
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</table>

<table>
<thead>
<tr>
<th>Sampled By:</th>
<th>C.O.C. No.:</th>
</tr>
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<tbody>
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</table>

### SURFACE SOIL

- **Sampled By:**
- **C.O.C. No.:**

### SUBSURFACE SOIL

- **Sampled By:**
- **C.O.C. No.:**

### SEDIMENT

- **Sampled By:**
- **C.O.C. No.:**

### OTHER

- **Sampled By:**
- **C.O.C. No.:**

### Q.A. SAMPLE TYPE

- **Sampled By:**
- **C.O.C. No.:**

### QA SAMPLE DATA

<table>
<thead>
<tr>
<th>Date:</th>
<th>Depth</th>
<th>Color</th>
<th>Description (Sand, Silt, Clay, Moisture, etc.)</th>
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<tbody>
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</tbody>
</table>

### COMPOSITE SAMPLE DATA

<table>
<thead>
<tr>
<th>Date:</th>
<th>Time</th>
<th>Depth</th>
<th>Color</th>
<th>Description (Sand, Silt, Clay, Moisture, etc.)</th>
</tr>
</thead>
<tbody>
<tr>
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</table>

### SAMPLE COLLECTION INFORMATION

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Container Requirements</th>
<th>Collected</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</table>

### OBSERVATIONS/NOTES:

- **Sampled By:**
- **C.O.C. No.:**

### Circle if Applicable:  

- **MS/MSD**

- **Duplicate ID No.:**

- **Signature(s):**
ATTACHMENT B
SPLIT-SPOON SAMPLER

A = 1.0 to 2.0 in. (25 to 50 mm)
E = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.83 ± 0.13 mm)
D = 1.50 ± 0.05 - 0.00 in. (38.1 ± 1.3 - 0.0 mm)
G = 0.10 ± 0.02 in. (2.54 ± 0.06 mm)
F = 2.00 ± 0.05 - 0.00 in. (50.8 ± 1.3 - 0.0 mm)
H = 16.0° to 22.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 18-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.
### TEST PIT LOG

<table>
<thead>
<tr>
<th>Depth (ft)</th>
<th>Lithology Change (Depth/ft)</th>
<th>MATERIAL DESCRIPTION</th>
<th>U S C S</th>
<th>Remarks</th>
<th>PID/PID READING</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Soil/Waste Characteristics (lithology, density, color, etc.)</td>
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### TEST PIT CROSS SECTION AND/OR PLAN VIEW

**REMARKS:**

**PHOTO LOG:**
ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING

![Diagram showing components of the remote sample holder: Right-angle adapter, sample bottle, steel conduit, hose clamp.]

- Right-angle adapter
- Sample bottle
- Steel conduit
- Hose clamp
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ATTACHMENTS

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

**Hazardous Material** - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

**Hazardous Waste** - Any substance listed in 40 CFR, Subpart D (261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

**Marking** - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

**n.o.i.** - Not otherwise indicated (may be used interchangeably with n.o.s.).

**n.o.s.** - Not otherwise specified.

**Packaging** - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

**Placard** - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

**Common Preservatives:**
- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH
Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na₂S₂O₃

**Normality (N)** - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

**Reportable Quantity (RQ)** - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

**Field Operations Leader** - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

**Field Samplers** - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 **Sample Containers**

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 **Sample Preservation**

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological...
changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO₃, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

<table>
<thead>
<tr>
<th>Acid/Base</th>
<th>Dilution</th>
<th>Concentration</th>
<th>Estimated Amount Required for Preservation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric Acid (HCl)</td>
<td>1 part concentrated HCl: 1 part double-distilled, deionized water</td>
<td>6N</td>
<td>5-10 mL</td>
</tr>
<tr>
<td>Sulfuric Acid (H₂SO₄)</td>
<td>1 part concentrated H₂SO₄: 1 part double-distilled, deionized water</td>
<td>18N</td>
<td>2 - 5 mL</td>
</tr>
<tr>
<td>Nitric Acid (HNO₃)</td>
<td>Undiluted concentrated HNO₃</td>
<td>16N</td>
<td>2 - 5 mL</td>
</tr>
<tr>
<td>Sodium Hydroxide (NaOH)</td>
<td>400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution</td>
<td>10N</td>
<td>2 mL</td>
</tr>
</tbody>
</table>

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:
- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

### 5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).
To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS “Shipping Hazardous Materials” training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)
- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.
- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.
- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.
- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).
- Seal (i.e., tape or tie top in knot) large liner bag.
- 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 the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.
- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.
Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES


International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.


### ATTACHMENT A

#### GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

<table>
<thead>
<tr>
<th>Sample Type and Concentration</th>
<th>Container</th>
<th>Sample Size</th>
<th>Preservation</th>
<th>Holding Time</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>WATER</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Organics (GC&amp;GC/MS)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>VOC</td>
<td>Low</td>
<td>Borosilicate glass</td>
<td>2 x 40 mL</td>
<td>Cool to 4°C HCl to ≤ 2</td>
</tr>
<tr>
<td>Extractables SVOCs and pesticides(PCBs)</td>
<td>Low</td>
<td>Amber glass</td>
<td>2 x 2 L or 4 x 1 L</td>
<td>Cool to 4°C</td>
</tr>
<tr>
<td>Extractables SVOCs and pesticides(PCBs)</td>
<td>Medium</td>
<td>Amber glass</td>
<td>2 x 2 L or 4 x 1 L</td>
<td>None</td>
</tr>
<tr>
<td>Inorganics</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>Low</td>
<td>High-density polyethylene</td>
<td>1 L</td>
<td>HNO₃ to pH ≤ 2</td>
</tr>
<tr>
<td>Cyanide</td>
<td>Low</td>
<td>High-density polyethylene</td>
<td>1 L</td>
<td>NaOH to pH&gt;12</td>
</tr>
<tr>
<td>Cyanide</td>
<td>Medium</td>
<td>Wide-mouth glass</td>
<td>16 oz.</td>
<td>None</td>
</tr>
<tr>
<td>Organic/Inorganic</td>
<td>High Hazard</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>None</td>
</tr>
<tr>
<td><strong>SOIL</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Organics (GC&amp;GC/MS)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>VOC</td>
<td>Low</td>
<td>EnCore Sampler</td>
<td>(3) 5 g Samplers</td>
<td>Cool to 4°C</td>
</tr>
<tr>
<td>Extractables SVOCs and pesticides(PCBs)</td>
<td>Low</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>Cool to 4°C</td>
</tr>
<tr>
<td>Extractables SVOCs and pesticides(PCBs)</td>
<td>Medium</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>Cool to 4°C</td>
</tr>
<tr>
<td>Inorganics</td>
<td>Low/Medium</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>Cool to 4°C</td>
</tr>
<tr>
<td>Organic/Inorganic</td>
<td>High Hazard</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>None</td>
</tr>
<tr>
<td>Dioxin/Furan</td>
<td>All</td>
<td>Wide-mouth glass</td>
<td>4 oz.</td>
<td>None</td>
</tr>
<tr>
<td>TCLP</td>
<td>All</td>
<td>Wide-mouth glass</td>
<td>8 oz.</td>
<td>None</td>
</tr>
<tr>
<td><strong>AIR</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volatile Organics</td>
<td>Low/Medium</td>
<td>Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID</td>
<td>100 L air</td>
<td>Cool to 4°C</td>
</tr>
</tbody>
</table>

1. All glass containers should have Teflon cap liners or septa.
2. See Attachment E: Preservation and maximum holding time allowances per 40 CFR 136.
**ATTACHMENT B**

**ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES**

<table>
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<th>Parameter Number/Name</th>
<th>Container(1)</th>
<th>Preservation(2)(3)</th>
<th>Maximum Holding Time(4)</th>
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</thead>
<tbody>
<tr>
<td>Acidity</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>14 days</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>14 days</td>
</tr>
<tr>
<td>Ammonia - Nitrogen</td>
<td>P, G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Biochemical Oxygen Demand (BOD)</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td>Bromide</td>
<td>P, G</td>
<td>None required</td>
<td>28 days</td>
</tr>
<tr>
<td>Chemical Oxygen Demand (COD)</td>
<td>P, G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Chloride</td>
<td>P, G</td>
<td>None required</td>
<td>28 days</td>
</tr>
<tr>
<td>Chlorine, Total Residual</td>
<td>P, G</td>
<td>None required</td>
<td>Analyze immediately</td>
</tr>
<tr>
<td>Color</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td>Cyanide, Total and Amenable to Chlorination</td>
<td>P, G</td>
<td>Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid(5)</td>
<td>14 days(5)</td>
</tr>
<tr>
<td>Fluoride</td>
<td>P</td>
<td>None required</td>
<td>28 days</td>
</tr>
<tr>
<td>Hardness</td>
<td>P, G</td>
<td>HNO3 to pH 2; H2SO4 to pH 2</td>
<td>6 months</td>
</tr>
<tr>
<td>Total Kjeldahl and Organic Nitrogen</td>
<td>P, G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Nitrate - Nitrogen</td>
<td>P, G</td>
<td>None required</td>
<td>48 hours</td>
</tr>
<tr>
<td>Nitrate-Nitrite - Nitrogen</td>
<td>P, G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Nitrite - Nitrogen</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td>Oil &amp; Grease</td>
<td>G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Organic Carbon (TOC)</td>
<td>P, G</td>
<td>Cool, 4°C; HCl or H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Orthophosphate</td>
<td>P, G</td>
<td>Filter immediately; Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td>Oxygen, Dissolved-Probe</td>
<td>G Bottle &amp; top</td>
<td>None required</td>
<td>Analyze immediately</td>
</tr>
<tr>
<td>Oxygen, Dissolved-Winkler</td>
<td>G Bottle &amp; top</td>
<td>Fix on site and store in dark</td>
<td>8 hours</td>
</tr>
<tr>
<td>Phenols</td>
<td>G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Phosphorus, Total</td>
<td>P, G</td>
<td>Cool, 4°C; H2SO4 to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Residue, Total</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 days</td>
</tr>
<tr>
<td>Residue, Filterable (TDS)</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 days</td>
</tr>
<tr>
<td>Residue, Nonfilterable (TSS)</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 days</td>
</tr>
<tr>
<td>Residue, settleable</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td>Residue, Volatile (Ash Content)</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 days</td>
</tr>
<tr>
<td>Silica</td>
<td>P</td>
<td>Cool, 4°C</td>
<td>28 days</td>
</tr>
<tr>
<td>Specific Conductance</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>28 days</td>
</tr>
<tr>
<td>Sulfate</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>28 days</td>
</tr>
</tbody>
</table>

(1) Container: P = Polyethylene, G = Glass

(2) Preservation: Cool, 4°C; H2SO4 to pH 2; HNO3 to pH 2; HCl or H2SO4 to pH 2

(3) H2SO4 to pH 2

(4) Analyze immediately

(5) 0.6 g ascorbic acid
**ATTACHMENT B**
**ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES**

### PAGE TWO

<table>
<thead>
<tr>
<th>Parameter Number/Name</th>
<th>Container(1)</th>
<th>Preservation(2)(3)</th>
<th>Maximum Holding Time(4)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>INORGANIC TESTS (Cont'd):</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sulfide</td>
<td>P, G</td>
<td>Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9</td>
<td>7 days</td>
</tr>
<tr>
<td>Sulfite</td>
<td>P, G</td>
<td>None required</td>
<td>Analyze immediately</td>
</tr>
<tr>
<td>Turbidity</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>48 hours</td>
</tr>
<tr>
<td><strong>METALS:</strong>(7)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chromium VI (Hexachrome)</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>24 hours</td>
</tr>
<tr>
<td>Mercury (Hg)</td>
<td>P, G</td>
<td>HNO₃ to pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Metals, except Chromium VI and Mercury</td>
<td>P, G</td>
<td>HNO₃ to pH 2</td>
<td>6 months</td>
</tr>
<tr>
<td><strong>ORGANIC TESTS:</strong>(8)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Purgeable Halocarbons</td>
<td>G, Teflon-lined septum</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>14 days</td>
</tr>
<tr>
<td>Purgeable Aromatic Hydrocarbons</td>
<td>G, Teflon-lined septum</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>14 days</td>
</tr>
<tr>
<td>Acrolein and Acrylonitrile</td>
<td>G, Teflon-lined septum</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>14 days</td>
</tr>
<tr>
<td>Phenols(11)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Benzidine(11),(12)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction(13)</td>
</tr>
<tr>
<td>Phthalate esters(13)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Nitrosamines(13),(14)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; store in dark; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>PCBs(15)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Nitroaromatics &amp; Isophorone(17)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Polynuclear Aromatic Hydrocarbons (PAHs)(11),(14)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃; store in dark</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Haloethers(18)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
<tr>
<td>Dioxin/Furan (TCDD/TCDF)(19)</td>
<td>G, Teflon-lined cap</td>
<td>Cool, 4°C; 0.008% Na₂S₂O₃</td>
<td>7 days until extraction; 40 days after extraction</td>
</tr>
</tbody>
</table>
(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₅ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₅.
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<td><a href="HTTP://INTRANET.TTNUS.COM">HTTP://INTRANET.TTNUS.COM</a> CLICK ON FIELD LOG SHEETS</td>
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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 initialed 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.
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 Rinse 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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<td>Low Flow Purge Data Sheet</td>
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<td>QA Sample Log Sheet</td>
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ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: ___________________ DATE: ____________________

SITE LEADER: _______________________
PERSONNEL: ___________________________________
TtNUS DRILLER SITE VISITORS
__________________________________________  ____________  _______________________
__________________________________________  ____________  _______________________
__________________________________________  ____________  _______________________

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

ACTIVITIES:

1. Steam jenney 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
### ATTACHMENT B

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2. RELINQUISHED BY DATE TIME 2. RECEIVED BY DATE TIME
3. RELINQUISHED BY DATE TIME 3. RECEIVED BY DATE TIME

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

CHAIN-OF-CUSTODY SEAL

[Signature]    [CUSTODY SEAL]    [Signature]

[Date]    [Date]    [Date]
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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, drill/drive 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/solils.

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.