965550

WORK PLAN FOR DECISION UNIT SOIL SAMPLING

USS LEAD OPERABLE UNIT 1 ZONE 1 SITE
EAST CHICAGO, INDIANA

PREPARED FOR:
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SEPTEMBER 2020

HULL
Environment / Energy / Infrastructure

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1.0 INTRODUCTION

1.1 General

This Work Plan has been prepared by Hull & Associates, LLC (Hull) on behalf of Industrial Advantage Advantage LLC (IDA). IDA is contemplating acquisition of a portion of the U.S. Smelter and Lead Refinery, Inc. (USS. Lead) Superfund Site Operable Unit 1 (OU1) Zone 1 for redevelopment as Class A warehouse and distribution facility. To the extent possible, IDA plans on incorporating remediation during development of the land. This Work Plan describes an approach to collect data to support decisions on how to potentially manage arsenic and lead impacted soils during remediation and development.

The USS Lead Superfund Site was placed on the National Priorities List (NPL) in April 2009. The 50.3-acre portion of the USS Lead OU1 Zone 1 area to be remediated and developed is located in East Chicago, Lake County, Indiana (Site). The Site formerly contained the West Calumet Housing Complex, Goodman Park, and a utility corridor. Primary chemicals of concern in soils at the Site were determined to be arsenic and lead. As part of a time-critical removal action funded by the U.S. Environmental Protection Agency (EPA), five residences at the Site were cleaned up in October 2011. Guided by a July 2012 Remedial Investigation/Feasibility Study, EPA issued a Record of Decision (ROD) for OU1, including the Site, selecting an excavation and off-Site disposal remedy for impacted soils. On October 28, 2014, EPA, the state of Indiana, Atlantic Richfield Company, The Chemours Company FC, LLC and E. I. du Pont de Nemours and Company entered into a Consent Decree for cleanup of residences and wetland areas, including the Site.

In November 2018, EPA issued a Proposed ROD Amendment followed by a March 2020 ROD Amendment for the Site recognizing the potential change in land use from residential to commercial and/or industrial uses. In light of the potential change in land use, the ROD Amendment contemplated modifications to excavation remedies where, for instance, the depth of excavation could be set to 12 inches below existing grade. On May 26, 2020, the East Chicago City Council approved rezoning the Site to an industrial land use.

1.1 Site Location and Disposition

The OU-1, Zone 1 Site is located in East Chicago, Indiana and generally bound by the former Carrie Gosch Elementary School to the north, East 151st Street to the south, McCook Avenue to the east and the Indiana Harbor canal to the west. A Site location map is presented as Figure 1.

Following demolition of the West Calumet Housing Complex and surface infrastructure during the summer of 2018, the Site no longer contains structures or drives with the exception of a maintenance building and associated parking lot owned by the East Chicago Housing Authority in the east-central portion of the Site.

1.2 Objectives

The objectives of activities described in this Work Plan are to:

1. define, within discrete decision units across the Site, soils within the upper one foot of surface grade meet or exceed decision criteria for arsenic (26 mg/kg) and lead (800 mg/kg), thereby allowing soils to remain on-Site or requiring off-Site disposal;

2. identify soils that will require stabilization treatment prior to off-Site disposal; and

3. quantify volumes of granular, engineered subbase materials for former roads that will be placed beneath the future hardscape areas (i.e., building slab and pavement).

All work will be completed consistent with the Final Quality Assurance Project Plan (QAPP), prepared by Hull in September 2020.

2.0 SAMPLING AND ANALYSIS

2.1 Sampling Layout

Per guidance provided by EPA, IDA proposes to employ a Geospatial Sample/Incremental Composite Sampling (ICS) Design to acquire data to support decisions relative to management of Site soils within the upper foot below grade. Specifically, as shown on Figure 2, the Site has been divided into 55 decision units. Each decision unit has a grid-based designation (i.e., 1A through 9F). Forty-five of the decision units, covering most of the Site, have approximately equal dimensions and are each roughly one acre in area. The ten decision units along the eastern margin of the Site, within the utility corridor, are roughly half the area of decision units to the west.

Also as shown on Figure 2, sub-sample locations are, where possible, arrayed in a “five on a die” pattern that will provide for compositing five (5) 0.0 to 1.0-foot depth interval sub-samples within each decision unit. As the maintenance building and parking lot in the east-central portion of the Site will remain post-development and soil beneath the hardscape materials will not be excavated, the sampling pattern has been modified such that sub-samples will be collected from existing green space areas in decision units F6 and F7.

In a strategy to provide repeated measures of the mean and variance within portions of the decision unit array (data statistics are described in more detail in section 3.2), triplicate sub-samples will be collected within nine of the decision units. Specifically, sub-samples will be collected as shown on Figure 2 from decision units selected for triplicate sampling. A second of the triplicate sub-sample set will be collected three feet east of each base sampling location. The third of the triplicate sub-sample set will be collected three feet south of each sub-sample sampling location.

The decision units selected for triplicate sampling are F1, A2, E3, B4, D5, C6, F7, A8 and E9.

2.2 Soil Sampling Procedures

2.2.1 Layout of Sampling Points

The composite sample in each decision unit will have five sub-sample locations (i.e., a five-point composite sample). Proposed sub-sample sampling locations will be located in the field using a global positioning system (GPS), marked with lathe or flagging, and each marker will be labeled with the sampling location designation as shown on Figure 2 (i.e., A1-1 through F9-5). Off-sets for triplicate sampling locations within selected decision units listed in section 2.1 will be offset three feet from the primary sub-sample locations using a measuring tape and marked with lathe or flagging. Markers for triplicate locations off-set east of base sub-samples will be labeled with an appended “a” (e.g., F1-1a) and triplicate locations off-set south of base sub-samples will be labeled with an appended “b” (e.g., F1-1b).

2.2.2 Sample Collection and Logging

All samples will be collected using AMS™ slide hammers (or equivalent) equipped with a one-foot-length split core sampler. The sampler will be opened in the field to allow Hull’s sampling personnel to log in a field notebook or pre-prepared field form:

1. soil or fill type(s) recovered in the sample;
2. percentage recovery for the sampling run; and
3. other observations such as odors, unusual discoloration, etc.

Any aggregate material for road subbases or in former building areas, large roots or anthropogenic materials (plastic, metals, etc.) will be discarded from the sample and not prepared for shipment to a laboratory for analysis. Only material below the aggregate material will be submitted to the laboratory. If the sample is made up solely of aggregate material, the base sampling location will be moved (along with triplicate sampling locations, as applicable) until sufficient non-aggregate sample volumes can be obtained. Off-set sampling location coordinates will be documented by a GPS unit after sampling is completed.

Prior to each day of sampling and prior to collection of each sample, the split core sampler will be thoroughly decontaminated consistent with Hull Standard Operating Procedure (SOP) F1000R1. SOP F1000R1 is contained in Appendix A.

All investigation derived wastes (IDW – e.g., used acetate sleeves and unused sample material, excepting aggregate subbase material) will be placed in 55-gallon Department of Transportation drums. Pending characterization, the IDW will be disposed of at an appropriately licensed disposal facility.

2.2.3 Field Quality Assurance/Quality Control (QA/QC)

In light of the triplicate composite sampling proposed for nine of the decision units, Hull does not propose collection of duplicate QA/QC samples.

2.2.4 Sample Preparation

Each sub-sample and triplicate sub-sample will be placed in two four-ounce or one eight-ounce laboratory supplied wide-mouth glass jar(s), or one-gallon sealable bag for analysis of total lead and total arsenic.

Additional sample material from sub-sample locations E2-3, C5-3 and B8-3 will be submitted to the laboratory in laboratory-supplied four-ounce or eight-ounce wide mouth containers for disposal characterization described in section 2.3. Each sub-sample will receive a designation that will be recorded on a laboratory chain-of-custody and sub-sample container along with date and time sampled, sampler and requested analyses. The sub-sample designation format will be as follows:

IDA00#:Grid_Sample_Location:SDepth_Interval

where:

- IDA00# is Hull's project number for the work;
- the grid sample location will be, for example, A1-1;
- "S" is the soil matrix; and
- the depth interval will be expressed in terms of feet and tenths of feet (i.e., 0010 is a sample interval of 0.0 to 1.0 feet below ground surface).

Laboratory samples will be placed in shipping containers sent to ALS Environmental USA (ALS) in Holland, Michigan under chain-of-custody for processing and analyses described in section 2.3. All analyses will be requested under a 14-day turnaround. The chain of custody will be filled out consistent with Hull Standard Operating Procedure (SOP) F3014 (Appendix A).

2.3 Soil Sample Processing and Analyses

Upon receiving and logging soil samples designated by chains-of-custody for total lead and arsenic analyses, ALS, dry and homogenize the sub-samples pursuant to SOP No. HN-QS-008-R06 in Appendix B. ALS will then sieve out larger debris in the homogenized samples. The sieving process will follow ALS Standard Operating Procedure (SOP) No. HN-MET-013-R03 (Particle Size Analysis), provided in Appendix B.

The above samples will be tested at the ALS laboratory for total lead and arsenic by U.S. EPA Method SW6010D. Additional composited sample will be held for toxicity characteristic leaching procedure (TCLP) extraction and testing for lead and arsenic to assist with determining the need for on-Site treatment (stabilization) prior to disposal, as applicable. Additional sample material from sample locations E2-3, C5-3 and B8-3 will be subjected to the following analyses to support off-Site disposal:

1. TCLP volatile organic compounds (VOCs);
2. TCLP semi-volatile organic compounds (SVOCs);
3. Ignitability by SW1030; and
4. pH by 9045C.

Laboratory analytical reports will be presented by ALS as Level IV data packages.

2.4 Laboratory QA/QC

Laboratory QA/QC will be performed pursuant to QAPP Worksheet #28 in the Final QAPP (Hull, 2020).

3.0 DATA EVALUATIONS

3.1 Decision Unit Composite Data

Upon receiving laboratory electronic data deliverable for composited total lead and arsenic, Hull will perform the following data evaluations:

1. In each of 46 of the 55 decision units (within which only one five-point composite sample was collected), the representative concentration of each analyte (i.e., lead and arsenic) in each decision unit will be the concentration as reported from the single five-point composite sample.

2. In each of nine of the 55 decision units (within which three five-point composite samples were collected), the representative concentration of each analyte (i.e., lead and arsenic) in each decision unit will be the arithmetic mean of the concentrations reported from the three five-point composite samples.

3. The representative concentration of each analyte in each decision unit will be compared to the Site-specific Decision Criterion Threshold Concentration (DCTC) for each analyte (i.e., lead and arsenic).

4. The Site-specific DCTC for each analyte will be based upon the Site-specific Decision Criterion (DC) for each analyte, adjusted to account for the 95% upper confidence interval (95% UCI) of the mean concentration, such that:

    a. The Site-specific DCTC for lead ($DCTC_{Pb}$) will be equal to the DC for lead (DC_{Pb}) minus the Site-specific 95% UCI for lead (95% UCI_{Pb}); and

    b. The Site-specific DCTC for arsenic ($DCTC_{As}$) will be equal to the DC for arsenic (DC_{As}) minus the Site-Specific 95% UCI for arsenic (95% UCI_{As}).

5. The Site-specific DC_{Pb} has been established as 800 mg/kg, based upon the US EPA Regional Screening Level for lead in industrial soil (May 2020)[1]; the Site-specific DC_{As} has been established as 26 mg/kg, the "upper-bound of the naturally-occurring arsenic concentration at OU1," calculated on the basis of the 95% upper tolerance limit the log-transformed data set of arsenic in native soils at the Site[2].

6. The 95% UCI will be calculated separately for each analyte, based upon the variance (mg/kg) in the concentration determined from the triplicate five-point composite samples collected in nine decision units, such that:

    a. The Site-specific variance in analyte concentrations will be calculated on the basis of the standard deviation (mg/kg) in the data set ($n = 27$, i.e., the reported concentrations reported from each of the three triplicate five-point composite samples in each of the nine decision units) for each analyte (SD_{Pb}, SD_{As})[3];

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[1] US EPA, Regional Screening Levels Generic Tables for Composite Worker Soil, value for Industrial Soil, Lead Compounds at HQ1 or HQ0.1, May 2020; available on-line at: https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables (accessed August 12, 2020).

[2] USS Lead, Remedial Investigation, Work Assignment No. 154-RICO-053J, June 2012, Section 8.4.2, pp. 184-186.

[3] U.S. EPA, 2020. Proposed USS Lead Zone 1 Sample Design, memorandum from John Canar, Chuck Roth and Tom Alcamo, April 30, 2020.

b. The 95% UCI for each analyte will be calculated using a standard factor for normally-distributed data sets (i.e., 1.706)[4], which when multiplied to the standard deviation for each analyte results in the 95% UCI for each analyte as follows:

   i. $95\%\ UCI_{Pb} = SD_{Pb} \times 1.706$; and
   ii. $95\%\ UCI_{As} = SD_{As} \times 1.706$.

c. In summary, the DCTC (mg/kg) for each analyte will be calculated as follows:

   i. $DCTC_{Pb} = DC_{Pb} - (SD_{Pb} \times 1.706)$; and
   ii. $DCTC_{As} = DC_{As} - (SD_{Pb} \times 1.706)$.

A report will be prepared containing a narrative of field efforts and any deviations from this Work Plan. The report will also present tabulated data, sampling locations, statistical methods used, decisions per decision unit (presented in both tabular form and on one or more maps) and recommendations based on the findings.

3.2 TCLP Lead and Arsenic Data

Upon determining which decision units will require excavation and off-Site disposal based on findings from evaluations described in section 3.1, Hull will direct ALS to conduct TCLP extraction and analysis of leachate for all retained samples collected from decision units requiring remediation. At locations where lead and/or arsenic exceed TCLP limits of 5.0 mg/L, Hull will determine the efficacy for stabilizing the materials on-Site to provide for transport a licensed disposal as a non-hazardous waste. It is anticipated that, at some point, treatability studies will be completed to facilitate an analysis of the efficacy of on-Site treatment. Hull will ultimately develop and submit to EPA a Remediation Work Plan that provides tabulated and map-referenced analytical data and for describes strategies for excavation, on-Site treatment and disposal of Site soils exceeding decision criteria.

3.3 Additional Disposal Characterization Data

In addition to TCLP lead and arsenic data described above in section 3.2, results for TCLP VOCs, TCLP SVOCs, ignitability and pH in soils collected from E2-3, C5-3 and B8-3 will be tabulated, retained and used to support a soil disposal profile for the licensed disposal facility that will receive excavated soils from planned remedial efforts at the Site.

3.4 Data Validation

Consistent to the Final QAPP (Hull, 2020), all laboratory analytical data generated pursuant to this Work Plan will be subjected to third party data validation.

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[4]. t-distribution value for one-tailed test, based on $t_{0.95}$, $\alpha = 0.05$, df = 26 (n = 27), from *t*-tables at: https://www.sjsu.edu/faculty/gerstman/StatPrimer/t-table.pdf (accessed August12, 2020).

4.0 REFERENCES

SurTRAC. *Remedial Investigation Report – US Smelter and Lead Refinery (USS Lead) Superfund Site, Lake County, Indiana.* June 2012.

Hull & Associates, LLC. *Final Quality Assurance Project Plan for the USS Lead Site, Operable Unit 1, Zone 1 Site, East Chicago, Lake County, Indiana.* September 2020.

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United States Environmental Protection Agency. *Unilateral Administrative Order for Remedial Action for Defined Properties in Operable Unit 1 of the U.S. Smelter and Lead Refinery, Inc., Superfund Site.* March 2019.

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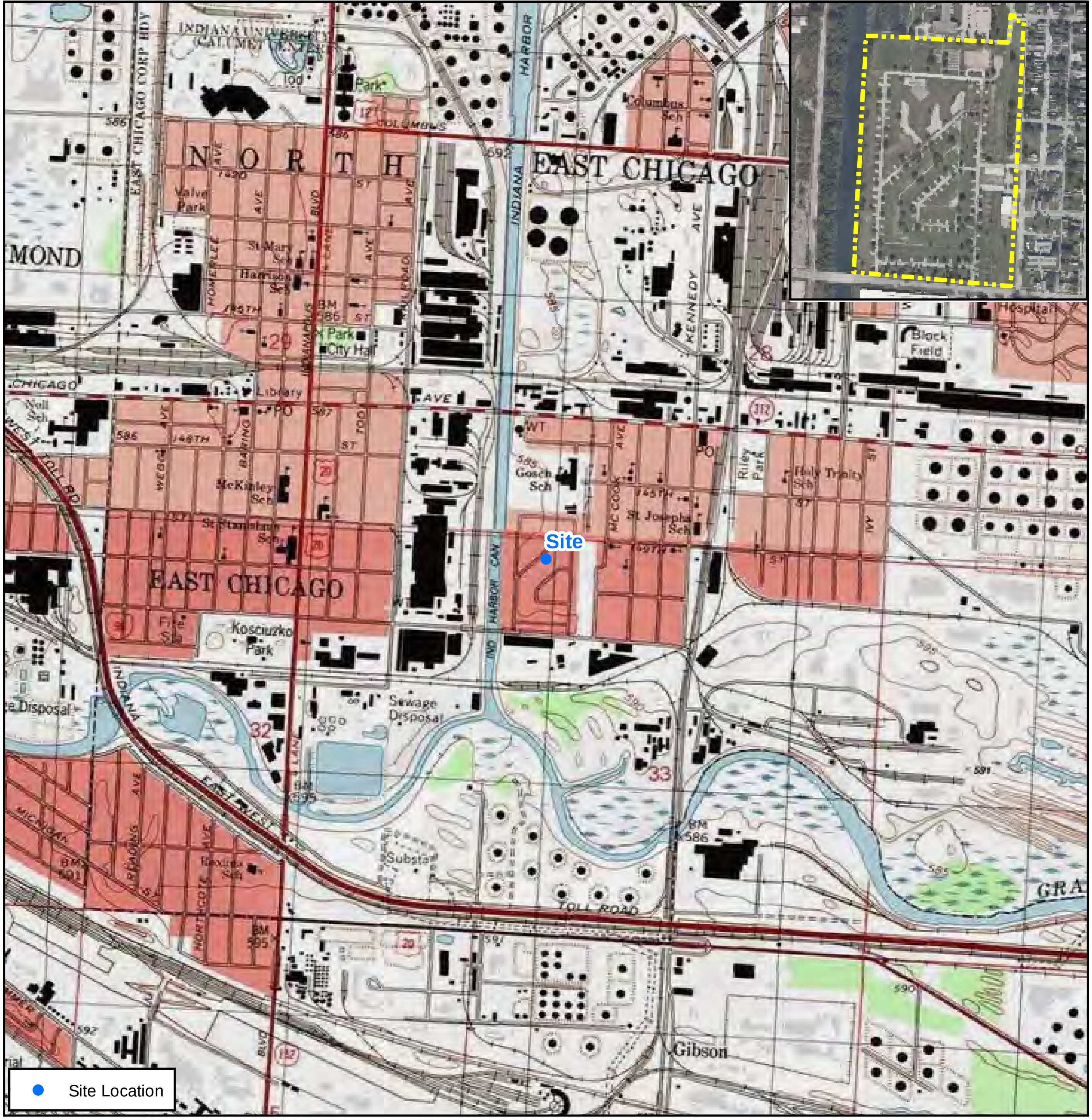
FIGURES



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Quad: Whiting and Highland

Source: The topographic map was acquired through the USGS Topographic Map web service.

The aerial photo was acquired through Bing Maps.
Aerial photography dated 2020.

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Work Plan for Decision Unit Soil Sampling
Zone 1 East Chicago
Industrial Development Advantage, LLC

Site Location Map

East Chicago, Lake County, Indiana

Date:
August 2020

File Name:
IDA001_12_Fig01_SLM.mxd

Edited: 8/11/2020 By: rkaip

Figure
1

Produced using ArcGIS 10.7

Note:
The aerial photo was acquired through Bing Maps.
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October 2020

Quality Assurance Project Plan
USS Lead Operable Unit 1 Zone 1
Industrial Development Advantage, LLC

Proposed Sampling Layout

East Chicago, Lake County, Indiana

Figure
2

Produced using ArcGIS 10.7

File Name: IDA001_13_Fig02_PropSamps.mxd

Edited: 10/29/2020 By: rkaip

APPENDICES

APPENDIX A

HULL SOP

HULL & ASSOCIATES, INC.
STANDARD OPERATING PROCEDURE

Title: Decontamination of Field Equipment

Effective Date: 1/22/14

Document Number: HULLSOP.F1000.R1

Author

Name: Monica Williamson
Title: Sr. Scientist

Signature: Date: 1/22/14

Approvals

Name: Bill Dennis
Title: Sr. Project Manager

WH Dennis III

Signature: Date: 1/22/14

REVISION HISTORY

The table below summarizes changes to this document over time. The most recent version is presented in the top row of the table. Previous versions of the document (if any) are maintained in the archive portion of the Hull SOP library.

History	Effective Date
HULLSOP.F1000.R1 – created in January 2014 to update the September 2013 version. Modifications to the previous version include: • Pre-installation decontamination of monitoring well materials; and • Specifying that potable water may be used in place of distilled water for final rinsing of all decontaminated equipment.	January 22, 2014
HULLSOP.F1000.R0 – created in September 2013 as part of overall reorganization of Hull's SOP program. While this version supercedes F1000-12REV, the content is substantially equivalent to the 2012 version; only minor editorial modifications were made for additional clarity, and the document name was changed for consistency with Hull's updated SOP nomenclature.	September 10, 2013
F1000-12REV – created in September 2012 to replace 1999 version; superceded in September 2013.	September 2012
F1000-99REV – created in 1999; superceded in September 2012.	1999 (month not recorded)

HULLSOP.F1000.R1
DECONTAMINATION OF FIELD EQUIPMENT

1.0 Purpose

This Hull & Associates, Inc. (Hull) Standard Operating Procedure (SOP) describes the minimum procedures that will be followed when decontaminating field equipment. Equipment may include soil sampling devices, bailers, trowels, shovels, hand augers, drilling rigs, or any other type of reusable equipment used during field investigations.

Decontamination will be performed as both a quality assurance measure and as a safety precaution. Specifically, the purpose for these decontamination procedures is to minimize the potential for cross contamination between sampling locations and prevent potentially contaminated materials from being transported off-site.

2.0 Equipment and Materials

Equipment and materials required for decontamination of field equipment may include, but will not necessarily be limited to:

- power-washer or high-pressure steam cleaner;
- cleaning fluids: non-phosphatic soap and/or detergents, potable water, distilled/deionized water; dilute nitric acid (as applicable);
- shovels and brushes;
- paper towels;
- disposable gloves;
- waste storage containers: plastic bags, drums, boxes;
- cleaning containers: plastic buckets, etc.;
- plastic sheeting; and
- personal protective equipment.

3.0 General

A. All decontamination will be performed under the assumption that the equipment is contaminated. At a minimum, clean, unused vinyl or nitrile gloves will be worn during all decontamination activities. Additional personal protective equipment will be worn as required by the site-specific health and safety plan.

B. An adequate supply of all decontamination equipment and materials will be available on site.

C. All equipment will be decontaminated before leaving the site.

D. Decontamination of vehicles or large equipment will generally be conducted in a designated area. Smaller equipment may be decontaminated near the sampling location.

E. All decontamination materials that cannot be re-used will be properly packaged for disposal based on the nature of contamination.

4.0 Procedures

The following sections present the minimum procedures that will be used to decontaminate field equipment. If different or more extensive procedures are required, they will be pre-approved by the Project Manager and/or Quality Assurance Officer, as appropriate.

4.1 Drilling Rig and Associated Equipment

A. Equipment coming in contact with potential contamination, both as part of subsurface equipment advancement and aboveground contact with drilling fluids, extracted soils, ground water, drill rig lubricants and fuels, etc., will be decontaminated prior to use. At the discretion of the Project Manager, decontamination of the entire drilling rig may be required due to the adherence of foreign substances as a result of operations, transportation from off-site, or travel between soil boring locations.

B. A high-pressure steam cleaner will be used to clean the inside and outside of drilling equipment that will potentially come into contact with test samples. Decontamination of sampling equipment (e.g., split-spoon samplers) is described in section 4.2.

C. All liquid and solid material produced from this operation will be collected and properly contained until such time as it can be properly disposed.

D. The date, time, and decontamination procedure used will be recorded on the boring log, daily field report or in a field notebook, as appropriate.

4.2 Sampling Equipment (split spoons, trowels, etc.)

Sampling equipment will be decontaminated between sample locations and sample intervals to minimize the potential for cross-contamination.

A. The sampler will be completely disassembled and any adhered soil will be removed.

B. The sampler will be placed in a bucket containing a non-phosphatic soap and water (e.g., *Liquinox*™) and scrubbed until visibly clean. The soap and water will be changed as necessary.

C. The sampler will then be thoroughly rinsed with potable water until all soap solution is removed. All rinse water will be collected and containerized.

C. As required by the site-specific work plan, the sampling equipment may be rinsed with a dilute nitric acid solution if metals are analytes of interest.

D. The sampler will be reassembled and given a final rinse with potable water.

E. If the sampler is not to be used immediately, it must be stored in a location or manner that will prevent it from becoming re-contaminated.

4.3 Groundwater Pumps

This procedure will be employed to decontaminate the non-dedicated pumps that are used during well purging, development, and sampling operations.

A. Any dedicated tubing that was used with the pump will be removed and properly discarded.

B. All exterior surfaces will be wiped with clean paper towels and any extraneous materials will be removed using a stiff brush.

C. The pump and all associated downhole equipment will be placed in a suitably sized container of non-phosphatic soap (e.g., *Liquinox*™) and potable water. If the tubing on the pump is to be re-used, the pump will be turned on to circulate the solution through the pump and tubing.

D. The pump will then be thoroughly rinsed with potable water. If the tubing on the pump is to be reused then the pump will be turned on until the internal portions of the pump and tubing are free of cleaning solution. The last rinse applied to the pump system will always be potable water.

F. The pump and associated downhole equipment will be properly stored to ensure that the system remains clean during transportation to other well heads. The pump will not be allowed to come in contact with the ground at any time during handling and transportation. If this occurs, the pump and associated downhole equipment will be re-cleaned.

G. All liquids and waste materials produced during this operation will be properly stored and disposed of as determined by the Project Manager.

4.4 Bailers

Hull's default/preferred procedure is to employ clean, single-use, disposable bailers such that decontamination is not required. In the event that reusable bailers are employed, they will be decontaminated as described below.

A. The bailer will be scrubbed with non-phosphatic soap and water solution. The inside of the bailer will be scrubbed with a cylinder brush to ensure that interior walls are thoroughly cleaned.

B. The bailer will be rinsed with potable water until it is free of the soap solution.

C. As required by the site-specific work plan, the bailer may be rinsed with a dilute nitric acid solution if metals are analytes of interest.

D. A final rinse of potable water will then be applied.

E The bailer will be properly stored if it is not to be immediately used. For proper storage, the entire bailer will be placed in its dedicated storage tube or wrapped in inert material (e.g., *Saran* wrap, aluminum foil, etc.).

F. All liquids and waste materials produced during this operation will be properly stored and disposed of as determined by the Project Manager.

4.5 Well Casing and Screen Pre-Installation Decontamination Procedures

All polyvinyl chloride (PVC) casing and screen materials contained in clean, sealed packaging direct from the manufacturer may be constructed by personnel wearing clean and unused vinyl or nitrile gloves and directly installed. In the event that visual inspection indicates torn packaging or the potential for contamination of well materials, a power washer or high pressure steam cleaner should be used to clean the material prior to assembly and installation. All wells consisting of Type 304 stainless steel should be cleaned with a high pressure steam cleaner prior to assembly and installation.

4.6 Interface Probe and Water Level Indicator

The entire length of the probe and tape that was inserted into the well will be decontaminated by washing with a non-phosphate detergent (e.g., *Liquinox*™) and then rinsing with potable water.

5.0 Documentation

The procedure(s) employed, date(s), and time(s) will be recorded on the appropriate documentation (e.g., daily field reports, field notebooks, boring logs, etc.). Deviations must be approved by the Project Manager and/or Quality Assurance Officer and documented in the field notebook or field logs.

6.0 Special Notes

None

HULL & ASSOCIATES, INC.
STANDARD OPERATING PROCEDURE

Title: Surface and Subsurface Soil Sampling

Effective Date: 11/4/13

Document Number: HULLSOP.F3029.R0

Author

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Signature:
Date: 11/4/13

Approvals

Name: W. Lance Turley
Title: Principal/Sr. Consultant

Signature: W. Lance Turley
Date: 11/4/13

HULL & ASSOCIATES, INC. i SEPTEMBER 2013
STANDARD OPERATING PROCEDURE HULLSOP.F3029.R0

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REVISION HISTORY

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History	Effective Date
HULLSOP.F3029.R0 – created in November 2013 as part of overall reorganization of Hull's SOP program. While this version supercedes F3029, the content is substantially equivalent to the 2012 version; only minor editorial modifications were made, and the document name was changed for consistency with Hull's updated SOP nomenclature.	November 4, 2013
F3029 – created in September 2012; superceded in November 2013. Created to combine a number of soil sampling-related SOPs into a comprehensive procedure. The revisions to the previously-existing documents were relatively extensive and are not individually enumerated herein.	September 2012
F2026-11REV – Use of Sonic Drilling Techniques for Soil and Bedrock Sampling and Monitoring Device Installation – created in 2011; superceded in September 2012.	2011 (month not recorded)
F2021-10REV – Soil and Groundwater Sampling Using Direct Push Techniques – created in 2010; superceded in September 2012.	2010 (month not recorded)
F3001 – Subsurface Soil Sampling Utilizing a Split Spoon Sampler – created in 1995; superceded in September 2012.	1995 (month not recorded)
F3001 – Subsurface Soil Sampling Utilizing a Thin-Walled Shelby Tube – created in 1995; superceded in September 2012.	1995 (month not recorded)

HULLSOP.F3029.R0
SURFACE AND SUBSURFACE SOIL SAMPLING

1.0 Purpose

The purpose of this Hull & Associates, Inc. (Hull) Standard Operating Procedure (SOP) is to describe the procedures and methods to be used for collection of soil samples for field screening, laboratory analysis or geotechnical characterization.

Environmental soil samples can be collected using manual and/or mechanical methods including the following:

1. Manual collection methods (such as hand auger systems, sampling trowels, surface grab sampling, spatulas, and core samplers);
2. Earth moving equipment such as backhoes and excavators;
3. Direct-push equipment; and
4. Drilling equipment.

The specific procedures for each activity should be developed on a site-specific basis, taking into consideration the data quality objectives of the project.

2.0 SOP-specific Terminology

Any language or specific terms related to this SOP are defined as they are presented within the procedure.

3.0 Sample Types

There are two main types of soil sampling: grab and composite. The type of sample to be collected is determined by the intent of the testing (i.e., spatial sampling, waste profiling/characterization, etc.) and characteristics of the study area such as the soil type, depth to saturation and project area size.

Grab samples are most commonly used to collect a discrete, representative soil sample to characterize the physical and/or environmental conditions of the soil at that particular location and sample depth. Grab sampling is the recommended soil sampling type for most projects and circumstances. Soil sample types should be verified with the Project Manager (PM), with support from any overseeing regulatory agencies. Sample types should also be specifically stated within the project-specific Scope of Work.

4.0 Special Considerations

4.1 Sampling for Volatile Organic Compounds (VOCs)

If it is anticipated the soil sample collected will be analyzed for VOCs, the use of a Terra Core™ field preservation kit is highly recommended. These kits use a methanol and/or sodium bisulfate preservation liquid to preserve these samples for a period ranging from 48 hours to 14 days, depending on the preservative, kit type and laboratory. Sampling for VOCs in soil using a glass, laboratory-supplied jar is an alternative method for soil sampling, but permission from the PM and regulatory agency should be granted prior to sample collection. The only exceptions for VOC sampling with jars is for waste characterization, Synthetic Precipitation Leaching Procedure SPLP testing, certain bench testing procedures for treatment technologies or composite sampling, which are both explained later in this SOP.

4.2 Sampling Equipment Determination

Care should be taken to select equipment that is consistent with the data quality objectives for the sampling. Stainless steel or *Teflon* equipment may be used for all analytes. Plastic and rubber devices should generally be avoided and specifically not used for VOC analyses unless they are single-use, disposable devices and will only be used at one specific sampling location. The selection of mechanical-based sampling equipment, such as drill rigs, direct push rigs or excavators, should be based on both the project area and project type.

4.3 General Decontamination Procedures

The decontamination process will be repeated after each use and between all discrete sample locations. If compositing strategies are used, decontamination may only be required between composite samples (i.e., not between discrete samples that form a single composite). Sample gloves shall be changed in between each location. Specific decontamination procedures for all field-related activities as specified in Hull's current SOP for equipment decontamination should be followed at all times.

5.0 Health and Safety

Proper safety precautions must be observed when collecting soil samples. Field personnel are responsible for reviewing the site-specific Health and Safety Plan (HASP) for guidelines on safety precautions. The appropriate level of personal protection equipment (PPE) must be worn at all times during sampling, as defined in the site-specific HASP. In addition, when applicable, utilities located within the study area should be clearly marked prior to the implementation of field activities.

6.0 Equipment and Materials

Depending on the sampling type and technique used, the following equipment may be needed:

1. Disposable nitrile sampling gloves;
2. Laboratory-supplied sample containers, which may include En Core®, Terra Core™ or equivalent kits (these typically come with a single-use, disposable, pre-calibrated sample coring device);
3. Photoionization Detector (PID) equipped with an appropriate bulb (10.6, 11.7 or 11.8 eV);
4. Flame Ionization Detector (FID) equipped with hydrogen gas;
5. PID/FID calibration gas;
6. 1 qt. re-sealable bag such as Zip-loc® or equivalent for field screening;

7. Stainless steel or disposable sampling spoon, trowel, or scoop (cleaned, and not plated or painted);
8. Stainless steel mixing bowl, *Teflon* tray or re-sealable bag;
9. Measuring tape;
10. GPS Unit;
11. Utility knife (for opening liners; when possible, always have subcontractor cut liners);
12. Stainless steel sampling knives/putty knife;
13. Decontamination materials including, but not limited to clean potable and distilled water, steam cleaner, buckets, brushes, and non-phosphatic detergent such as *Alconox*® or equivalent (refer to Hull's current SOP for equipment decontamination);
14. Paper towels;
15. Waste storage containers (i.e., bags, drums, boxes, buckets, etc.);
16. Plastic sheeting/*Visqueen*;
17. Coolers;
18. Ice;
19. Chain of Custody records, Custody seals;
20. Hull Soil Boring/Test Pit Logs; and
21. Logbook/Waterproof Pen.

7.0 Procedures

7.1 Manual Soil Sampling

These procedures will be followed when collecting unsaturated surface or shallow subsurface soil via manual sampling techniques.

7.1.1 Stainless Steel Spoons

Spoons are appropriate for soft soil samples collected within six inches of the surface. Stainless steel spoons may also be used in conjunction with other methods of soil sampling (e.g., transferring of soil to a sample jar, compositing of soils). When using a stainless steel spoon to collect a shallow soil sample, a trowel, spade or shovel can be used to remove the soil from the ground. The stainless steel spoon can then be used to collect the sample chosen for submittal, taking care not to contact the spoon directly with the trowel, spade or shovel. When collecting a composite sample, a trowel, spade or shovel can be used to remove the soil from the ground from each location that will be composited. Once all of the soil has been combined into the stainless steel bowl, *Teflon* tray, resealable bag or equivalent, the spoon can then be used to mix and transfer the soil into the sample container. It is not recommended that composite samples be analyzed for VOCs.

7.1.2 Hand Augers

A hand auger is an appropriate sampling device that is often used for the collection of shallow soil samples (usually up to 5 feet). A hand auger consists of a stainless steel tube with angled teeth on the end that attaches to stainless steel rods and a handle. The auger portion, often called a bucket, is typically 2.5 to 4 inches in diameter and advances into the soil by twisting the handle. The bucket fills with soil as the tool is advanced. This method is most appropriate for the collection of near-surface samples; however, hand auger systems have rod extensions that can be used to advance the auger deeper into the subsurface if the soil is soft enough. A hand auger is also an appropriate tool to use if underground utilities in the area of the desired sampling location are unknown, are too close to the desired sampling location, or if hand clearance is required at the subject site.

When sampling with a hand auger, all soils removed from the hole should be contained, unless approved by the PM. Once the desired depth interval has been reached by the hand auger (should confirm with a tape measure), the auger should be extracted from the ground and a small amount of soil placed into a clean, resealable plastic bag for headspace screening as discussed in Section 7.7 below. The remainder of the soil within the bucket should then be used to fill the appropriate sample containers for the sample location. Should VOCs be analyzed and a 5035 kit be used, soil for the kit should be collected directly from the sampling device, and in this case, from the hand auger.

When utilizing manual soil sampling methods, decontamination procedures following Hull's current SOP for equipment decontamination should be completed in between each sampling location to minimize the potential for cross-contamination.

7.2 Soil Sampling by Use of Test Pits

These procedures will be followed for the safe and effective collection of soil samples and/or visual inspection of near surface soil by use of a backhoe, excavator, or equivalent heavy machinery. Shallow subsurface excavations allow for sidewall and floor conditions to be analyzed while removing potentially contaminated soil. The primary hazard of trenching and excavation is employee injury from collapse. Hull field personnel are **never** permitted to enter a trench or excavation that is greater than five feet deep, unless appropriate shoring and/or benching has been conducted and inspected by an Occupational Safety and Health Administration (OSHA) competent person and the Hull personnel has been given permission by the PM and Hull Health and Safety Officer. **Current OSHA trench and excavation regulations will be followed by all Hull licensed subcontractors and Hull field personnel.**

7.2.1 Discrete/Composite Test Pit Soil Sampling

Using a backhoe, excavator or equivalent, soils should be excavated and stockpiled to the depth specified by the PM. Once the desired depth interval is reached, the soil sample should be collected directly from the bucket of the excavator, taking care not to contact the sample container with the bucket itself. A small amount of soil should also be placed into a clean resealable bag for headspace screening. If soils are to be field-screened and collected at numerous depths, new sampling gloves and resealable baggies should be used at each different sampling interval. If a composite sample is desired, samples collected from the bucket of the excavator can be combined, mixed and sampled as outlined in Section 7.2.1 above.

7.2.2 Remedial Excavation Test Pit Confirmatory Soil Sampling

When remedial excavations or tank removals are conducted on a job site, a backhoe, excavator or other heavy machinery is usually used to remove the soil and/or tank. In this case, excavation sidewall samples and/or floor samples are usually collected by use of a bucket. There are no differences from the sampling techniques described above in Section 7.2.1, with the exception that numerous samples can be collected within one test pit and/or excavation. Using input from the PM, care should be taken to document sample identifications including depth and spatial location, so that sample locations can be easily replicated.

7.3 Soil Sampling Using Direct Push Method

This section documents the procedures to advance direct push samplers (i.e., Geoprobe™) in unconsolidated material to collect soil samples for chemical and geotechnical analyses. There are two primary methods for soil sampling using direct push methods; dual tube and macrocore, both of which are discussed below. Factors to consider when choosing a direct push sampling method include the following:

1. The known or potential presence of shallow soil or groundwater contaminants which could cross-contaminate underlying soil or groundwater samples, especially in swelling soils;

2. The known or potential presence of multiple saturated zones in the soil interval to be investigated;

3. The known or potential presence of a confined groundwater zone, which, when penetrated, could result in the rise of groundwater in the boring and the contact of uncontaminated groundwater and overlying contaminated soils or the contact of contaminated groundwater and overlying uncontaminated soils;

4. Appropriate regulatory agencies should be consulted before using direct-push technology to collect soil or groundwater samples for chemical analyses because of potential problems with sample integrity. In addition, chemical analytical results from soil or groundwater samples collected using direct-push technology may not be acceptable to certain regulatory agencies;

5. Certain site characteristics and circumstances may preclude the use of direct-push technology. These characteristics and circumstances include shallow soils with coarse gravel, cobbles or boulders which may be difficult to penetrate; loose, saturated granular soils or soft, wet clayey

soils which are not retained well by the sampler (i.e., very poor sample recovery); access to specific sampling locations; and required sampling depths which may exceed the practical use of direct-push equipment, which is generally in the range of twenty to thirty feet in dense clay-rich soil; and

6. The amount of soil required for submittal to the laboratory will be dependent on the potential chemicals of concern and/or the geotechnical analyses required as part of the investigation. Typically, dual tube samplers do not provide as much soil for sample collection as a macrocore sampler.

7.3.1 Dual Tube Sampling Method

Using direct push equipment equipped with dual tube technology is the preferred method when using direct push methods. This system allows soil sampling to progress while simultaneously casing off the borehole. This prevents cave-in and reduces the potential to drag down potential contaminants from upper zones. When using the dual tube system, the drilling subcontractor will provide the direct push equipment, dual tube sample collection rods with disposable acetate liners, drive rods and a utility knife for liner cutting. The following procedures should be followed by the subcontractor for sample collection:

1. A clean acetate liner is inserted into the decontaminated stainless steel sampler;

2. The sampler is then attached to the drive head on the direct-push drilling rig and is hydraulically pushed/driven to the desired depth (generally, the samplers are pushed in two, four or five-foot intervals);

3. The drive rods on the inside of the outer roads, which are attached to the acetate sample liner, are then pulled from the boring using the direct push rig. On occasion, especially when drilling through fill material or granular soils, sand or fine gravel particles may become wedged between the outside of the liner and the inside of the stainless steel sampler, making removal of the acetate liner difficult. If this occurs, the acetate liner should be removed from the sampler with pliers. Attempting to push the acetate liner and sample out of the macro-core with a drive rod or other object may deform or cross-contaminate the soil sample;

4. The acetate liners shall be opened by placing them on a level and stable surface (usually the bed of a pickup truck or a table), and cutting them lengthwise with a utility knife or equivalent. Two cuts approximately 180° apart should be made so that the upper half of the liner can be removed for logging and soil sampling and the

lower half of the liner remains in place to protect the soil sample from cross-contamination with the stable surface.

A decontaminated sampler with a new acetate liner shall be used for each consecutive sampler; decontamination of the drive rods between samples is not necessary. Most samplers are equipped with a tip which helps prevent the introduction of material from the sides of the open boring, caved materials, or heaved materials into the sampler. The tip is sometimes referred to as a "piston tip" and is usually retrievable (the tip is pushed to the top of the inside of the acetate liner as the sample is collected). The tip shall be used for each sample interval except for the initial (ground surface) sample. Please note that use of the tips will not necessarily prevent the inclusion of caved or heaved soil at the top of the samples, so careful inspection and description of the soil sample is very important!

7.3.2 Macrocore Sampling Method

Use of the macrocore sampling method with direct push technology is preferred when installing shallow soil borings (up to 4-5 feet) where the sampler only needs to be installed in the borehole one time. Additionally, the macrocore sampling method can also be used when the soil type is non-granular and consists primarily of unsaturated clays and silts. However, soil type should never be assumed on a site; this information should be based on previously existing documentation. It is imperative that the borehole remain open if using the macrocore sampling method, as there is no equipment remaining in the ground in between sampling intervals to keep the hole open.

Sampling procedures using the macrocore method are the same as listed above for the dual tube method. Typically, the acetate liner diameter is slightly larger with a macrocore; therefore, recovery is better and the amount of soil removed is greater.

Following installation of the soil boring using the dual tube or macrocore sampling method, the soils should be logged appropriately using the methods outlined in HULLSOP.F1006.R0 (or current version). After each direct-push sample is retrieved, the total depth of the boring should be checked with a water level indicator or an interface probe. Measuring the total depth will evaluate the amount of material that may have fallen into the hole and help to determine what portion of the next sample is to be considered *in-situ* soil. Typically, issues with caving are mitigated through the use of the dual tube system.

Before logging the soil, the entire sample should be split lengthwise with a decontaminated stainless steel soil knife or equivalent to evaluate what portion of the sample may be caved materials or boring heave. After logging the soil, place a representative portion of the soil (typically in two-foot intervals) into a clearly labeled clean re-sealable bag (such as *Zip-loc* or equivalent) for headspace screening. Replace the top portion of the acetate liner to minimize volatilization of the remaining soil sample. Sampling intervals may be predetermined or can be based on elevated soil headspace screening or visual impact. Once the desired sampling interval is determined, distribute the soil into appropriate laboratory supplied sampling containers. When duplicate samples are to be collected, an equal amount of sample will be collected from each of the “split” samples. Samples will be collected in order of volatility (greatest to least). VOCs in soil will be collected in accordance with Section 7.6 of this SOP.

If an obstruction is encountered which cannot be penetrated during drilling, the direct-push boring will be decommissioned as described in Section 9.0 below.

7.4 Soil Sampling Using Rotary Drilling Methods

This section documents the procedures for using a rotary drill rig equipped with hollow-stem augers to advance a collection device (i.e., split spoon) which may be used to collect surface and subsurface soil samples. Although a standard practice for geotechnical sampling, Hull does not recommend the use of solid stem augers for environmental sampling, unless prior approval has been granted by the PM. The driller shall provide a drilling rig capable of use with hollow-stem augers appropriate for the tasks requested by the PM. The inside diameter (I.D.) of the hollow-stem augers will be specified in the work plan or Scope of Work. The following equipment will be on-site and provided by the drilling contractor.

1. drilling rig with appropriate drilling capacity; drill rods, minimum size equivalent to the "A" rod, with an outside diameter (O.D.) of 1-5/8 inches and an I.D. of 1-1/8 inches;
2. hollow-stem augers;
3. hollow-stem auger center plug;
4. hydraulic drive hammer, ~140 pounds (+ 5 pounds);
5. 18- or 24-inch-long split-spoon barrel samplers or five-foot length continuous samplers;
6. 24- or 30-in-long thin-walled Shelby tubes;
7. water tank and pump;
8. Decontamination equipment including, but not limited to a steam cleaner, 5-gallon buckets, brushes, and nonphosphate detergent;
9. Decommisioning materials including sodium-bentonite slurry or cement/bentonite grout; and
10. Concrete and/or asphalt.

The boring will be installed by rotating the hollow-stem augers to the desired depth to collect soil samples. While drilling, the hollow-stem auger center plug and drill rods will be inserted in the auger stem to minimize soil cuttings from entering the augers. Waste soil/overburden material from drilling will be properly contained and disposed of unless otherwise directed by the PM.

The driller will be prepared to minimize the effects of heaving sands during drilling operations by using auger finger baskets in place of a center plug to minimize heave into the augers, or by removing heaving sand from the augers with a steel bailer. Potable water can be added to the augers to control heave only if approved by the PM. Any other methods for removing or controlling heaving sands should also be approved by the PM.

If an obstruction is encountered, the driller will attempt to penetrate the obstruction using a tri-cone roller bit, coring device, or other acceptable equipment as directed by the PM. If attempts to penetrate the obstruction are unsuccessful, the boring will be decommissioned in accordance with HULLSOP.F2022.R0 (or current version). Depending on the depth of the obstruction, the boring may be reinstalled at a known or inferred upgradient location.

Hollow-stem augering should always be employed **without** the use of drilling fluids unless approved by the PM. When water is used in the drilling operations, it will not be recirculated into the boring unless approved by the PM. Water produced from this operation will be properly contained and disposed of as directed by the PM.

There are three main types of collection devices when using rotary drilling methods equipped with hollow-stem augers, as discussed below.

7.4.1 Split Spoon Sampling

Once the desired depth has been reached using hollow-stem augers equipped with a center plug, the plug and drive rods are removed and a clean decontaminated split spoon sampler (18-inch or 24-inch) is attached to the drive rods. Typically, a cable winch system is used to lower the sampler and drive roads to the base of the augers. Once the sampler and drive rods are in place, the sampler is driven to the desired bottom depth using a hydraulic drive hammer (generally, the samplers are pushed in two-foot intervals, recording consecutive blow counts every six inches). Older rigs occasionally utilize a cathead hammer system with a rope. Prior approval should be granted from the PM prior to using a cathead system due to variances in hammer weight and safety concerns.

After the sampler is pulled from the boring using the drilling rig, the split spoons shall be opened by placing them on a level and stable surface (usually the bed of a pickup truck or table), and opened using a pipe wrench. Once one side of the split spoon sampler is removed, the other side should maintain its position between the soil and the level surface to prevent the soil sample from cross-contamination. A decontaminated sampler shall be used for each consecutive sample; decontamination of the drive rods between samples is not necessary.

7.4.2 Shelby Tube Sampling

In circumstances where grain size analysis or permeability analysis of soils are desired, a thin-walled Shelby tube may be driven. Shelby tubes are installed and driven in a manner similar to that described above for a split spoon, although typically, the drive head is lowered slowly to advance the Shelby tube, instead of using the hydraulic hammer. This is to avoid damaging the sampler. Blow counts should be analyzed prior to attempting the installation of a Shelby tube. If the material is very hard or contains a high gravel content, the tube may be crushed or damaged during installation. Once the Shelby tube has been driven to the desired bottom depth, the tube is removed in the same manner as a split spoon using the drill rig. Typically, in order to preserve the sample, once the Shelby tube has been removed, the ends of the tube are dipped in wax and the ends are capped with plastic caps.

7.4.3 Continuous Macrocore Sampling

Occasionally, a macrocore system can be attached the end of the drive rods and driven by the use of the hydraulic hammer. These macrocores are typically four or five feet long. Upon removal, these soils should be logged and sampled in a manner consistent with that described above in Section 7.3.2.

Similar to that described above in Section 7.3, soil logging procedures, consistent with HULLSOP.F1006.R0 (or current version) should always be followed, keeping in mind that blow counts need to be logged using this method.

7.5 Soil Sampling Using Sonic Drilling Methods

This section documents the procedures for sonic drilling techniques for environmental and geotechnical soil and rock sampling and installing various subsurface monitoring points including: groundwater monitoring wells; piezometers; groundwater extraction wells; soil vapor monitoring wells and soil vapor extraction wells. Sonic drilling can be used in both unconsolidated and consolidated formations. Sonic drilling uses high frequency vibration aided by down pressure and rotation to advance drilling tools and sampling devices through various geologic formations. The advantages of sonic methods over other methods include:

1. reduced soil cuttings and fluid production, thus reducing disposal costs;
2. rapid formation penetration reducing drilling time;
3. continuous core collection improving lithologic interpretation;
4. reduces casing bridging and lock up;
5. reduces well development time; and
6. rapid ability to telescopically case off formations.

The driller shall provide a sonic drilling rig properly equipped for the tasks requested by the PM. The sample core and casing diameters will be specified in the work plan or Scope of Work. The following equipment will be on-site and provided by the drilling contractor:

1. sonic drilling rig with appropriate drilling capacity;
2. drill rods that are used to propel and recover the sample barrels (typically in 2.0 and 4.0-inch O.D. and lengths of 2.0, 5.0, 10.0 and 20.0 feet);
3. casing with diameters that can range from 0.5 to 14 inches depending on project requirements;
4. sample/core barrels that can be either solid tubes or split barrels of various diameters and lengths to match the drill casing. The barrel is fitted with a drill bit or cutting shoe that holds the borehole alignment as it

passes through the outer casing into the formation. Sample barrels are typically in 5, 10 or 20-foot lengths;
5. water tank and pump;
6. if potable water is used during drilling, containment bins must be used to collect drilling fluids for appropriate disposal; and
7. steam cleaner, 5-gallon buckets, brushes, and non-phosphate detergent.

Occasionally, sonic drills can be modified to use split spoon samplers or collect thin-walled Shelby tubes for geotechnical applications. The use of a sonic rig for geotechnical applications should only be done under the approval of a PM and after an inspection of the rig. In these rare cases, the sonic rig would need to be equipped with:

1. drive hammer, 140 pounds (+ 5 pounds);
2. 18- or 24- inch-long split-spoon barrel samplers; and
3. thin-walled Shelby tubes.

7.5.1 Core Barrel Sampling

The boring will be installed by advancing the sonic tooling to the desired depth to collect soil samples or install a well, piezometer, or probe. While drilling, care should be taken to keep the outer casing in place and reduce the potential for the borehole caving or slough. Waste cuttings and samples from drilling will be properly disposed of as directed by the PM. Soil samples will be collected by advancing the sample barrel ahead of the casing. The casing is driven to the top of the desired sampling interval and the sample barrel (5-foot, 10-foot or 20-foot) is advanced through the casing into undisturbed soil/overburden material. Once the sample barrel has been advanced to the desired depth the outer casing is then advanced to the same depth. This process is repeated until the target depth is achieved. The inner sampling barrel will then be removed from the ground with the outer casing left in place. Soils are typically removed from the barrel using a reverse vibration. Soils are extracted from the barrel into clean, plastic sleeves and placed on a flat surface. The bags are then cut open and logged, following the same procedures outlined in Sections 7.3 and 7.4 above, maintaining consistency with HULLSOP.F1006.R0 (or current version). Alternatively, split spoon samples can be used in accordance with ASTM International (ASTM) D1586, and Shelby tube samples can be collected in accordance with ASTM D1587, but only with a modified sonic rig and PM approval.

If an obstruction is encountered, the driller will attempt to penetrate the obstruction using the appropriate coring device, or other acceptable equipment as directed by the project manager. If attempts to penetrate the obstruction are unsuccessful, the boring will be decommissioned in accordance with HULLSOP.F2022.R0 (or current version), and, if possible, reinstalled at an assumed or inferred upgradient location.

Sonic drilling will be employed without the use of drilling fluids unless compatible with the study data quality objectives and approved by the PM. If water is used in the drilling operations, it will not be recirculated into the boring unless approved by the PM. Water produced from this operation will be properly contained and disposed of as directed by the PM. If a well, piezometer or probe is not installed, each boring will be decommissioned in accordance with Section 9.0 of this SOP.

7.6 Soil Sampling for USEPA SW-846 Method 5035

This section details the procedures to be followed when collecting soil samples for United States Environmental Protection Agency (U.S. EPA) SW-846 Method 5035. These procedures are applicable for soil samples requiring volatile organic analyses (VOA) by U.S. EPA SW-846 Update III methods (or subsequent revisions). Specifically, these procedures will be performed for all VOA analyses requiring preparation by SW-846 Method 5035 [e.g., 8260, 8021, and 8015-Gasoline Range Organics (GRO)]. Samples may either be collected in the field using En Core® samplers (or equivalent) and subsequently preserved at the laboratory or the samples may be collected and preserved in the field in a Terra Core™ or equivalent kit.

The following equipment and materials should be used when collecting samples for Method 5035:

1. Disposable nitrile sample gloves; and
2. Decontamination equipment and supplies.

Option 1 – field collection with subsequent laboratory preservation

1. 5 or 25 gram disposable En Core® Samplers; and
2. En Core® T-Handle.

Option 2 – field preservation

1. Field preservation kits (e.g., Terra Core™ or equivalent) consisting of either pre-weighed and pre-preserved sample vials (occasionally, labs send pre-weighed vial and an aliquot of methanol to add to the vial); and
2. Single-use, pre-calibrated sample coring device.

The following procedure should be followed when collecting samples for Method 5035 using an En Core® system:

1. Put on sample gloves and remove En Core® Sampler from the zipper bag or containment device. Keep the zipper bag for storing the sampler after soil sample collection;

2. Ensure that the plunger on the sampler moves freely by holding the coring body and pushing the plunger rod down until the small o-ring rests against the tabs;

3. Depress the locking lever on the En Core$^{®}$ T-Handle and place the sampler into the open end of the T-handle (plunger end first). When inserting the sampler, make sure the slots in the coring body are aligned with the locking pins in the T-handle. Lock the sampler in place by twisting the coring body clockwise until the pins lock in the slots;

4. Turn the assembly upright (T-handle up and coring body down) to ensure that the plunger bottom is flush with the bottom of the coring body;

5. Push sampler into soil until coring body is completely full. The small o-ring will be centered in the T-Handle viewing hole when the sampler is full;

6. Remove the sampler from the soil and wipe any excess soil from the coring body;

7. Cap the sampler while it is still attached to the T-handle. Push the cap over the flat areas of the ridge on the coring body and twist to lock the cap in place;

8. Separate the sampler from the T-handle by depressing the handle’s locking lever while twisting and pulling the sampler;

9. Lock the plunger in place by rotating the extended plunger rod counterclockwise until the wings rest firmly against the tabs on the coring body;

10. Complete and attach the small tear-away sample label (located on the zipper bag) to the cap on the coring body and return the sampler to the zipper bag. Seal the bag and complete the large sample label on the outside of the zipper bag;

11. Place the sealed zipper bags inside an iced cooler;

12. A bulk soil sample must also be submitted for dry weight determination. This sample will be collected in a small glass or plastic container supplied by the laboratory. Note: If the sample location will also be analyzed for non-volatile parameters [i.e., polyaromatic hydrocarbons (PAHs), metals, etc.] then no additional sample volume is required for dry weight determination; and

13. **Note: Unpreserved samples must be shipped to the laboratory the day they are collected as there is a 48-hour holding time for preservation. The laboratory must be notified in advance that they will be receiving samples requiring preservation.**

Keep in mind that when using En Core$^{®}$ samplers, a minimum of three samples are required for analysis. Additional samples may also be required for laboratory matrix spikes/matrix spike duplicates (MS/MSD) as defined by the data quality objectives for the project. Additional sample volume must be submitted for the purposes of determining dry weight. If other analyses are to be run by the laboratory through the analysis of other sample containers from the same interval, then additional soil is not needed.

The following procedure should be followed when collecting samples for Method 5035 using a Terra Core™, or equivalent field preservation kit:

1. Put on sample gloves and obtain a complete sample kit (Terra Core™ or equivalent). A sample kit will consist of the following:

   i. One vial preserved (may come pre-preserved or may require preservation in the field) with methanol for high level analysis (depending on the laboratory, this vial may be amber or clear, but should be labeled with the preservative);
   ii. Two clear vials pre-preserved with sodium bisulfate for low level analysis (occasionally, these may be preserved with deionized water instead); and
   iii. One unpreserved clear vial for evaporative loss and dry weight analysis.

2. Remove the caps from the four vials;

3. With the single use 5-gram pre-calibrated coring device supplied with the kit, collect a full core of soil and transfer to each vial by depressing the plunger;

4. In the case of Terra Core™ kits, all preservatives should already be contained in the included vials. In the event an equivalent kit is used and the methanol is not already included in the vial, transfer the pre-measured 5 milliliter (mL) aliquot of methanol into the appropriate pre-marked vial;

5. Tightly cap all the vials;

6. Complete the sample label on the kit; and

7. Place the kit upright into an iced cooler.

7.7 Soil Sample Headspace Screening with a PID or FID

Headspace screening of soil samples will be completed according to Hull SOP F4008-00REV (or current version).

8.0 Documentation and Sample Handling

All samples will be properly labeled and packed in the shipping cooler prior to leaving the project site. A number of different documents will be completed and maintained as part of the sampling effort. The documents must provide a summary of the sample-collection procedures and conditions, shipment method, the analyses requested, and the custody history. The following is a list of the documents that must be filled out:

1. soil sample log/collection field data sheet/field notebook;
2. sample labels;
3. chain-of-custody records; and
4. shipping receipt (e.g., *Federal Express* receipt).

Once samples are properly labeled and documented, samples should be packaged and shipped according to Hull SOP F1013 (2012-REV) or current version.

9.0 Special Notes

On the occasion that field personnel determine that a representative soil sample must be obtained through an alternative method than the procedures described in this SOP due to access, safety concern or other reason, the alternate procedure used should be documented in the field log book, along with a description of the circumstances requiring its use. Sampling personnel should also get approval from the PM and/or regulatory agencies involved prior to sample collection.

The decontamination process outlined in HULLSOP.F1000.R0 (or current version) will be repeated after each use of sampling equipment and in between all discrete sampling locations. Should disposable equipment be used, the equipment will be changed out between all sampling locations. Disposable sampling gloves will be changed following the collection of each discrete sample.

Following completion of a soil boring, a borehole should always be decommissioned following guidance from HULLSOP.F2022R0 (or current version), unless the borehole is to be converted to a well or vapor probe.

10.0 Applicable Standards and References

ASTM D1452, *Standard Practice for Soil Investigation and Sampling by Auger Borings*.

ASTM D1586, *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*.

ASTM D1587, *Standard Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes*.

ASTM D5784, *Guide for Use of Hollow-Stem Augers for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices.*

ASTM D6282 - 98(2005), *Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations*.

Hull & Associates, Inc., *Standard Operating Procedure No. F1000-12REV or current version. Decontamination of Field Equipment*.

Hull & Associates, Inc., *Standard Operating Procedure No. F1006-12REV or current version. Field Classification and Description of Soils*.

Hull & Associates, Inc., *Standard Operating Procedure No. F1013-2012REV or current version. Packaging and Shipping of Non-Hazardous Samples*.

Hull & Associates, Inc., *Standard Operating Procedure No. F2024-11REV or current version. Procedure for Proper Containment/Storage of Used Drilling Fluids, Decon Fluids, and Purged Groundwater Investigative-Derived Solid and Selected Excavated Materials*.

Hull & Associates, Inc., *Standard Operating Procedure No. F2022-2001REV or current version. Decommissioning of Borings, Wells and Piezometers*.

Hull & Associates, Inc., *Standard Operating Procedure No. F4008-2000REV or current version. Soil/Water Sample Headspace Screening with a Photoionization Detector or Flame Ionization Detector*.

U.S. EPA, *Characterization of Hazardous Waste Sites, A Methods Manual - Vol. II, Available Sampling Methods,* 2nd Ed. 12/84. EPA/600/4-84/076.

U.S. EPA, *U.S. EPA Region 4 Science and Ecosystem Support Division. Operating Procedure for Soil Sampling. SESDPROC-300-R2*, December 2011, Retrieved from http://www.epa.gov/region4/sesd/fbqstp/Soil-Sampling.pdf

ASTM D6914-04 (2010), *Standard Practice for Sonic Drilling for Site Characterization and the Installation of Subsurface Monitoring Devices*.

HULL & ASSOCIATES, INC.
STANDARD OPERATING PROCEDURE

Title: Chain-of-Custody Procedures – Environmental Samples

Effective Date: 11/4/13	**Document Number: HULLSOP.F3014.R0**

Author

Name: Monica Williamson
Title: Sr. Scientist

Signature: Date: 11/4/13

Approvals

Name: Ray Kennedy
Title: Sr. Project Manager

Signature: Raymond Kennedy Date: 11/4/13

REVISION HISTORY

The table below summarizes changes to this document over time. The most recent version is presented in the top row of the table. Previous versions of the document (if any) are maintained in the archive portion of the Hull SOP library.

History	Effective Date
HULLSOP.F3014.R0 – created in November 2013 as part of overall reorganization of Hull's SOP program. While this version supercedes F3014-12REV, the content is generally equivalent to the 2012 version; only minor editorial modifications were made and the document name was changed for consistency with Hull's updated SOP nomenclature.	November 4, 2013
F3014-12REV – created in 2012; superceded in September 2013.	2012 (month not recorded)
F3014-02REV – created in 2002; superceded in 2012.	2002 (month not recorded)

HULLSOP.F3014.R0
CHAIN-OF-CUSTODY PROCEDURES
ENVIRONMENTAL SAMPLES

1.0 Purpose

This Hull & Associates, Inc. (Hull) Standard Operating Procedure (SOP) documents the chain-of-custody (COC) procedures that will be employed during all environmental sampling activities.

2.0 Equipment and Materials

- Indelible ink ball-point pens
- Chain-of-custody records
- One-gallon size *Zip-Loc* (or equivalent) storage bags

3.0 General

A completed COC record must accompany every sample from the point of collection to delivery to the laboratory. A single COC record may accompany several samples as long as all the samples are contained in a single unit (e.g., cooler, box, etc.). If a single COC is to be used for multiple samples in multiple coolers, then a photocopy of the original COC must be placed in each cooler. All COCs will be kept in one-gallon *Zip-Loc* bags, or equivalent to prevent damage from melting ice, broken samples, and bad weather. A copy of every completed COC record will be retained in the project files.

4.0 Procedures

4.1 Completion of COC Record

A. The COC record is initiated in the field by the sampler(s) immediately after a sample is collected. Figure F3014-1 illustrates a properly completed COC.

B. The sample identification number will be recorded on the COC. Each sample number consist of three distinct data fields. These data fields include; Project Number, Sample Location, and Sample Type. A space for each data field is provided on the COC.

C. The number of containers that makes a complete sample will be recorded in the box labeled "No. of Containers." A sample may consist of multiple containers depending upon the analytical procedures requested.

D. If the sample is to be analyzed for metals, the box labeled "Metals" shall be completed to indicate whether the sample fractions for metals have been filtered. A "F" will be used to indicate that the metals were filtered and a "N" will indicate that they were not filtered. Occasionally, some samples may require metal fractions to be filtered and not filtered (e.g., analyses for dissolved and total metals). In this case, "B" will be used to indicate that the sample contains both filtered and non-filtered fractions. If the sample does not require analyses for metals a single line will be drawn through this box.

E. The date and time (military) of sample collection will be recorded in the box labeled "Sampling Date/Time." It is very important to note the time each sample was collected even if samples are collected a few minutes apart.

F. The requested analytical methods will be recorded in the diagonal spaces provided under the box labeled "Analyses." The preservatives added to the containers for each analytical method will be indicated by recording the letter in the box labeled "Preservatives" that corresponds to the preservative added. The preservatives and corresponding letters are listed near the top of the COC record. Finally, a check mark(s) will be made under each analysis for which a particular sample will be analyzed.

G. Any comments relating to the collected sample(s) can be recorded in the box labeled "Comments." These comments may indicate special handling or analytical instructions for the laboratory (e.g., compositing instructions, confirm MTBE, etc.) or may be used to indicate the location of sample collection.

H. Additional information required on the COC record includes the person the analytical reports should be sent to, client, site, project description, project number, names of all samplers involved in sample collection, where the samples are to be delivered, method of delivery, and airbill number (if applicable).

I. In certain instances when a Hull COC is not available, it may be necessary to use a laboratory-supplied COC. The laboratory-supplied COC should contain the information outlined in Section 4.1; A through H.

4.2 Transfer of Custody

A. The COC record must document the transfer of custody each time the sample(s) changes hands. The National Enforcement Investigations Center (NEIC) of the United States Environmental Protection Agency (EPA) defines custody as:

1. the sample is in your physical possession;
2. the sample is within view after being in your physical possession;
3. the sample was in your possession and then you locked it or sealed it to prevent tampering; and/or
4. the sample is placed in a designated secure place with limited access to authorized personnel only.

B. When transferring custody of samples, the person in custody (e.g., the sampler) must sign the box labeled "Relinquished By" and fill in the date and time (military time) the custody of the samples was relinquished. The person accepting custody of the samples must then sign the box labeled "Received By" and complete the date and time (military time) the custody of the samples was accepted.

C. The above procedures must be followed until the samples are delivered to the laboratory. Both internal (within the same organization) and external (between different organizations) transfers need to be documented. In cases where a commercial courier (e.g., Federal Express) is used to deliver the samples, the person relinquishing custody to the courier should put the name of the courier in the "Received By" box and seal the COC inside the cooler. Most couriers have a policy against signing for custody of samples.

D. The pink copy (bottom) of the COC will be retained by the sampler before the samples are shipped and the remaining copies (white and yellow) of the COC are delivered to the laboratory. The pink copy will then be immediately given to the Project Manager or Quality Assurance Officer (QAO). The white copy will be returned by the laboratory with the final report.

5.0 Documentation

Chain-of-custody record

6.0 Special Notes

If samples are shipped via commercial courier on Friday the air bill needs to be checked for Saturday delivery and appropriate “Saturday Delivery” stickers (provided by the courier company) must be affixed to the container.

If samples are known to contain flammable or hazardous materials they need to be shipped accordingly. Check with the courier for specific shipping, labeling and packing requirements.

7.0 Applicable Standards and References

U.S. Environmental Protection Agency. NEIC Policies and Procedures. EPA-330/9-78-001-R. May 1978. (Revised February 1983.)

U.S. Environmental Protection Agency. User's Guide to the Contract Laboratory Program. Office of Emergency and Remedial Response. December 1986.

U.S. Environmental Protection Agency. A Compendium of Superfund Field Operations Methods. EPA/540/P-87/001, December 1987.

FIGURE

FIGURE 3014-1

PAGE 1 OF 1

CHAIN OF CUSTODY RECORD

NO. 8323

Hull & associates, inc.

Dublin, OH ☒	**Indianapolis, IN** ☐	**Mason, OH** ☐	**Bedford, OH** ☐	**Toledo, OH** ☐	**Pittsburgh, PA** ☐
6397 Emerald Pkwy Suite 200 Dublin, OH 43016 P: (614) 793-8777 F: (614) 793-9070	6435 Castleway W. Dr. Suite 119 Indianapolis, IN 46250 P: (800) 241-7173 F: (614) 793-9070	4770 Duke Dr. Suite 300 Mason, OH 45040 P: (513) 459-9677 F: (513) 459-9869	4 Hemisphere Way Bedford, OH 44146 P: (440) 232-9945 F: (440) 232-9946	3401 Glendale Ave. Suite 300 Toledo, OH 43614 P: (419) 385-2018 F: (419) 385-5487	300 Business Center Dr. Suite 320 Pittsburgh, PA 15205 P: (412) 446-0315 F: (412) 446-0324

REPORT TO: K. WILDMAN

Client: ABC COMPANY

Site: ANYTOWN, USA

Project #: ABC001 Phase: 01.SMP

Samplers: J. SMALL, T. LARGE

SAMPLE TYPES	PRESERVATIVES		METALS
A - AIR	A - Cool only, <4 deg. C	H - EDTA	F - Filtered
C - ASBESTOS	B - HNO_3 pH<2	I - 5ml 1:1 HCL	N - Not filtered
D - SEDIMENT	C - H_2SO_4 pH<2	J - None	B - Both
G - GROUNDWATER	D - NaOH pH>12	K - Stored in dark	
P - PRODUCT	E - ZnAcetate + NaOH, pH>9	L - NH_4Cl	
S - SOIL	F - $Na_2S_2O_3$ (0.008%)	M - Methanol	
W - WATER	G - HCL pH <2	S - Sodium bisulfate	
Z - OTHERS			

All samples are kept at 4 degrees Celsius.

ANALYSES

					PRESERVATIVES	A	A	G	B							
PROJECT NO.	SAMPLE LOCATION	SAMPLE TYPE & ID	NO. OF CONT.	METALS	COLLECTION DATE/TIME	VOCS - 8260	PAHS - 8310	VOCS - 8260	RCRA METALS							COMMENTS
ABC001	: MW-1	: S020030	2	—	11/4/13 / 0905	X	X									
ABC001	: MW-2	: S030035	2	—	11/14/13 / 1013	X	X									(HOLD SAMPLE)
ABC001	: MW-3	: S080100	2	—	11/14/13 / 1315	X	X									
ABC001	: MW-1	: G111413	3	F	11/14/13 / 1528			X	X							
ABC001	: FB-1	: W111413	2	—	11/14/13 / 1530			X								
ABC001	: TB-1	: W111413	2	—	11/14/13 / —			X								
	:	:			/											
	:	:			/											
	:	:			/											
	:	:			/											
	:	:			/											
	:	:			/											

RELINQUISHED BY: J. SMALL	DATE: 11/14/13 TIME: 1705	RECEIVED BY: FEDEX	DATE: 11/14/13 TIME: 1705
RELINQUISHED BY:	DATE: TIME:	RECEIVED BY:	DATE: TIME:
RELINQUISHED BY:	DATE: TIME:	RECEIVED FOR LAB BY:	DATE: TIME:

Deliver To: SOME LABORATORY, INC.

Method of Delivery: FEDEX

Airbill Number: WZ9832109X

NOTES: VAP PROJECT

COOLER TEMPERATURE AS RECEIVED: °C

DISTRIBUTION:
WHITE - LAB USE (MUST BE RETURNED WITH REPORT)
YELLOW - LAB USE
PINK - RETAINED BY HULL

TURN AROUND TIME: 14 DAYS

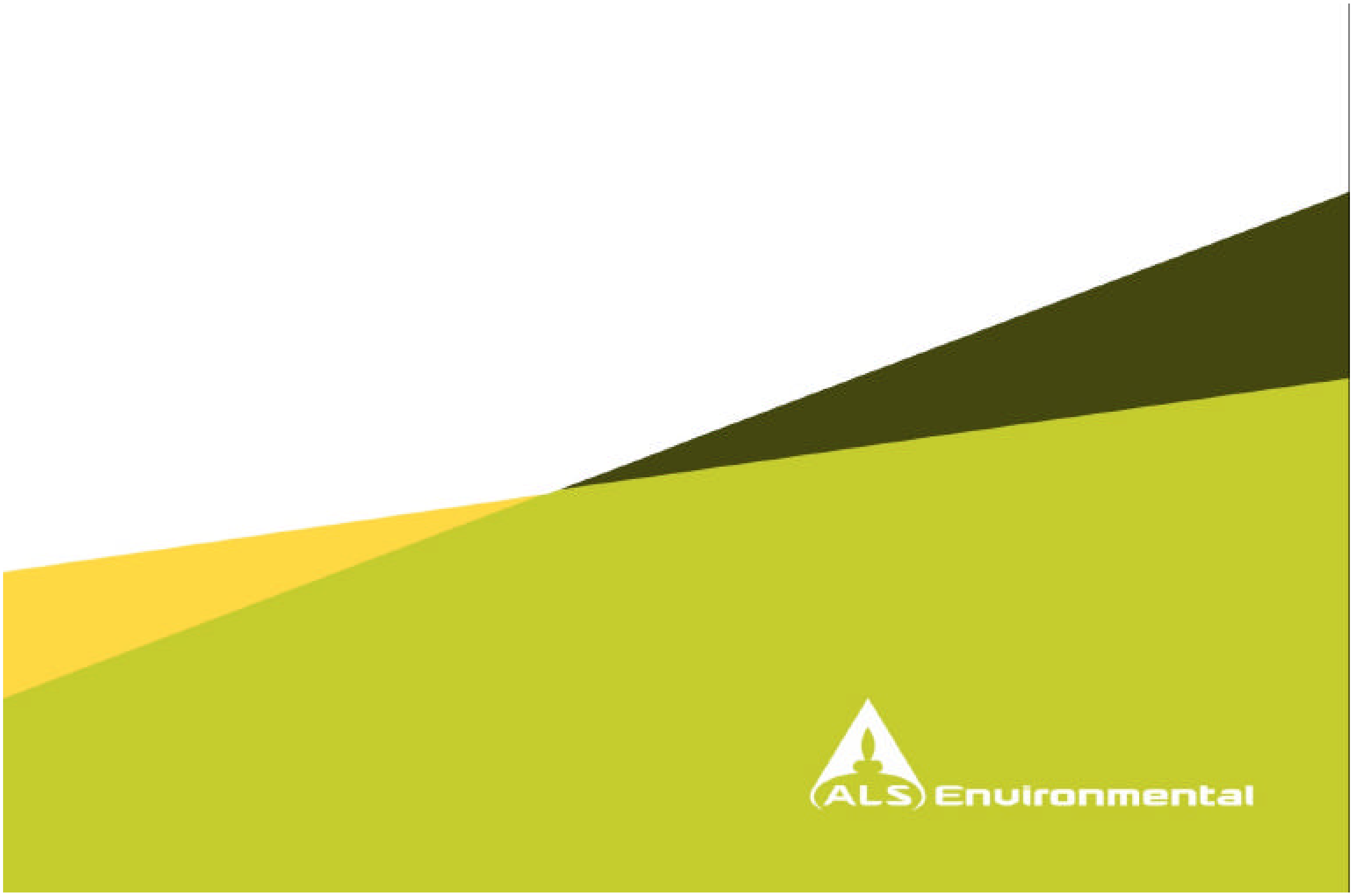
APPENDIX B

ALS SOPs

ALS Standard Operating Procedure

DOCUMENT TITLE: SAMPLE HOMOGENIZATION

REFERENCED METHOD: N/A
SOP ID: HN-QS-008
REV. NUMBER: R06
EFFECTIVE DATE: 06/01/2015



SAMPLE HOMOGENIZATION

SOPID: HN-QS-008 **Rev. Number:** R06 **Effective Date:** 06/01/2015

Approved By: Operations Manager Date: 5/18/15

Approved By: QA Manager Date: 5/18/15

Approved By: Laboratory Director Date: 5/18/15

Archival Date: Doc Control ID#: Editor:

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
	QA Manager	6/6/2017

TABLE OF CONTENTS

SAMPLE HOMOGENIZATION

1) Scope and Applicability

1.1 This SOP is designed to provide guidance on homogenizing samples in order to obtain a representative aliquot for further processing. It should be noted that specific samples may require special techniques due to problematic matrices or project specific requirements. Approaches found in this SOP are designed to address the following matrix groups and subgroups:

1.1.1 Liquids:

- Aqueous liquids as surface water, drinking water, groundwater, TCLP extracts, wastewaters, and leachates
- Non-aqueous liquids as organic solvents and/or oils

1.1.2 Solids: Soil, sediment, heterogeneous solid substances, concrete, paint chips, ash

1.1.3 Multiphase (Liquids/Solids) Sludge

- Aqueous sludge, liquid sludge, solid sludge, emulsions
- Non-aqueous sludge

1.2 This SOP is not designed for VOA, wipe, or air samples. Duplicate samples should be collected if multiple sample aliquots will be required.

1.3 Preparation of sample composites is generally performed at the request of a client. When the client requests sample composites, equal or designated portions of homogenized samples will be combined as instructed by the client.

1.3.1 For soils, equal or designated weights will be combined.
1.3.2 For aqueous samples, equal or designated volumes are used.
1.3.3 Composite samples are either assigned new sample identifications as part of a new work order number or assigned fraction IDs as part of an existing work order.

2) Summary of Procedure

2.1 Dependent upon matrix characteristics, samples are homogenized prior to sub-sampling for applicable analytical procedures. The homogenization process can entail simple shaking for aqueous matrices to particle size reduction or separation for solid matrices.

3) Definitions

3.1 Aliquot: A sub-sample taken by an analyst that constitutes a representative portion of a sample. Also, the process of sub-sampling or taking a representative sub-sample.

3.2 Dissolved Material: A filterable residue that is defined by a specified filtration procedure (e.g. dissolved metals are those metals passing through a 0.45µm filter).

3.3 Friable: A solid material that can be easily crumbled, pulverized or broken into small fragments, particles or powders with minimal pressure or mechanical action.

3.4 Multiphasic Sample: a sample that contains more than one solid or liquid phase.

3.5 PSR: Particle size reduction - usually performed by manual or mechanical grinding.

3.6 PSS: Particle size separation - generally performed by sieving.

4) Health and Safety Warnings

4.1 All activities must be performed using appropriate personal protective equipment.

4.2 Homogenization and sub-sampling should be performed in a properly operating laboratory fume hood.

5) Personnel Qualifications and Responsibilities

5.1 General Responsibilities – These procedures are restricted to use by or under the supervision of analysts experienced in their use. Analysts may not perform these procedures until they have completed training from supervisory personnel.

5.2 Analyst - It is the responsibility of the analyst(s) to:

5.2.1 Document homogenization activity according to this SOP or applicable contractual requirements using designated controlled logsheets.

5.3 Department Supervisor - It is the responsibility of the Department Supervisor to:

5.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform homogenization procedures according to this SOP.

5.4 Project Manager - It is the responsibility of the Project Manager to:

5.4.1 Ensure that all contractual requirements for a client sample homogenization or compositing, if different than described in this SOP, are relayed to and understood by the analytical staff prior to initiating sample receipt.

6) Procedure

Inspect each sample to determine if the sample is a liquid, solid, or a combination (multiphasic). Depending upon the matrix, refer to sections 6.1, 6.2, and/or 6.3, respectively. For multiphasic samples received, client contact via project management must be made to confirm the procedure meets the client needs.

6.1 Liquids: Evaluate the liquid sample, looking for suspended matter, multiple phases, or any other features that may require specific measures to obtain a representative sub-sample. If the samples contain no detectable suspended matter, follow guidance presented in section 6.1.1. Samples containing suspended matter, but requiring analysis as a single liquid phase should follow the guidance presented in section 6.1.2. Multiphase samples should follow guidance established in section 6.3.

6.1.1 Aqueous Liquids Homogenization - Surface Water, Groundwater, Drinking Water, TCLP Extracts, Wastewater, and Leachates

- Inorganics, Metals, and Extractables (Total Analysis)

  - Make sure sample lid is securely attached to sample container.
  - Invert, or shake the sample in an up and down fashion a minimum of 7 times.
  - Remove lid and quickly yet smoothly transfer desired aliquot into an appropriately sized graduated cylinder or digestion container. If the required sample volume is small, a serological or volumetric pipette should be used to obtain sample.
  - Transfer the sample from the pipette or graduated cylinder into an appropriate container used to either process further or to ship to a contract laboratory. The sample can be transferred directly into the process apparatus or testing vessel, if appropriate (e.g., metals digestion cups).
  - When sample results are not affected by volume additions, a rinse of the delivery glassware may be done with a small volume (1-2 mL) of reagent water. This will ensure a quantitative transfer.
  - If the cylinder or pipette used to sub-sample is to be reused for other samples, thoroughly clean the transfer apparatus appropriately and include a method blank utilizing the same procedures.

- Dissolved Inorganics and Dissolved Metals

When dissolved analytes are required, field filtration followed by appropriate preservation is preferred. If this is not practical, project personnel may elect to send unpreserved samples to the laboratory for filtration and preservation. The appropriate regulatory authorities should be consulted and concur with this approach.

  - Generally, the only difference between this and 6.1.1 is the sample is filtered through a 0.45µm filter before processing the filtrate using appropriate procedures. The residue is discarded.
  - Mark as filtered with a "F"
  - The filtering device needs to be of known cleanliness and thoroughly decontaminated in between uses.

- Volatile Organics:

VOA vials should never be opened prior to analysis.

6.1.2 Liquid Samples with Suspended Matter

Sampling procedures for liquid samples that contain suspended matter are very similar to the procedures outlined in section 6.1.1 with minor deviations.

- Inorganics, Metals, and Extractable Organics

  - Invert, or shake the sample in an up and down fashion a minimum of 7 times, or until the sample appears uniformly mixed.
  - Evaluate whether the rate that the suspended matter settles allows sufficient time to acquire a representative aliquot.
  - If the suspended matter settles slowly, the sample is shaken repeatedly and an aliquot taken immediately.

  - If the suspended matter settles rapidly, the sample is shaken repeatedly and immediately transferred to a large beaker. A PTFE coated magnetic stir bar is added and the sample is magnetically stirred until uniformly mixed. While stirring continues, an aliquot is taken by pipette or other sub-sampling means.
  - Refer to remaining protocol presented in section 6.1.1.
  - If the amount of suspended material is excessive, consult with the Project Manager and consider the sample as bi-phasic (see Section 6.3).

- Dissolved Inorganics and Dissolved Metals

  - Due to the physical separation of the suspended matter, refer to the procedures outlined in section 6.1.1 “Dissolved Metals”.

- Volatile Organics

  - Refer to Section 6.1.1 “Volatile Organics”.

6.1.3 Non-aqueous Liquids-Oils:

- The need for mixing of these non-aqueous liquids must be investigated. Verify whether minor amounts of particles are present which would require redistribution into the liquid prior to sub-sampling. Samples with substantial amounts of separated phases should refer to section 6.3.
- Due to the different viscosities, densities, coating properties, etc., the weight of the sub-sample should typically be determined, rather than attempting to express the sub-sample in terms of volume. If it is difficult to volumetrically measure a sample because it adheres to glassware walls, weight must be used.
- If an aliquot needs a specific and accurate weight, a disposable pipette or syringe is a good option for sub-sampling. Aspirate the sample, and then carefully transfer the sample into a tarred vessel, controlling the addition by finger pressure on the top of the pipette.

6.2 Solids Homogenization - Soil, Sediment, Concrete, Paint Chips, Ash

When homogenizing these matrices, first examine the particle size distribution. A decision must be made as to whether a representative sub-sample can be obtained without prior sample manipulation. This is determined visually and based on the physical attributes of the sample. Variables to be considered include:

- ➢ Is there a significant amount of oversized material (be it either naturally occurring [rocks] or artificially introduced material [debris])?
- ➢ Are there obvious soil, chemical, or ash inclusions where indiscriminate sub-sampling might bias the analytical results or caution against specific protocols (e.g., lead chunks, tar balls, solid (grayish-white) explosives material)?
- ➢ Does the sample tend to segregate into various size fractions easily?

6.2.1 Inorganics, Metals, and Extractable Organics

- Even if the material received appears homogeneous, the entire sample must be thoroughly mixed using an inert, non-contaminating spatula or rod.
  - Perform by emptying container contents onto a clean surface (stainless steel or, if metal analysis, glass)
    - Decant and discard any water layer on sediment samples.
    - For cohesive material, the bulk material should be size reduced using a stiff bladed utensil such that the average size of any clump is approximately pea-sized (approximately 6mm), if possible.
  - Discard any foreign objects such as sticks, leaves, and rocks.
  - Thoroughly mix the sample using a clean spatula or mixing device
  - Return mixed sample to the container and mark with an "M" to indicate homogenization (mixing) has been completed. Record an “M” within prep batch comments to document that mixing has been performed.

6.2.2 Volatile Organics

- Volatile samples must not be opened or exposed to the environment prior to analysis.
- If bulk samples are supplied per contractual requirement, weigh the required amount of soil, place immediately into methanol, DI water, or the sparging device, and proceed with analysis. Analytical results should be qualified as to potential bias due to sample exposure during the preparation process.

6.2.3 Heterogeneous Material

If a solid sample is determined to be heterogeneous, a decision must be made as to the correct homogenization procedure. This decision must be made in conjunction with the Project Manager who submitted the samples for analysis and must be appropriately narrated.

6.3 Sub-sampling Multiphase (Liquids/Solids) Sludge

This section is meant to address samples considered multiphasic based on physical characteristics (mixtures of solids and/or liquids). The choice of the procedure for handling multiphasic samples is highly dependent on project needs. There are no specific procedures describing how samples with certain volume distributions of liquid/solid are handled. It is essential that the Project Manager be consulted before selecting one of the approaches outlined below, and that the approach used is adequately documented. Three approaches are provided in the following subsections:

6.3.1 Sub-sampling of samples analyzed as a Single Mixed Phase (as received). This approach will not provide information on the abundance of analytes in the individual phases other than what can be implied by solubility.

- The sample is mixed sufficiently so as to create a homogenous sample. This is usually assessed on a visual basis. A single analysis will then be performed.

  - The manner in which the sample is mixed is highly dependent on sample consistency and how easily the phases mix. Some samples can simply be shaken, while others will require a spatula or mixing rod, or laboratory blender. If this is necessary, the device used to mix the sample must be clean, inert, and easily decontaminated. Usually a glass or Teflon-coated device is appropriate.

- The sample is then poured, sampled with a scoop, or transferred by some other physical means into a tarred vessel and the weight of sample is recorded. This transfer is dependent upon sample viscosity/consistency.

6.3.2 Segregation as Separate Phases

- When the phases of a multiphasic sample are to be tested individually, the phases are separated by physical means (i.e., filtration [either pressurized or non-pressurized], centrifugation, settling, or use of a separatory funnel). The technique used is dependent on items such as laboratory capabilities, sample characteristics, and analytes of interest. If an aliquot of a sample is to be phase separated, this aliquot must be representative of the original sample. This means that the solid and liquid ratio of the aliquot needs to be the same as the original sample.
- To accomplish this, follow the procedures listed in Section 6.3.1. Items to consider when performing the phase separation include:

  - Device is non-contaminating.
  - Device is non-absorbent for analytes of interest.
  - Device does not cause the loss of analytes through other means other than noted above.

- Once the phases are separated, the solid and liquid ratios are recorded. The two portions are transferred into either sample preparation vessels, testing vessels, or into appropriate storage containers for later analysis. If the liquid portion is subsequently processed, follow procedures listed in 6.1. If the solid portion is subsequently processed, follow the solids handling procedures described in 6.2.

6.4 When phases are separated and analyzed separately, the final concentration for the total sample is calculated by using:

$$Final\ Analyte\ Concentration = \frac{(V_1)(C_1)+(V_2)(C_2)}{V_1+V_2}$$

6.4.1 The final analyte concentration is expressed as µg/mL or µg/g, where:

- V_1 = volume (mL) or weight (g) of first phase
- C_2 = concentration of analyte in first phase (µg/mL or µg/g)
- V_2 = volume (mL) or weight (g) of second phase
- C_2 = concentration of analyte in second phase (µg/mL or µg/g)

7) Equipment and Supplies

7.1 The specific equipment used for homogenization is dependent on the analyses and methods to be performed. (Specific methods should be consulted prior to performing these activities.) Specific equipment should be selected from the options presented below:

7.1.1 Spatulas, spoons, scoops, PTFE-coated or stainless steel, various sizes
7.1.2 Stirring rods, glass
7.1.3 Magnetic stirrers, PTFE-coated magnetic stir bars
7.1.4 Laboratory trays, metal, plastic or glass.
7.1.5 Laboratory beakers, PTFE or glass
7.1.6 Class A Pipette, serological and volumetric (to deliver), various sizes
7.1.7 Class A Graduate Cylinders (to deliver), various sizes up to 2L
7.1.8 Weigh boats, plastic
7.1.9 Laboratory drying oven
7.1.10 Balance, top-loader with readability to 0.01-g, or analytical with readability to 0.1 mg.
7.1.11 Laboratory grinders (mortar/pestle)
7.1.12 Sieves, various sizes: the sieve size required is dependent upon the analyses performed by the lab.
7.1.13 Centrifuge – Bench top or floor unit
7.1.14 Separatory Funnel (Teflon or glass)
7.1.15 Filtering apparatus - Glass Microanalysis, 47 mm, 0.45µm filters, Fisher cat# 09-753-1c or equivalent.
7.1.16 Syringe filters with Teflon filtering discs
7.1.17 Syringes, glass, air-tight, various sizes, with Luer-Lok valves
7.1.18 Kimwipes, or equivalent
7.1.19 5035 Coring device

8) Quality Assurance and Quality Control

8.1 Care must be exercised so that the introduction of contamination or the potential for cross-contamination from equipment used to manipulate the sample is minimized. Full decontamination protocols must be performed on equipment used between each use.

8.2 Aqueous samples containing hydrophobic analytes, e.g., petroleum hydro-carbons, should not be sub-sampled. The entire bottle contents should be used to avoid loss of analytes to container walls or cap.

8.3 When processing samples, be sure to fully document the actual procedure used (describing the manipulations as completely as possible), and any irregularities or observations made into the appropriate logbook, or bench sheet.

8.4 Check to make sure sub-sample weights/volumes have been properly recorded in sample preparation logbooks.

8.5 Implement the appropriate quality control samples to allow an assessment of the potential cross-contamination (blanks), and overall effectiveness of the sub-sampling techniques (duplicates).

8.6 If ineffective homogenization or sampling is indicated, the procedure used must be reevaluated, appropriate revisions implemented, and all affected samples reprocessed.

9) Summary of Changes

Table 9.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R05	07/01/2013	CES	Formatting
R06	06/01/2015	CES	Document Revision Criteria.

10) References and Related Documents

10.1 Test methods for Evaluating Solid Wastes, SW-846, Third Edition, through Update II, September 1994.

10.2 Kratochvil, B, and Taylor, J.K., Sampling for Chemical Analysis, Analytical Chemistry, American Chemical Society, July 1981, pp. 924-938.

10.3 Lewis, T.E., et.al., Soil Sampling and Analysis for Volatile Organic Compounds, U.S.EPA, Office of Solid Waste and Emergency Response, EPA/540/4-91/001, February 1991

PARTICLE SIZE ANALYSIS

ASTM D422

SOPID:	HN-MET-013	Rev. Number:	R03	Effective Date:	10/15/2017

Approved By: ______________________ Date: 9/21/17
Department Supervisor

Approved By: ______________________ Date: 9/21/17
Quality Assurance

Approved By: ______________________ Date: 9/21/17
Laboratory Director

Archival Date:	________	Doc Control ID#:	________	Editor:	________

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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PARTICLE SIZE ANALYSIS

1) Scope and Applicability

1.1 This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75um is determined by sieving, while the distribution of particle sizes smaller than 75um is determined by a sedimentation process using a hydrometer.

1.2 This procedure is based upon and compliant with ASTM D422-63

2) Summary of Procedure

2.1 A soil sample is dried, ground, and passed through a no. 10 sieve.

2.2 Material retained in the no. 10 sieve is then passed through larger sieves and recorded.

2.3 Material passing the no. 10 sieve is then determined for particle size in two ways.

2.3.1 The distribution of particle sizes smaller than 75um is determined by a sedimentation process using a hydrometer for data collection.
2.3.2 The distribution of particle sizes greater than 75um is determined by sieving through a series of test sieves.

3) Definitions

3.1 DI Water- water purified via reverse osmosis and mixed bed polishing, conforming to ASTM type 2 specifications.

3.2 Particle Size – a physical characteristic representing the diameter of a particle, expressed in um.

3.3 Hydrometer Analysis – the use of an appropriately calibrated hydrometer device, meeting ASTM specifications, for the purpose of data collection.

3.4 Test sieve – a device composed of square-mesh woven-wire cloth, conforming to ASTM specifications for the separation of a particular particle size at a given diameter.

3.5 Coarse Material – portion of sample not passing through a No. 10 test sieve.

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.
4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.

4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.

4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Take care in the operation of mechanical agitation apparatus. Follow all manufacturers' guidelines taking appropriate precautions during the operation of such equipment.

6) Interferences

6.1 Thorough homogenization/pulverization of the dried sample is critical in getting an accurate representation of the particle size distribution. Elimination of aggregate clumps of material must be performed utilizing grinding in a mortar and pestle.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities – This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst – It is the responsibility of the analyst(s) to:

7.2.1 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.2.2 Complete the required demonstration of proficiency before performing this procedure without supervision.

7.2.3 Create and populate a data entry batch in LIMS for review by the Supervisor.

7.3 Section Supervisor – It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.

7.3.3 Ensure the production of contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager – It is the responsibility of the Project Manager to ensure that all contractual requirements for a client requiring this procedure are understood prior to initiating this procedure for a given set of samples.

8) Sample Collection, Handling, and Preservation

8.1 Samples should be collected in 16 ounce jars.

8.2 Preserve the samples with refrigeration at 4 ± 2 °C from the time of collection until analysis.

8.3 The samples must be analyzed within 90 days of collection.

9) Equipment and Supplies

9.1 Balance capable of weighing to 0.01g.

9.2 Stirring Apparatus

9.2.1 Hamilton Beach H-4260A commercial mixer or equivalent, capable of 10,000 rpm.
9.2.2 No. 18 chrome plated stirring paddle

9.3 Hydrometer- ASTM 151H, 280MM, 0.995-1.038

9.4 Sedimentation Cylinder – approx. 18” tall by 2.5” diameter and marked for 1000 mL.

9.5 Thermometer – Accurate to 1˚ F (0.5˚ C)

9.6 Test Sieves

9.6.1 3.0”, 1.5”, 0.75”, 0.375”, No. 4, No. 10, No. 16, No. 30, No. 50, No. 60, No. 100, and No. 200.
9.6.2 Additional test sieves may also be included upon client request.

9.7 Water bath – capable of being maintained at room temperature (at or near 68˚ F)

9.8 Beaker – 250mL capacity

9.9 Volumetric flask – 1 liter capacity

9.10 Magnetic stirrer

9.11 Timing device – watch or clock with a second hand

9.12 Mortar and pestle, rubber – suitable for breaking up aggregations of soil within the sample

9.13 Drying Oven - capable of maintaing a temperature of ~105˚ C.

9.14 Graduated cylinder, 25 mL

10) Standards and Reagents

10.1 DI water, meets ASTM type 2 specifications.

10.2 Sodium Hexametaphosphate, powder, ACS grade

10.3 Sodium Hexametaphosphate Dispersing Agent

10.3.1 Add approx. 500ml of DI water to a 1 liter volumetric flask. Add 40g sodium hexametaphosphate.
10.3.2 Mix utilizing a magnetic stirrer
10.3.3 Bring to a final volume of 1 liter with DI water.
10.3.4 Assign an appropriate BPL and label accordingly

10.3.5 Solution must be prepared monthly

11) Method Calibration

11.1 Perform all routine calibration necessary for equipment to meet ASTM specifications. See SOPs HN-EQ-001,002, and 003 (current revisions) for further guidance.

12) Sample Preparation/Analysis

12.1 Prepare the test sample according to guidelines set forth in ASTM D421.

12.1.1 Obtain a wet weight of an aliquot of sample large enough that upon passing through a No. 10 sieve, a minimum of 115g dried sample is present for analysis.
12.1.2 Air Dry the sample at ambient temperature for a minimum of 24 hours. NOTE: Samples with considerable amounts of moisture may longer to dry.
12.1.3 Record the dry weight of the sample.
12.1.4 Grind the dried sample with a mortar and rubber pestle to break up any aggregations.
12.1.5 Obtain 15g of air dried sample and proceed with section 12.3.
12.1.6 Obtain and record the weight of ~ 10g of air dried sample in a pre-weighed aluminum tin. Dry in an oven overnight at 105˚C. Remove the sample from the oven and place in a desiccator to cool. Weigh the oven dried sample and tin. Subtract the weight of the tin from the combined weight to obtain the weight of the oven dried sample. Use this value to calculate the Hydroscopic Moisture Correction Factor (HMCF).
12.1.7 Pass the remaining air dried sample through the No. 10 sieve, collecting the fraction of sample passing the sieve. This portion will be analyzed for particle size via hydrometer analysis and/or test sieve analysis. Proceed to section 12.2 for this procedure.
12.1.8 The portion of sample not passing the No. 10 sieve is washed thoroughly with tap water until the water runs clear.
12.1.9 Dry the washed sample in the oven until a constant mass is achieved. This portion constitutes the Coarse Material.
12.1.10 Pass the dried sample through the 3”, 1.5”, 0.75”, 0.375”, No. 4, and No. 10 sieves. Record the weight of sample left on each sieve.
12.1.11 Use this data to generate the graph as discussed in section 15.

12.2 Hydrometer and Sieve analysis of portion passing the No. 10 sieve

12.2.1 If hydrometer analysis is not required per client instruction, perform sections 12.2.2 through 12.2.4 and then proceed to section 12.2.10.
12.2.2 Weigh out approximately 115g of dried material that passed the No. 10 sieve from section 12.1.5 into a 250 mL beaker. Record the weight of this material.
12.2.3 Add 125 ml Sodium Hexametaphosphate Dispersing Agent to the beaker and mix to thoroughly wet the sample. Add DI water to ensure the sample is completely covered.
12.2.4 Let the sample soak for a minimum of 16 hours.
12.2.5 Transfer the sample to the baffled cup include with the stirring apparatus. Quantitatively transfer the sample to the cup using aliquots of DI water. Ensure the cup is more than half full.
12.2.6 Mix the sample for a period of 1 minute.

12.2.7 Immediately after dispersion, transfer the slurry to the sedimentation cylinder. Add DI water to a final volume of 1000 mL.

12.2.8 Place parafilm on the mouth of the cylinder, cover with a gloved hand, and mix the sample by inversion for a period of 1 minute.

12.2.9 Record hydrometer readings at the following intervals

- 12.2.9.1 2 minutes, **place the cylinder in the water bath after this initial reading**
- 12.2.9.2 5 minutes
- 12.2.9.3 15 minutes
- 12.2.9.4 30 minutes
- 12.2.9.5 60 minutes
- 12.2.9.6 250 minutes
- 12.2.9.7 1440 minutes
- 12.2.9.8 After each reading, record the water bath temperature at each interval.

12.2.10 Sieve analysis of material passing the No. 10 sieve.

- 12.2.10.1 Transfer the material from the hydrometer reading, or if no hydrometer analysis was required from section 12.2.4, to a No. 200 test sieve.
- 12.2.10.2 Wash the sample with tap water until the water runs completely clear.
- 12.2.10.3 Transfer the material remaining on the No. 200 sieve to a suitable container and dry to a constant weight.
- 12.2.10.4 Pass the dried sample through No. 16, 30, 50, 60, 100, and 200 test sieves. Additional sieves may be used at the client’s request.
- 12.2.10.5 Record the weight of sample retained on each sieve.
- 12.2.10.6 Use this data to generate the graph as discussed in Sec. 15.

12.3 Specific gravity determination for soils

- 12.3.1 Place approximately 15g of air dried soil into a 25mL graduated cylinder.
- 12.3.2 Add 15mL of DI water.
- 12.3.3 Tap the sample repeated to remove any air pockets or bubbles.
- 12.3.4 Record the displacement of water after soil addition.

13) Troubleshooting

13.1 N/A

14) Data Acquisition

14.1 Record all data in the designated Particle Size logbook.

14.2 Transfer the data collected in the logbook to the Excel spreadsheet template.

14.3 Save the template under the sample Work Order ID.

14.4 Record the data generated through the Excel spreadsheet into LIMS to be reported.

14.5 Generate a graph of the data as described in section 15.

15) Calculation, and Data Reduction Requirements

15.1 The percentage of soil remaining in suspension at the time of the hydrometer reading is calculated as follows

$$P= [(100000/W) \times G/(G-G_1)](R-G_1)$$

Where:

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied,

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed, g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15.2 The diameter of a particle corresponding to the percentage indicated by the hydrometer reading is calculated according to Stokes' law as follows

$$D=k\sqrt{[L/T]}$$

Where:

k = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K are given in Table 2. The value of K will not change for a series of readings.

L = Effective depth is the distance from the surface of the suspension at the density is measured, cm. This is taken from the actual hydrometer reading, and can be found in Table 1.

T = interval of time from beginning of sedimentation to the time at which measurement is taken.

15.3 When the hydrometer and sieve analyses are complete, a semi log graph may be constructed, plotting the diameters of the particles on a logarithmic scale on the x-axis, and the percentages passing on an arithmetic scale on the y-axis.

15.4 Specific gravity of the soil, from 12.3, is calculated as follows

G = volume of water added in ml/volume of water displaced in ml

16) Quality Control, Data Assessment and Corrective Action

16.1 There are no quality control criteria outlined for this method.

17) Data Records Management

17.1 All data shall be stored both electronically and in hard copy.

17.1.1 Hard copy documentation must be maintained via logbooks for standard/chemical tracking, sample preparation procedures, and instrument maintenance.

17.1.2 All raw data and logbooks must be maintained for a period of no less than 10 years.

17.1.3 Electronic records shall be maintained for a period of no less than 10 years.

17.2 Prior to uploading into LIMS, the primary analyst must review all raw data for acceptability. The primary analyst must date/initial the hardcopy documentation verifying this review and the data acceptability.

17.3 A peer review of uploaded analytical data must be completed prior to validation. The secondary analyst must date/initial the hardcopy documentation verifying the secondary review and acceptability.

17.4 Finalized data shall be stored in a designated section of the laboratory. After approximately two months from analysis date, data shall be transferred to the QA department for archival.

18) Quality Assurance and Quality Control

18.1 Lab notebooks must be reviewed monthly by the department supervisor.

18.2 Lab notebooks must be reviewed quarterly by the QA Staff.

18.3 The QA Staff must conduct periodic audits to evaluate compliance with this SOP.

19) Contingencies for Handling Out of Control Data

19.1 All analysts must report sufficient comments in LIMS such that project management can narrate and ensure appropriate data qualifiers are properly assigned.

19.2 Non-conformances must be documented in the data checklist for the analytical run.

19.3 Deviations from this procedure or the quality control criteria outlined within must be documented via the NC/CA database for further evaluation.

20) Method Performance

20.1 Additional method performance criteria have not been evaluated.

21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R01	11/15/12	CES	New SOP
R02	02/15/15	CES	Formatting
R03	04/15/17	CES	Updated procedure for determination of HMCF
R03	04/15/17	CES	Updated section 12.1 to include air drying

22) References and Related Documents

22.1 Method D422-63, Standard Test Method for Particle-Size Analysis in Soils, Annual Book of ASTM Standards, Volume 04.08, March 2002.

22.2 Method D421-85, Standard Practice for Dry Preparation of Soil Samples for Particle Size Analysis and Determination of Soil Constants, Annual Book of ASTM Standards, Volume 04.08, March 2002.

22.3 Quality Assurance Manual, ALS Environmental, Revision (most current)

22.4 Table 1: Values of Effective Depth

22.5 Table 2: Values of k

Table 1. Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specific Sizes

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L (cm)	Actual Hydrometer Reading	Effective Depth, L (cm)	Actual Hydrometer Reading	Effective Depth, L (cm)
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5	36	10.4
1.006	14.7	6	15.3	37	10.2
1.007	14.4	7	15.2	38	10.1
1.008	14.2	8	15.0	39	9.9
1.009	13.9	9	14.8	40	9.7
1.010	13.7	10	14.7	41	9.6
1.011	13.4	11	14.5	42	9.4
1.012	13.1	12	14.3	43	9.2
1.013	12.9	13	14.2	44	9.1
1.014	12.6	14	14.0	45	8.9
1.015	12.3	15	13.8	46	8.8
1.016	12.1	16	13.7	47	8.6
1.017	11.8	17	13.5	48	8.4
1.018	11.5	18	13.3	49	8.3
1.019	11.3	19	13.2	50	8.1
1.020	11.0	20	13.0	51	7.9
1.021	10.7	21	12.9	52	7.8
1.022	10.5	22	12.7	53	7.6
1.023	10.2	23	12.5	54	7.4
1.024	10.0	24	12.4	55	7.3
1.025	9.7	25	12.2	56	7.1
1.026	9.4	26	12.0	57	7.0
1.027	9.2	27	11.9	58	6.8
1.028	8.9	28	11.7	59	6.6
1.029	8.6	29	11.5	60	6.5
1.030	8.4	30	11.4		
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				
1.039	5.9				

Table 2. Values of **k** for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.0394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.01323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01269	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149