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URS

Nydia Burdick
SC DHEC
Office of Environmental Laboratory Certification
2600 Bull Street
Columbia, SC 29201

Re: Philip Services Site – Preliminary Design Investigation QAPP
Former PSC Site – Rock Hill, SC

Dear Ms. Burdick:

On behalf of the PRP Group for the Philip Services Site, URS Corporation (URS) is providing the *Quality Assurance Project Plan* (QAPP) revised according to comments provided by South Carolina Department of Health and Environmental Control (SCDHEC) in February 2013. The QAPP has been prepared in accordance with the SCDHEC Office of Quality Assurance Bureau of Environmental Services Environmental Quality Control. The QAPP is prepared as a Class 3 for the Preliminary Design Investigation (PDI) scope of work, as agreed upon by SCDHEC. .

Please feel free to contact me with any questions. Thank you for your continued help in this matter.

Best Regards,

URS Corporation

A. Brett Berra, PE
Senior Project Manager

cc: Lucas Berresford, SCDHEC
PRP Group Steering Committee
PRP Group Technical Committee
William W. Toole
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Preliminary Design Investigation Quality Assurance Project Plan

May 2014

Former Philip Services Corporation Site
Rock Hill, South Carolina

Prepared For:
Philip Services PRP Group
Mr. William M. Toole
Robinson, Bradshaw & Hinson, PA
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QUALITY ASSURANCE PROJECT PLAN FOR THE PRELIMINARY DESIGN INVESTIGATION AT FORMER PHILIP SERVICES CORPORATION SITE ROCK HILL, SOUTH CAROLINA

Prepared for:
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Approved by:

Name	Title	Organization	Signature	Date
Brett Berra	Project Manager	URS Corporation	A. Brett Berra	5/30/14
Martha Meyers-Lee	Project QA Officer	URS Corporation	Martha Meyers-Lee	5/30/14
William M. Toole	Group Project Coordinator	PSC PRP Group	W. William M. Toole	5/29/14
Ken Hayes	Offsite Laboratory Project Manager	TestAmerica Nashville	Ken Hayes	05/29/2014
Steve Miller	Offsite Laboratory QA Officer	TestAmerica Nashville	Steve Miller	5/29/14
Todd Romero	Onsite Laboratory Project Manager	KB Labs, Inc.	Todd Romero	5/27/14
Bob George	Onsite Laboratory QA Officer	KB Labs, Inc.	Bob George	5/27/14
Nydia Burdick	SQAMO Designee	SCDHEC		
J. Lucas Berresford	Project Manager	SCDHEC		

Prepared May 2014
Revision 0

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

LIST OF ACRONYMS AND ABBREVIATIONS

°C	Degrees Celsius
µg/L	Micrograms per Liter
bgs	Below Ground Surface
CLP	Contract Laboratory Program
CoC	Chain-of-Custody
COC	Constituent of Concern
CPR	Cardiopulmonary Resuscitation
DO	Dissolved Oxygen
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
EDD	Electronic data deliverables
FL	Florida
FS	Feasibility Study
GIS	Geographic Information System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
HSO	Health and Safety Officer
IDW	Investigation-derived Waste
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LIF	Laser Induced Fluorescence
LIMS	Laboratory Information Management System
LNAPL	Light Non Aqueous Phase Liquid
LQL	Lower Quantitation Limit
MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goals
MD	Maryland
MDL	Method Detection Limit
MNA	Monitored Natural Attenuation
MPC	Measurement Performance Criteria
MPE	Multiphase Extraction
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAP	National Environmental Laboratory Accreditation Program

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

NFG	National Functional Guidelines
NIST	National Institute of Standards and Technology
NTU	Nephelometric Turbidity Units
ORP	Oxidation-reduction Potential
OSHA	Occupation Safety and Health Administration
PARCCS	Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PDI	Preliminary Design Investigation
PID	Photo Ionization Detector
PM	Project Manager
PPM	Parts Per Million
PSC	Philip Services Corporation
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RBSL	Risk Based Screening Levels
RCRA	Resource Conservation and Recovery Act
RG	Remediation Goals
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SC DHEC	South Carolina Department of Health and Environmental Control
SESD	Science and Ecosystem Support Division
SOP	Standard Operating Procedures
SQAMO	State Quality Assurance Management Office
SVE	Soil Vapor Extraction
TNI	National Environmental Laboratory Accreditation Conference Institute
TSS	Total Suspended Solids
TN	Tennessee
UCL	Upper Control Limit
URS	URS Corporation
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
VT	Vermont
WP	Work Plan

1.0 INTRODUCTION

The project team executing the Preliminary Design Investigation (PDI) activities at the Former Philip Services Corporation (PSC) site in Rock Hill, South Carolina, consists of URS Corporation (URS) staff along with subcontractors as described in the following section.

1.1 Distribution List

The following individuals will receive a copy of the approved Quality Assurance Project Plan (QAPP) and all subsequent revisions.

Individual and Title	Organization	Telephone Number	Fax Number	E-mail Address
J. Lucas Berresford Project Manager	SC DHEC	(803) 896-0747	(803) 896-0980	berresjl@dhec.sc.gov
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Brad Weichert Onsite Lab Director	KB Labs, Inc.	(352) 367-0073	(352) 378-6491	bradw@kbmobilelabs.com
Todd Romero Onsite Lab Project Manager	KB Labs, Inc.	(352) 367-0073	(352) 378-6491	toddr@kbmobilelabs.com
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Amelia Kennedy Offsite Lab Director	TestAmerica Nashville	(615) 726-0177	(615) 726-3404	amelia.kennedy@testamericainc.com
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Pat Beaver Offsite Lab Director	Beaver Engineering, Inc.	(615) 350-8124	(615) 350-8149	pat@beaverengineering.com

Individual and Title	Organization	Telephone Number	Fax Number	E-mail Address
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Chris Beaver Offsite Lab QA Officer	Beaver Engineering, Inc.	(615) 350-8124	(615) 350-8149	chris@beaverengineering.com

The QAPP will be read by all essential staff participating in the work effort. The QAPP will be in the possession of the field teams and in all laboratories performing analytical services. All contractors and subcontractors will be required to comply with the procedures documented in the QAPP in order to maintain comparability and representativeness of the data produced.

1.2 Project/Task Organization

This QAPP addresses the PDI activities at the Former PSC site. The specific individuals participating in the project are presented in **Figure 1-1**, and the various quality assurance (QA) and management responsibilities of key project personnel are described below.

SC DHEC Project Manager

Mr. Lucas Berresford of South Carolina Department of Health and Environmental Control (SC DHEC) is the Project Manager (PM) and has oversight responsibility for all phases of this work effort.

SC DHEC SQAMO Designee

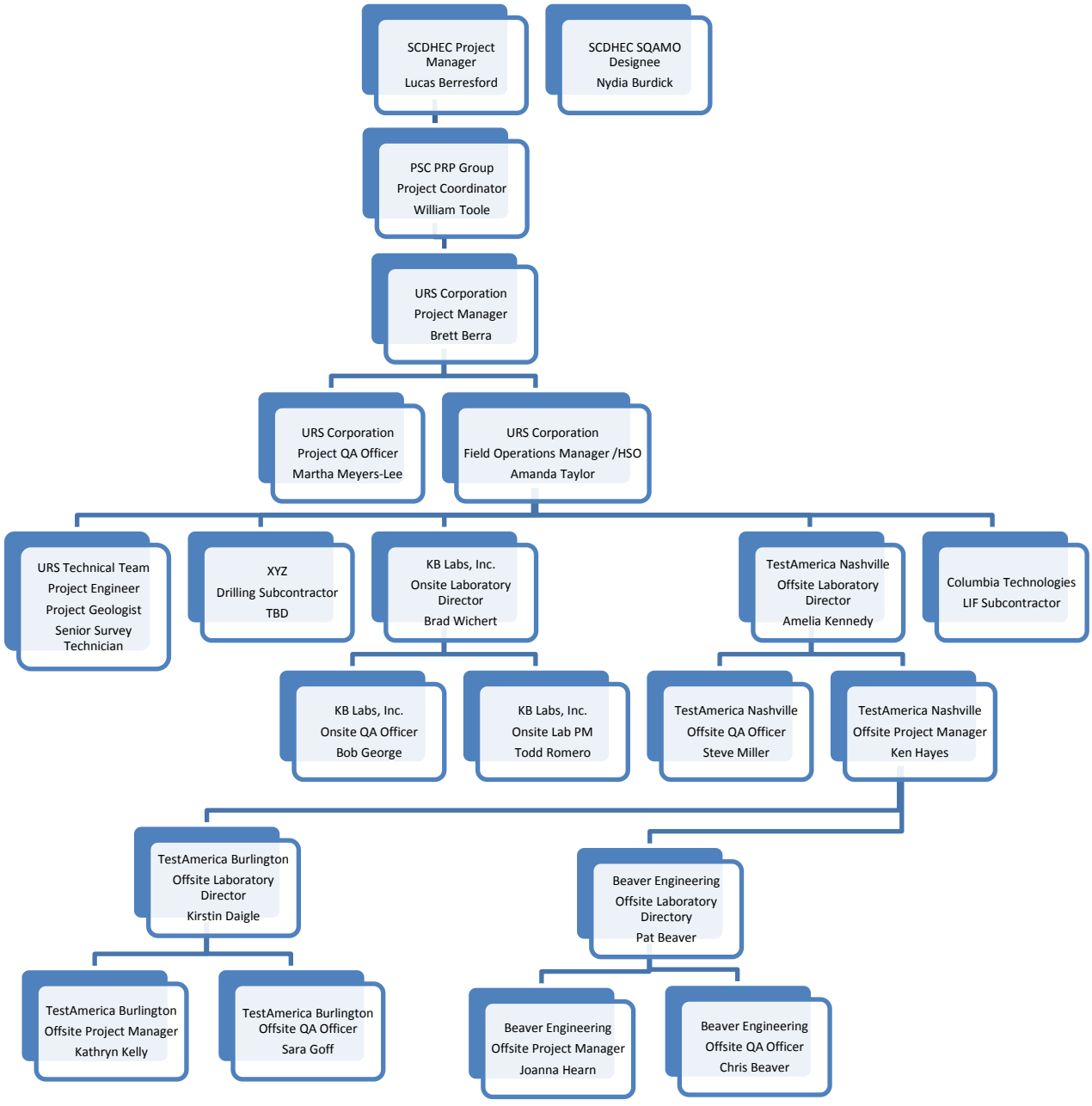
Ms. Nydia Burdick, the SC DHEC State Quality Assurance Management Office (SQAMO) Designee, has the responsibility to review and approval all QAPPs.

URS Project Manager

Mr. Brett Berra, P.E., URS PM, will be responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The primary function of the URS PM is to ensure that technical, financial, and scheduling objectives are achieved successfully. The PM will:

- Identify appropriate personnel and subcontractors to accomplish objectives efficiently
- Delegate applicable authority to various team members to complete certain tasks
- Monitor conformance to project scope and, within his authority, adapt the scope to fit site conditions as permitted by the Work Plan (WP), budget, and schedule

Figure 1-1. Project Organization Chart



- Provide senior-level technical review
- Coordinate preparation of project plans and approve deliverables before submittal to SC DHEC
- Maintain the official, approved QAPP for the project
- Ensure that the project is completed on schedule and within budget
- Ensure adequate communications between all project stakeholders is maintained, corresponding with SC DHEC on technical issues at critical points during each task

URS Field Operations Manager/Health and Safety Officer

Ms. Amanda Taylor will serve as the Field Operations Manager during field activities. Ms. Taylor will be responsible for leading and coordinating the day-to-day activities of the various resource specialists.

Additionally, Ms. Taylor also serves as the Health and Safety Officer (HSO) and is responsible for the health and safety of URS personnel on site. Specific responsibilities include:

- Provide day-to-day coordination with the URS PM on technical issues
- Develop and implement field-related WPs, adhere to schedule and management-developed study requirements
- Coordinate and manage field staff including URS and subcontractor personnel during sampling, drilling, and in-field analysis activities
- Implement quality control (QC) procedures for technical data provided by the field staff, including field measurement data
- Identify problems at the field team level, discuss possible resolution actions with the URS PM, and provide communication between the field team and management
- Participate in preparation of reports

URS Senior Technical Advisors

Mr. Rob MacWilliams, Senior Hydrogeologist, and Mr. Bob Lunardini, Lead Engineer, will serve as senior technical advisors. They will be responsible for the technical direction of the project, developing scientifically based assumptions and providing support for the ultimate design of the site remediation.

URS Project QA Officer and Senior Chemist

Ms. Martha Meyers-Lee is a Senior Chemist that will serve as the Project QA Officer. She has overall responsibility to independently assure that the planning, implementation, and reporting of PDI activities fulfill the objectives for data use. This includes data evaluation/validation implementation, database information flow, and identification and communication of problems that affect data quality and schedule. The Project QA Officer will have reporting responsibility outside of the project organization to assure independence in decision-making and in recommending corrective actions. The Project QA Officer is responsible for assuring that needed corrective actions are implemented.

URS Technical Team

URS technical staff will be used to collect samples, gather and analyze data, and prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

URS staff will collect groundwater field measurements, including pH, temperature, specific conductance, dissolved oxygen, and oxidation reduction potential (ORP). However, URS is not certified by SC DHEC for the analysis of these field parameters and the data collected will be used for informational purposes only.

Each project deliverable will be reviewed by senior-level URS technical personnel. The intent of the reviews will be to check for completeness, consistency, and overall quality of the data interpretations. The senior technical staff to conduct the review will be selected by the URS PM. These people will be selected based upon their experience in the specific disciplines applicable to the document under review.

1.2.1 Subcontractors

Analytical Laboratories

KB Labs, Inc. of Gainesville, FL, will analyze soil samples that are collected for a volatile organic analysis (VOA) by the onsite laboratory using United States Environmental Protection Agency (USEPA) Methods 5035 and 8260B. Mr. Todd Romero will serve as the Laboratory PM for the project. The laboratory is certified by SC DHEC under license number 96040001 for those compounds that are drivers for PDI decision making purposes. KB Labs will also screen soil for four volatile organic compounds (VOCs) (i.e., acetone, benzene, methylene chloride, and 1,2,4-trichlorobenzene) that they are not certified for as April 2014, and the data will be flagged accordingly. KB Labs is in the process of obtaining SC DHEC laboratory certification

for benzene, methylene chloride, and 1,2,4-trichlorobenzene by SW-846 Methods 5035 and 8260B.

TestAmerica of Nashville, TN, will analyze groundwater and surface water samples that are collected as part of the PDI. Samples will be shipped to the laboratory via overnight courier for analysis. The TestAmerica Nashville laboratory will analyze samples for VOCs by USEPA 5030B/8260B, nitrate by USEPA 9056A, and iron by USEPA 6010C. Mr. Ken Hayes will serve as the Laboratory PM for the project, and is responsible for coordination, oversight, and reporting of all geotechnical work that is being conducted by their sister and subcontract laboratories. The laboratory is certified by SC DHEC to conduct the required analyses under license number 84009001.

TestAmerica of Nashville, TN, will subcontract all soil geotechnical analyses, except percent saturation, to TestAmerica of Burlington, VT. Percent saturation determinations will be conducted by Beaver Engineering, Inc., of Nashville, TN. Ms. Kathryn Kelly of TestAmerica Burlington and Ms. Joanna Hearn of Beaver Engineering will serve as the Laboratory PMs.

Specific responsibilities of laboratory personnel are as follows:

Laboratory Project Manager

- Ensure all resources of the laboratory are available on an as-required basis
- Perform initial review of a project's analytical scope of work and manage the project while samples are being analyzed in the laboratory
- Review the final report

Laboratory Departmental Managers

- Coordinate laboratory analyses within the department
- Schedule sample analyses
- Conduct detailed data review at the departmental level
- Prepare laboratory Standard Operating Procedures (SOPs)

Laboratory QA Officer

- Supervise laboratory quality assurance
- Supervise QA/QC documentation
- Review data for corrective actions, if required

Laboratory Sample Custodian

- Receive and inspect the incoming sample containers
- Record the condition of the incoming sample containers
- Sign appropriate documents
- Verify chain-of-custody (CoC) and its correctness
- Notify the PM and laboratory analysts of sample receipt and inspection
- Assign a unique identification number and customer number, and enters each number into the Laboratory Information Management System (LIMS)

Driller

A SC DHEC certified drilling contractor will be contracted to perform rotosonic and DPT drilling at the site. URS will provide SCDHEC with the drilling supervisor and SCDHEC drilling license as an amendment to this QAPP.

Laser Induced Fluorescence Investigation Subcontractor

Columbia Technologies of Baltimore, MD, will be contracted to perform laser induced fluorescence (LIF) investigation at the site.

1.3 Problem Definition/Background

This section presents a brief background and overview of remedial activities for the site. Additional information may be found in Section 1 of the PDI WP (URS, 2012).

1.3.1 Site Description and Background

The PSC site in Rock Hill, South Carolina, is a former Resource Conservation and Recovery Act (RCRA) hazardous waste treatment, storage, and disposal facility. Operations began at the site in 1966 and continued until the bankruptcy of PSC in December 2003, at which time the SC DHEC assumed the environmental management responsibilities of the site. Several previous investigations at the site have identified chemical releases to soil and groundwater, and some remediation has been performed. Current remediation consists of groundwater extraction through three vertical and one horizontal extraction well, treatment using liquid phase carbon, and discharge to the local sanitary sewer.

The PSC site is located at 2324 Vernsdale Road, approximately 4.5 miles southwest of the City of Rock Hill in South Carolina (refer to **Figure 1-2**). Robertson Road borders the industrial portion of the property to the northeast, and the Norfolk Southern Railroad forms the northwestern boundary. Wildcat and Fishing Creeks border the industrial property on the

southeast and southwest, respectively. The site consists of approximately 44.5 acres of industrial property on the west side of Wildcat Creek and approximately 108 acres of undeveloped woodland on the east side of Wildcat Creek. Additional information pertaining to site location may be found in Section 1.1 of the PDI WP.

1.3.2 Site History

Section 1.2 of the PDI WP presents the site history and potential source areas for contamination.

1.3.3 Site Constituents of Concern

Several media and constituents of concern (COCs) are associated with the site; however, the primary COCs are VOCs. Additional information is provided in Section 1.2 and Table 6 of the PDI WP.

1.3.4 Problem Definition

Several previous investigations at the site have identified chemical releases to soil and groundwater, and some remediation has been performed. The feasibility study conducted in March 2011 (CDM, 2011) for the site recommended a remedial action alternative for the source area that involved a combined soil and groundwater remedy of thermal-enhanced multi-phase extraction (MPE) and in-situ thermal treatment. Under the anticipated remedy, in-situ thermal treatment of soil and regolith groundwater will be applied to the areas of higher VOC concentrations to rapidly reduce the contaminant mass and significantly reduce the time required to complete remediation. The location of the thermal treatment application will be better defined following the PDI. The area of treatment will be refined during the remedial design based on one or more of the following factors: PDI results, fate and transport modeling, and pilot-scale test results.

The in-situ thermal treatment will not be applied at all locations where VOC concentrations exceed the remediation goals (RGs) for soil and groundwater. However, this technology will accomplish remediation beyond the treatment zone through enhanced degradation and volatilization. As a result, post treatment monitored natural attenuation of groundwater, at a minimum, will be necessary for both the regolith and bedrock zones.

Additional data collection is necessary to accurately define the thermal treatment footprint of the source areas.

1.4 Project/Task Description and Schedule

1.4.1 Summary of Work and Objectives

To effectively design a treatment area for the proposed thermal remedy, additional data is needed. The primary objective of the PDI will be to generate additional data to identify "hotspot" locations within the delineated source areas that will potentially require treatment. The following data collection activities have been identified:

1. Source area soil sampling and analysis to refine source areas potentially requiring treatment. Existing soil data will be supplemented to design the placement, configuration, and duration of thermal treatment.
2. Burn Pit Area soil sampling and analysis to determine if Soil Vapor Extraction (SVE) or another form of soil treatment is required to address any residual soil impacts potentially acting as a continuing source of groundwater impact.
3. Aquifer testing to more accurately define the hydraulic characteristics of the regolith/transition zone to support the design of thermal treatment. The information will supplement existing aquifer performance data to calibrate anticipated future fate and transport modeling.
4. Comprehensive baseline groundwater water levels to prepare accurate and updated potentiometric maps for each hydraulic zone.
5. Comprehensive groundwater and surface water sampling and analysis to establish a baseline of COCs prior to pilot-test and remedy implementation.
6. Surface water flow measurements and stream gauging for model calibration.

Additional details on the planned scope of work, including sampling collection activities, are presented in the Section 3 of the PDI WP (URS, 2012) and Section 2.1 of this QAPP. Field activities will be conducted using methods and procedures described in the PDI WP and Appendices A and B in this plan. Analytical work will be conducted in accordance with each laboratory's Quality Manual and the SOPs that are presented in Appendix C. The laboratory will have a quality program in place that is comparable to the USEPA Contract Laboratory Program (CLP) or alternatively meets National Environmental Laboratory Accreditation Program (NELAP)/National Environmental Laboratory Accreditation Conference Institute (TNI) standards.

1.4.2 Project/Task Schedule

The schedule for the PDI activities is presented in Section 4.3 of the PDI WP. A general outline of the schedule is as follows:

Activity	Duration (weeks)	Anticipated Start Date	Anticipated Completion Date
Regulatory review and approval of the QAPP	4 weeks	May 2014	June 2014
Preparation for field activities	3 weeks	June 2014	June 2014
Implementation of field activities, including on-site mobile laboratory analyses	8 weeks	July 2014	August 2014
Off-site commercial laboratory analyses	2 weeks after sample receipt	July 2014	August 2014
Data validation	3 weeks after receipt of final laboratory deliverable	August 2014	September 2014
Report development, review, and submittal to SCDHEC	6 weeks	September 2014	November 2014

Implementation of the PDI will begin following approval of this QAPP and final PDI WP by SC DHEC. A delay in schedule due to time and resource constraints is not anticipated; however, if a delay occurs, SC DHEC will be notified.

1.5 Quality Objectives and Criteria for Measurement Data

This section provides internal means for control and review so that environmentally related measurements and data collected by URS are of a known quality. Data collected on this project will be used to further delineate impacted areas and establish pre-remediation baseline conditions for groundwater and surface water.

When conducting the PDI, all measurements will be made so that results reflect the medium and conditions being measured. Prior to all environmental measurement activities, site-specific Data Quality Objectives (DQOs) and measurement performance criteria will be determined. DQOs are qualitative and quantitative statements that specify the quality of the environmental monitoring data required to support decisions. The subsections below describe the DQOs (Section 1.5.1) and data measurement objectives (Section 1.5.2).

1.5.1 Data Quality Objectives

EPA's DQO process was used to develop the data collection design, which is discussed in detail in the Sections 3 and 4 of the PDI WP. The seven steps of the DQO process (EPA 2006) have been adapted to meet the requirements of this project and create the data collection design. The steps are discussed below:

1.5.1.1 Step 1: State the Problem

The purpose of this step is to describe the problem to be studied so that the focus of the study will be unambiguous. Based on the Feasibility Study (CDM, 2011) and review of historical data, several data gaps have been identified:

- Source areas have been delineated, but the locations within them that potentially require treatment are unknown
- Burn Pit Area residual soil impacts potentially acting as a continuing source of groundwater impact are unknown
- Hydraulic characteristics of the regolith/transition zone are unknown
- A current potentiometric map for each hydraulic zone is not available as the last gauging event occurred in 2007
- A baseline of COCs prior to pilot-test and remedy implementation is not available as the last sampling event occurred in 2007
- Surface water flow and stream gauging data for model calibration are not current

1.5.1.2 Step 2: Identify the Decision

This step identifies the questions the study will attempt to resolve and the actions that may result. The principal study components and related questions are:

Question 1 What is the extent of soil, groundwater, and surface water contamination that potentially requires treatment at the site?

Question 2 What is an appropriate design for a remediation program using thermal treatment and possibly other technologies for specific purposes (e.g., SVE for the Burn Pit Area)?

Based on the questions above, the following scenarios and possible actions have been identified:

Scenario 1

Soil, groundwater, and surface water data collected during the investigation fully define the nature and extent of contamination. Source areas potentially requiring treatment will be identified.

- The model is calibrated and an accurate potentiometric map is developed based on current site conditions
- Anticipated future fate and transport modeling can be performed
- A remediation program using thermal treatment and possibly other technologies is designed

Scenario 2

The extent of contamination in the soil, groundwater, and surface water are not fully defined during the investigation.

- With incomplete data, the remedial design for the thermal treatment footprint of the individual source areas will be inaccurate

1.5.1.3 Step 3: Identify the Inputs to the Decision

The purpose of this step is to identify the information and data that need to be obtained and the measurements that need to be taken to resolve the decision statement. Based on the problem stated in Step 1, the following information is required:

- VOC concentrations in surface and subsurface soil to delineate the nature and extent of contamination.
- LIF probing data from approximately 39 locations to identify potential zones of free product in the subsurface.
- Single-well hydraulic test data for a subset of existing wells to obtain estimates of hydraulic conductivity of the saprolite, transition zone, and the fractured bedrock as well as to evaluate variations in site transmissivity and groundwater velocity. This data will also be used to calibrate future predictive groundwater modeling if required for the site.
- Groundwater elevations from existing monitoring wells to provide current data from which predictive groundwater flow and transport modeling can be completed and efficacy of future remedial actions can be measured.

- Chemical analysis of groundwater samples for site-specific COCs (i.e., VOCs) and monitored natural attenuation (MNA) parameters to establish pre-remediation baseline conditions.
- Chemical analysis of surface water from seven (7) locations at Fishing and Wildcat Creeks to provide a baseline of possible COCs and flow measurements of the creeks prior to implementation of a remedy. In addition, surface water measurements are needed to calibrate the groundwater fate and transport model discussed previously.

Source area data will be used to identify "hotspot" locations within the delineated source areas that will potentially require treatment. Soil sample VOC results will be compared to Industrial Soil Screening Levels, *Regional Screening Levels for Chemical Contaminants at Superfund Sites* (USEPA, May 2013). SC DHEC risk-based screening levels (RBSLs), which are the same as EPA Maximum Contaminant Levels/Maximum Contaminant Level Goals (MCLs/MCLGs) are the RGs for groundwater. Surface water screening values (chronic) presented in the *USEPA Region 4 Ecological Risk Assessment Bulletin* (USEPA, 2001) are the RGs for surface water.

1.5.1.4 Step 4: Define the boundaries of the Study

This step defines the spatial and temporal boundaries of the study.

The horizontal spatial boundaries of the study area include the approximately 45 acres of industrial property on the west side of Wildcat Creek and the approximately 108 acres of undeveloped woodland on the east side of Wildcat Creek. The industrial portion of the property is bounded by Robertson Road to the northeast and Vernsdale Road to the northwest. Wildcat and Fishing Creeks border the industrial property on the southeast and southwest, respectively. The vertical spatial boundaries are from ground surface to the depth of the deepest onsite monitoring well (bedrock zone). This study focuses on current conditions and, therefore, temporal boundaries include the period for the Remedial Investigation/Feasibility Study (RI/FS) (2006 to 2007) (CDM, 2008) as well as the FS (CDM, 2011). The main data used for decision-making will be collected from current conditions although the contamination may have originated at any time over the past 40 years (approximately).

The data needed to support decision making for this investigation include the chemical concentrations (including both detected and non-detected values) for all media sampled and analyzed. Constraints that could potentially interfere with data collection are physically inaccessible sampling locations and a limited number of sampling events.

1.5.1.5 Step 5: Develop a Decision Rule

The purpose of this step is to define the parameter of interest, specify the action level, and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions. The parameters of interest are the concentrations of constituents identified in each media. These concentrations should estimate the true values of the constituents and may be used on an individual (e.g., trichloroethylene) basis or cumulatively (e.g., total VOCs).

The development of decision rules for the site involves using lithological, hydrological, and analytical data collected during the investigation to effectively design a treatment area for the proposed thermal remedy. The decision rule is:

- If data from the PDI allows for an accurate design of the thermal treatment footprint and probable treatment duration, then it will be possible to proceed with fate and transport modeling and subsequent selection of locations for thermal treatment at the site.

1.5.1.6 Step 6: Specify Tolerable Limits on Decision Errors

In general, decision errors for projects involving environmental sampling fall into two categories: false positive (Type I) and false negative (Type II). For this project, a Type I decision error would result in deciding that contaminant concentrations in various media present an unacceptable risk when they do not. A Type II decision error would result in deciding that contaminant levels do not present an unacceptable risk to human health and the environment when they actually do.

Type II decision errors are more serious than Type I errors because they could possibly mask contaminant levels that may pose a risk. In order to manage the possibility of Type II errors, quality assurance procedures (field and laboratory) and data validation will be performed as outlined in this QAPP.

1.5.1.7 Step 7: Optimize the Design for Obtaining Data

This step identifies a resource effective data collection design for generating data that are expected to satisfy the DQOs.

The data collection design (sampling program) is described in detail in the Section 3 and 4 of the PDI WP.

1.5.2 Data Measurement Objectives

The precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) of the sampling and analytical procedures must be adequate to allow the data to be used to effectively design a treatment area for the proposed thermal remedy. Every reasonable attempt will be made to obtain a complete set of usable field measurements and analytical data. If a measurement cannot be obtained or is unusable for any reason, the URS PM and URS Project QA Officer will evaluate the effect of the missing data. This evaluation will be reported to SC DHEC with a proposed corrective action.

The measurement performance criteria (MPC) define the quality elements monitored and the acceptable performance for these elements. Tables 1-1 through 1-7 describe the MPC for precision, accuracy, and sensitivity for the VOAs. MPC for all other parameters will be per the analytical methods and laboratory SOPs. For sensitivity, achievement of limits listed in Tables 1-5 through 1-7 is sufficient to achieve the project objectives for soil, groundwater, and surface water, respectively.

1.5.2.1 Precision

Precision is the agreement between a set of replicate measurements without assumption and knowledge of the true value. Both field and analytical precision is assessed based on the results of field duplicate sample analyses. Analytical precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. This is accomplished in the laboratory by calculating the relative percent difference (RPD) between laboratory duplicate sample results, or the RPD between a spike and spike duplicate results (i.e., matrix spike (MS) and matrix spike duplicate (MSD), or laboratory control sample (LCS) and laboratory control sample duplicate (LCSD)). RPD is calculated using one of the following methods:

$$RPD = \frac{(R_1 - R_2)\,x\,100}{R_{Bar}} \quad \text{or} \quad RPD = \frac{(S_1 - S_2)\,x\,100}{S_{Bar}}$$

where:

R_1 and R_2 are the first and duplicate results, respectively

R Bar is the average of the two duplicate results

S_1 and S_2 are the spike and duplicate spike results, respectively

S_{Bar} is the average of the two duplicate spike results.

Historical laboratory limits for RPD are determined from pairs of either replicates or spikes. The RPDs will be greater than zero to determine upper warning and control limits.

Field duplicate samples will be collected at the frequency indicated in Tables 1-8 through 1-10. Laboratory duplicate analyses will be performed as recommended by USEPA methods. The required level of precision for each matrix is designated in Table 1-1.

1.5.2.2 Accuracy

Accuracy is the nearness of a measurement or the mean of a set of measurements to the true value. Accuracy is assessed by the analysis of reference samples and recovery of spiked samples. Sample matrix accuracy is determined by comparing the recovery of target analytes that are spiked into a sample as a MS or MSD to laboratory control limits. Analytical method accuracy is measured by comparing the recovery of target analytes that are spiked in the LCS of the same matrix as the samples, to a control limit. The percent recovery will be calculated using the following equation:

$$P = 100 \times (A - B)/T$$

where:

P is the percent spike recovery

A is the concentration determined on spiked sample

B is the concentration determined on original unspiked sample

T is the true value of spike added.

Accuracy requirements are listed for each method in Table 1-1.

1.5.2.3 Representativeness

Representativeness is defined for each sampling and analysis task and is a function of the investigative objectives. Representativeness is achieved through use of the standard field, sampling, and analytical procedures. Representativeness is determined by appropriate program design, with consideration of elements such as proper sampling locations, procedures, and target species.

1.5.2.4 Comparability

Comparability is the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard

and comprehensive reporting formats. Complete field documentation using standardized data collection forms will support the assessment of comparability. Historical comparability will be achieved through consistent use of methods and documentation procedures throughout the project.

1.5.2.5 Completeness

Completeness is calculated for the aggregation of data for each analytical group measured for any particular sampling event or other defined set of samples. Completeness is calculated and reported for each analytical method and sample matrix. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an "R" flag (refer to Section 4.2 for an explanation of validation flagging criteria). For any instances where samples could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, spoiled samples, etc.), the numerator of this calculation becomes the number of possible results minus the number of possible results not reported. The formula for calculation of completeness is presented below:

$$\% \text{ Completeness} = \frac{\text{Number of valid results (i.e., non-R-flagged data)}}{\text{Number of possible results}} \times 100$$

The completeness goal for this project will be 90% based on the planned samples for each sample matrix, and will be determined separately for each analytical method. If the goal is not met, the effect of not meeting the goal will be discussed with the URS PM.

1.5.2.6 Field Measurement Data QA Objectives

DQOs for field measurements are discussed in Section 4.1 of the PDI WP.

Horizontal and vertical accuracy requirements for survey data are presented in Section 3.4.1 of the PDI WP.

Accuracy and precision requirements for all field measurements (i.e., temperature, pH, turbidity, dissolved oxygen, specific conductivity, and oxidation-reduction potential) obtained during low-flow groundwater sampling are presented in Section 3.4.2.2 of the PDI WP. Accuracy specifications for the parameters analyzed by the Color Tec® methods are presented in Appendix B. The accuracy and precision of field measurements involves operating equipment in accordance with operating manuals, conducting daily calibrations, and checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard. A copy of the field equipment operating manuals is provided in Appendix B.

1.5.2.7 Laboratory Analytical QA Objectives

The DQOs developed for data collection during the PDI are intended to ensure that the analytical data generated during the design project are of sufficient quality to support their respective intended uses. DQOs for analytical data generated by the onsite and offsite laboratories are discussed in Section 4.1 of the PDI WP and Table 1-4. MPC are presented in Tables 1-1 through 1-3. Tables 1-5, 1-6, and 1-7 define the DQO level required for each type of sample to be collected during the PDI. These DQO levels are consistent with those presented in the PDI WP.

To achieve DQO Level IV, all laboratory data packages associated with groundwater and surface water sampling events will be provided in a "CLP-like" format that contains all supportive data necessary to reconstruct analytical work (e.g., raw data). The onsite mobile and geotechnical laboratories will provide a data package that presents a summary of sample and associated laboratory QC results with control limits.

1.5.3 Special Training Requirements and Certifications

Qualified personnel will perform all investigations, characterizations, and sampling. Staff assigned to perform sampling, field data collection, and provide scientific or engineering opinions will have the requisite education and certification (as necessary) to perform these tasks.

The Site-specific Health and Safety Plan (HASP) requires that personnel working on project-related field tasks be trained in accordance with the Occupational Safety and Health Administration (OSHA) regulations. This takes the form of URS supplied training (e.g., cardiopulmonary resuscitation [CPR], First Aid, OSHA Hazardous Waste Operations and Emergency Response [HAZWOPER]), work area specific training (e.g., work zone safety around roads and highways), and client-/site-specific training. This training is documented both on cards given to individual employees and on training record forms, and is tracked in a database.

The URS Field Operations Manager will be supported by personnel that are familiar with South Carolina protocols and have field experience related to sample collection, packing and shipment, decontamination, and documentation.

Laboratories are to be certified by SC DHEC for each method and parameter (except acetone, and field and geotechnical parameters) to be performed for the PDI. KB Labs, Inc. will analyze soil for VOCs by SW-846 Methods 5035 and 8260B using an on-site mobile laboratory. The laboratory is certified by SC DHEC under license number 96040001 for those compounds that

are drivers for PDI decision making purposes. KB Labs will also screen soil for four VOCs (i.e., acetone, benzene, methylene chloride, and 1,2,4-trichlorobenzene) that they are not certified for as of April 2014, and the data will be flagged accordingly. KB Labs is in the process of obtaining SC DHEC laboratory certification for benzene, methylene chloride, and 1,2,4-trichlorobenzene by SW-846 Methods 5035 and 8260B.

1.6 Documentation and Records

This section presents the procedures for documentation and records management.

1.6.1 Project Documentation and Records

The URS PM will be responsible for distributing the most current approved version of the QAPP to those individuals on the distribution list and documenting distribution in the project file. The QAPP will be distributed electronically to the team members listed on the distribution list. If QAPP revisions are required and the changes only involve a few pages, the pages may be sent out with directions explaining which pages to replace in the QAPP.

Instructions and procedures for documenting field activities are outlined in Section 4.2.2 of the WP.

Project documents will be controlled through an organized project filing system. Analytical/technical files will include work products generated during the project. Field books, field observations, photographs, and other field related documents will be prepared and placed in the project file. Subcontract deliverables will be reviewed for accuracy and completeness.

Data received from the field, subcontractors, or private sources will be tabulated on a spreadsheet or database and will be subject to QC procedures, including comparing raw data to the original source, verifying calculations, and confirming data summaries. Data distribution will not occur until data review has been completed.

Work products will be checked before final use. This includes checking calculations, reports, plans, etc. with various levels of review. The URS PM will review the work as an element of his project responsibilities. Also, all deliverables/work products will be subject to a discipline-specific technical review.

1.6.2 Laboratory Data Package Deliverables

The laboratory will provide an electronic copy (PDF format) of each laboratory data package to the URS Project Chemist or designee. All data packages associated with groundwater and surface water sampling will be provided in a “CLP-like” format, containing all supportive data necessary to reconstruct analytical work (e.g., raw data).

All onsite mobile laboratory deliverables will presents a summary of sample and associated laboratory QC results with control limits. The following information is required to be included in each laboratory data package, at a minimum:

- Laboratory Certification Number
- Facility name & address
- Date of report preparation
- Case Narrative
- Summary of Analytical Results, including:
  - Sample identification and the corresponding laboratory identification
  - Date of sample collection and laboratory receipt
  - Sample matrix
  - Dates of and methods of preparation/extraction and analysis
  - Weight or volume of sample used for preparation/extraction and analysis
  - Dilution or concentration factor for the samples
  - Definitions of all data qualifiers and acronyms
  - Method detection limits (MDLs) and Reporting Limits (RLs), adjusted for sample preparation activities
  - Analytical results
  - Units of measure
- Summary of QC Results, including the following as applicable by the method:
  - Laboratory blank results
  - Surrogate recoveries and control limits
  - LCS results with percent recoveries and control limits for all target analytes
  - Laboratory duplicate results with RPD and control limits for all target analytes
  - MS and MSD results, recoveries, RPD, and control limits for all target analytes

For each QC measurement, the theoretical value, the quality objective, and the calculated error (in terms of the quality objective for the measurement) will be maintained in a permanent record. It will be clear from the QC data report that the correct QC measurements have been made for the method employed and what the outcome was. Relevant QC measurement data will be reported with the results for each sample.

MDLs and sample results will be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise.

Soil sample results are to be reported on a dry weight basis. The percent moisture is to be included in the laboratory report.

Two electronic data deliverables (EDDs) will be required for the project database. One EDD will be formatted in Microsoft Excel for the EnviroData program (version 1.6 data transfer standard), and will include both field and laboratory QC (i.e., method blank, LCS, MS, MSD, and surrogate) results. The second EDD will be a field sample result cross-tabular summary that is formatted in Microsoft Excel.

1.6.3 Electronic and Digital Data Storage

URS will employ an environmental information management system for the site. Project environmental data will be managed using EnviroData©, a software application that integrates a relational database with graphing, mapping, and reporting. The relational database can be used to electronically import historical and current data from the field and analytical laboratories, as well as manually input and edit data. Capabilities include a powerful and flexible data validation/verification sequence based upon laboratory and project limits that are created and entered by the data management team. Project information, using Enviro Data©, can also be directly connected to ArcGIS, a Geographic Information System (GIS), for data visualization, presentation, and analysis.

Data and information that may be stored on a computer or network may include calibration, maintenance records, raw field data, reports, procedures, or subcontracted analytical data. Any data is stored either on individual company-owned computers or the corporate network. In either case, access to the computer or network is limited, by use of password, to authorized personnel only. If desired, access can be accommodated on a project-by-project basis, by the Project Manager. All network data and database data are backed-up regularly. The office network has redundant server storage drives. All new files are backed up on tape daily. Full tape backups are made bi-weekly and archived securely off-site. Electronic documentation associated with the project may be copied to CDs, DVDs, or other portable electronic media and placed with physical project documentation.

All computers and operating systems will be operated in conditions within the specifications of their manufacturer.

1.6.4 Document Control

The term document control refers to the maintenance and inspection of project files. The project file will be maintained in a secure area. All official and original documents relating to the PDI will be placed in the project file. The following documents will be placed in the project file:

- WP
- HASP
- QAPP
- CoC records
- Field logbooks
- Records obtained during the investigation
- Laboratory deliverables
- Data validation reports
- PDI report
- Detail checking reports
- QA audit reports
- Official correspondence received from or sent to SC DHEC relating to the investigation
- Photographs associated with the project

Records and documents that relate to the site will be preserved and retained for a minimum of ten years after the work has been completed. The URS PM will review the files at the conclusion of the project to ensure that they are complete. Any transfer of results or records will be done in a way to ensure that client confidentiality is maintained.

Records may be maintained in a hard copy (physical) or an electronic format in a centralized file. All electronic records are maintained on the office's local area network. A complete read-only electronic copy of the final deliverable is stored in an assigned location on the local area network (LAN) in a restricted folder. Physical records are stored in file cabinets and/or other repositories in an accessible location, with suitable security.

2.0 MEASUREMENT / DATA ACQUISITION

This section of the QAPP describes all data collection activities to be conducted during the PDI at the PSC site. Refer to Section 3.0 of the PDI WP for additional details on sampling procedures and requirements.

2.1 Sampling Process (Experimental Design)

Although the site has been the subject of several sampling investigations and some remediation, additional source area data are required to identify "hotspot" locations within the delineated source areas that will potentially require treatment. Rationale for soil boring locations is presented in Table 5 of the PDI WP. The data will be used to design remediation program using thermal treatment and possibly other technologies for specific purposes (e.g., SVE for the Burn Pit Area). As discussed in Section 3.1.3 of the PDI WP, proposed subsurface sampling locations are presented on Figures 12 and 13 of the PDI WP.

2.1.1 Source Area Soil Sampling

Source area soil sampling will be conducted to refine the vertical and horizontal dimensions of the area requiring treatment. The sampling approach and proposed sample locations are identified in Section 3.2.1 of the PDI WP and presented on Figures 14 and 15 of the PDI WP. Field work will be conducted according to procedures in this plan's Appendices A and B. Initially, thirty-two (32) soil samples will be collected to determine if site-specific VOCs are present at concentrations greater than the RGs. Additional soil samples may be collected to further define potential treatment areas. Six (6) soil samples will also be collected for the analysis of geotechnical parameters (i.e., grain size, bulk density, wet unit weight, specific gravity, air-filled porosity, and percent saturation). To expedite receipt of results, samples will be analyzed for all site-specific VOCs. The laboratory is certified by SC DHEC for those compounds that are drivers for PDI decision making purposes. KB Labs will also screen soil for four VOCs (i.e., acetone, benzene, methylene chloride, and 1,2,4-trichlorobenzene) that they are not certified for as April 2014, and the data will be flagged accordingly. KB Labs is in the process of obtaining SC DHEC laboratory certification for benzene, methylene chloride, and 1,2,4-trichlorobenzene by SW-846 Methods 5035 and 8260B. Soil samples requiring geotechnical analyses will be shipped via overnight courier to the laboratory.

Soil samples will be analyzed for site-specific VOCs by USEPA Method 5035/8260B, as indicated in Table 1-5. The methods for determining geotechnical parameters are also provided in Table 1-5. Volatile organic soil sample results will be reported by the onsite laboratory on a dry-weight basis. QC samples will be collected at the frequency indicated in Table 1-8.

Investigation-derived waste (IDW) will be managed according to procedures provided in Section 4.2.4 of the PDI WP and Appendix A of this plan.

2.1.2 Light Non-Aqueous Phase Liquid Delineation

Further delineation of the lateral and vertical extent of Light Non-Aqueous Phase Liquid (LNAPL) will be completed using Direct Push Technology (DPT) to advance a LIF probe at approximately 39 locations within the known LNAPL plume. The LIF investigation will be conducted and results reported in accordance with Section 3.2.3 of the PDI WP and Appendix A of this QAPP.

2.1.3 Hydraulic Testing

Single-well hydraulic testing will be performed on a subset of existing wells to obtain estimates of hydraulic conductivity of the saprolite, transition zone, and the fractured bedrock. A system shutdown/recovery test will also be conducted. All hydraulic tests will be conducted in accordance with Section 3.3 of the PDI WP and Appendix A of this QAPP.

2.1.4 Plume Evaluation/Monitoring

A well location/elevation survey will be conducted and groundwater samples collected and analyzed from existing monitoring wells. The survey will provide current groundwater quality data from which predictive groundwater flow and transport modeling can be completed and the efficacy of future remedial actions can be measured. The approach and methodology for conducting the plume evaluation and comprehensive groundwater monitoring event is presented in Section 3.4.2 of the PDI WP (Table 4 and Figure 2 of the PDI WP).

Groundwater samples will be collected from existing monitoring wells and analyzed for select site-specific COCs. All groundwater samples will be analyzed by an offsite laboratory, which is certified by SC DHEC, for the Table 1-6 VOCs by USEPA Methods 5030B/8260B. Although bis(2-ethyl hexyl) phthalate is listed as a VOC in Table 6 of the WP, groundwater samples will not be collected for this compound because it is a semivolatile organic compound. In addition, groundwater from twelve (12) wells will be analyzed for nitrate and sulfate by USEPA Method 9056A and total iron by USEPA Methods 3010A and 6010C by a SC DHEC certified laboratory. QC samples will be collected at the frequency indicated in Table 1-9.

2.1.5 Surface Water Assessment

A surface water assessment will be conducted at Fishing and Wildcat Creeks in accordance with Section 3.5 of the PDI WP to provide a baseline of possible COCs in surface water and flow measurements prior to implementation of a remedy. Surface water measurements are also required for calibration of the groundwater fate and transport model. The approach for the

collection of surface water elevations and samples, and identification of proposed sample locations are presented in Section 3.5.1 of the PDI WP. The proposed surface water sample locations are presented in Figure 18 of the PDI WP. Surface water sampling procedures are also outlined in Appendix A of this plan.

Surface water samples will be collected from seven (7) locations for the analysis of site-specific VOCs and Total Suspended Solids (TSS). All samples will be analyzed by an offsite SC DHEC certified laboratory for Table 1-7 VOCs by USEPA Methods 5030B and 8260B and TSS by Standard Methods 2540D. QC samples will be collected at the frequency indicated in Table 1-10.

2.1.6 Portable Sampling and Monitoring Equipment

Hand-held portable monitoring equipment may be used during site investigation or monitoring events as sampling equipment or in addition to other sampling methodologies. All equipment utilized during sampling activities will be operated in accordance to respective operating manuals (refer to Appendix B). Equipment will be inspected and calibrated daily prior to use.

2.1.7 Performance Modifications

The URS PM or designee is responsible for all site activities. In this role, the PM is required to adjust the site program to accommodate site-specific needs. When it becomes necessary to modify the program addressed in this QAPP, the responsible person notifies the URS PM, who will then notify SCDHEC of the anticipated change. The change in the program will be conducted by the URS Field Operations Manager. SCDHEC will approve all changes in writing or verbally prior to field implementation, if feasible. If not approved, the action taken will be evaluated to determine the significance of any departure from established program practices.

Any nonconformance with the established procedures in the QAPP will be identified and corrected in accordance with this plan. A corrective action program will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the URS PM and Project QA Officer. No staff member will initiate corrective action without prior communication of findings through the proper channels. Implementation of corrective action will be confirmed in writing through the same channels. If corrective action is insufficient, work may be stopped by the URS PM or Project QA Officer.

The URS PM is responsible for the controlling, tracking, and implementing the identified changes to the plans. SCDHEC will be requested to approve all identified changes. Reports on all changes will be distributed to all affected parties, including the SCDHEC PM. The SCDHEC PM will be notified whenever program changes in the field are made. Changes to the QAPP will

be provided in an amended QAPP to all those on the distribution list. If the changes are major, a complete QAPP with new signature page and a revision number will be distributed.

2.2 Sampling Methods Requirements

The PDI WP and this QAPP are designed to ensure that samples are collected consistently between sampling locations, with no contamination being introduced. The use of standardized methods and trained personnel will also help ensure data quality and consistency.

Sampling and decontamination procedures and investigation-derived waste management for the PDI are provided in the Sections 3 and 4.2.4 of the WP, and Appendix A to this plan. A summary of sample container, preservation, storage, and holding time requirements is presented in Table 2-4.

2.3 Sampling Handling and Custody

The U.S. EPA Region IV sample custody, or CoC, protocols are described in the *Field Branches Quality System and Technical Procedures, Sample and Evidence Management* (EPA, January 2013).

A sample or evidence file is under your custody if it:

- It is in the actual possession of an investigator
- It is in the view of an investigator, after being in their physical possession
- It was in the physical possession of an investigator and then they secured it to prevent tampering
- It is placed in a designated secure area

2.3.1 Field Sample Collection Documentation Procedures

Field documentation procedures are provided in Section 4.2.2 of the PDI WP. All entries will be made in indelible ink with a ballpoint pen and will be written legibly. Entry errors will be crossed out with a single line, dated, and initialed by the person making the correction. A diagonal line and initial will be added at the end of unfilled logbook pages. Field logbooks and data sheets will be reviewed periodically during the course of the project by the URS Project QA Officer or designee.

2.3.2 Sample Preservation, Container Specification, and Holding Time Requirements

The sample container, preservation, storage, and holding time requirements are presented in Table 2-.

2.3.3 Field Sample Handling and Custody

Field CoC procedures are as follows.

Sample Labelling and Identification

Sample labelling and identification procedures are presented in Section 4.2.3 of the PDI WP. Per the WP, the first portion of the Sample ID will be a one- to three-letter alphabetic code that identifies the type of sample location followed by a sequential numeric code that specifies the location of the sample horizontally and, if appropriate, vertically. In addition, the sample date (i.e., numerical month, day, and year, as MMDDYY) will be a suffix to the Sample ID with a sequential number added to indicate if more than one sample is collected in a single day. For example, PSC1BR-CGW50-52-0217142 represents the second groundwater sample collected from a depth of 50 to 52 feet at a rock core location from new bedrock well PSC-1BR on February 17, 2014.

Sample Seals

Prior to sample shipment, sample coolers will be sealed as soon as possible following collection utilizing a custody seal. The sample collector will write the date and their signature or initials on the seal. The use of custody seals may be waived if field personnel keep the samples in their custody from the time of collection until the samples are delivered to the laboratory analyzing the samples.

Chain of Custody Record

The field CoC record is used to record the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence and sample sets will be accompanied by a CoC record. This CoC record documents transfer of custody of samples from the sample collector to another authorized person, to the laboratory, or other organizational units. To simplify the CoC record, as few people as possible should have custody of the samples during the investigation. The CoC record also serves as a sample logging mechanism for the laboratory sample custodian. A CoC record will be completed for all samples collected during the PDI.

The following information will be entered on the CoC record:

- Project number
- Project name and location
- Contact information
- All samplers' signatures in the designated signature blocks(s)

The sample identification, date and time of sample collection, grab or composite sampling designation, and sample matrix will be included on each line. One sample will be entered on each line, and a sample will not be split among multiple lines.

The required analysis will be entered in the appropriate location, as indicated on the CoC Record. The preservation and storage conditions for each analysis will also be identified on the COC record. The total number of sample containers will be listed in the "Number of Containers" column for each sample. The number of individual containers for each analysis will also be listed. There will not be more than one sample type per sample.

The sample custodian and subsequent transferee(s) will document the transfer of the samples listed on the CoC record. The person who originally relinquishes custody will be the sample custodian. Both the person relinquishing the samples and the person receiving them will sign the form. The date and time this occurred will be document in the proper space on the CoC record. Usually, the last person receiving the samples will be the laboratory sample custodian or their designee(s).

The CoC record is a serialized document. Once the record is completed, it becomes an accountable document and will be maintained in the project file. An example CoC record is provided in Appendix D.

Transfer of Custody with Shipment

All samples will be accompanied by the CoC record. The original record will be placed in a plastic bag inside the secured shipping container if the samples are shipped.

When shipping samples via common carrier, the "Relinquished By" box will be filled in; however, the "Received By" box will be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the CoC record. The original CoC record will be transmitted to the laboratory. A copy of the completed CoC record will be included in the laboratory deliverable, and this copy will become a part of the project file.

Samples will be packaged for shipment according to the following procedures:

- All samples will be stored in conditions that maintain integrity. Samples will be stored in accordance with requirements provided in Table 2-4 during collection and shipment. Fresh ice will be double-bagged and placed in the cooler prior to shipment.
- Absorbent packing material and insulation is to be placed at the bottom of each cooler. Sample containers that are enclosed in Zip-loc® bags are to be placed right

side up in the cooler. Each cooler used for sample shipment should also contain a temperature blank. The temperature blank should be packed in with samples as close to the center of the cooler as possible to obtain a fair representation of sample temperature upon laboratory receipt.

- Ice, sealed in double bags and preferably wet, will be placed above, below, between, and around sample containers in the cooler. Individual sample containers will be wrapped with and void cooler space will be filled with additional protective packing material (e.g., bubble wrap) to prevent breakage. The amount of ice placed in coolers will be increased in warmer months.
- When shipping samples with dry ice, special instructions are to be followed. Employees engaged in shipping samples on dry ice will follow guidance outlined in URS Safety Management Standard 48 Hazardous Materials/ Dangerous Goods Shipping (SMS 48) found in Appendix A. Containers shipped with dry ice will have the appropriate Department of Transportation Labels, which includes one specifically for dry ice giving the weight of dry ice in the container and a second label for "Cargo Aircraft only" as applicable. The URS employee in charge of handling shipments of such materials will complete the hazardous materials/dangerous goods shipping course conducted by URS or complete an outside equivalent course.
- Sample transfer requires the individuals relinquishing and receiving the samples to sign, date, and note the time on the CoC record. An example CoC record is included in Appendix D.
- After conducting a final inventory check of the cooler's contents, the completed COC record is to be sealed in a plastic Zip-loc® bag and included in the cooler prior to sample shipment.
- The cooler is secured by completely wrapping it with strapping tape around both ends. All seams on the cooler will also be taped to retain coldness within and to prevent leakage. If there is a drain on the cooler, it will also be taped shut on the inside and outside.
- Custody seals, which will be signed and dated, are to be affixed to the cooler to preclude tampering.
- Samples are to be shipped in accordance with International Air and Transport Association and United States Department of Transportation shipping regulations.

2.3.4 Laboratory Sample Handling and Custody

On arrival at the laboratory, all samples will be inspected thoroughly to confirm that the integrity of the samples and containers has not been compromised. The temperature of the cooler contents will be determined and recorded. If the temperature does not meet storage requirements, it will be reported to the Laboratory PM who will immediately notify the URS Project Chemist. The exception to this will be if samples are hand delivered from the site to the laboratory on the day of collection. In this circumstance, the cooler temperature and samples may not have cooled during transport and elevated temperatures will be considered acceptable as long as ice is present in the cooler. The individual sample containers will be inspected to verify that each has a sample label. The condition of the samples (e.g., intact, multiple phases, etc.) will be noted on the CoC record and/or laboratory sample receipt paperwork that will be included in the laboratory deliverable.

Samples will be checked against the information on the CoC record for discrepancies. If discrepancies exist, they will be reported to the LPM, who will immediately notify the URS Project Chemist. The problem will be resolved, in writing, before analytical work begins.

After the Laboratory Sample Custodian has determined that the samples are in satisfactory condition and the documents are in order, all sample information will be entered into a tracking system and unique analytical sample identifiers will be assigned. A copy of this information will be reviewed by the laboratory for accuracy and completeness. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Samples not preserved or analyzed in accordance with these requirements will be resampled and analyzed.

Specific instructions concerning the analysis specified for each sample will be communicated to the analysts. Analytical batches will be created, and laboratory QC samples will be introduced into each batch.

While in the laboratory, samples will be stored in a limited-access, temperature-controlled area. Refrigerators and freezers will be monitored for temperature 7 days/week; coolers will be monitored each working day. Soil requiring geotechnical analyses will be frozen on receipt at the laboratory. The acceptance criterion for the temperatures of the freezers is less than 0°C. All cold storage areas will be monitored by thermometers that have been calibrated with a National Institute of Standards and Technology (NIST)-traceable thermometer. As indicated by the findings of the calibration, correction factors will be applied to each thermometer. Records that include acceptance criteria will be maintained. Samples will be stored after analysis until disposed of in accordance with applicable local, state, and federal regulations. Disposal records will be maintained by the laboratory.

SOPs describing sample control and custody will be maintained by the laboratories.

2.4 Analytical Methods

Samples collected during the PDI activities will be analyzed by field instruments and an onsite, mobile laboratory as well as an offsite, fixed-based laboratory. Samples that will be sent to an offsite laboratory will be submitted via local laboratory courier or FedEx in accordance with standard COC procedures. Additional details regarding analytical and field screening method requirements are provided in the following subsections.

2.4.1 Field Measurement Procedures

Field measurements will be collected according to procedures provided in Section 3.4.2.2 of the WP, Appendices A and B of this plan, and the following subsection. Dedicated equipment will be used during sample collection as discussed in Sections 3.3.1.2, 3.4.2.2, and 3.5.1 of the PDI WP. Equipment requiring decontamination is discussed in Sections 3.2.1.1 and 3.4.2.2 of the PDI WP, with procedures for decontamination provided in Appendix A (Section 4). Investigation derived waste (IDW) is discussed in Section 4.2.4 of the PDI WP and Appendix A (Section 5). URS is not a SC certified laboratory and field measurements will be collected for informational purposes only.

Specific Conductivity, Temperature, pH, Dissolved Oxygen, and Oxidation-Reduction Potential (ORP)

Measurement of specific conductivity, temperature, pH, dissolved oxygen, and ORP in groundwater and surface water samples are discussed in Section 3.4.2.2 of the WP, and Appendices A (Section 2.3.1) and B1. URS personnel will rent a YSI-556 or equivalent water quality meter to be used onsite. Calibration, operation and decontamination of the instruments will be in accordance with current EPA Region IV Science and Ecosystem Support Division (SESD) Operating Procedures.

Turbidity

A nephelometer is used in comparing the turbidity of liquids by viewing light through them and determining how much light is eliminated. Turbidity measurements are reported in nephelometric turbidity units (NTUs). URS personnel will rent a LaMotte 2020 Portable Turbidity Meter or equivalent to be used onsite. The instrument is calibrated daily by using a sample cell containing organic free or deionized water and the provided cells containing pre-made standards. All findings are recorded and anomalies are noted. The turbidity meter will be periodically checked by using the standard provided and a post calibration will be performed at the end of day. Additional information for measuring turbidity is provided in Section 3.4.2.2 of the WP, and Appendices A (Section 2.3.1) and B1.

Organic Vapor Analyzer (e.g., Photoionization Detector [PID])

URS personnel will rent an organic vapor analyzer (i.e., MiniRae 2000 or equivalent) to screen samples for VOCs. The organic vapor analyzer will be operated and calibrated according to the manufacturer's instruction manual. PIDs typically operate with a 10.6 eV lamp; however, 9.5 and 117 eV lamps are available as options. The detector is capable of measuring concentrations down to about 1 ppm sensitivity for certain compounds.

LIF Probe

LIF technology will be used to delineate the depth and horizontal extent of LNAPL. Prior to each working day, the instrument will be tuned and calibrated by the subcontractor according to manufacturer's recommendations (refer to Appendix B2).

2.4.2 Laboratory Procedures

Analytical work is to be conducted in accordance with laboratory SOPs that are referenced in Tables 1-8 through 1-10 and copies of which are included in Appendix C. Volatile organic MPC, which defines the quality elements monitored and acceptance performance for these elements, are presented in Table 1-1 and Sections 1.5.2 and 2.5. MPC for all other parameters will be per the analytical method and laboratory SOPs and control limits.

Tables 1-5 through 1-7 present performance standards, RL objectives for undiluted samples, and laboratory RLs and MDLs for each analyte. Laboratory RLs are less than performance standards for all target analytes, except 1,2-dichloroethane, cis-1,2-dichlorethene, methylene chloride, 1,1,2-trichloroethane, trichloroethene, and vinyl chloride in soil. Since soil samples are being collected in the source area and elevated concentrations of site-specific VOCs are anticipated, and sample results are to be reported to sample-specific MDLs, achievement of those limits listed in Table 1-5 are sufficient to meet project objectives.

All VOC sample results will be reported at or above the MDL values, which is to be adjusted for sample preparation activities, dilution, and percent moisture, as appropriate. However, for those results falling between the MDL and the RL, a "J" flag will be applied to the results, indicating that such values are estimated. Sample results will not be reported below the MDL.

Onsite real time analysis of soil samples for site-specific VOCs will be conducted by the mobile laboratory. Preliminary data reports will be provided daily onsite in an electronic spreadsheet format to the URS Field Operations Manager or designee. The final laboratory deliverable will be provided within five days of sample analysis.

"CLP-like" laboratory deliverables are to be distributed within twenty-one (21) calendar days of sample receipt, while all other offsite laboratory deliverables are to be provided within fourteen (14) calendar days of sample receipt.

2.5 Quality Control Requirements

This section presents QC requirements relevant to field and laboratory data generated by field staff and laboratories. The purpose of this QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of both field and laboratory QC materials.

2.5.1 Field Collection Methods of Quality Control

This section presents QC requirements relevant to field data that will be generated by field staff and laboratories.

Trip Blank

Trip blanks, which consist of organic/analyte-free water, are prepared and provided by the laboratory when samples are to be collected for a VOA. Trip blanks results are used to assess the potential for contamination of samples due to contaminant migration during sample shipment, storage, and analysis. Trip blanks will be submitted to the laboratory at the frequency indicated in Table 1-2. When an analyte is detected in the trip blank, the appropriate validation flag, as described in Section 4.0, may be applied to results for those samples that were shipped to the laboratory with the affected trip blank, based on professional judgment used during the validation process.

Equipment Blank

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures. An equipment blank is a sample of organic/analyte-free water poured into or over the sampling device, collected in a sample container, and transported to the laboratory for analysis.

Per Table 1-2, equipment blanks will be collected from any equipment used in sample collection or processing that is re-used for more than one sample location and is not equipped with a liner. Equipment blanks will be collected immediately after the equipment has been decontaminated. Equipment blanks will be collected in sample containers appropriate for an aqueous matrix, and preserved in accordance to the pertinent analytical method. The blank will be analyzed for all laboratory analyses requested for the environmental samples collected using the equipment.

Acceptance criteria are provided in Table 1-2 for the equipment blank. When an analyte is detected in the equipment blank, the appropriate validation flag, as described in Section 4.0, may be applied to results from samples collected with the affected equipment, based on professional judgment used during the validation process.

Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The frequency of collection for field duplicate samples and acceptance criteria are provided in Table 1-2. Field duplicates with quantifiable results that exceed criteria presented in Table 1-2 will be validation flagged as indicated in Section 4.0.

2.5.2 Laboratory Methods of Quality Control

This section presents QC requirements relevant to analysis of samples that will be followed by laboratories producing analytical data.

Holding Time Compliance

Sample preparation and analysis will be completed within the method-required holding times. The holding time for a sample begins at the time of sample collection. The preparation holding time is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the applicable method, prior to any necessary extract cleanup and/or volume reduction procedures. If no preparation (e.g., extraction) is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation prior to analysis, the analysis holding time is calculated from the time of preparation completion to the time of completion of all analytical runs, including dilutions, and any required reanalyses.

If holding times specified in Table 2-1 are exceeded, sample results will be flagged during validation according to the procedures described in Section 4.0.

Laboratory Control Sample

For USEPA Method 8260B, the performance of the LCS will be evaluated against the QC acceptance limits presented in Table 1-3. The LCS will be spiked with VOCs indicated in Table 1-4. The performance of the LCS will be evaluated against the QC acceptance limits presented in Table 1-3. Whenever the analyte in an LCS is outside these acceptance criteria, corrective action is required. All samples in the associated extraction batch will be reanalyzed if

the LCS fails to meet the acceptance criteria, unless the LSC fails high and associated sample results are non-detects. When the LCS recovery is high and associated sample results are non-detect, data may be reported with narration. When an analyte in an LCS exceeds the upper control limit (UCL) or lower control limit (LCL) and no corrective action is performed or the corrective action was ineffective, the data will be qualified as described in Section 4.0 during data validation.

Matrix Spike/Matrix Spike Duplicate

A MS and MSD is an aliquot of sample spiked with known concentrations of VOCs indicated in Table 1-4. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS and MSD will be spiked at a level less than or equal to the midpoint of the calibration curve. The MS and MSD results are used to document potential matrix effects associated with a site. The performance of the MS and MSD is evaluated against the QC acceptance limits given in Table 1-3. If MS and MSD results do not meet QC acceptance criteria, the result in the parent sample will be qualified according to the data flagging criteria in Section 4.0 during data validation.

Surrogate Compounds

The performance of USEPA Method 8260B is monitored using surrogate compounds indicated in Table 1-4. Surrogates are added to each sample prior to analysis to monitor efficiency throughout the analytical process. Acceptance criteria are provided in Table 1-3. If more than one surrogate does not meet these criteria or if any surrogate has less than 10% recovery, the lab will take corrective action, except in those cases where the surrogate was diluted out of the sample or when surrogate recoveries are high and the analyte is not detected in the sample. The sample will be re-analyzed. If the surrogate still fails, the lab will document matrix interference for the sample in the case narrative. Sample results with surrogate recoveries that do not meet acceptance criteria will be qualified per data validation procedures discussed in Section 4.0.

Method Blank

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. A method blank will be included in every analysis batch.

As indicated in Table 1-3, the presence of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action will be performed to eliminate the source of contamination prior to proceeding with analysis. After the

source of contamination has been eliminated, all samples containing the analyte found in the method blank above the RL will be reanalyzed. No analytical data will be corrected for the presence of analytes in blanks. When an analyte is detected above the MDL in the method blank and in the associated samples, and corrective actions (for samples with detections above the RL) are not performed or are ineffective, the laboratory will qualify results. During the data validation process, the data will be qualified with the appropriate validation flag, as described in Section 4.0.

2.6 Instrument/Equipment Testing, Inspection, Maintenance Requirements

Field equipment for this project includes water level meters, water quality meters, and organic vapor analyzers. The Field Operations Manager is responsible for oversight of testing, inspection, and maintenance of field instrumentation. The field analyst/staff is responsible for conducting the field instrumentation testing, inspection, and maintenance, and the name of the field analyst/staff and results with will be recorded in the field notebook. Equipment testing, inspection, and preventative maintenance procedures to be followed for field equipment will be in accordance with EPA Region 4 SESD Operating Procedures (refer to Appendix B1). Where appropriate, new batteries and other spare parts will be purchased and kept with the field equipment to facilitate immediate replacement (refer to Appendix B1). Any deficiencies in testing, inspection, or calibration of field measurement equipment will be reported by field staff/analyst to the Field Operations Manager, who will notify the PM and QA Officer. Corrective actions and its effectiveness will be documented by the Field Operations Manager per Section 3.1.4 of the QAPP.

As part of the laboratory's Quality Manual, a routine preventative maintenance program will be conducted by the laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees will regularly perform routine scheduled maintenance and repair of (or coordinate with the vendor for the repair of) all instruments. All laboratory instruments will be maintained in accordance with manufacturer's specifications. The preventive maintenance program should include:

- An inventory of replacement and spare parts for instruments that require maintenance.
- Maintenance logbooks for each instrument with information on routine and non-routine procedures. The logbook records will include the instrument number, description of malfunction or problem, date of maintenance activity, the type of activity performed, and final resolution.

- Training of laboratory staff in the maintenance requirements of the instruments. Preventive maintenance schedules and activities will be outlined in the laboratory SOPs.

2.7 Instrument Calibration and Frequency

Analytical instruments will be calibrated in accordance with laboratory SOPs, which are provided in Appendix C. All analytes reported will be present in the initial and continuing calibrations, and these calibrations will meet the acceptance criteria specified in this Section as well as Section 4.0.

Results outside the calibration range are unsuitable for quantitative work and will only give an estimate of the true concentration. Records of standard preparation and instrument calibration will be maintained. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials. All calibration criteria will satisfy SW-846 requirements at a minimum. Multipoint calibrations will contain the minimum number of calibration points specified in the method with all points used for the calibration being contiguous. The only exception to this rule is that a standard that has been statistically determined as being an outlier can be dropped from the calibration, providing any method requirement for a minimum number of standards is met.

Other laboratory equipment such as refrigerators, balances, and ovens required for the storage and preparation of samples will be calibrated and/or monitored according to the following guidelines:

- Equipment will be checked daily and these records kept in a logbook or calibration specific log
- The laboratory will document clearly the acceptance criteria for all such equipment (e.g., refrigerator temperature) and corrective actions will be taken for any out-of-control situation as described in the laboratory's Quality Manual
- The equipment will not be used after corrective action until it has been recalibrated or verified through the successful analysis of a check standard
- Calibrations of other miscellaneous analytical equipment (e.g., automatic pipettes) will be performed according to manufacturer's recommendations

Implementation of the laboratory calibrations will be the responsibility of the Laboratory Manager and the analysts performing the procedures.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables

The laboratory analysts will inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis will be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents will be monitored by analysis of LCSs. An inventory and storage system for these materials will ensure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.

Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples will be those normally used by the analytical laboratory. The laboratory, however, is responsible for maintaining standard accuracy and traceability in accordance with good laboratory practice. The standard materials will be current, and the lab will insure that all expiration dates are current for all standard materials. Expired standard materials will be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory will label standard and QC materials with expiration dates.

A second source standard will be used to independently confirm initial calibration. A second source standard is a standard purchased from a different vendor than the vendor supplying the material used in the initial calibration standards. The second source material will be used for the calibration verification standards or for the LCS.

2.9 Data Acquisition Requirements for Non-direct Measurements

In addition to the data generated as part of the current investigation, data that were collected during previous investigations will be used in the PDI. Results from the previous investigations will be used in combination with the newly collected data to refine the vertical and horizontal dimensions of the actual area requiring thermal treatment.

2.10 Data Management

Data management requirements are an essential part of every investigation. Data management ensures that procedures are in place to document, track, and manage all field and laboratory data generated during the course of field activities. Records management is discussed in Section 1.6 of this plan. The URS PM has overall responsibility for data management. The PM ensures that all field and laboratory information are collected and accurately recorded, and that inspections relating to the generation, collection, and storage of data are conducted.

3.0 ASSESSMENT OVERSIGHT

3.1 Assessment and Response Actions

The purpose of this section is to describe the methods which will ensure that the data collected for the PDI activities comply with the DQOs as described in Section 1.5. To meet these DQOs, a combination of statistical procedures and qualitative evaluations will be used to check the quality of the data. These procedures will be used by the laboratory while generating the data.

Results for QC samples, including field and laboratory blanks, spikes, and duplicates, will be evaluated using the equations in the validation guidelines to determine the validity and usability of the data. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, contamination during sampling, contamination in the laboratory, and sample preservation and storage anomalies (i.e., sample holding time or analytical instrument problems).

3.1.1 Field Audits

An internal audit of field activities, including sampling and field observations, may be conducted by the URS Project QA Officer (or designee) early in the sampling event to verify that all established procedures are being followed.

Technical staff and field project personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any field collection or observation activity by recording in field notes and reporting the situation to the URS PM or designee. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated.

The URS Project QA Officer will be responsible for ensuring that the corrective action for nonconformance is initiated by:

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a log of nonconformances
- Reviewing nonconformance reports and corrective actions taken
- Ensuring nonconformance reports are included in the final site documentation in project files

Corrective actions will be implemented and documented in the field record book. Documentation of corrective actions will include:

- A description of the circumstances that initiated the corrective action
- The action taken in response
- The final resolution
- Any necessary approvals

No staff member will initiate a corrective action without prior communication of findings through the proper channels.

Any corrective actions resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The URS Project QA Officer or designee will identify deficiencies and recommend corrective action to the URS PM. Implementation of field audit corrective actions will be performed by the Field Operations Manager and field team.

If appropriate, the URS PM will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

If a corrective action warrants a change in the program protocols, the change will be documented and signed by the Field Operations Manager and URS PM (or designee).

3.1.2 Internal Laboratory Audits

In each laboratory analytical section, the analyst performing the tests will review 100% of the data. After the analyst's review is complete, 100% of the data will be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria.

The laboratories participating in this program are required to have a written policy specifying corrective actions to be taken when an analytical error is discovered or the analytical system is determined to be out of control. These policies require documentation of the corrective action and notification by the analyst about the errors and corrective procedures. Corrective action for each laboratory is described in each laboratory's Quality Manual.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis and the event. Laboratory corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy
- Blanks containing analytes of interest, as listed in the QAPP, are above acceptable levels
- Undesirable trends are detected in MS recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by the Laboratory QA Department during internal or external audits or from the results of performance evaluation samples
- Inquiries concerning data quality are received

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors; checks the instrument calibration, spike, and calibration mixes, instrument sensitivity, etc. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager, and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the laboratory QA department.

Corrective action may include:

- Re-analyzing the samples, if holding time criteria permits
- Re-sampling and analyzing
- Evaluating and amending analytical procedures
- Accepting data and acknowledging the level of uncertainty, as documented in the laboratory data package case narrative and flagging of affected sample results with laboratory data qualifiers

If re-sampling is deemed necessary due to laboratory problems, the URS PM will identify the necessary approach including cost recovery for the additional sampling effort.

3.1.3 Data Package Technical Systems Audit

Assessment of the analytical information will be accomplished by the joint efforts of the URS QA Officer, URS Project Chemist, and URS PM. The data assessment will be based on the criteria that the samples were properly collected and handled according to the QAPP.

The URS Project Chemist will conduct a systematic review of the data for compliance with the established QC criteria based on the laboratory QC results (e.g., spikes, duplicates, and blanks, etc.). The data validation, based on criteria set forth in this QAPP, will be performed and

included in the sampling event report. An example data validation checklist is provided as Appendix E.

The URS Project Chemist will identify any out of control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the URS PM based on the extent of the deficiencies and their importance in the overall context of the project.

3.1.4 Assessment Findings and Corrective Action Responses

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of QC performance, which can affect data quality and usability. Corrective actions may be required for two classes of problems: analytical and equipment problems and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems (e.g., non-compliance with USEPA methods or QC defined in the QAPP) a formal corrective action will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the URS Project QA Officer. A description of the problem and the corrective action implemented will be confirmed in writing via email, facsimile, or technical memorandum.

Any nonconformances with the established QC procedures in the QAPP will be identified and corrected on an ongoing basis throughout the course of the project.

The need for corrective action may be identified at any time during the analytical process. Potential types of corrective action may include resampling by the field team or reinjection/re analysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected are necessary to meet the required QA objectives. If URS QA Officer or Project Chemist identifies a corrective action situation during the data assessment or validation, the URS PM will be responsible for informing the appropriate personnel. All corrective actions of this type will be documented by the URS PM or designee.

3.2 Reports to Management

Data quality information collected during the project will be maintained in the project file. This includes, but is not limited to, field instrument calibration records, field sampling data records, and data validation, audit, and corrective action reports. The QA information generated during the project and included in these reports may include the achievement of project-specific DQOs, uncertainties in the data used and their effect on the data usage, and a summary of corrective

actions implemented, as necessary, as it may have affected results. The URS PM will verify completeness of the reports, and that problems and deficiencies have been appropriately addressed.

4.0 DATA VALIDATION AND USABILITY

This section describes specific QA activities that occur after data collection. This includes the methods and equations used to assess the quality of the data with regard to precision, accuracy, representativeness, completeness, and comparability.

The procedures used to assess the DQOs as outlined in this QAPP were developed to generate data which meets the project's needs. Through the systematic method of data assessment, data of known quality will be produced and then applied to specific needs based on the actual quality of the data. By subjecting the data to standard calculations and validation guidelines, the usability of the data is enhanced when comparison against past, present, or future data is necessary. Actual use of any data for specific purposes will be determined by the URS PM in coordination with the URS QA Officer, based on the required data quality needs for a particular data set (i.e., matrix, concentration level, intended data use, quantification accuracy, and precision needs).

4.1 Data Review, Validation, and Verification Requirements

The parameters that will be assessed and the criteria used to review and validate data objectively and consistently are provided in Section 1.5. The MPC define the quality elements monitored and the acceptable performance for these elements. Tables 1-1 through 1-4 describe the MPC for precision, accuracy, and sensitivity for the VOAs. MPC for all other parameters will be per the analytical methods and laboratory SOPs. For sensitivity, achievement of limits listed in Tables 1-5 through 1-8 is sufficient to achieve the project objectives.

4.2 Validation and Verification Methods

The data verification and validation procedures described in this section will ensure (1) complete documentation is maintained, (2) transcription and data reduction errors are minimized, (3) the data are reviewed and documented, and (4) the reported results are qualified, if necessary.

4.2.1 Procedure to Verify Field Data

The procedures to verify accuracy and completeness of field information include checking for transcription errors, ensuring that field measurement equipment was properly calibrated, and review of field logbooks. Historical data from previous assessments will be compared to newly generated data. These assessments will be conducted by the Field Operations Manager or designee.

4.2.2 Procedure to Verify Laboratory Data

All laboratory deliverables will be reviewed by the Project Chemist for completeness to ensure that all pertinent data has been included, reporting limit objectives met, and discrepancies/transcription errors do not exist. The Project Chemist will contact the laboratory to resolve all laboratory deliverable issues.

4.2.3 Data Validation

Laboratory data will be assessed for usability, completeness, and adherence to key QA/QC objectives for this project. The data will be validated using a procedure that is modeled after the *USEPA CLP National Functional Guidelines (NFG) for Superfund Organic Methods Data Review* (USEPA, June 2008) and *USEPA NFG for Inorganic Superfund Data Review* (USEPA, January 2010) with changes and allowances made to conform to analytical methodology. The MPC for PARCCS are presented in Section 1.. Sample results will be qualified using flags presented in Table 4-1 and based on the flagging criteria presented in Table 4-2 and professional judgment to alert the user to limitations of data use. A data validation report will be completed that provides a summary of all data quality issues and rationale for data qualification. An example data validation checklist is provided as Appendix E.

4.3 Overall Assessment of Environmental Data

Data assessment will involve data evaluation and usability to determine if the data collected are of the appropriate quality, quantity, and representativeness to support the PDI. The effect of the loss of data deemed unacceptable for use, for whatever reason, will be discussed and decisions made on corrective action for potential data gaps. The QC results associated with each analytical parameter for each matrix type will be compared to the objectives presented in the QAPP. Only data generated in association with QC results meeting these objectives and the data validation criteria will be considered usable.

Questions to be answered in the overall data assessment based on the DQOs in this QAPP and the data evaluation by the URS Project Chemist or designee will include, but not necessarily be limited to, the following:

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP and/or WP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP and/or WP?
- Were samples obtained from all proposed sampling locations?

- To any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were all laboratory data evaluated according to the validation protocols, including project specific QC objectives as defined in this QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data evaluation results?
- Which data sets were found to be useable, but estimated (qualified as "J" or "UJ"), based on the data evaluation results?
- Have sufficient data of appropriate quality been generated to support the project?
- Were all issues requiring corrective action, if any, fully resolved?
- Have any remaining data gaps been identified and summarized in the final report?

4.4 Data Usability/Reconciliation with Project Quality Objectives

The goal of this project is to produce data that can be used to effectively design a treatment area for the proposed thermal remedy. As such, the data generated must meet the data user's needs as defined in the project DQOs in the QAPP and WP. In summary, the primary objectives for assessing the usability of the data are: (1) to collect data that are representative of site conditions that can be combined and compared with prior data and (2) to identify "hotspot" locations within the delineated source areas that will potentially require treatment.

For data subject to validation, the URS Project Chemist or designee will apply USEPA data qualifiers from the NFGs to indicate the level of uncertainty in the associated sample result. In general, data that are left unqualified, or qualified with a "U," "J," and "UJ" are considered valid and usable for project objectives. Data that are qualified "R" due to severe deviations from QC requirements will be considered invalid and unusable.

The goal of this program is to generate valid, usable data. However, in environmental sampling and analysis, some data may be lost due to sampling location access constraints, field or laboratory errors, or matrix effects that may cause the rejection of results for some compounds. The overall goal for completeness of collection of valid data is 90%. The URS Project Chemist or designee will assess the completeness of the overall data generation against the project goal of producing 90% of the planned data as valid and usable results. If this goal is not met, data gaps may exist that compromise the intended use of the data.

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TABLES

Table 1-1
Volatile Organic Measurement Performance Criteria
Former PSC Site, Rock Hill, South Carolina

Parameter	Matrix	Methods	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
VOCs	Soil	USEPA Methods 5035/8260B	Overall Precision	Within limits on Table 1-2	Field Duplicates	S&A
			Precision - Laboratory	Within limits on Tables 1-2 & 1-3	MS and MSD, and LCS and LCSD, if MS and MSD not conducted	S&A
					Calibration	A
			Accuracy - Laboratory	Within limits on Tables 1-2 & 1-3	MS and MSD, and Surrogates	S&A
			Accuracy - Laboratory	Within limits on Table 1-3	LCS and LCSD, if MS and MSD not conducted	A
					Calibration	
			Accuracy - Contamination	Within limits on Tables 1-2 and 1-3	Method, Trip, and Equipment Blanks	S&A
			Sensitivity	Calibration to RL. Sample results reported to MDL.	Verify low calibration standard is at RL and that MDL is less than RL.	A
			Representativeness	Holding time	Verify that samples were analyzed within USEPA-established holding time	A

Table 1-1 (Continued)
Volatile Organic Measurement Performance Criteria
Former PSC Site, Rock Hill, South Carolina

Parameter	Matrix	Methods	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
VOCs	Water	USEPA Methods 5030B/8260B	Overall Precision	Within limits on Table 1-2	Field Duplicates	S&A
			Precision - Laboratory	Within limits on Tables 1-2 & 1-3	MS and MSD, and LCS and LCSD, if MS and MSD not conducted	S&A
					Calibration	A
			Accuracy - Laboratory	Within limits on Tables 1-2 & 1-3	MS and MSD, and Surrogates	S&A
			Accuracy - Laboratory	Within limits on Table 1-3	LCS and LCSD, if MS and MSD not conducted	A
					Calibration	
			Accuracy - Contamination	Within limits on Tables 1-2 & 1-3	Method, Trip, and Equipment Blanks	S&A
			Sensitivity	Calibration to RL. Sample results reported to the MDL.	Verify low calibration standard is at RL and that MDL is less than RL.	A
			Representativeness	Holding time	Verify that samples were analyzed within USEPA-established holding times	A

A Analysis
DQI Data quality indicator
USEPA United States Environmental Protection Agency
LCS Laboratory control limit
LCSD Laboratory control sample duplicate
MDL Method detection limit
MS Matrix spike
MSD Matrix spike duplicate
QC Quality control
RL Reporting limit
S Sampling
VOC Volatile organic compound

Table 1-2
Measurement Performance Criteria for Field QC Samples
Former PSC Site, Rock Hill, South Carolina

QC Sample	Frequency	Measurement Performance Criteria[1]	Corrective Action (CA)	Persons Responsible for CA	Data Quality Indicator
Field Duplicate[2]	1 per medium per 10 field samples collected	Meets requirement in Table 1-4	Compare to matrix duplicates, check for possible matrix interferences or improper sample collection procedure, qualify data	Data Validator / QA Officer	Evaluate precision and representativeness taking into account variability of sample matrix
Trip Blank	1 per 20 field samples collected or 1 per sample shipment, whichever is more frequent	Meets requirement in Table 1-4	Qualify data.	Data Validator / QA Officer	Evaluate contamination potentially introduced during sampling, shipping, and analysis.
Equipment Blank[3]	As required[3]	Meets requirement in Table 1-4	Qualify data.	Data Validator / QA Officer	Evaluate cleanliness of sample containers and sample handling and collection procedures
Cooler Temperature Blanks	One per cooler	Above freezing (soil and water only), $\leq$6°C	Qualify data. Reject data or resample for excessively high temperatures.	Data Validator / QA Officer / Project Manager	Evaluate representativeness and bias
MS/MSD	1 per 20 field samples will be designated for MS/MSD analysis and additional samples volume will be provided to the laboratory for this QC analysis.	Meets %R and %RPD requirements in Table 1-4 when native sample concentration is $\leq$4x spiking level	Check for possible matrix interferences, review laboratory procedures for variations or improper sample collection procedure, qualify data	Laboratory Analyst / Data Validator	Evaluate precision and representativeness taking into account variability of sample matrix and laboratory practices.

[1] Provisions for wider acceptance limits may be based on professional judgment during data review/validation.
[2] A field duplicate is a split sample with both portions sent to the same lab.
[3] Equipment blanks will be collected from any equipment used in sample collection or processing that is re-used for more than one sample location and is not equipped with a liner. Equipment blanks are prepared by pouring laboratory-supplied, analyte-free water over decontaminated equipment. The equipment blanks will be used to check decontamination methods.

Table 1-2 (Continued)

Measurement Performance Criteria for Field QC Samples
Former PSC Site, Rock Hill, South Carolina

%R	Percent recovery
%RPD	Percent relative percent difference
°C	Degrees Celsius
CA	Corrective Action
MS	Matrix spike
MSD	Matrix spike duplicate
QA	Quality Assurance
QC	Quality Control
VOA	Volatile Organic Analysis

Table 1-3
Measurement Performance Criteria for Laboratory QC Samples
Former PSC Site, Rock Hill, South Carolina

Type	Frequency	Measurement Performance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	Data Quality Indicator
Method Blank	Minimum of 1 per preparation batch	≤RL	Re-analyze; if blank still exceeds criteria, clean and recalibrate system; document corrective action, evaluate/ prepare/reanalyze samples[2]	Laboratory Analyst / Area Manager	Evaluate cleanliness of sample preparation and analysis procedures
MS	At least 1 per preparation/ analytical batch[3] or as requested on CoC record	Meet requirements in Table 1-4 for project-specific samples	Re-analyze samples if necessary. Qualify data if criteria are still not met.	Laboratory Analyst / Area Manager	Evaluate accuracy and representativeness taking into account variability of sample matrix
MSD	At least 1 per preparation/ analytical batch[3] or as requested on CoC record	Meet requirements in Table 1-4 for project-specific samples	Re-analyze samples if necessary. Qualify data if criteria are still not met.	Laboratory Analyst / Area Manager	Evaluate precision, accuracy, and representativeness taking into account variability of sample matrix

[1] Provisions for wider acceptance limits may be based on professional judgment during data review/validation.
[2] Unless there are no positive sample results or if sample results are >10x the amount detected in the blank
[3] Batch QC samples may be used to meet requirement when a project-specific sample is not identified on the COC record

Table 1-3 (Continued)
Measurement Performance Criteria for Laboratory QC Samples
Former PSC Site, Rock Hill, South Carolina

Type	Frequency	Measurement Performance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	Data Quality Indicator
LCS	At least 1 per preparation batch, containing 20 samples or less. LCSD required in cases where insufficient sample available for the MS and MSD analysis	Meet requirements specified in Table 1-4	Check if MS/MSD acceptable to compare for matrix effects. Evaluate the bias in relation to sample result. Re-analysis may be required. Data may require qualifiers.	Laboratory Analyst / Area Manager	Evaluate accuracy
Calibration	Per Laboratory SOPs	Per Lab SOPs and Section 2.7 of this QAPP	Check system; recalibrate	Laboratory Analyst	Establish instrument response and linearity.
Surrogates	All samples requiring a GC analysis	Recoveries as specified in Table 1-4	Evaluate data; samples may require reanalysis and/or qualification	Laboratory Analyst / Area Manager	Evaluate accuracy of sample preparation and effect of matrix on preparation

CA Corrective Action
CoC Chain-of-Custody
GC Gas Chromatograph
LCS Laboratory Control Sample
LCSD Laboratory Control Sample Duplicate
MS Matrix Spike
MSD Matrix Spike Duplicate
QAPP Quality Assurance Project Plan
RL Reporting Limit
SOP Standard Operating Procedure

Table 1-4

Analytical Laboratory DQOs for Precision and Accuracy
Former PSC Site, Rock Hill, South Carolina

Parameter	Matrix	Compounds	Field Duplicate Precision[1] (% RPD)	Laboratory Precision[2,3] (% RPD)	Blanks	LCS/D' Accuracy[2] (%R)	MS/D Accuracy[2] (%R)	Surrogate Accuracy[2] (%R)
VOCs	Soil	Benzene	≤50%RPD when sample concentrations >5x RL; absolute difference ≤ 2x RL when sample concentrations ≤5x RL	≤30	≤RL	71-130	71-123	NA
		Chlorobenzene		≤30		70-139	65-135	NA
		1,4-Dichlorobenzene		≤30		71-126	65-124	NA
		1,2-Dichloroethane		≤30		58-153	65-138	NA
		cis-1,2-Dichloroethene		≤30		59-142	51-147	NA
		trans-1,2-Dichloroethene		≤30		51-148	48-145	NA
		Ethylbenzene		≤30		74-134	69-125	NA
		Methylene Chloride		≤30		34-161	44-135	NA
		Tetrachloroethene		≤30		58-145	54-140	NA
		Toluene		≤30		70-134	67-130	NA
		1,2,4-Trichlorobenzene		≤30		71-136	66-134	NA
		1,1,1-Trichloroethane		≤30		57-150	65-135	NA
		1,1,2-Trichloroethane		≤30		53-159	67-137	NA
		Trichloroethene		≤30		69-132	64-134	NA
		Vinyl Chloride		≤30		37-158	38-145	NA
		m,p-Xylene		≤30		70-146	63-144	NA
		o-Xylene		≤30		71-139	74-125	NA
					-			
		Surrogates:			-			
		Dibromofluoromethane	NA	NA	NA	NA	NA	73-141
		1,2-Dichloroethane-D4	NA	NA	NA	NA	NA	63-156
		Toluene-D8	NA	NA	NA	NA	NA	76-119
		4-Bromofluorobenzene	NA	NA	NA	NA	NA	69-129

[1] Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.
[2] Limits are based on annual limits supplied by KB Labs and TestAmerica Nashville for soil and water samples, respectively. Actual limits will vary with the historical limits established by each individual laboratory.
[3] Lab duplicate or LCSD analyses should be conducted, if MS and MSD analyses unavailable.

Table 1-4 (Continued)
Analytical Laboratory DQOs for Precision and Accuracy
Former PSC Site, Rock Hill, South Carolina

Parameter	Matrix	Compounds	Field Duplicate Precision[1] (% RPD)	Laboratory Precision[2,3] (% RPD)	Blanks	LCS/D' Accuracy[2] (%R)	MS/D Accuracy[2] (%R)	Surrogate Accuracy[2] (%R)
VOCs	Water	Benzene	≤30%RPD when sample concentrations >5x RL; absolute difference ≤ RL when sample concentrations ≤5x RL	≤17	≤RL	80-121	75-133	NA
		Carbon Tetrachloride		≤19		64-147	62-164	NA
		Chlorobenzene		≤14		80-120	80-129	NA
		1,2-Dichlorobenzene		≤15		80-121	79-128	NA
		1,4-Dichlorobenzene		≤15		80-120	78-126	NA
		1,2-Dichloroethane		≤17		77-121	64-136	NA
		1,1-Dichloroethene		≤17		79-124	70-142	NA
		cis-1,2-Dichloroethene		≤17		76-125	68-138	NA
		Ethylbenzene		≤15		80-130	79-139	NA
		Isopropylbenzene (cumene)		≤16		80-141	80-153	NA
		Methylene chloride		≤17		79-123	64-139	NA
		Tetrachloroethene		≤16		80-126	72-145	NA
		Toluene		≤15		80-126	75-136	NA
		1,2,4-Trichlorobenzene		≤19		63-133	60-136	NA
		1,1,1-Trichloroethane		≤17		78-135	76-149	NA
		1,1,2-Trichloroethane		≤15		80-124	74-134	NA
		Trichloroethene		≤17		80-123	73-144	NA
		Vinyl chloride		≤17		68-120	56-129	NA
		Xylenes (Total)		≤15		80-132	74-141	NA
					-			
		Surrogates:			-			
		Dibromofluoromethane	NA	NA	NA	NA	NA	70-130
		1,2-Dichloroethane-d4	NA	NA	NA	NA	NA	70-130
		Toluene-d8	NA	NA	NA	NA	NA	70-130
		4-Bromofluorobenzene	NA	NA	NA	NA	NA	70-130

%R Percent Recovery
D Duplicate
DQO Data Quality Objectives
LCS Laboratory Control Sample
LCSD Laboratory Control Sample Duplicate
MS Matrix Spike
MSD Matrix Spike Duplicate
NA Not applicable
RL Reporting Limit
RPD Relative Percent Difference
VOC Volatile Organic Compound

Table 1-5
Parameters to be Measured for Soil Samples
Former PSC Site, Rock Hill, South Carolina

Analyte[1]	CAS #	Performance Standards - Industrial SSL[2] (mg/Kg)	RL Objectives (mg/Kg)	Laboratory RL[3] (mg/Kg)	Laboratory MDL[4] (mg/Kg)	DQO Level[5]
VOC (USEPA Methods 5035/8260B)						
Acetone	67-64-1	63,000	63,000	0.100	NA	II
Benzene	71-43-2	5.4	5.4	0.002	0.00058	II
Chlorobenzene	108-90-7	140	140	0.002	0.00058	II
1,4-Dichlorobenzene	106-46-7	12	12	0.002	0.00045	II
1,2-Dichloroethane	107-06-2	2.2	2.2	0.002	0.00052	II
1,2-Dichloroethene (Total)	540-59-0	920	920	0.004	0.002	II
cis-1,2-Dichloroethene	156-59-2	200	200	0.002	0.00057	II
Ethylbenzene	100-41-4	27	27	0.002	0.00040	II
Methylene chloride	75-09-2	310	310	0.002	0.00099	II
Tetrachloroethene	127-18-4	41	41	0.002	0.00059	II
Toluene	108-88-3	4500	4500	0.002	0.00056	II
1,2,4-Trichlorobenzene	120-82-1	27	27	0.010	0.00039	II
1,1,1-Trichloroethane	71-55-6	3800	3800	0.002	0.00060	II
1,1,2-Trichloroethane	79-00-5	0.68	0.68	0.002	0.00053	II
Trichloroethene	79-01-6	2	2	0.002	0.00060	II
Vinyl chloride	75-01-4	1.7	1.7	0.002	0.00087	II
Xylenes (Total)	1330-20-7	270	270	0.006	0.001	II
Geotechnical Parameters						
Fractional Organic Carbon (ASTM D2974)	NA	NA	NA	0%	NA	IV
Grain-size Distribution (ASTM D422)	NA	NA	NA	NA	NA	IV
Bulk Density (ASTM D2937)	NA	NA	NA	NA	NA	IV
Wet Unit Weight (ASTM D2216-05)	NA	NA	NA	NA	NA	IV
Specific Gravity (ASTM D854)	NA	NA	NA	NA	NA	IV
Air-filled Porosity (NA[6])	NA	NA	NA	0.1 porosity units	NA	IV
Percent Saturation (NA[7])	NA	NA	NA	NA	NA	IV

%	Percent
ASTM	American Society for Testing and Materials
CAS	Chemical Abstracts Service
DQO	Data Quality Objectives
MDL	Method detection limit
mg/Kg	Milligrams per Kilograms
NA	Not available/applicable
NE	Not established
QA	Quality Assurance
QC	Quality Control
RL	Reporting limit
SESD	Science and Ecosystem Support Division
SSL	Soil Screening Level
USEPA	United State Environmental Protection Agency
VOC	Volatile Organic Compounds

[1] Preparation and analytical methods are in parentheses after analyte. The methods used should be the most recent, USEPA-approved update of the above-mentioned methods

[2] *Regional Screening Levels for Chemical Contaminants at Superfund Sites*, United States Environmental Protection, May 2013

[3] All units of measure in milligrams per kilogram, unless indicated otherwise

[4] Laboratory MDLs subject to change

[5] DQOs and QA/QC frequencies per Region 4 SESD Field Branches Quality System and Technical Procedures, which are available at http://www.epa.gov/region4/sesd/fbqstp/. Level I = Field Screening; Level II = Field Analyses; Level III = Screening Data with Definitive Confirmation; Level IV = Definitive Data.

[6] Based on a calculation using specific gravity (ASTM D854) and density (ASTM D698) results

[7] Based on calculations using specific gravity (ASTM D854), density (ASTM D698), grain size (ASTM D422), and water content (ASTM D2216) results

Table 1-6
Parameters to be Measured for Groundwater Samples
Former PSC Site, Rock Hill, South Carolina

Analyte[1]	CAS #	Performance Standards MCL[4] (µg/L)	RL Objectives (µg/L)	Laboratory RL (µg/L)	Laboratory MDL[2] (µg/L)	DQO Level[3]
VOC (USEPA Methods 5030B/8260B)						
Benzene	71-43-2	5.0	5.0	1.00	0.200	IV
Carbon Tetrachloride	56-23-5	5.0	5.0	1.00	0.180	IV
Chlorobenzene	108-90-7	100	100	1.00	0.180	IV
1,2-Dichlorobenzene	95-50-1	600	600	1.00	0.190	IV
1,4-Dichlorobenzene	106-46-7	75	75	1.00	0.200	IV
1,2-Dichloroethane	107-06-2	5.0	5.0	1.00	0.200	IV
1,1-Dichloroethene	75-35-4	7.0	7.0	1.00	0.250	IV
cis-1,2-Dichloroethene	156-59-2	70	70	1.00	0.210	IV
Ethylbenzene	100-41-4	700	700	1.00	0.190	IV
Isopropylbenzene (cumene)	98-82-8	NE	1.00	1.00	0.330	IV
Methylene chloride	75-09-2	5.0	2.0	2.00	0.220	IV
Tetrachloroethene	127-18-4	5.0	5.0	1.00	0.140	IV
Toluene	108-88-3	1000	1000	1.00	0.170	IV
1,2,4-Trichlorobenzene	120-82-1	70	70	1.00	0.200	IV
1,1,1-Trichloroethane	71-55-6	200	200	1.00	0.190	IV
1,1,2-Trichloroethane	79-00-5	5.0	5.0	1.00	0.190	IV
Trichloroethene	79-01-6	5.0	5.0	1.00	0.200	IV
Vinyl chloride	75-01-4	2.0	2.0	1.00	0.180	IV
Xylenes (Total)	1330-20-7	10000	10000	3.00	0.580	IV
Monitoring Natural Parameters						
Nitrate (USEPA Method 9056A)	14797-55-8	NA	100	100	60	IV
Sulfate (USEPA Method 9056A)	18787-72-3	NA	1000	1000	600	IV
Iron (USEPA Methods 3010A/6010C)	7439-89-6	NA	100	100	10	IV

µg/L Micrograms per Liter
CAS Chemical Abstracts Service
DQO Data Quality Objectives
MCL Maximum Contaminant Level
MDL Method detection limit
NA Not applicable
NE Not established
QA Quality Assurance
QC Quality Control
RL Reporting limit
SESD Science and Ecosystem Support Division
USEPA United State Environmental Protection Agency
VOC Volatile organic compounds

[1] Preparation and analytical methods are in parentheses after analyte. The methods used should be the most recent, USEPA-approved update of the above-mentioned methods
[2] Laboratory MDLs subject to change
[3] DQOs and QA/QC frequencies per Region 4 SESD Field Branches Quality System and Technical Procedures, which are available at http://www.epa.gov/region4/sesd/fbqstp/. Level I = Field Screening; Level II = Field Analyses; Level III = Screening Data with Definitive Confirmation; Level IV = Definitive Data.
[4] MCLs per National Primary Drinking Water Regulations, USEPA 816-F-09-0004, May 2009

Table 1-7
Parameters to be Measured for Surface Water Samples
Former PSC Site, Rock Hill, South Carolina

Analyte[1]	CAS #	Performance Standards Screening Value[4] (µg/L)	RL Objectives (µg/L)	Laboratory RL (µg/L)	Laboratory MDL[2] (µg/L)	DQO Level[3]
VOC (USEPA Methods 5030B /8260B)						
Benzene	71-43-2	53	53	1.00	0.200	IV
Carbon Tetrachloride	56-23-5	352	352	1.00	0.180	IV
Chlorobenzene	108-90-7	195	195	1.00	0.180	IV
1,2-Dichlorobenzene	95-50-1	15.8	15.8	1.00	0.190	IV
1,4-Dichlorobenzene	106-46-7	11.2	11.2	1.00	0.200	IV
1,2-Dichloroethane	107-06-2	2000	2000	1.00	0.200	IV
1,1-Dichloroethene	75-35-4	303	303	1.00	0.250	IV
cis-1,2-Dichloroethene	156-59-2	NE	1.00	1.00	0.210	IV
Ethylbenzene	100-41-4	453	453	1.00	0.190	IV
Isopropylbenzene (cumene)	98-82-8	NE	1.00	1.00	0.330	IV
Methylene chloride	75-09-2	1930	1930	2.00	0.220	IV
Tetrachloroethene	127-18-4	84	84	1.00	0.140	IV
Toluene	108-88-3	175	175	1.00	0.170	IV
1,2,4-Trichlorobenzene	120-82-1	44.9	44.9	1.00	0.200	IV
1,1,1-Trichloroethane	71-55-6	528	528	1.00	0.190	IV
1,1,2-Trichloroethane	79-00-5	940	940	1.00	0.190	IV
Trichloroethene	79-01-6	NE	1.00	1.00	0.200	IV
Vinyl chloride	75-01-4	NE	1.00	1.00	0.180	IV
Xylenes (Total)	1330-20-7	NE	3.00	3.00	0.580	IV
General Chemistry (SM 2540D)						
Total Suspended Solids	NA	NA	1000	1000	700	IV

µg/L Micrograms per Liter
CAS Chemical Abstracts Service
DQO Data Quality Objectives
MDL Method detection limit
NA Not applicable
NE Not established
QA Quality Assurance
QC Quality Control
RL Reporting limit
SESD Science and Ecosystem Support Division
SM *Standard Methods for the Examination of Water and Wastewater*, APHA-AWWA-WEF
USEPA United State Environmental Protection Agency
VOC Volatile organic compounds

[1] Preparation and analytical methods are in parentheses after analyte. The methods used should be the most recent, USEPA-approved update of the above-mentioned methods
[2] Laboratory MDLs subject to change
[3] DQOs and QA/QC frequencies per Region 4 SESD Field Branches Quality System and Technical Procedures, which are available at http://www.epa.gov/region4/sesd/fbqstp/. Level I = Field Screening; Level II = Field Analyses; Level III = Screening Data with Definitive Confirmation; Level IV = Definitive Data.
[4] Surface Water Chronic Screening Values, *Region 4 Waste Management Division Freshwater Surface Water Screening Values for Hazardous Waste Sites*, http://www.epa.gov/region04/superfund/programs/riskassess/ecolbul.html#tbl1

Table 1-8
Quality Control Summary for Soil Sampling
Former PSC Site, Rock Hill, South Carolina

Analytical Parameter	Preparation Method[1]/ SOP Reference	Analytical Method[1]/ SOP Reference	No. of Trip Blanks[2]	No. of Field Duplicate Samples[3]	No. of Equipment Blanks[3]	No. of MS/MSD[2]
VOCs[4]	USEPA Method 5035/ KB Labs SOP01VOC	USEPA Method 8260B/ KB Labs SOP01VOC	1 per 20 samples	1 per 10 samples	As required[5]	1 per 20 samples
Percent Solids	NA	ASTM D2216-10/ KB Labs SOP KB-INORG-001	NA	NA	NA	NA
Fractional Organic Carbon	NA	ASTM D2974/ TestAmerica Burlington SOP BR-GT-019	NA	NA	NA	NA
Grain-size Distribution	NA	ASTM D422/TestAmerica Burlington SOP BR-GT-006	NA	NA	NA	NA
Bulk Density	NA	ASTM D2937/ TestAmerica Burlington SOP BR-GT-018	NA	NA	NA	NA
Wet Unit Weight	NA	ASTM D2216-05/ TestAmerica Burlington SOP BR-GT-016	NA	NA	NA	NA
Specific Gravity	NA	ASTM D854/TestAmerica Burlington SOP BR-GT-004	NA	NA	NA	NA
Air-filled Porosity	NA	NA[6]	NA	NA	NA	NA
Percent Saturation	NA	NA[7]	NA	NA	NA	NA

API American Petroleum Institute
ASTM American Society for Testing and Materials
MS Matrix spike
MSD Matrix spike duplicate
NA Not applicable
SOP Standard Operating Procedure
USEPA United States Environmental Protection Agency
VOC Volatile Organic Compounds

[1] The method used should be the most recent, US EPA-approved update of the above-mentioned methods
[2] Laboratory quality control
[3] Field quality control
[4] VOC analyte list per Table A-1 unless otherwise specified
[5] Equipment blanks will be collected from any equipment used in sample collection or processing that is re-used for more than one sample location and is not equipped with a liner.
[6] Based on a calculation using specific gravity (ASTM D854) and density (ASTM D698) results
[7] Based on calculations using specific gravity (ASTM D854), density (ASTM D698), grain size (ASTM D422), and water content (ASTM D2216) results

Table 1-9
Quality Control Summary for Groundwater Sampling
Former PSC Site, Rock Hill, South Carolina

Analytical Parameter	Preparation Method[1]/ SOP Reference	Analytical Method[1]/ SOP Reference	No. of Trip Blanks[2]	No. of Field Duplicate Samples[3]	No. of Equipment Blanks[3]	No. of MS/MSD[2]
VOCs[4]	USEPA Method 5030B/ TestAmerica SOP 5030/NV05-107	USEPA Method 8260B/ TestAmerica SOP 8260/NV05-77	1 per shipment[5]	1 per 10 samples	As required[6]	1 per 20 samples
Nitrate	NA	USEPA Method 9056A/ TestAmerica SOP 9056/NV12-119	NA	1 per 10 samples	As required[6]	1 per 20 samples
Sulfate	NA	USEPA Method 9056A/ TestAmerica SOP 9056/NV12-119	NA	1 per 10 samples	As required[6]	1 per 20 samples
Iron	USEPA Method 3010A/ TestAmerica SOP 3010/NV06-18	USEPA Method 6010C/ TestAmerica SOP 6010 / NV06-44	NA	1 per 10 samples	As required[6]	1 per 20 samples

MS Matrix spike
MSD Matrix spike duplicate
NA Not applicable
SOP Standard Operating Procedure
USEPA United States Environmental Protection Agency
VOC Volatile Organic Compound

[1] The method used should be the most recent, US EPA-approved update of the above-mentioned methods
[2] Laboratory quality control
[3] Field quality control
[4] VOC analyte list per Table A-2 unless otherwise specified
[5] A "shipment" refers to sending samples from the field to the laboratory
[6] Equipment blanks will be collected from any equipment used in sample collection or processing that is re-used for more than one sample location and is not equipped with a liner.

Table 1-10
Quality Control Summary for Surface Water Sampling
Former PSC Site, Rock Hill, South Carolina

Analytical Parameter	Preparation Method[1]/ SOP Reference	Analytical Method[1]/ SOP Reference	No. of Trip Blanks[2]	No. of Field Duplicate Samples[3]	No. of Equipment Blanks[3]	No. of MS/MSD[2]
VOCs[4]	USEPA Method 5030B/ TestAmerica SOP 5030 / NV05-107	USEPA Method 8260B/ TestAmerica SOP 8260 / NV05-77	1 per shipment[5]	1 per 10 samples	As required[6]	1 per 20 samples
TSS	NA	SM2540D/ TestAmerica SOP SM2540 D / NV07-63	NA	1 per 10 samples	As required[6]	1 per 20 samples

MS Matrix spike
MSD Matrix spike duplicate
NA Not applicable
SM *Standard Methods for the Examination of Water and Wastewater*, APHA-AWWA-WEF
SOP Standard Operating Procedure
TSS Total Suspended Solids
USEPA United States Environmental Protection Agency
VOC Volatile Organic Compound

[1] The method used should be the most recent, US EPA-approved update of the above-mentioned methods
[2] Laboratory quality control
[3] Field quality control
[4] VOC analyte list per Table A-3 unless otherwise specified
[5] A "shipment" refers to sending samples from the field to the laboratory
[6] Equipment blanks will be collected from any equipment used in sample collection or processing that is re-used for more than one sample location and is not equipped with a liner.

Table 2-1
Sample Container, Preservation, Storage, and Holding Time Requirements
Former PSC Site, Rock Hill, South Carolina

Matrix	Parameter[1]	Sample Container(s)	Recommended Sample Size	Preservative	Storage Conditions	Holding Time
Water	VOCs (USEPA Method 8260B)	40 mL Glass VOA vials (3), PTFE septum; no headspace **Note:** Small bubbles may occur during shipping and handling. Samples with bubbles < 6 mm in diameter (pea sized) are acceptable.	120 ml (3 x 40 ml vials)	HCl to pH ≤2	Cool, ≤6°C	14 days (preserved) 7 days (unpreserved)
	Nitrate (USEPA Method 9056A)	Glass/HDPE bottle	250 ml[2]	None	Cool, ≤6°C	48 hours
	Sulfate (USEPA Method 9056A)	Glass/HDPE bottle	250 ml	None	Cool, ≤6°C	28 days
	Iron (USEPA Method 6010C)	HDPE bottle	250 ml	HNO_3 to pH ≤2	None	180 days
	TSS (SM 2540D)	HDPE bottle	1 L	None	Cool, ≤6°C	7 days
Soil	VOCs – Medium/High Concentrations (USEPA Method 8260B)	Wide-mouth glass jar with Teflon-lined screw cap	2 oz.	None	Cool, ≤6°C	48 hours, if unpreserved and cooled to ≤6 °C 14 days (preserved[3])
	VOCs – Low Concentration (USEPA Method 8260B)	40 mL Glass VOA vials (2), PTFE-lined septum; each vial containing 5 ml of organic free water	5 g (5 g x 2)	None	Cooled to ≤6 °C and frozen within 48 hours of collection	14 days
	Percent Solids (ASTM D2216-10)	Wide-mouth glass jar with Teflon-lined screw cap	2 oz.	None	Cool, ≤6°C	7 days
	Fractional Organic Carbon (ASTM D2974)	Wide-mouth glass jar with Teflon-lined screw cap	125 ml	None	Cool, ≤6°C	28 days

[1] Analytical method is in parentheses.
[2] All parameters may be collected in a single 250 mL bottle and submitted to the laboratory for analysis.
[3] Sample preserved with methanol in accordance with USEPA Method 5035 by onsite laboratory within 48-hours of collection

Table 2-1 (Continued)
Sample Container, Preservation, Storage, and Holding Time Requirements
Former PSC Site, Rock Hill, South Carolina

Matrix	Parameter[1]	Sample Container(s)	Recommended Sample Size	Preservative	Storage Conditions	Holding Time
Soil	Grain-size Distribution (ASTM D422)	Capped, acetate liner placed on two layers of bubble wrap in cooler	1 Liner	None	Ship on dry ice	NA
	Bulk Density (ASTM D2937)					
	Wet Unit Weight (ASTM D2216-90)					
	Specific Gravity (ASTM D854)					
	Air-filled Porosity					
	Percent Saturation					

ºC Degrees Celsius
ASTM American Society for Testing and Materials
g Grams
HCL Hydrochloric acid
HNO_3 Nitric acid
HDPE High density polyethylene
L Liter
mL Milliliter
NA Not applicable
Oz. Ounce
PTFE Polytetrafluoroethylene (Teflon®)
SM *Standard Methods for the Examination of Water and Wastewater*, APHA-AWWA-WEF
USEPA United States Environmental Protection Agency
VOA Volatile organic analysis
VOC Volatile organic compounds

Table 4-1
Data Validation Qualifiers
Former PSC Site, Rock Hill, South Carolina

Qualifier	Description
J	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.
N	There is presumptive evidence that the analyte is present, but it has not been confirmed. The analyte is "tentatively identified." There is an indication that the reported analyte is present, however, all quality control requirements necessary for confirmation were not met.
R	The sample results are unusable due to the quality of the data generated because certain criteria were not met. Resampling and analysis are necessary to confirm or deny the presence of the analyte.
U	The analyte should be considered not detected at the reported value for the reasons discussed in the validation report. This is distinct from the laboratory U qualifier, which means that the analyte was simply not detected.
UJ	The analyte was not detected and the limit of detection is estimated.

Table 4-2
Data Validation Flagging Conventions
Former PSC Site, Rock Hill, South Carolina

QC Requirement	Criteria	Flag		Flag Applied To
		Positive	Non-detect	
Holding Time	Time exceeded for extraction or analysis	J	UJ[1]	All analytes in the sample
Sample Storage	> 6°C	J	UJ[1]	All analytes in the sample
GC/MS Initial Calibration (5-Point)	%RSD exceeds 40 for poor performers and method specified limit for all others	J	UJ	The specific analyte in all samples associated with the initial calibration
	Average RRF <0.050 (<0.010 for poor performers)	J[2]	UJ[2]	
Calibration Verification	CL (e.g., %D, RF, etc.) exceeded with positive bias	J	None	The specific analyte in all samples associated with the calibration verification
	CL (e.g., %D, RF, etc.) exceeded with negative bias	J	UJ	The specific analyte in all samples associated with the calibration verification
Retention Time	Retention time of analyte outside of established retention time window	R	R	The specific analyte in the sample
LCS	%R > UCL	J	None	The specific analyte(s) in all samples in the associated batch (preparation or analytical)
	10 ≤%R < LCL	J	UJ	
	%R <10%	J	R	
	%D > CL	J	UJ	
Method Blank	Analyte detected ≥ MDL	If result <RL, U If result ≥RL, U if ≤5x Blank (≤10x Common Blank Contaminants[3])	None	The specific analyte(s) in all samples in the associated batch (preparation) with results less than the action level.
Trip Blank			None	The specific analyte(s) in all samples associated with the trip blank with results less than the action level.
Equipment Blank			None	The specific analyte(s) in all samples with the same sampling date as the equipment blank with results less than the action level.
Surrogates	If surrogate %R is: o > UCL OR	J	None	Results for all analytes in the sample, **unless the sample was diluted by a factor of four or greater due to high analyte concentration or interference**
	o ≥ 20, but < LCL	J	UJ	
	If any surrogate %R < 20	J	R	
Field duplicates	RPD > CL and both field duplicate sample results > 5x RL	J	UJ	The specific analyte(s) in all samples collected on the same sampling date.
	Absolute difference between field duplicate results does not meet criteria in Table B-8	J	UJ	

[1] Professional judgment may be used to reject non-detect results
[2] Professional judgment may be used to accept data without qualification based on bias and associated sample results
[3] Site-specific VOCs: acetone and methylene chloride

Table 4-2
Data Validation Flagging Conventions
Former PSC Site, Rock Hill, South Carolina

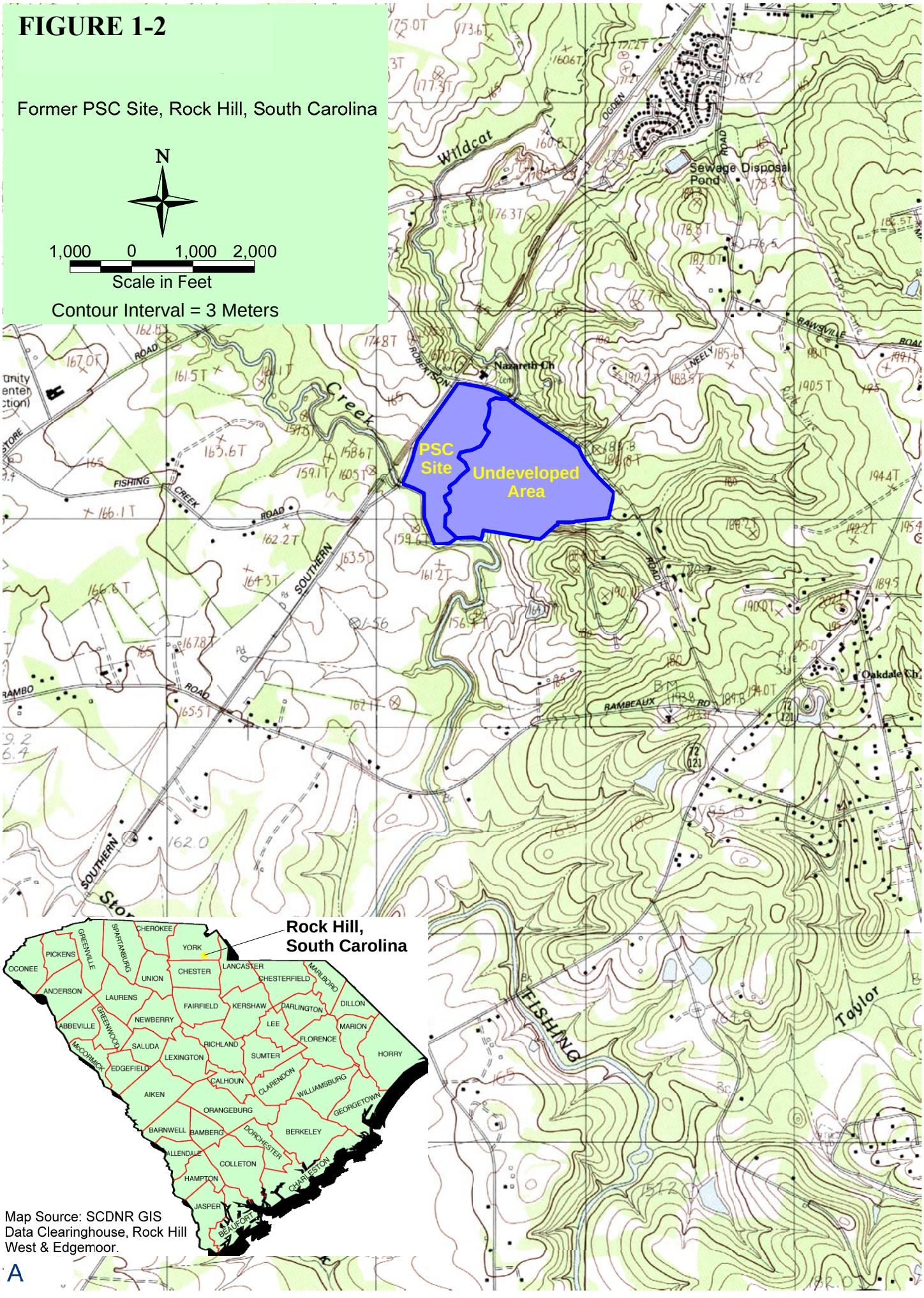
QC Requirement	Criteria	Flag		Flag Applied To
		Positive	Non-detect	
MS/MSD	MS or MSD %R > UCL	J	None	The specific analyte(s) in parent sample.
	MS or MSD %R ≥20%, but < LCL	J	UJ	
	MS or MSD %R ≤`20%	J	R	
	RPD > CL	J	UJ	
Quantitation (by laboratory)	< MDL	U	None	The specific analyte(s) in the sample.
	≥ MDL < RL	J	None	
	≥ High standard / linear range	J	None	
Percent Moisture (Soil only)	70 ≤ %Moisture <90	J	UJ	All analytes in sample
	%Moisture ≥90	J	R	All analytes in sample

°C Degrees Celsius
%D Percent difference
%R Percent recovery
CL Control limit
LCL Lower control limit
LCS Laboratory control limit
MDL Method detection limit
MS Matrix spike
MSD Matrix spike duplicate
NA Not applicable
ND Not detected
QC Quality Control
RL Reporting limit
RPD Relative percent difference
RSD Relative standard deviation
UCL Upper control limit

FIGURES

FIGURE 1-2

Former PSC Site, Rock Hill, South Carolina



Map Source: SCDNR GIS Data Clearinghouse, Rock Hill West & Edgemoor.

A

APPENDIX A

FIELD SAMPLING PROCEDUES

- A1: Field Sampling Plan
- A2: URS Safety Management Standard 48 Hazardous Materials/ Dangerous Goods Shipping

A1: Field Sampling Plan

Quality Assurance Project Plan
Appendix A
Field Sampling Plan

May 2014

Former Philip Services Corporation Site
Rock Hill, South Carolina

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TABLE OF CONTENTS

1.0 INTRODUCTION

This Field Sampling Plan (FSP) provides the methods and procedures that will be employed to conduct the field activities for the Preliminary Design Investigation (PDI) Work Plan (URS, 2012) at the former Philip Services Corporation (PSC) Site in Rock Hill, South Carolina. Section 2.0 of this plan includes the sampling procedures for the PDI. Section 3.0 details the subsurface investigation procedures for the PDI. Section 4.0 documents the decontamination procedures to be used. Section 5.0 describes how investigative derived waste will be managed. Section 6.0 details field management and reporting procedures, and Section 7.0 includes all procedures incorporated by reference in this plan. Additional details regarding the site, PDI scope, and quality control procedures are available in the PDI Work Plan (URS, 2012) and the Quality Assurance Project Plan (QAPP) (URS, 2014).

2.0 SAMPLING PROCEDURES

This section details the sampling procedures and investigation methods that will be used during the PDI. Details regarding sample collection locations and analyses are presented in the PDI Work Plan (URS, 2012).

2.1 Soil Sample Collection

Soil sampling procedures utilizing direct-push technology (DPT) or sonic drilling will follow those documented in the current version of the United States Environmental Protection Agency (USEPA) Region IV Science and Ecosystem Support Division (SESD) Operating Procedure Soil Sampling (USEPA, 2011a). The principal equipment used for the collection of surface and subsurface soil samples by DPT (i.e., Geoprobe®) or sonic drilling will be constructed of material compatible with sampling for trace concentrations of volatile organic compounds (VOCs). The drilling rigs will be inspected for fuel or hydraulic system leaks prior to mobilizing the equipment to the sampling location. In addition to following the USEPA SESD Operating Procedure for Soil Sampling, the following procedures will be completed.

1. Samples collected from each boring that exhibit the highest OVA reading will be collected in accordance with Table 2-1 of the QAPP and submitted to the onsite mobile laboratory for analysis. If all OVA readings within a borehole are within 10 % of each other or non-detect (i.e. 0 ppm), the sample at the surface and the sample directly above the saturated zone will be submitted to the onsite mobile laboratory for analysis.
2. Physical parameter (i.e., geotechnical) samples will be collected using the drill rig to advance Shelby tubes. Samples will be capped and shipped on dry ice via standard chain of custody (CoC) procedure to the laboratory.
3. Complete all required documentation, including field logbook entries, soil description, sample container label, and chain-of-custody form. Instructions and procedures for documenting field activities are outlined in Section 4.2.2 of the WP. Document control procedures are outlined in section 1.6 of the QAPP.
4. Flag or stake the sample collection locations.
5. Decontamination procedures are described in Section 4.
6. Investigation Derived Waste (IDW) management procedures are described in Section 5.

2.2 Monitor Well Sampling

Groundwater sampling procedures for monitor wells will follow those documented in the current USEPA Region IV SESD Operating Procedure Groundwater Sampling (USEPA, 2013a). Low Stress/Low Flow Sampling and No Purge (i.e. Hydrasleeve) sampling will be conducted as part of the PDI scope of work. Hydrasleeves® are no-purge samplers used to collect samples for analysis of, not only VOCs, but also inorganics (i.e., metals, natural attenuation parameters). The Hydrasleeves® sampler collects a discrete sample from within the screened interval of the monitoring well without purging and relies on groundwater advection to transport constituents from the aquifer into the well.

In addition to following the USEPA Region IV SESD Operating Procedure for Groundwater Sampling, the following specific procedures will be completed for low flow/low stress sampling:

1. Field parameters as listed below will be measured with a flow-through cell during low flow/low stress purging until three consecutive measurements meet the following criteria:
   - pH remains constant within 0.1 standard pH units;
   - Specific conductance does not vary by greater than ±5 percent;
   - Dissolved oxygen when greater than 1 milligram per liter (mg/l) does not vary by more than 10 percent or by greater than ±0.2 mg/l when less than 1 mg/l; and
   - Turbidity is reduced to 10 NTUs or less, if this is unattainable, turbidity must stabilize to ±10% NTUs.
   - Temperature does not vary by more than 1 degree Celsius (^{0}C)
   - Oxidation Reduction Potential (ORP) does not vary by more than ±10 millivolts (mV).
2. Complete all required documentation, including field logbook entries, field parameters, sample container labels, and chain-of-custody form. Instructions and procedures for documenting field activities are outlined in Section 4.2.2 of the WP. Document control procedures are outlined in section 1.6 of the QAPP.
3. Decontamination procedures are described in more detail in Section 4.
4. IDW procedures are described in more detail in Section 5.

Procedures for Hydrasleeves® No-Purge Sampling will be completed as follows:

1. Lower a Hydrasleeves® No-Purge Bag Sampler into the well using a tether. If the Hydrasleeve® is pulled up while lowering into the well, the Hydrasleeve® top check valve will open and the sample will not be a representative sample. If this occurs, a new Hydrasleeves® will need to be deployed.
2. Secure the tether at the top of the well with either the well cap on top of the casing and over the tether, or by using clips, cable ties, or other devices to a hook on the bottom of the well cap.
3. After a minimum of 24 hours, return to the Site. Don new, uncontaminated latex or nitrile gloves.
4. Gauge the water level in the well using an electronic oil/water interface probe with a probe accuracy of ±0.01 feet.
5. Remove the Hydrasleeve® from the well by pulling the tether up faster than 1 foot per second in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval.
6. Once the Hydrasleeve® has been recovered, remove the tether. Tip the sleeve over to remove any waste water that has been captured outside of the sleeve.
7. Place the groundwater collected in the Hydrasleeve® in the sample container and then place the sample container in the iced cooler.
8. Complete all required documentation, including field logbook entries, sample container label, and chain-of-custody form. Instructions and procedures for documenting field activities are outlined in Section 4.2.2 of the WP. Document control procedures are outlined in section 1.6 of the QAPP.
9. Decontamination procedures are described in Section 4.
10. IDW procedures are described in Section 5.

2.3 Surface Water Sampling

Surface water sampling procedures will follow those proscribed in the current version of USEPA Region IV SESD Operating Procedure Surface Water Sampling (USEPA, 2013b).

3.0 SUBSURFACE INVESTIGATION

Subsurface investigation will proceed concurrently with soil sampling and monitor well sampling. The results will provide guidance for soil and groundwater sampling and sample locations.

3.1 LIF Procedures for Soil

Laser induced fluorescence probe (LIF) procedures for soil will follow those outlined below and provided in Appendix B2. LIF sampling is to be performed concurrently with DPT sampling in order to delineate the horizontal and vertical extent of a free product plume. It is a fiber optic system with a probe and laser which reads the fluorescence created by ultraviolet light, allowing for a more detailed soil analysis.

The required equipment is summarized below:

- Direct-Push Technology Drilling Rig that supports LIF
- LIF System (i.e. fiber optic cables, laser laboratory, generator)
- Field Logbook
- GPS Unit

Procedure

1. Verify sample collection location, sample code, and required analyses.
2. Record GPS location.
3. Tune and calibrate the LIF according to manufacturer's recommendations.
4. Record readings taken by LIF in field book.
5. Set the rate of LIF readings dependent on amount of free product present.
6. Advance the LIF until no further fluorescence is logged or refusal.
7. Backfill the borehole using a cement-bentonite grout slurry or similar sealing material.
8. Flag or stake the sample collection locations.
9. Complete all required documentation, including field logbook entries, and LIF description,
10. Clean sensory portion of LIF between boreholes.
11. Decontamination procedures are described in Section 4.
12. IDW procedures are described in Section 5.

3.2 Groundwater Treatment System Recovery Test

Groundwater recovery testing procedures for monitoring the shutdown of the existing pump and treat system are outlined below. The required equipment is summarized below:

- Pressure Transducers
- Water Level Meter
- Latex or Nitrile Gloves
- Field Logbook

Procedure:

1. Record well construction and geologic information (i.e., well diameter, borehole diameter, screen length, total well depth, and the depth interval of the geologic unit screened by the well).
2. Don new, uncontaminated latex or nitrile gloves.
3. Record water levels in existing site monitoring wells with a water level meter, record depth to water in the field logbook or field data sheets.
4. Lower the pressure-sensitive transducer/data loggers in select wells including the existing pumping wells as outlined in the appropriate work plan.
5. Lower the pressure-sensitive transducer/data logger in the well to approximately 1 foot off the bottom of the well. Secure the transducer at the wellhead and record the depth of the transducer in the well. Allow the water level within the well to stabilize to equilibrium conditions (could take upwards of 24 hours).
6. Record the water level sensed by the transducer in the field notebook.
7. Set up the transducer for automatic continuous data logging. Start data logging. Data will be downloaded from the transducer based on storage capacity of the specific transducer or monthly at a minimum.
8. Shutdown the pump and treat system per operations and maintenance guidelines for temporary shutdown status (completed by the onsite system operator – third party contractor).
9. Collect water level measurements with the water level meter periodically to verify the transducer is functioning properly. This can be conducted when data is downloaded from the transducers.

Field activities will be documented per Section 4.2.2 of the WP and Section 1.6 of the QAPP.

3.3 Aquifer Testing

Aquifer testing (i.e. slug testing) procedures for monitor wells will follow those outlined below. Falling and rising head tests will be performed in existing wells screened in the saprolite, transition zone material, and bedrock in order to obtain estimates of hydraulic conductivity at the Site. A falling head test is only applicable to wells with fully submerged screens. Therefore, if a well screen is only partially submerged, the falling head test will be omitted and only a rising head test will be run.

The required equipment is summarized below.

- Pressure Transducer/data logger
- Bailer with string
- Water Level Meter
- Latex or Nitrile Gloves
- Field Logbooks

Procedure:

1. Record well construction and geologic information (i.e., well diameter, borehole diameter, screen length, total well depth, and the depth interval of the geologic unit screened by the well).
2. Don new, uncontaminated latex or nitrile gloves.
3. Lower the pressure-sensitive transducer/data logger in the well to approximately 1 foot off the bottom of the well. Secure the transducer at the wellhead and record the depth of the transducer in the well. Allow the water level within the well to stabilize to equilibrium conditions (could take upwards of 24 hours).
4. A pre-test static water level is to be measured and recorded using an electronic water level meter in the well.
5. Record the water level sensed by the transducer in the field notebook.
6. Collect water level measurements with the water level meter periodically to verify the transducer is functioning properly.
7. Activate the data logger and rapidly lower the bailer into the well (Falling head test). Note, a falling head test is only applicable for wells with fully submerged screens. If the screen is partially submerged, omit the falling head test and proceed to the rising head test by inserting the bailer immediately after the transducer is inserted and

allowing the water level to return to static conditions. Upon achieving static conditions, activate the data logger and continue with step 11.

8. Record water level measurements (with transducer) using the fast linear mode of recording to collect a sufficient number of data points for test analysis.
9. When the water level has recovered approximately 95 percent or after a maximum of 60 minutes, stop the test.
10. After the falling head test has been stopped and when the water level has recovered to the static water level, activate the data logger for a rising head test.
11. Rapidly withdraw the bailer from the well (Rising head test) and perform steps 8 and 9.
12. Download the data at the end of the day.
13. Decontamination procedures are described in Section 4.
14. IDW procedures are described in Section 5.

Field activities will be documented per Section 4.2.2 of the WP and Section 1.6 of the QAPP.

4.0 DECONTAMINATION PROCEDURES

Decontamination procedures will follow those proscribed in the USEPA Region IV SESD Operating Procedure Field Equipment Cleaning and Decontamination (USEPA, 2011b). Additional decontamination steps are identified for equipment that may potentially come in direct contact with concentrated materials.

Decontamination procedures will be performed in a designated area. The decontamination area will be selected on the basis of the following criteria:

1. Accessibility to heavy equipment
2. Accessibility to distilled water
3. Accessibility to treatment area

4.1 Sample Collection Equipment Contaminated with Concentrated Materials

All equipment used to collect samples of media materials (i.e. soil, groundwater, surface water) from the Site must be field cleaned before reuse or leaving the Site. The following procedure for decontamination is provided below:

1. Wash with phosphate free detergent (i.e. Alconox, Liquinox).
2. Rinse with tap water.

If this is not possible, the following procedure will be followed:

1. Leave with facility for proper disposal;
2. If possible, containerize, seal, and secure the equipment and leave onsite for later disposal; and/or
3. Containerize, bag or seal the equipment so that no odor is detected and return to the temporary field office until the procedure out lined above can be followed.

It is the responsibility of the URS Field Operations Manager to evaluate the sample material and determine the appropriate cleaning procedures for the equipment that is used to sample.

4.2 Sample Collection Equipment Contaminated with Sample Media

All equipment used in sample collection that will contact potentially contaminated sample media shall be decontaminated prior to and after use. The sample collection equipment procedure for decontamination is provided below.

1. Wipe the equipment clean.
2. Tap water rinse the equipment.
3. Wash the equipment in phosphate free detergent (i.e., Alconox or Liquinox) and water followed by a tap water rinse.
4. For grossly contaminated equipment, follow procedures in 5.1.

After decontamination, all persons handling the equipment should use new latex or nitrile gloves in order to limit cross contamination of the equipment. The equipment should be moved upwind immediately from the contaminated area in order to prevent possible recontamination. If the newly decontaminated equipment will not be immediately reused, cover the equipment in plastic wrap or aluminum foil to prevent contamination, and store the equipment in an uncontaminated area.

4.3 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

For equipment that is used to sample for trace organic or inorganic compounds, the following procedure should be followed:

1. Clean with tap water and Liquinox® or Alconox®. If necessary, use a brush to remove any additional film or matter that is adhering to the equipment. Additionally, equipment can be steam cleaned at least two feet off the decontamination pad. Do not steam clean PVC or plastic items.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with non-organic water and place on a clean, foil wrapped surface to air dry.
4. Before storing, cover and secure with clean, unused plastic wrap or aluminum foil.

4.4 Water Level Meter or Oil/Water Interface Probe

For well sounders or tape, such as those included in a water level meter or oil/water interface probe, the following procedure should be followed:

1. Clean with tap water and Liquinox or Alconox®. If necessary, use a brush to remove any additional film or matter that is adhering to the equipment.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with deionized water.

4.5 Decontamination Pads

Decontamination pads for field cleaning of equipment must meet the following:

1. Constructed in an area known to be free of surface contamination;
2. Leak free;
3. Constructed on a level, paved surface, and should facilitate the removal of wastewater, if sumps or pits are used for water removal they must be lined;
4. Equipment should be on sawhorses or racks while being cleaned and high enough above the ground to prevent equipment from being splashed and further contaminated;
5. Wash water should be removed from the pad frequently;
6. Lined with a water impermeable material without seams that is easily disposed, replaced, or repaired.

At the completion of the PDI, the decontamination pad(s) should be disposed of properly. Any sumps or pits should be backfilled after all water has been removed for disposal. If excessive water leakage has occurred, soil sampling of the area may be necessary.

4.6 Downhole Drilling Equipment

For drilling equipment used for downhole drilling activities that involve the collection and sampling of soil for trace organic and inorganic constituents, the following cleaning procedures should be followed:

1. All equipment should be cleaned, steam cleaned, and wire brushed, as needed, before being arriving onsite.
2. Equipment should be inspected for fluid leaks prior to mobilization to the sampling locations.
3. All portions of the drill rig that will be placed over the borehole should be steam cleaned (detergent and high pressure (2500 pounds per square inch (PSI) or greater) hot water (200 degrees Fahrenheit plus)) between borehole locations.

Decontamination procedures:

1. Clean with tap water and Liquinox or Alconox®. If necessary, use a brush to remove any additional film or matter that is adhering to the equipment. Steam clean if necessary. If steam cleaning is necessary, put equipment at least 2 feet above the

decontamination pad using sawhorses or racks. Any hollow equipment or equipment that has holes should be cleaned on the inside with heavy brushing.

2. Rinse thoroughly with tap water.
3. Once it is rinsed, remove it from the decontamination pad and cover with clean, unused plastic wrap. If stored overnight, make sure the plastic wrap is secured.

For DPT equipment, additional procedures should be followed, including:

1. All threaded drilling tool joints should be removed and cleaned per section 4.2.
2. Equipment that comes in contact with the sample media and is cleaned in the field for reuse should be cleaned per section 4.3.
3. Equipment that does not directly contact the sample media and is cleaned in the field for reuse can be cleaned per the previous cleaning and decontamination procedures listed.
4. Well casings, well screens, or split spoon samplers should be decontaminated per section 4.3.

5.0 INVESTIGATION-DERIVED WASTE MANAGEMENT

Uncontaminated waste and potentially contaminated liquid and solid waste materials will be generated during investigation activities. This section describes the methods for handling and disposing of the waste material, as proscribed by USEPA's SESD Operating Procedure for Management of Investigation Derived Waste (USEPA, 2010) and additionally outlined below.

5.1 Potentially Contaminated Solid Waste Material

Drill cuttings and other excess solid materials generated during boring activities and sampling activities will be placed in roll off containers or 55-gallon steel drums provided by a transportation, storage and disposal (TSD) subcontractor. URS will collect composite samples from the roll off containers or drums for Toxicity Characteristic Leaching Procedure (TCLP) analysis of VOCs, semi-volatile organic compounds (SVOCs), and metals.

Based on the results from these analyses, solid waste material will be transported and disposed of as non-hazardous or hazardous waste at an appropriate facility.

5.2 Potentially Contaminated Water

The main sources of investigative derived wastewater are expected to include:

- Water from decontamination (pressure washing) of the drill equipment.
- Purge water from well development and sampling of groundwater monitor wells.

If possible, all waste water generated onsite will be treated by the onsite waste water treatment facility. If this is not possible, all investigative derived wastewater will be containerized in a DOT approved container with tight fitting lid. The containers will be identified and left onsite with permission of the onsite treatment system operator and the URS Field Operations Manager; otherwise arrangements will be made with the URS Field Operations Manager for testing and disposal.

Decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plan representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations.

5.3 PPE

Personal Protection Equipment (PPE) such as latex and nitrile gloves will be containerized in a plastic 5-gallon bucket with a tight fitting lid. The bucket will be identified and left onsite with

permission of the URS Field Operations Manager, otherwise it will be returned to the temporary field office for disposal.

5.4 Uncontaminated Waste

Uncontaminated waste material such as shipping boxes, wrapping paper, cement bags, and general trash will placed in a conventional trash bin onsite and serviced by a local vendor.

6.0 FIELD MANAGEMENT AND REPORTING

This section outlines the communication and reporting guidelines for the investigation phases of the PDI. Team member responsibilities and roles are summarized below.

6.1 Project Management

The URS project manager (PM) is responsible for the overall performance of the PDI and is accountable to the South Carolina Department of Health and Environmental Control (SCDHEC) PM. URS' PM will be supported during field operations primarily by the URS Field Operations Manager. The URS Field Operations Manager will be responsible for coordinating and executing field operations and will be supported by the URS field staff.

The URS Field Operations Manager will provide daily progress reports to the URS PM via e-mail that will include the work completed, schedule/ staffing plans, and actual or potential issues. Resolution of issues that may potentially affect the PDI technical objectives, field investigation schedule, or field investigation scope (tasks/ costs) will require input from the URS PM/Project Chemist and approval by the SCDHEC PM.

The PM is typically supported onsite by a hydrogeologist or technical representative during investigations. The hydrogeologist is essentially a counterpart to the URS Field Operations Manager, and each of these positions will be critical toward identifying options for issue resolution. Issues that do not affect the PDI technical objectives, field investigation schedule, or field investigation scope (tasks/ costs) may be resolved onsite by the URS Field Operations Manager and hydrogeologist and communicated in the daily reports. All Site personnel including URS field staff, representatives, and subcontractors are responsible for identifying potential field issues and notifying the URS Field Operations Manager or URS PM.

6.2 Field Staff

URS field staff will be assembled in teams and assigned to the various project activities. Each team will have a designated Task Leader. The task leader will be responsible for understanding and executing the associated task and ensuring that the required equipment/ supplies and sample containers for that task are available prior to starting the task. The URS Field Operations Manager will monitor each team's work progress and work quality. Task leaders will report issues to the Field Operations Manager as they arise.

6.3 Subcontractors

The Field Operations Manager will be responsible for coordinating work with the subcontractors (drilling, laboratory, surveying, etc.). The Field Operations Manager may delegate detailed

portions of subcontractor coordination to task leaders but will remain responsible. Significant changes to the subcontractor's scope of work must be approved by the URS PM. Minor field modifications of the subcontractor's technical approach may be approved by the URS Field Operations Manager.

6.4 Health & Safety

The URS Site Safety Coordinator is identified in the URS Health and Safety Plan (HASP). All field staff including onsite subcontractors will be required to read and understand the HASP, and to sign the plan prior to working onsite. Daily tailgate safety meetings will be required for all onsite staff to discuss safety issues related to work completed the previous day and work planned for that day. Any safety incidents or significant near misses will be reported to the URS Site Safety Coordinator and/or URS Field Operations Manager.

7.0 REFERENCES

URS, 2014. Quality Assurance Project Plan, May 2014.

URS, 2012. Preliminary Design Investigation Work Plan, September 2012.

USEPA, 2013a. SESDPROC-301-R3 Groundwater Sampling, USEPA Science and Ecosystem Support Division, Athens, Georgia, March 6, 2013.

USEPA, 2013b. SESDPROC-201-R3 Surface Water Sampling, USEPA Science and Ecosystem Support Division, Athens, Georgia, February 28, 2013.

USEPA, 2011a, SESDPROC-300-R2 Soil Sampling, USEPA Science and Ecosystem Support Division, Athens, Georgia, December 20, 2011.

USEPA, 2011b. SESDPROC-205-R2 Field Equipment Cleaning and Decontamination, USEPA Science and Ecosystem Support Division, Athens, Georgia, December 20, 2011.

USEPA, 2010. SESDPROC-202-R2 Management of Investigation Derived Waste, USEPA Science and Ecosystem Support Division, Athens, Georgia, October 15, 2010.

A2: URS Safety Management Standard 48 Hazardous Materials/ Dangerous Goods Shipping

URS SAFETY MANAGEMENT STANDARD

Hazardous Materials/Dangerous Goods Shipping

1. Applicability

This standard applies to URS Corporation (URS) and its subsidiary companies that ship hazardous materials (hazmat).

U.S. Department of Transportation (DOT) regulations for hazardous materials shipping (by air, ground, or water) and the International Air Transportation Association (IATA) regulations for dangerous goods shipping (by air) prohibit the shipment of certain materials unless they are packaged, marked, labeled, and accompanied with shipping documentation in a specified manner. Failure to adhere to these shipping requirements may result in fines to the company and disciplinary action to the employee(s) involved in the shipment.

Examples of Hazardous Materials/Dangerous Goods regulated by the DOT and IATA that may be encountered or used during URS projects may include, but are not limited to, certain field environmental samples, compressed gases (fire extinguishers, calibration gases, compressed air, and welding and cutting gases), ionizing radiation sources used to calibrate detection equipment or analytical equipment, nuclear-density meters, laboratory reagents, hazardous wastes, materials used for bench-scale and pilot plant operations, oils, greases, lubricating fluids, cleaning solvents, degreasing solvents, paints, spray paints, paint removers and/or strippers, diesel fuel, gasoline, pesticides, inks, glues, and other adhesives, battery fluids, ammonia cleaning solutions and peroxide solutions. When possible, only use ground carriers for transportation of hazardous materials.

The air shipment of environmental samples represents a significant percentage of hazardous materials/dangerous goods shipped by URS. Although most environmental samples (both water and soil) do not meet the definition of hazardous, extreme care must be taken to properly classify materials.

2. Purpose and Scope

The purposed of this standard is to prevent shipping-related incidents and violations, and prevent injuries to employees and members of the public.

3. Implementation

Implementation of this standard is the responsibility of the URS manager directing activities of the facility, site, or project location.

Project Managers' responsibilities include the following related to hazardous materials shipping:

A. Ensure that every employee and driver involved with shipping a hazardous material in commerce is trained and certified, and records are maintained in accordance with 49 Code of Federal Regulations (CFR) 172.704.

B. Ensure every driver of a truck that has a gross vehicle weight rating exceeding 26,000 pounds (11.8 kg), or as mandated by state, or hauling a placardable quantity of hazmat has a commercial driver's license (CDL) with proper endorsements (e.g., hazmat, tank, etc.) in accordance with 49 CFR 383.91 and 383.93, and has current DOT hazmat training in all required areas, in accordance with 49 CFR 172.704 and 177.816.

C. Ensure every truck hauling hazmat in regulated quantities carries a current DOT Hazardous Materials Certificate of Registration; and if required, a Federal Motor Carrier Safety Administration (FMCSA) Hazardous Materials Safety Permit and any other state-mandated registration.

D. Verify that insurance coverage includes transportation of hazardous materials over commercial roads (49 CFR 387.9).

E. Ensure every truck hauling hazmat has the proper documentation, including shipping paper, emergency response information, 24-hour emergency response telephone number, and the DOT Hazardous Materials Registration (if required). In addition, when using a third-party emergency response provided such as CHEMTREC, a Customer Contract Number (CCN#) must appear on the shipping paper.

F. If using CHEMTREC as the 24-hour emergency number, ensure that current Safety Data Sheets (SDSs) for each transported hazmat are submitted to CHEMTREC before transport.

G. Ensure all hazmat incidents are properly reported to the project safety supervisor, in accordance with URS reporting procedures.

H. Report hazardous material spills within 24 hours, including material spilled and estimated quantity.

4. Requirements

In order to minimize the potential for an improper shipment, Project Managers and Site Managers are required to ensure that an individual trained according to DOT Regulations in 49 CFR 172 Subpart H and, if applicable, IATA Dangerous Goods Regulations Subsection 1.5 is responsible for the correct classification, packaging, marking, labeling, and completion of shipping papers for any hazardous materials being shipped offsite. No hazmat shipments shall leave the site without prior inspection. The assigned person must have current DOT

hazmat certification and, if applicable, IATA certification. DOT requires recurrent training every 3 years, and IATA requires recurrent training every 2 years.

A. Staffing

1. Each project or site must ensure that DOT hazmat-trained individuals are involved in the process of preparing hazardous materials for shipment.

2. Each location where hazardous material shipping occurs or where hazardous material employees are assigned must identify a local or regional shipping specialist.

3. The assigned shipping specialist must have current certification of DOT hazmat, and if applicable, IATA training.

B. Hazmat Hotline

URS maintains a **shipping Hazmat Hotline** for hazardous materials/dangerous goods to provide answers to specific shipping questions.

1. **800-381-0664** in Canada and U.S.

2. **919-461-1227** for other countries

3. **Email: HazmatHotline@urs.com**

C. Shipper Training

All employees involved in the transportation of hazmat in commerce must be formally trained and certified in accordance with 49 CFR 172.704. Training must include the following components: general awareness, function-specific, safety, hazmat security, security plan (if applicable), and if applicable, driver training.

1. Training Requirements. Require employees who package, prepare paperwork, load and/or unload, and transport hazardous materials be trained to the appropriate level of activity:

   a. Training is required prior to performing hazardous material shipping activities.

   b. Training is required when regulatory changes impact current procedures, and every 2 years (IATA) or 3 years (DOT).

c. Regional or local hazmat shipping specialists must complete a 2-day hazardous material/dangerous goods shipping course conducted by URS, or complete an outside equivalent course.

d. Drivers may be exempt from function-specific training if the DOT's Materials of Trade (MOT) exception applies to the shipment (see Section 4.K.6 and SMS 048 AMER, Supplemental Information A).

e. Certain shipments of hazmat must have a Hazardous Materials Security Plan (see Section 4.I for more information).

2. Training Records. Employers are required to maintain training records for all hazmat employees during employment, and for 90 days after, including:

a. Hazmat employee's name;

b. Completion date of most recent training;

c. Training materials (copy, description, or location);

d. Name and address of hazmat trainer; and

e. Certification that the hazmat employee has been trained and tested.

D. Hazmat Driver Training

1. In addition to the training required by 49 CFR 172.704 (above), hazmat drivers must also be trained in the requirements of 49 CFR 177.816, or have a CDL with a hazmat endorsement.

2. CDL requirements are located in 49 CFR 383.

E. Hazmat Registration

1. Shippers or carriers who offer any of the following in commerce must have a hazmat registration in accordance with 49 CFR 107.601-620:

a. Any highway route–controlled quantity of a Class 7 (radioactive) material;

b. More than 55 pounds (25 kilograms) of a Division 1.1, 1.2, or 1.3 (explosive) material in a motor vehicle, rail car, or freight container;

c. More than 1 liter (1.08 quarts) per package of a material extremely toxic by inhalation (i.e., “material poisonous by inhalation,” as defined in 49 CFR 171.8, that meets the criteria for “hazard zone A,” as specified in 49 CFR 173.116(a) or 173.133(a));

d. A hazardous material in a bulk packaging having a capacity of 3,500 gallons for liquids or gases, or more than 468 cubic feet of solids;

e. A shipment in other than bulk packaging of 5,000 pounds gross weight or more of one class of hazardous material for which the transport vehicle requires placarding for which placarding of a vehicle, rail car, or freight container is required for that class; and

f. Except for certain farm-related activities, any quantity of materials requiring placarding.

In general, this includes Company fuel and lube trucks that travel on public roads.

2. The vehicle must keep a copy of the current Certificate of Registration in each truck used to transport hazmat.

3. In addition, a copy of the registration statement filed with the DOT and the Certificate of Registration must be maintained at the principal place of business for a period of 3 years.

4. This registration must be renewed each year.

F. FMCSA Hazardous Materials Safety Permits

1. Since January 2005, certain highway carriers of hazmat must obtain a hazmat safety permit from the FMCSA as required under 49 CFR 385.403, 390.3, and 390.19. In general, a safety permit is required if a motor carrier transports any of the following:

a. A highway route–controlled quantity of a Class 7 (radioactive) material;

b. More than 55 pounds (25 kilograms) of a Division 1.1, 1.2, or 1.3 (explosive) material or an amount of a Division 1.5 (explosive) material requiring placarding;

c. More than 1.08 quarts (one liter) per package of a “material poisonous by inhalation,” that meets the criteria for “Hazard Zone A.”

d. A “material poisonous by inhalation” that meets the criteria for “Hazard Zone B ” in a bulk packaging (capacity greater than 119 gallons [450 liters]);

e. A “material poisonous by inhalation” in a ”bulk packaging,” both defined in 49 CFR 171.8, that meets the criteria for “Hazard Zone C or “Hazard Zone D” in a packaging having a capacity equal to or greater than 3,500 gallons (13,248 liters); or

f. A shipment of compressed or refrigerated liquefied methane or liquefied natural gas, or other liquefied gas with a methane content of at least 85 percent, in a bulk packaging having a capacity equal to or greater than 3,500 gallons (13,248 liters).

G. Shipping Papers

1. With few exemptions, anyone who offers a hazmat for transportation must complete shipping papers that must be carried in the vehicle, within the driver’s immediate reach when restrained by a seat belt, and visible to a person entering the vehicle, or in a holder mounted on the inside of the driver’s door (49 CFR 172, Subpart C; and 49 CFR 177.817).

2. Shippers must retain copies of shipping papers for at least 2 years after the transporter accepts the material (49 CFR 172.201).

3. A motor carrier using a shipping paper without change for multiple shipments of one or more hazardous materials having the same shipping name and identification number may retain a single copy of the shipping paper, instead of a copy for each shipment made, if the carrier also retains a record of each shipment made, to include shipping name, identification number, quantity transported, and date of shipment.

4. Shippers and transporters of hazardous waste (as defined in 40 CFR 261) must retain copies of hazardous waste manifests for at least 3 years after the initial carrier accepted the material.

5. Upon request, hazmat shipping papers and hazardous waste manifests must be made available to federal, state, and local inspectors.

H. Emergency Response Information

1. DOT requires anyone who offers, transports, or handles hazmat to have emergency response information immediately available. (49 CFR 172.600). Safety Data Sheets (SDSs) and DOT's Emergency Response Guidebook are common reference sources for emergency response information.

2. In addition, persons who offer hazmat for transportation must provide a 24-hour emergency response telephone number that must be monitored by a knowledgeable person at all times while the material is in transit.

3. URS maintains an account with CHEMTREC for this service. Before using this service, URS must submit an SDS or Waste Safety Data Sheet to them. Contact the Hazmat Hotline (see Section 4.B) for more information.

I. Hazardous Material Transportation Security Plan

1. URS sites that transport or offer the following types or quantities of materials for transportation must have a Hazardous Material Transportation Security Plan on site and must ensure that all hazmat employees are trained in the plan, as required by 49 CFR 172.800.

   a. Any quantity of a Division 1.1, 1.2, or 1.3 material;

   b. A quantity of a Division 1.4, 1.5, or 1.6 material requiring placarding in accordance with subpart F;

   c. A large bulk quantity of Division 2.1 material;

   d. A large bulk quantity of Division 2.2 material with a subsidiary hazard of 5.1;

   e. Any quantity of a material poisonous by inhalation, as defined in 49 CFR 171.8;

   f. A large bulk quantity of a Class 3 material meeting the criteria for Packing Group I or II;

g. A quantity of desensitized explosives meeting the definition of Division 4.1 or Class 3 material requiring placarding in accordance with subpart F;

h. A large bulk quantity of a Division 4.2 material meeting the criteria for Packing Group I or II;

i. A quantity of a Division 4.3 material requiring placarding in accordance with subpart F;

j. A large bulk quantity of a Division 5.1 material in Packing Groups I and II; perchlorates; or ammonium nitrate, ammonium nitrate fertilizers, or ammonium nitrate emulsions, suspensions, or gels;

k. Any quantity of organic peroxide, Type B, liquid or solid, temperature controlled;

l. A large bulk quantity of Division 6.1 material (for a material poisonous by inhalation see paragraph (e) above);

m. A select agent or toxin regulated by the Centers for Disease Control and Prevention under 42 CFR 73 or the United States Department of Agriculture under 9 CFR 121;

n. A quantity of uranium hexafluoride requiring placarding under 49 CFR 172.505(b);

o. International Atomic Energy Agency (IAEA) Code of Conduct Category 1 and 2 materials including Highway Route Controlled quantities as defined in 49 CFR 173.403 or known radionuclides in forms listed as RAM-QC by the Nuclear Regulatory Commission;

p. A large bulk quantity of Class 8 material meeting the criteria for Packing Group I.

2. If a project or office determines that a hazmat security plan is required, contact the URS Hazmat Hotline.
3. A Hazmat Security Specialist will be assigned at each site required to have a hazmat security plan.
4. All Hazmat Security Plans will be reviewed annually and updated if required.

J. Hazardous Incident Report

1. A person in possession of a hazmat at the time of a reportable incident as outlines in 49 CFR 171.15 must immediately report the incident to the National Response Center. In addition, these incidents require filing a detailed written incident report within 30 days of the incident (see 49 CFR 171.16).

2. Incidents that do not trigger the immediate reporting as outlined in 49 CFR 171.15, but which meet any of the other incident criteria in 49 CFR 171.16 still warrant a detailed written report under 171.16 within 30 days of the incident.

3. All hazmat incidents must be reported in accordance with SMS 049 – Incident Reporting, Notifications & Investigation.

K. General Procedures

1. Select the best way to ship the hazardous material based on the quantity, hazard(s), and mode of transportation (e.g., air, land, water).

2. Ensure shipping containers are designed, constructed, filled, closed, secured and maintained so that, under normal conditions of handling and transport, there will be no accidental release of hazardous materials which could endanger public safety.

3. Ensure that a copy of the closure instructions provided by the package manufacturer is available for each UN specification shipping container type that is used at the facility.

4. Package, mark, label, and placard according to applicable regulations.

5. Complete the shipping documentation according to applicable regulations, which may include bill of lading, shipper's declaration, hazardous waste manifest, or other, as applicable.

6. Follow hazard communication requirements:

   a. Send a copy of the appropriate Emergency Response Guidebook page or MSDS with each shipment.

b. Include the 24-hour emergency response phone number (CHEMTREC 800-262-8200 domestic; 1-703-741-5500 international) on the shipping paperwork.

i. Any shipment of a hazardous material or hazardous waste requires that CHEMTREC be notified in advance of the shipment. CHEMTREC requires that either an SDS or Hazard Profile of the hazardous material being offered for shipment be provided to CHEMTREC. URS maintains a current contract with CHEMTREC to provide this required service and the right to use the CHEMTREC emergency phone number on shipping papers when notification of the shipment has been made. Contact the Hazmat Hotline (see Section 4.B) before contacting CHEMTREC.

7. URS also maintains current Hazardous Materials Certificate of Registrations with the U.S. Department of Transportation. Contact the Hazmat Hotline for more information.

8. DOT regulations include a "Materials of Trade" or "MOTs" exception. MOTs are hazmat, other than hazardous waste, that are carried on a motor vehicle:

- to protect the health and safety of the motor vehicle operator or passengers, such as insect repellant or a fire extinguisher;
- to support the operation or maintenance of a motor vehicle (including its auxiliary equipment), such as a spare battery or gasoline; or
- to directly support a principal business of a private motor carrier (including vehicles operated by a rail carrier) that is other than transportation by motor vehicle – for example, landscaping, pest control, painting, plumbing, or welding services.

URS operations may qualify under this exception. Refer to the exception requirements under 49 CFR 173.6. A hazmat-trained employee should make the determination as to whether this exception will apply to the shipment.

L. Special Requirements

1. Do not offer packages for shipment without knowing the contents and classifying the packages in accordance with the DOT, and, if applicable, IATA regulations. Do not ship potentially hazardous

materials using an unknown carrier or broker. A Hazardous Material Transportation Security Plan may be required for shipment of certain hazardous materials, and employee training is required to protect shipments of hazardous materials from theft and acts of terrorism.

2. Contact the applicable shipping company, shipping specialist, or the Hazmat Hotline if you are unsure or suspect there may be additional special requirements on a shipment.

3. Some transporters have more stringent requirements than DOT or IATA. For example, the United Parcel Service (UPS) publishes its own Guide for Shipping Ground and Air Hazardous Materials. URS shipping training and this program may not meet these additional requirements.

4. Some countries have more stringent requirements than DOT or IATA. Refer to the international hotline for assistance.

5. For international shipments, an expediter may be required to ensure needed materials are not held in customs. It may be advisable to purchase hazardous materials in the destination country.

6. The air shipment of environmental media samples represents a large percentage of potential hazardous materials/dangerous goods shipped by URS. Most environmental media samples (water and soil) typically do not meet the definition of a dangerous good (hazardous material) unless preservatives are added to make the sample a corrosive material. DOT exemptions may apply to allow air shipment as long as the samples are properly packaged and the package is properly marked; however, extreme care must be taken to properly classify, package, and mark the environmental samples to ensure compliance with the regulations.

7. Because more stringent requirements apply to air shipments, ground shipment (e.g., including use of a lab courier service) should be considered first for hazardous materials shipping.

8. Hazardous materials shipments must be loaded and secured in an appropriate shipping container (see 4.K.2 for additional information). The shipping container must also be loaded and secured on the means of transportation used for shipping in such a way as to prevent, under normal means of transport, damage to the

shipping container or to the means of transportation that could lead to an accidental release of the hazardous materials.

9. Where an accidental release of hazardous materials from its packaging/containment in excess of a prescribed quantity or concentration occurs or is imminent, any person who at the time has the charge, management or control of the means of containment shall report the occurrence or imminence of the release to the project safety supervisor. Every person required to make a report shall, as soon as possible in the circumstances, take all safe and reasonable emergency measures to reduce or eliminate any danger to public safety that results or may reasonably be expected to result from the release using the Safety Data Sheets (SDSs), DOT's Emergency Response Guidebook, or other resources as appropriate.

5. Documentation

The following documentation will be maintained in the project files:

A. Training Records

1. Employers are required to maintain training records for all hazmat employees during employment and for 90 days after, including hazmat employee's name; completion date of most recent training; training materials (copy, description, or location); name and address of hazmat trainer; and certification that the hazmat employee has been trained and tested. Ensure training records include:

   a. Hazmat employee's name;

   b. Completion date of most recent training;

   c. Training materials (copy, description, or location);

   d. Name and address of hazmat trainer; and

   e. Certification that the hazmat employee has been trained.

B. Shipping Documentation Records

1. Shippers must retain copies of shipping papers for at least 2 years after the transporter accepts the material. Shippers and transporters of hazardous waste must retain copies of hazardous

waste manifests for at least 3 years after the initial carrier accepted the material.

2. For each shipment:

   a. Copy of shipper's declaration for dangerous goods;

   b. Copy of applicable ERG or SDS accompanying shipment;

   c. Copy of information (MSDS or Hazard Profile) provided to CHEMTREC; and

   d. Supporting documentation related to the classification of the material.

C. Hazardous Materials Transportation Security Plan, if required and applicable to site or facility operations.

D. Hazardous Incident Report(s), if reportable incident(s) has occurred.

6. Resources

A. 49 Code of Federal Regulations, Parts 171-180, Subchapter C – Hazardous Materials Regulations

B. International Air Transport Association Dangerous Goods Regulations (DGR), updated and issued annually

C. International Maritime Dangerous Goods Code. International Maritime Organization, Amendment 29-98

D. DOT Office of Hazardous Materials Safety

E. URS Hazardous Materials Hotline: **800-381-0664**

F. SMS 049 – Incident Reporting, Notifications & Investigation

7. Supplemental Information

A. Materials of Trade Summary

APPENDIX B

FIELD EQUIPMENT OPERATIONS PROCEDURES

- B1: Field Operations Manual
- B2: LIF Manufacturer Operating Procedures and Technical Literature

B1: Field Operations Manual

Quality Assurance Project Plan Appendix B1 Field Equipment Operations Procedures

May 2014

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Measurement of field parameters during sampling activities will follow the current EPA Region IV SESD Operating Procedures listed below:

- SESDPROC-103 Field Turbidity Measurements;
- SESDPROC-100 Field pH Measurements;
- SESDPROC-113 Field Measurement of Oxidation-Reduction Potential;
- SESDPROC-106 Field Measurement of Dissolved Oxygen;
- SESDPROC-102 Field Temperature Measurement; and
- SESDPROC-101 Field Specific Conductance Measurement
- SESDPROC-105 Groundwater Level and Well Depth Measurement
- SESDPROC-111 In Situ Water Quality Monitoring

Specific instrument models that will be used to measure field parameters are identified in section 2.4.1 of the QAPP. The following table provides details regarding spare parts for field monitoring equipment.

Spare Parts for Field Equipment

Equipment	Spare Parts	Location
Portable water quality meter (YSI-556 or equivalent)	Standard buffer(s) Spare electrodes Spare battery Spare dissolved oxygen probe membrane kit with o-rings	Field equipment case or field office
Turbidity Meter (LaMotte 2020 Portable Turbidity Meter or equivalent)	Standards Spare sample cells Spare battery	Field equipment case or field office
Air monitoring equipment (MiniRae 2000 or equivalent)	Spare UV Lamps Isobutylene air calibration gas Regulators for gas Spare battery chargers	Field equipment case or field office
Electronic water level meter	Spare battery Weighted steel tape Chalk	Field equipment case or field office

Any deficiencies in testing, inspection, or calibration of field measurement equipment will be reported by field staff/analyst to the Field Operations Manager, who will notify the PM and Project QA Officer. Corrective actions will be implemented and its effectiveness documented by the Field Operations Manager per Section 3.1.4 of the QAPP.

B2: LIF Manufacturer Operating Procedures and Technical Literature

Laser-Induced Fluorescence Primer

Randy St. Germain, President , Dakota Technologies, Inc.

Excerpted in Applied NAPL Science Review, volume 1, issue 9, September 2011

Editors Note: Randy St. Germain is one of the world's leading scientists in the development and application of laser-induced fluorescence tools for the investigation of non-aqueous phase liquids (NAPLs).

Laser-induced fluorescence (LIF) employs laser light to excite fluorescent molecules contained in the majority of non-aqueous phase liquids (NAPLs) including petroleum fuels/oils, coal tars, and creosotes. Direct push logging of the NAPL's inherent fluorescence with depth provides rapid and cost-effective delineation of NAPL. Multiple LIF logs conducted at NAPL release sites provide a relatively non-subjective basis for a detailed NAPL conceptual site model.

[For purposes of simplicity this article focuses principally on ultraviolet (UV) LIF's application toward petroleum fuels/oils, not creosotes and coal tars, which demand visible wavelength LIF.]

BACKGROUND: Laser light is "clean" (narrow in wavelength) and relatively powerful. As such, laser light is readily transmitted with fiber optics, allowing scientists to deliver laser light remotely to samples and bring any light resulting from interaction with the laser back for analysis. Spectroscopists (those who study matter with light) discovered long ago that polycyclic aromatic hydrocarbons (PAHs) are highly fluorescent, and many of their spectra were catalogued decades ago[1]. To coax them into fluorescing you must excite PAHs into an electronically excited state by shining the correct color of light at them. They will absorb that light and, in a matter of nanoseconds, emit some light of their own as a way of getting rid of the excess energy they had gained.

The color of the emitted light is "Stokes shifted" (of lower energy or longer wavelength than the exciting light) and is dependent on the number of rings and degree of substitution of the PAH that emits the fluorescence. Petroleum NAPLs, as it turns out, usually contain enough PAHs to be detected with LIF (even gasoline). This is illustrated in the photos below showing layers of crude oil and diesel on wet sea sand, which demonstrate the utility of fluorescence for "seeing" petroleum NAPLs.

Notice the different fluorescence color being emitted. This is due to the two NAPL's differing PAH distribution/content. Notice also that the "core" of the NAPL is brightest and the edges get fainter. This is because fluorescence scales monotonically with the amount of oil/fuel in the pore spaces. There are many exceptions to this monotonicity, but it's typical for a single fuel/oil on a single soil type.

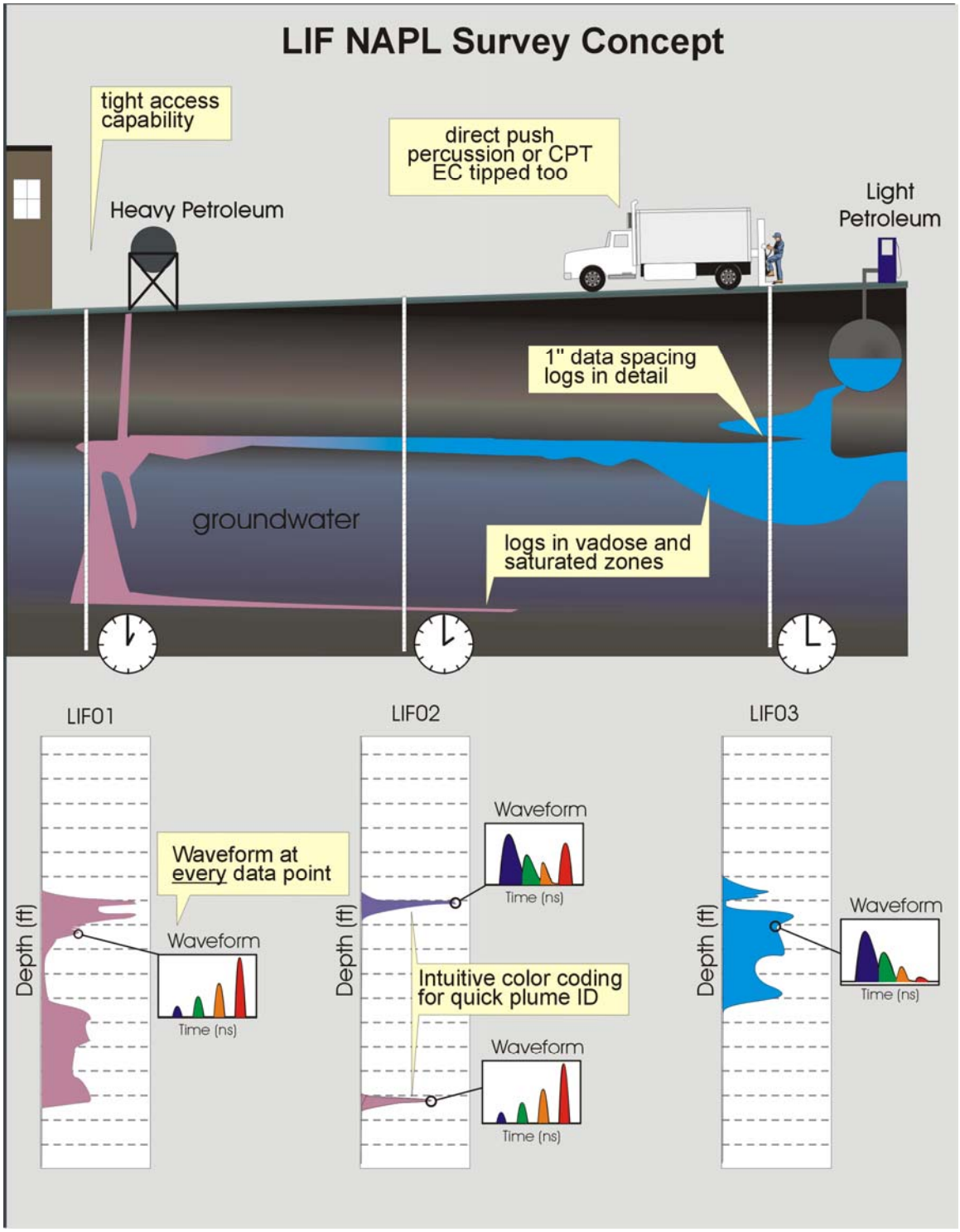
Finally, while the photographs can't capture lifetime information, the PAHs in the crude oil and the diesel are emitting varying colors of light over varying periods of time, allowing further differentiation by LIF systems that are designed to log both the spectral and temporal nature of the emitted light.

Photographs of crude oil (left) and diesel fuel (right) on water-saturated sand under room light (top) and long wavelength ultraviolet lamp excitation (bottom).

BASIC METHOD:
Commercially available LIF screening tools take advantage of the same phenomenon shown in the photograph. However, instead of the fuels/oils emitting light from behind the 'window' of the clear jar, a clear sapphire window is fitted into the side of a direct push probe that is pushed through the soil column (see illustration below). As this sapphire-windowed probe is advanced steadily into the soil column at ~2cm/second, pulses of laser light are sent down the rod string via fiber optics, where they exit the window and shine onto the face of the passing soil (without penetrating into the formation). Any resulting fluorescence and/or scattered laser light that comes back into the window is brought uphole by a second fiber, where the light is processed and analyzed in real time.

LIF systems log up to 100 ft below the surface with an "average" LIF log being about 35 ft and taking approximately 45 minutes start to finish. The logs indicate in real time exactly where

NAPL fluorescence is occurring, with major differences in the fluorescence color and lifetime due to differing NAPL types, weathering, or false positives. The goal is to quickly and non-subjectively survey the subsurface to generate a “machine vision” electronic data set used to develop a NAPL conceptual site model (CSM).



Concept diagram of NAPL site investigation with LIF. Direct push logging generates 300-400 ft of detailed information daily, spread across 8-14 locations, allowing for rapid assessment of the nature and extent of NAPL.

BASIC LIF DATA INTERPRETATION:

The simulated logging scenario in the illustration below contains many classic situations that are encountered during a typical LIF investigation. Four distinct mobile NAPL intervals in the vadose and saturated zones (far left saturation curves) and a layer of NAPL-free shell hash occur within two primary soil types: coarse high permeability soils (sands/gravel) and fine grain low-permeability soils (silts/clays). Notice the LIF probe traveling down through the soil column in the diagram's center. On the right is the resulting LIF log that would be generated in real time as the LIF probe is pushed/pounded steadily through the soil column.

The LIF log y-axis is depth below ground surface, while the x-axis is fluorescence intensity or Signal %RE. The fluorescence signal typically scales monotonically with NAPL pore saturation. The RE stands for Reference Emitter, which normalizes the LIF response similar to using a tank of 100 ppm isobutylene to normalize the response on a handheld PID. The RE is a cuvette filled with a stable fluorescent NAPL that is placed on the sapphire window and recorded prior to each and every LIF log. It serves as both a single point calibration and as an overall system check (RE should look 'correct' to the LIF operator).

The RE-labeled callout (waveform) at the upper right side of the figure is not attached to any depth of the log, because it wasn't acquired at depth, but right before logging began. These callout waveforms are a rather complicated hybrid of both spectral (color) and temporal (lifetime) fluorescence information. The full nature and utility of waveforms will be covered in detail in future ANSR articles. Until then, suffice it to say that for UV LIF, waveforms from fuels dominated by 2-ring PAHs (like naphthalenes in kerosene), are heavily weighted toward the first (blue) channel. Diesel has a broad distribution of 2, 3, and 4 ring PAHs, so it's lit up across all channels (blue, green, orange, and red). Bunker fuel's fluorescence is dominated by the larger 4 and 5 rings, so its fluorescence waveform is right-weighted (high in the orange and red channels).

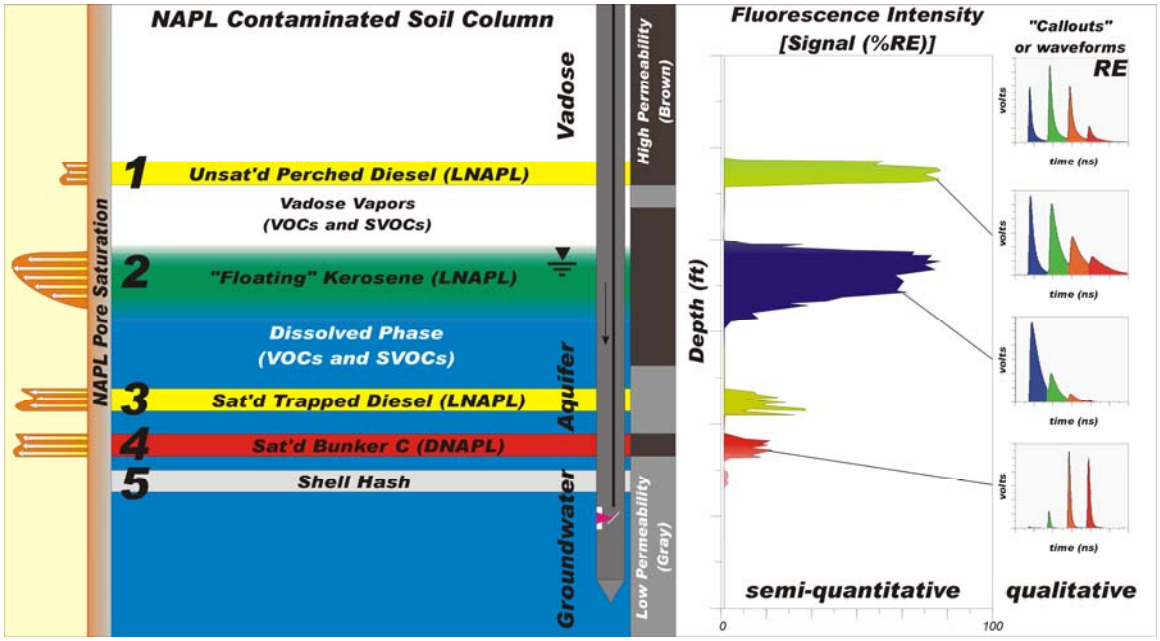
The x-axis (Signal %RE) of the LIF log is total fluorescence (area) of each of the hundreds of waveforms acquired during the log, divided by the total fluorescence under the RE waveform. Keep in mind that the voltages on the y-axes of the callout waveforms are typically not all scaled the same, so be careful of this when examining LIF logs. Operators usually "zoom in" on the y-scale of the waveforms so the waveform shape can be clearly seen - because the shape tells us a lot about the nature of the NAPL (or may indicate it is NOT NAPL).

As the LIF probe passes through the clean upper vadose zone of our soil column we see that the LIF fluorescence response is low (background) because most soils that are NAPL-free have near zero fluorescence of their own. That's because LIF measurement in/on opaque materials like soils does not have enough sensitivity to detect the relatively tiny quantity of PAHs that exist in soil pore gas, let alone the mono-aromatics or aliphatics that don't fluoresce.

As we enter level 1 and the LIF window encounters the ~20% pore saturation layer of perched diesel, we see a large "green" response on the x-axis of the LIF log because the diesel is fluorescing. You'll notice it approaches 80% RE, which is a fairly robust signal. See the second callout down? That single waveform was acquired at the depth shown tagged to this waveform with a line. Waveforms are acquired and stored every inch or so, the operator simply chose this one to represent the many "green" waveforms acquired while passing through the perched

diesel lens. The waveform shown is a ‘classic’ diesel waveform – very few NAPLs other than diesel have this waveform.

So why is the perched diesel’s fluorescence response filled with the color green? That is the result of the mixing of the 4 colored peaks or channels of the perched diesel’s waveform. This technique of fill-coloring the log allows us to see at a glance which depths have similar or differing fluorescence characteristics (different waveforms). The perched diesel occurs in the permeable sand/gravel layer, which enhances the fluorescence response because high permeability soils allow more diesel to press up against the sapphire LIF window (versus fine-grained soils which displace the NAPL), so a healthy 80% RE response is achieved.



Cross-sectional diagram showing the relationship between NAPL type, matrix, and saturation of pore spaces with NAPL vs LIF data.

Below the diesel-affected sand/gravel lens we pass through the clay layer on which the diesel was perched, and the fluorescence signal goes back to baseline in less than an inch. This is common, as LIF is a nearly instantaneous measurement.

As we pass through level 2 (a classic ‘floating NAPL’ layer) we notice that the LIF response rises and falls in concert with the NAPL pore saturation, and generates a response graph just like the NAPL saturation curve on the far left[2]. We’ve been seeing these “shark’s fin” curves at homogeneous course sand/gravel sites for decades. Notice the LIF log’s fill color is blue rather than green - and the waveform is very different. That’s due to naphthalenes (2-ring PAHs) which dominant the PAHs in kerosene. The blue fill is the resulting fill color calculated by the waveform’s blue channel dominance.

In addition, notice that even though the kerosene is at 100% pore saturation, its fluorescence is only registering the same Signal %RE as the 20% pore saturation diesel above it. That’s because kerosene is less fluorescent, by about a factor of 5, than diesel, due to kerosene’s lower PAH content. We simply have to live with the variable fluorescent nature of different fuels/oils.

As we exit level 2 we exit the diminishing kerosene NAPL and enter high dissolved-phase BTEX and PAHs (especially naphthalenes as they are the most soluble of all PAHs). But again, the LIF appears to be "missing" these too. That's because while PAHs are highly soluble in NAPL, the water can only hold 1/100th to 1/1,000,000th as much PAHs as the NAPL. LIF can and does detect dissolved phase PAHs in clear water, but not when it's mixed up in opaque muddy soil. So remember, LIF is generally <u>blind to dissolved phase</u>, it only responds to the NAPL.

As the probe enters level 3 the window passes by a macropore or fracture of saturated diesel that was pushed down into and subsequently trapped below the groundwater table. We see that the pore saturation (in that macropore fracture anyway) reaches near 100%, but the fluorescence is only 20% RE, which is much less than the 80% RE fluorescence the LIF system logged when the probe window was in only 20% pore saturation diesel in layer 1. That's because fines, especially clays, "hide" the NAPL from the laser/optics, causing up to a factor of 10 reduction of fluorescence response in clays than in sands/gravels. Many sites exhibit a response only in the sands/gravels because that's where the NAPLs can travel.

Notice that while the diesel response is lower than in level 1, the greenish color is the same, suggesting the diesel waveform (not shown, but it would be the same regardless of soil type). It's worth noting here that trapped fuels/oils such as layer 3 are actually common, especially in fine-grained, low permeability soils like clays with fractures and glacial tills. In many cases LIF has discovered entire NAPL plumes 5-10 feet below the current and/or historical groundwater table lows. Remember that NAPL is where you find it, and relatively few "sandbox" sites exist where the shark's fin saturation curve can form in a homogenous soil column. One of LIF's gifts to the industry is the eye-opening data it has generated showing that many NAPL CSMs based on boring log samples and wells are simply wrong.

Next the LIF probe passes into level 4 where saturated bunker fuel (DNAPL) sank down and penetrated laterally into a sand lens. The UV LIF being used here responds to the bunker, but barely, and not monotonically. Visible wavelength LIF should be used here (and for all coal tars and creosotes) if bunker is the main target of the investigation. This is because the visible LIF system responds monotonically to these "heavies" by design. Something called energy transfer quenches the fluorescence when UV is used, so the response is simply MUCH lower for heavies with UV fluorescence than one would expect. The very narrow lifetimes (skinny channels in the waveform) are direct evidence of the energy transfer quenching the LIF process.

Virtually all heavies show this right-sided waveform dominance, short lifetimes, and orange-red color-fill on the LIF log. But energy transfer and heavies are another advanced ANSR topic to address in a later edition. In any event, the response is unexpectedly low, even though the bunker is 100% pore saturated and it contains substantially higher PAH content than the other fuels. When you see this waveform and color-fill you should realize that you <u>might</u> have much more NAPL down there than the small %RE suggests.

Finally we pass into level 5, a layer of shell hash. Notice that the LIF log contains a "bump" in the fluorescence at this depth, even though the shell hash is clean of NAPL. That's because sea shells, calcareous sands, peats, wood, and other atypical organic soils can and do fluoresce (typically < 20% RE). Notice the fill-color is an odd pink that doesn't match any of the other fill-colors on the log.

Had we highlighted one of the shell hash waveforms with a callout we also would have seen the waveform itself was "different" from the other waveforms, signaling to us that this material is not

similar to the other NAPLs we encountered and that it may be a false positive material we pushed through at that depth. It is often the case that these false positives, while they make LIF interpretation less straightforward, can be identified as such and are later confirmed with a sample or two in depths identified precisely in the LIF log.

SUMMARY:

So let's review some key elements of LIF screening tools:

- LIF detects the PAHs in NAPL and this is how it logs for NAPL vs depth
- LIF is compatible with both cone penetration test (CPT) and percussion based direct push drilling technologies
- LIF is a mature technology, with hundreds of miles of LIF logging at hundreds of sites in the last 20 years
- LIF is logged continuously with depth (2cm/second) - no data gaps or partial recovery
- Typical LIF production is 300-400 ft/day - spread across 8-14 locations
- Experience has taught us there is almost never pulldown or sloughing of NAPL on the probe except when probing through the very softest of soils (pudding)
- Waveforms (and color-fill logs derived from waveforms) help us differentiate between NAPL types and differentiate NAPLs from false positives
- LIF detects NAPL equally well in both the vadose and saturated zones
- LIF's detection limit ranges between 10 and 1000 mg/kg (TPH), depending on fuel type and sol matrix
- LIF does not respond to dissolved phase VOCs or SVOCs
- LIF does not detect BTEX (excitation wavelength for BTEX is incompatible with fiber optics)
- Soil matrix affects fluorescence - sands/gravels have ~10x higher response than clay/silt
- LIF's potential false positives include shell hash, meadow mat, peat, wood, and calcareous sands, but waveforms usually identify these as suspect
- LIF does not detect chlorinated solvent DNAPL because they aren't fluorescent molecules – exception is chlorinated DNAPL that contains enough fluorophores (degreasing, industrial waste, etc.) - which is about 25% of DNAPLs tested recently
- UV LIF should NEVER be used to delineate coal tars and creosotes, use visible LIF instead – some of these materials don't fluoresce at all or have non-monotonic responses
- Tracer dye injections can and have been delineated with LIF

While it does have limitations that need to be understood and managed, LIF is a very rapid and thorough method of determining nature and extent of a petroleum site's source term NAPL. LIF surveys almost always reveal that previous NAPL CSMs based on standard sampling or monitoring well apparent NAPL thicknesses are deficient, if not grossly flawed, in comparison.

REFERENCES:

1. Berlman, Handbook of Fluorescence Spectra of Aromatic Molecules, Second Edition, ACADEMIC PRESS, 1971
2. ITRC's Internet-based Training Program, LNAPL Training Part 2: LNAPL Characterization and Recoverability – Improved Analysis, slides 29, 49. http://www.cluin.org/conf/itrc/LNAPLcr/prez/ITRC_LNAPL_Part2_052711ibtbw.pdf

Standard Parts

The following figure provides the standard parts for the UVOST. Prior to using the UVOST, these parts or satisfactory substitutions should be on hand or readily available when needed.

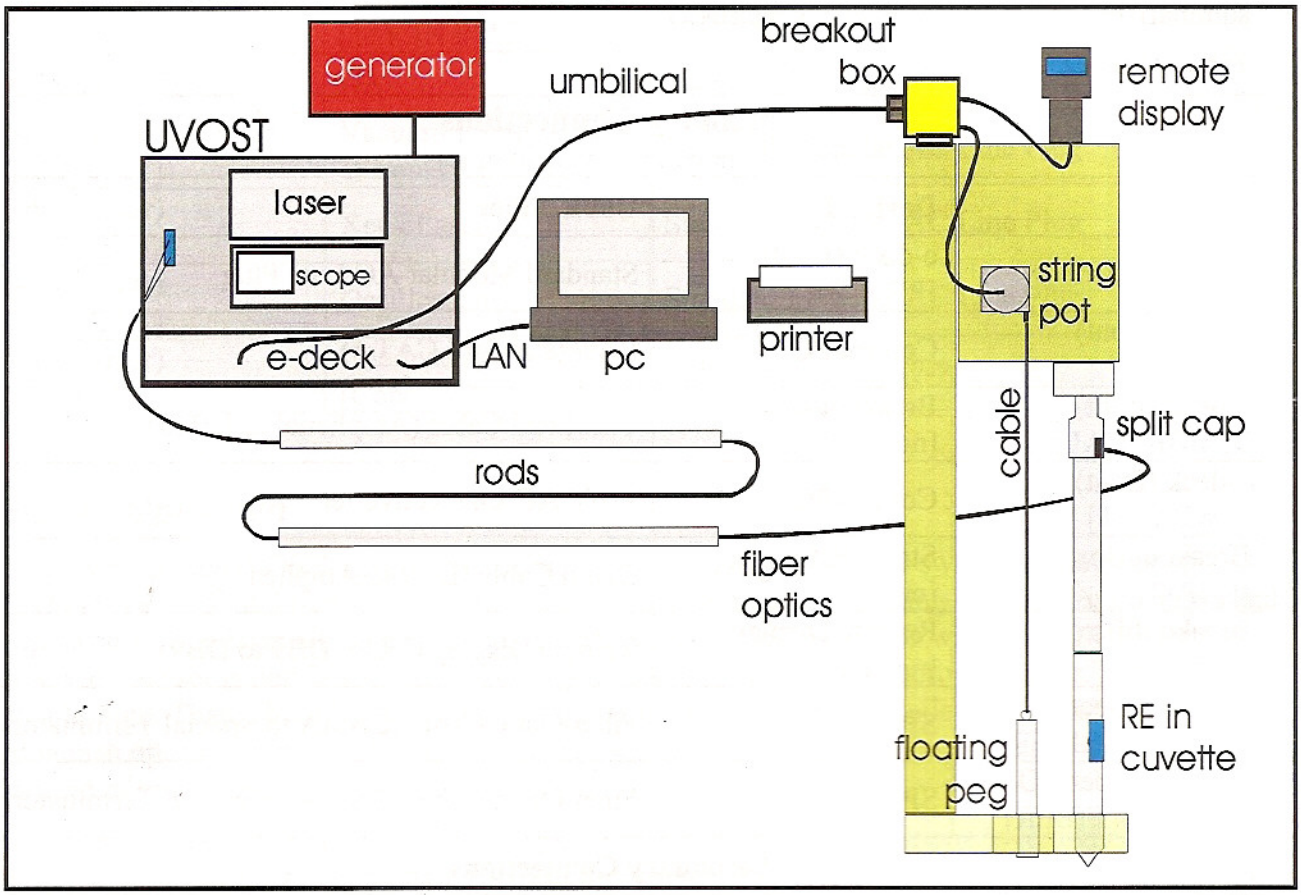


Figure 1. Standard UVOST parts.

General Operating Procedures

Set Up

Prior to operation, all the sub-systems require proper connections/cabling and power. Table 2 summarizes the proper connections/cabling.

Table 2. Cabling/Connections

Primary Connections [Connection labels in blue]		
Device 1	**Device 2**	**Cable/Fiber**
Power/Generator	e-deck (front) [PWR IN]	Standard Modular AC Line Plug
e-deck (front) [NET]	Control PC	LAN (standard CAT 5)
e-deck (front) [UMBILICAL]	Breakout Box [no label]	Umbilical Cable (Amphenol to DB15)
e-deck (front) [PWR OUT]	Control PC	110V AC Line converter
Breakout Box [DEPTH]	String Pot [no label]	Depth Cable (DB9 to Amphenol)
Breakout Box [DISLAY]	Remote Display [no label]	Remote Display Cable (DB9 to DB9)
UVOST Fiber I/O [LAUNCH FIBER]	SPOC	Fiber Optic Cable (2 SMA to Special Terminator)
UVOST Fiber I/O [RETURN FIBER]	SPOC	Fiber Optic Cable (2 SMA to Special Terminator)
Secondary Connections		
e-deck (front) [GPS]	GPS NMEA Output	DB9 RS-232 (Serial – usually integral to GPS)
e-deck (front) [AUX COM]	AD4 Quadrature	DB9 RS-232 (Serial)
e-deck (front) [12V AUX]	Generic 12V Accessory	Power Plug 0.1" (Switchcraft 761K)
Breakout Box [AUX]	NA (future use)	DB15 to (yet defined)

Permanent Connections		
Device 1	**Device 2**	**Cable/Fiber**
UVOST Fiber I/O (lower backside)	Detection Module (FIBER RETURN)	Single Fiber Optic (SMA-SMA)
UVOST Fiber I/O (upper backside)	Laser Launch Optics	Single Fiber Optic (High Power SMA-SMA) (standard fiber can be used as backup)
Trigger Photodiode	Oscilloscope [Ch1]	Coaxial Cable (SMA-BNC)
Emission Module [SIGNAL OUT]	Oscilloscope [Ch2]	Shielded Coax (PMT-BNC)

Trigger Photodiode	XeCl Laser (vessel)	Single Fiber Optic (SMA-SMA)
UVOST e-deck (rear) [NET SCP]	Oscilloscope	LAN (CrossOver CAT 5)
UVOST e-deck (rear) [12V PMT]	Detection Module [12V IN PMT]	12V supply (SMA-SMA)
AC Line (external)	NA	Standard Modular AC Line Plug
e-deck (rear left-most) [PWR OUT]	Vacuum Pump (switch at front of e-deck)	Standard Modular AC Line Plug
e-deck (rear) [PWR OUT]	XeCl Laser	Standard Modular AC Line Plug
e-deck (rear) [PWR OUT]	Oscilloscope	Standard Modular AC Line Plug
e-deck (rear) [Cond.]	Conductivity Module [Cond. Out]	12V Signal Cable (?-?)

Power Up/Down

To power up the UVOST, simply switch the power on using the power switch on the front of UVOST's e-deck. All peripheral devices are powered through the cabling – minimizing tangles and trip hazards. The laser takes several minutes of warm-up and Wait LED will then light. Once warmed the user is notified by the Ready light. Push the On button to activate lasing. Lasing LED should light. There should be a small rectangular yellow glow on the yellow glass indicator at front of launch optics assembly. The oscilloscope should display Trig'd – if laser has sufficient output (not in need of recharge).

Set laser rep rate to between 63-65 Hz. If powering up from cold conditions (overnight, etc.), make sure you have laser running at least 10-15 minutes prior to attempting your first RE calibration. We recommend running heaters overnight if in sub-freezing conditions to minimize warm-up times in the morning. Extremely high or low temperatures negatively affect laser power. If used in extreme conditions one should attempt to house/store the UVOST system in a warmer/cooler environment to assure proper operation. There are no hard/fast rules for this – since case temperatures/heaters can assist but a lot depends on winds, ventilation, direct sun, etc.

To power down the UVOST, first Stop the laser pulsing, then switch off the power button.

Boot PC and Check Software Function

Make sure all drivers are loaded and ready. Start the OST system software. Indicators in the software will assist in alerting you to problem connections and general status of the components (Hardware Tab). See software manual for specifics on OST software.

Proper System Function

Once the software is started and functional you can proceed to check the depth encoding and associated peripheral functions. Actuating the probe (or hand advancing the string pot) should show Current Depth changing on the OST software (Depth tab). The Remote Display should be functional and show status. Activate Info tab and make sure your job information is updated for storage with each LIF log.

SPOC Setup

A detailed discussion is available under SPOC Assembly heading. Carefully examine mirror and window for ANY trace grease, lint, and moisture. They must be very clean. Assure that all o-rings, seals, and adapters are in correct order – including Teflon tape, and associated hardware. With SPOC tip left off of SPOC, dry the air inside the SPOC, and quickly screw in window. You can check for moisture condensing inside window using an ice cube. If there is condensation you must dry the SPOC air better. Slightly tighten the mirror and fiber optic Swagelock seals (just snug). Adjust fiber terminator up/down to achieve proper distance from mirror to collimate the laser beam (use white paper – you may have to "up" energy for this).

Place RE in front of window and adjust laser energy (Fiber I/O block screw) to achieve approximately ¾ scale with oscilloscope's CH2 on 50 mV/div. Adjust the mirror (using window pick/hook) to image only the sapphire window – not epoxy or SPOC barrel (no clipping – full circle image on paper). This occurs approximately 1/3 of the way down from top of window.

Clean/polish window and then make sure that background does not exceed ~2.5mV peak signals. If background is high, carefully inspect for imaging of sides/epoxy or contamination (lint, cotton fibers, fuel, moisture, grease, etc.) An unacceptably high background can make interpretation extremely difficult.

Once you're certain the mirror/fiber/window system is achieving proper results you can tighten the Swagelocks securely. Use ONLY the supplied wrenches to hold the SPOC securely during tightening. This is most readily assured by laying SPOC down and only handling wrenches. Use the mirror pick/hook to hold the mirror firmly in place during tightening to prevent rotation. Make sure laser beam stays in centered in the window (side to side) and 1/3 down from the top (toward first rod).

With window/mirror/fiber terminator all secured, proceed with attaching drive tip, adapter, extension rod, and tighten extremely well with 2 pipe wrenches or pipe wrench and vice. Teflon tape helps reduce loosening from rattling/vibration.

Background

Wipe window clean and acquire a Background (blank) waveform with the Acq BckG command. A perfect system would yield no waveform at all – only white noise. But there is always trace fluorescence from mirror/window, fiber-generated Raman, and contamination. Try to achieve <2mV peak signal in any one channel. You simply want it as small as you can get it. A severely jagged/noisy background indicates possible pickup of the large laser EMF (Electric and Magnetic Fields) into the trigger and signal coax cables. Loose grounds, connections, misrouting of cables, etc. can induce this. If the first channel (350 nm) is considerably large than the other three, there is a chance that you have excessive backscatter of laser light into the system (350nm filter is near laser wavelength) or the laser rejection filter (inside I/O block) may be damaged or malfunctioning. Channels 3 and 4 being high/narrow is a classic lint signature. A background waveform that looks like your current contaminant of interest suggests leakage and contamination of the internal SPOC mirror/window OR simply a dirty window. Clean with methanol or solvent if soap/water doesn't work.

RE Calibration

Calibration should be done as immediately preceding each UVOST logging event. Don't calibrate with RE, then spend time monkeying around with push rig, etc. Wait until the direct push rig is ready to go. Pre-push with dummy tip if obstructions are likely or getting a "straight hole going" is

difficult. Place RE holder on window (making sure window is very clean). Immediately acquire RE with Acq RE command. Extended exposure to laser light can form excimers and photodegradation – causing a morph in waveform shape/intensity. If you have changed fiber optic lengths the software may correct the delay time to achieve proper position in window. Make sure the RE signal level exceeds a 10, 000 pVs minimum but does not exceed 20,000 pVs with 14,000-15,000 pVs about optimum. Try to be consistent (± 1000 pVs) – especially when on the same project/site. Make sure the RE waveform shape "looks right". Compare it to the reference waveform displayed on the scope during the RE acquisition. Extremely noisy/jagged REs, misshapen REs, and missing/low channel contributions indicate damaged or loose fiber optics/filters/detector.

Logging

Follow these steps to acquire a UVOST log:

Step 1. With proper RE and background acquired, pertinent log information recorded, and probe in position (window just below (~1 inch) ground surface), activate the Record command.

Step 2. If you failed to acquire a recent RE the OST software will alert you that it's not recent (at least one log event old). Proceed with you recent (perhaps you just aborted a "false start"/crooked log) – or cancel out and acquire the RE you forgot to acquire. You can "rescue" an RE if it's for a rational purpose (such as an accidentally aborted log and you want to continue logging and probe is under ground, under water during a barge project, etc.) DO NOT purposefully continue logging without a new RE for each and every log if you're having problems acquiring a new RE due to a problem. FIX the problem, acquire a good RE, then proceed. Failure to acquire a new RE for each log will generate inaccurate data.

Step 3. Choose a directory and name for your log. UVOST auto-suggests the name sequentially in an attempt to reduce typing. In order to absolutely avoid accidental overwrite of any OST file, the OST software creates a unique time/date name and uses that name in place of overwrites (even though you said "OK" to the overwrite. If you want to risk it, you can always delete a file from the Save File dialog after you click on it once, but before hitting OK. That prevents the Windows software from reporting an overwrite to the OST and cueing the unique filename routine. The safest method is to choose OK to overwrite – and rename files later.

Step 4. Once the name is chosen you are asked to choose whether or not to "zero" the depth. For normal logs you always choose Yes and zero out depth. If you're continuing an aborted log that you want to continue (accidental termination) – choose No. Log should continue at depth where you left off.

Step 5. As the log progresses, it is your responsibility to make sure the system is operating properly. Observe the oscilloscope or OST display to watch for unusual events such as:

A. Try to keep the probe advancing at approximately 0.75 inch/sec – your company may choose less – but we do not recommend faster

B. Strange background drifts several feet under (possible fogging), etc.

C. Broken depth cable or poor connection will result in jumps in depth or a loss of depth increase – even though the operator is advancing the probe

D. Incorrect depths would indicate a possible rod length or string pot cal factor mismatch

E. Sudden loss of waveform (flatline) indicates possible fiber optic break due to broken probe

F. depth is advancing – but no new waveform updates aren't showing up – this indicates poor triggering – is Trig'd showing up on oscilloscope every second or so? if not – hit Trigger

50% button on scope or look for other cause such as Stop button on laser being accidentally pushed.

Step 6. Once refusal is reached – or target depth is reached – activate the End command. All pertinent data is stored and the oscilloscope scale is automatically returned to the default 50mV/div scale in preparation for next RE.

Step 7. Inspect the probe, window, etc. for leaks, breaks, and loose parts in preparation for next the next logging event (push).

Printing/Exporting LIF Logs

Once the push is complete the log can be viewed (a log can be also opened from file and viewed with the OST software) it is necessary to print the log to paper or export it to an electronic image (JPG file). Prior to print/export it is most often desirable to select callout waveforms. Select single waveforms by clicking the log at any depth – which creates a stats bar. Transfer single logs by dragging/dropping the stats bar or with the < bar next to each callout box. Select the average of a region of waveforms along a log by clicking the log, holding down, then releasing at a second depth along the log. Transfer average zone waveforms by dragging/dropping the bottom stats bar or with the < bar next to each callout box. Reasons to select certain depths/regions include:

- Bracketing what appear to be continually affected zones - this helps the client/consultant "summarize" the general NAPL zones and easily jot down depths for future validation sampling, project design, discussion with site owner, etc.
- It's best to bracket large zones of homogenous NAPL - do not span different products
- Highlighting unusual signatures – perhaps to suggest sampling there or to "flag" things the client needs to investigate or discount
- Maybe a background here/there to remind viewer what "clean" looks like
- Any potential "false positives" such as mineral/plant/urban background/highly degraded NAPL – the different waveform should help client understand that "it's nothing to worry about"
- Use caution when highlighting single waveforms from the rising edge of NAPL hits – the waveforms in these area are usually saturated because the oscilloscope scaling wasn't able to fully respond – they are morphed and ugly and cause unnecessary confusion and alarm
- You do not have to start with top and work down – pick a callout "straight across" for neater appearance
- Avoid "crossing" of the depths of multiple callouts as this looks messy/confusing

It is best that the UVOST operator and the client discuss depth/RE scales, depths of interest, etc. ahead of time to hopefully avoid lots of "reprints".

It is suggested that you annotate the callouts (text box under each waveform) in order to guide the client. If it's the usual product you expect then leave it blank – but if it's unusual, significant, or out of the ordinary, guide the viewer with a brief description.

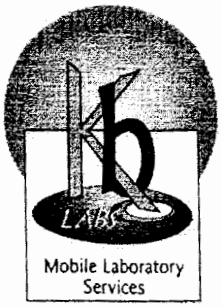
Each time you print/export the settings are saved in a lif.plt (plot) file. That way the same callouts and depths are available later. The OST software (and we) suggest that the very first print/export a log in the filed you save it as field. That way you always know what the client received originally. Subsequent print schemes are saved as well. Later, upon opening, you can choose which of the various schemes to open the file with.

APPENDIX C

LABORATORY SOPs

- C1: KB Labs, Inc. SOPs
- C2: TestAmerica Nashville SOPs
- C3: TestAmerica Burlington SOPs
- C4: Beaver Engineering, Inc. SOPs

C1: KB Labs, Inc. SOPs



ANALYTICAL STANDARD OPERATING PROCEDURE KB-INORG-001

Percent Solids

APPROVALS

Robert E. George
Quality Assurance Officer — Date: 1/29/14

Bradley A. Weichert
Technical Director — Date: 1/28/14

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

Copy No. _____ distributed on _____ by _____ and is ____CONTROLLED or _X_ UNCONTROLLED

PERCENT SOLIDS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of moisture present in a client sample for the purpose of dry weight correction. The method is based on ASTM D2216-10.

2.0 SUMMARY OF METHOD

2.1 A sample is dried at 110 ± 5°C. The change in weight over time is used to calculate % solids for use in dry weight correction of samples.

3.0 DEFINITIONS

3.1 A list of terms and definitions are provided in Appendix A of the KB Labs Quality Assurance Manual.

4.0 INTERFERENCES

4.1 Not applicable to this SOP.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures described in KB Labs' *Health and Safety Manual* and this document. This procedure may involve the handling of hazardous materials, operations and equipment. This document is not designed to address all of the safety problems associated with its use. All samples and reagents shall be handled under the assumption that they are potentially hazardous.

5.2 Eye protection and gloves must be worn while performing the analyses. Samples shall be handled in a well ventilated area. The laboratory area has an eyewash kit, and fire extinguisher.

5.3 A reference file of material safety data sheets (MSDSs) is available to all personnel.

6.0 EQUIPMENT AND SUPPLIES

6.1 Top-loading balance: OHAUS Scout Pro SP202, capable of weighing 0.01 grams.

6.2 Aluminum weighing dishes

6.3 Oven capable of maintaining temperature 110 ± 5°C

6.4 Desiccators

7.0 REAGENTS AND STANDARDS

7.1 Not applicable to this SOP

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Samples should be collected in glass containers and stored on ice prior to analysis.

9.0 ANALYTICAL PROCEDURE

9.1 Dry Weight procedure for non-DOD clients.

9.1.1 Tare a clean weigh dish and record the dish weight on % solids logbook.

9.1.2 Place approximately 5 to 10g of sample in the weighing dish. Record the initial weight of the wet sample plus dish on the % solids logbook.

9.1.3 Place the samples in the drying oven for a minimum of 1 hour preferably 2 hours.

9.1.4 Remove the samples from the oven and allow to cool in a desiccators for at least 15 minutes.

9.1.5 Weigh the dry sample plus dish and record the final weight on the % solids logbook.

9.1.6 Calculate % solids.

9.2 Dry Weight procedure for DOD clients

9.2.1 Tare a clean weigh dish and record the dish weight on the % solids logbook.

9.2.2 Place approximately 5 to 10g of sample in the weighing dish. Record the initial weight of the wet sample plus dish on the % solids logbook.

9.2.3 Place the sample in the drying oven for a minimum of 2 hours.

9.2.4 Remove the sample from the oven and allow to cool in a desiccators for at least 15 minutes.

9.2.5 Weigh the dry sample plus dish and record the final weight on the % solids logbook.

9.2.6 Return the dish to the oven for an additional hour.

9.2.7 Remove the sample from the oven and allow to cool in a desiccators for at least 15 minutes.

9.2.8 Weigh the dry sample plus dish and record the final weight on the % solids logbook. The weight need to agree within 0.02g of the initial value.

9.2.9 If the weights do not agree, return the dish to the oven for an additional hour and repeat until the weights agree within 0.02g.

9.2.10 Calculate % solids.

10.0 DATA ANALYSIS AND CALCULATIONS

Calculate % solids as follows:

% Solids = ((C-A)/(B-A)) x 100

Where: A = dish weight
B = Wet sample plus dish weight
C = Dry sample plus dish weight

11.0 QUALITY CONTROL

11.1 Not applicable to this SOP.

12.0 DATA ASSESSMENT, QC CRITERIA, AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

12.1 Not applicable to this SOP.

13.0 EQUIPMENT MAINTENANCE AND TROUBLE SHOOTING

13.1 The lab maintains an instrument maintenance logbook. All maintenance performed on the instruments must be recorded. Additionally, any modifications to the instrument settings shall be noted in the specific instrument logbook. Maintenance procedures provided in the equipment manual shall be followed.

14.0 METHOD PERFORMANCE

14.1 Not applicable to this SOP.

15.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

15.1 All laboratory waste must be managed, stored, and disposed in accordance with all federal and state laws and regulations. Additional information can be found in KB Labs' *Standard Operating Procedure KB-WASTE-001* and KB Labs' *Health and Safety Manual.*

16.0 REFERENCES

16.1 KB Labs' Quality Assurance Manual, current revision.

16.2 KB Labs' Health and Safety Manual, current revision.

16.3 KB-QA-007, *Sample Receipt and Acceptance*, July 2013, Revision 2.

16.4 ASTM D2216-10, *Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*

17.0 TABLES ATTACHMENTS APPENDICES, etc.

17.1 Attachment 1 - % Solids Logbook

18.0 REVISION HISTORY

Revision No.	Revision Summary	Revision Date
0	Initial SOP	October 2013
1	Fixed Calculations	November 2013
2	Clarified constant weight procedure	January 2014

ATTACHMENT 1

KB LABS, INC.
% Solids Logbook

Mobile Unit: ________
Date: ______________ **Project Name:** ____________________
Initials: ____________

Balance Check (3 weights)

Target (g)	1.00g	50.00g	200.00g
Acceptable Range (g)	0.98-1.02	49.00-51.00	196.00-204.00
Actual (g)			

Sample ID	Dish Weight (grams)	Dish+Wet (grams)	Dish+Dry (grams)	% Solids	In	Out	2nd Dish+Dry (grams)	% Solids	In	Out

KB Labs, Inc.

ANALYTICAL STANDARD OPERATING PROCEDURE No. 1

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY PURGE & TRAP GAS CHROMATOGRAPHY/MASS SPECTROMETRY – METHOD 8260B

Signature of Approving Authority: _____

Michael G. Winslow
Quality Assurance Officer

Effective Date: June 2010

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY PURGE & TRAP GAS CHROMATOGRAPHY/MASS SPECTROMETRY – METHOD 8260B

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water and soil samples by purge and trap/gas chromatography/mass spectrometry.

1.2 The following compounds may be determined by this method:

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethyhlbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Chlorotoluene
4-Chlorotoluene
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
c-1,2-Dichloroethene
c-1,3-Dichloropropene
Carbon tetrachloride
Chlorobenzene
Chloroethane
Chloroform
Chloromethane
Dibromochloromethane
Dibromomethane
Dichorodifluoromethane
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
m&p-Xylene
Methylene chloride
MtBE
Naphthalene
n-Butylbenzene
n-Propylbenzene
o-Xylene
p-Isopropyltoluene
sec-Butylbenzene
Styrene
t-1,2-Dichloroethene
t-1,3-Dichloropropene
tert-Butylbenzene
Tetrachloroethene
Toluene
Trichloroethene
Trichlorofluoromethane
Vinyl chloride

2.0 SUMMARY OF METHOD

2.1 The VOCs are introduced into the gas chromatograph by the purge-and-trap technique as described in EPA SW 846 Method 5030B for waters and EPA SW846 Method 5035 for soils. Samples are purged with helium and the volatile components are collected on a solid-phase adsorption trap.

2.2 After purging is complete, the adsorption trap is heated and back-purged with helium to desorb the trapped components into a gas chromatograph for separation on a narrow bore

capillary column. Components eluted from the capillary column are introduced directly into a mass spectrometer for qualitative and quantitative determination based on EPA SW846 Method 8260B.

2.3 The individual volatile components are measured against appropriate standards. Identification of the target compounds is accomplished by comparing their mass spectra with the electron impact mass spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a five-point calibration curve.

2.4 The estimated quantitation limits (EQL) or reporting limits established by KB Labs for this method are 1 ug/L for low-level water samples and 2 - 10 ug/kg wet-weight for soil samples. The actual limits are dependent upon individual compound purging efficiency, the amount of sample used.

3.0 DEFINITIONS

Refer to Sec 5.0, Chapter 1, *Test Methods for Evaluating Solid Wastes, Fourth Edition, SW-846*.

4.0 INTERFERENCES

4.1 The analyst must be careful not to introduce major sources of VOC contamination into the laboratory. These sources include organic extraction solvents, impurities in the purging gas and sorbent trap, and the use of non-PTFE sealants, plastic tubing, or flow controllers with rubber components. A method or reagent blank should be analyzed to determine whether contaminants are present. Subtracting blank values from sample results is not permitted.

4.2 Contamination can occur when a sample containing low concentrations of VOCs is analyzed immediately after one containing high concentrations of VOCs. The analyst should rinse the sample transfer syringe with two portions of reagent water after each sample transfer into the autosampler purging chambers. If time allows, reagent water blanks can be placed between samples in the autosampler device.

4.3 To reduce the chances of sample and system contamination, all samples are screened prior to analysis by GC/MS. Screening is performed by analyzing sample headspace using GC/FID.

5.0 SAFETY

Refer to procedures described in KB Labs' *Health and Safety Manual*.

6.0 EQUIPMENT AND SUPPLIES

6.1 Purge-and-trap concentrator: Tekmar Model LSC 3000

6.2 Purge-and-trap autosampler: Varian Arcon

6.3 Gas chromatograph/mass spectrometer (GC/MS) system: Hewlett-Packard (HP) 6890A GC/HP5973A MS / Chem Station

6.4 Gas chromatograph/flame ionization dectector: HP 5890A with a HP 3396 integrator.

6.5 GC column for GC/MS: Rtx – 624, 20 m x 0.18mm, 1.0mm film thickness

6.6 Syringes: 10 uL, 100 uL, 1 mL (gas tight), and 10 mL,

6.7 Volumetric flasks: 10 mL, 100 mL

6.8 Glass vials: 40 mL with PTFE-lined septum screw caps.

6.9 PTFE-lined screw cap vials: 2 mL

6.10 Disposable pipets: 1 mL Pasteur.

6.11 Top-loading balance: Ohaus SC2020, capable of weighing 0.01 grams.

7.0 REAGENTS AND STANDARDS

7.1 Methanol, purge and trap grade.

7.2 Reagent water: VOC free (determined from method blank analysis)

7.3 Stock Calibration Standard Solutions

7.3.1 Volatiles (54 components): 200 ug/mL in methanol, purchased from Accustandard.

7.3.2 Gases (6 components): 200 ug/mL in methanol, purchased from Accustandard.

7.4 Stock Calibration Verification Standard Solutions (second source)

7.4.1 Volatiles (54 components): 200 ug/mL in methanol, purchased from Restek.

7.4.2 Gases (6 components): 200 ug/mL in methanol, purchased from Restek

7.5 Stock Internal Standard and Surrogate Solutions

7.5.1 7 Components: 2000 ug/mL, purchased from Accustandard.

7.6 Preparation of Calibration Standards

7.6.1 A *calibration standard spiking solution* is first prepared by diluting 0.5 mL of each 200 ug/mL stock calibration standard (Accustandard volatile and gas) to 10 mL with methanol. This working stock has a concentration of 10 ug/mL.

7.6.2 *Calibration standards in reagent water* are then prepared from the working stock solution according to the dilution scheme outlined below:

Volume of Calilbration Standard Spiking Solution Added (uL)	Volume of Reagent Water (mL)	Concentration of Calibration Standard (ug/L)
0.5	5	1
2.5	5	5
5	5	10
10	5	20
25	5	50
50	5	100

7.7 Preparation of *Calibration Verification Standard*

7.7.1 A *calibration verification standard spiking solution* is prepared by diluting 0.5 mL of each stock calibration standard (Restek volatile and gas) to a 10 mL volumetric flask and bringing to volume with methanol. This *calibration verification standard spiking solution* has a concentration of 10 ug/mL.

7.7.2 A *calibration verification standard in water* is prepared by adding either 10 or 25 uL of the calibration verification standard spiking solution to 5 mL of reagent water contained in a 10-mL gas-tight syringe. The concentration of the *calibration verification standard* is 20 ug/L or 50 ug/L.

7.8 Preparation of *Internal Standards* and *Surrogate Standards*

7.8.1 An intermediate *internal standard/surrogate stock solution* is prepared by diluting 1 mL of the 2000 ug/mL stock standard into 10 mL of methanol. This intermediate stock solution has a concentration of 200 ug/mL.

7.8.2 A *internal standard/surrogate spiking solution* is prepared by adding 1 mL of the intermediate internal standard/surrogate stock solution to 10 mL of methanol. This spiking solution has a concentration of 20 ug/mL.

7.8.3 5 uL of the internal standard/surrogate spiking solution is added to all 5 mL water standards and samples prior to analysis. This will give a 20 ug/L concentration for internal standard and surrogate compounds.

7.9 Storage of Standards

7.9.1 All standards will be stored in a freezer @ -5 ^{o}C or less.

7.9.2 All unopened stock standards in methanol have an expiration day assigned by the manufacturer.

7.9.3 All standards prepared in methanol will be stored in 2 mL-PTFE-lined screw cap vials without headspace in the vial. Standards with partial headspace in the vial will not be retained.

7.9.4 VOC stock standards in methanol with permanent gases that are stored long term in the office VOC freezer expire one week after opening unless acceptability of the standard can be documented.

7.9.5 VOC standards in methanol with non-gaseous compounds that are stored long-term in the office VOC freezer expire 6 months after opening unless acceptability of the standard can be documented.

7.9.6 Secondary standards in methanol have a one-week holding time.

7.9.7 Vials expire 7 days from when the septum is punctured.

7.9.8 All standards will be stored separately from samples

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection

8.1.1 Sample containers are purchased pre-cleaned and with a Certificate of Quality Assurance/Analysis from an approved vendor and will be supplied to the client by KB Labs prior to sampling.

8.1.2 Collection of Water Samples

8.1.2.1 Each water sample is collected in two 40-mL glass vials with open-top screw caps fitted with PTFE-lined septa. The duplicate vial is potentially used for MS/MSD analysis. Once the sample is collected, the septum must be placed with the PTFE side towards the water sample and the open-top cap tightened finger tight.

8.1.2.2 Water samples must be collected without headspace (no bubbles).

8.1.3 Collection of Soil Samples

8.1.3.1 KB Labs provides the sampling team with sampling kits, each of which contains two (2) sealed pre-weighed 40-ml vials containing 10 ml of reagent water, sample preservative, and a stir bar; a 40-ml vialcontaining 10 mL of Methanol; an empty 2-ox soil jar, and a Terra Core sampling toolfor use as a sample coring device. Sample vials are pre-weighed in the laboratory prior to supplying them to the sampling team using a balance that reads to 0.01 grams. They are then reweighed with the sample after receipt in the laboratory. Sampling instructions will be supplied with the kits.

8.1.3.2 Approximately five grams of sample is collected in each of the two pre-weighed 40-mL glass vials containing the 10 mL of reagent water, sodium bisulfate preservative, and a stir bar. Another five gram sample is placed in the 40-mL vial containing 10 mL of Methanol. Once a sample is placed in a vial, the septum must be placed with the PTFE side towards the soil sample and the open-top cap tightened finger tight.

8.1.3.3 In order to accommodate for the determination of percent moisture and headspace screening, the 2 o-oz soil jar should be filled to the top with the soil sample.

8.1.4 The label on each sample container must be have a distinguishing field identification for each sample and be accompanied by a properly completed chain-of-custody form. The weight of each prepared vial should be recorded on the label to the nearest .01 grams. Refer to KB Labs' Standard Operating Procedure (SOP) No. 007 for a description of sample receipt and acceptance procedures prior to sample analysis.

8.2 Sample Preservation and Storage

8.2.1 Even though samples are generally received and analyzed in the mobile lab soon after collection by the client, they must still be received on ice. Samples will be preserved with HCl to pH<2, which will be added to sample vials prior to going to the field.

8.2.2 If a sample is not processed immediately after receipt, it will be stored in an iced cooler at 4 $\pm$ 2 ^{o}C until ready for processing. The sample should be allowed to come to room temperature before processing for analysis.

8.2.3 Because samples are generally analyzed in the mobile lab the same day as receipt, holding times should not be an issue. The regulatory holding times for water

samples refrigerated at 4 $\pm$ 2 ^{o}C is 7 days and for soil samples 14 days. Soils samples collected in water, if not analyzed within 48 hrs, must be frozen – after freezing they have a 14-day holding time from the time of collection. Samples preserved with sodium bisulfate in the field have a 14-day holding time.

9.0 QUALITY CONTROL

9.1 Initial Demonstration of Capability (IDOC)

9.1.1 A new analyst will perform an IDOC prior to using any test method for the analysis of client samples.

9.1.2 An IDOC will also be performed whenever a new instrument or method is implemented.

9.1.3 The IDOC will be performed in a clean and applicable matrix.

9.1.5 Four replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest at 10 – 50 times the method detection limits for the analytes.

9.1.6 The concentrations for each analyte are then experimentally determined using the standard operating procedures for the analytical method.

9.1.7 The mean recovery and standard deviation of the found concentrations for the replicates is then calculated for each analyte, and these are then compared to the corresponding acceptance criteria for accuracy and precision established by the lab from historical data. Acceptance criteria established by the lab may not exceed 70 – 130%.

9.2 Quality control procedures for the operation of the GC/MS include:

9.2.1 The GC/MS system must be *tuned* to meet specified BFB criteria described in Section 10.1.

9.2.2 *Initial calibration* of the GC/MS system must be performed as described in Section 10.2.

9.2.3 *Calibration verification* procedures must be performed every 12 hours of instrument operation as described in Section 10.3 and the CCC, SPCC, and IS criteria must be met.

9.2.4 A *laboratory reagent blank (method blank)* must be analyzed in order to monitor the cleanliness of the analytical system. The method blank must be analyzed after the calibration standard(s) and before the samples and the results must

demonstrate that the analytical system contains less than 20% of the reporting level for all target compounds and is free of any contaminates that might interfere with the analysis of the target compounds. Method blanks may be analyzed at a higher frequency if deemed necessary by the operator.

9.2.5 All samples, including standards and method blanks, must be fortified with *surrogate* and *internal standards***.** The percent recovery of each surrogate compound is calculated in order to evaluate the performance of the analytical system and to help determine the potential for sample matrix effects. Surrogate compounds include 1,2-dichloroethane-d4, 1,4-difluorobenzene, toluene-d8, and 4-bromofluorobenzene. Surrogate control limits are set at $\pm$ 3 standard deviations of the laboratory average historical recoveries. Quantitation is performed with the internal standards which include pentafluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4.

9.2.6 Duplicate *matrix spike (MS/MSD)* samples must be analyzed for every 20 samples analyzed and for any daily sample batch that is less than 20 samples in order to monitor the performance (precision and accuracy) of the target compounds in the actual matrix. The accuracy (% Recovery) and precision (%RPD) is calculated for every pair of spikes and a statistical analysis is performed on at least the last 20 samples analyzed in order to calculate and update control limits. The matrix spike samples are prepared by spiking 5-mL aliquots of a selected water sample (or 5 gram soil sample) with the appropriate uL amounts of the matrix spiking standard, which are prepared from different stock standards than the calibration standards.

9.2.7 A single *laboratory control sample (LCS)* must be analyzed for every 20 samples analyzed and for any daily sample batch that is less than 20 samples in order to monitor the recovery of the target compounds from a clean sample matrix. An accuracy (% Recovery) is calculated and a statistical analysis is performed on at least the last twenty samples analyzed in order to calculate and update control limits.

10.0 ANALYTICAL PROCEDURE

10.1 Calibration and Standardization

10.1.1 *Bromofluorobenzene (BFB) tuning* criteria must be met at the beginning of each day and every 12 hours thereafter as long as analyses are performed. The following tuning criteria from EPA Method 8260 must be met before any samples or standards are analyzed.

M/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95

75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9 % of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9 % of m/z 176

10.1.2 *Initial calibration* is performed when the instrument is started up when the instrument response has drifted out of calibration in order to demonstrate that the instrument is capable of acceptable performance at the beginning of the analytical run and is producing a linear calibration.

10.1.2.1 Five or six calibration standards @ 1,5,10,20,50, and 100 ug/L are analyzed.

10.1.2.2 The percent relative standard deviation (%RSD) of each target analyte must be $\leq$ 15 %.

10.1.2.3 The system performance check compounds (SPCCs) must pass the following minimum mean response factor (RF) criteria:

Chloromethane	0.10
1.1-Dichloroedthane	0.10
Bromoform	0.10
Chorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

10.1.2.4 The following calibration check compounds (CCCs) must meet minimum RSD criteria of $\leq$ 30%

1,1-Dichloroethane	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropene	Vinyl chloride

10.1.3 A *calibration verification standard (CVS)* at least every 12 hours in order to verify initial calibration.

10.1.3.1 A 20 ug/L or 50 ug/L standard must be analyzed.

10.1.3.2 The SPCCs must pass the minimum mean RF criteria as in initial calibration.

10.1.3.3 The percent difference of the CCCs must be $\leq$ 20% of initial calibration, or if not included in the target compounds, all analytes must be $\leq$ 20@ of initial calibration.

10.1.3.4 The internal standard (IS) retention times must be < 30 secs from those in the midpoint standard of the most recent initial calibration.

10.1.3.5 The IS responses must be within –50% and +100% of those in the midpoint standard for the most recent initial calibration.

10.2 Sample Screening:

10.2.1 All samples (waters and soils) are screened by GC/FID before preparation for loading onto the GC/MS purge and trap system. The screening procedure prevents contamination from high-level samples from being introduced into the instrumentation. Dilution levels are determined from the sample screening, allowing for a more rapid concentration estimation and limiting reruns for dilutions.

10.2.2 Water samples: Approximately 1 mL of the sample is removed from the sample vial with a disposable pipette and placed into a 40-mL glass vial/PTFE-lined open-top screw cap. The cap is finger tightened with the PTFE-lined screw cap facing toward the sample. The vial is shaken for about 30 seconds. The sample from which 1 mL has been removed must be analyzed within 24 hrs.

10.2.3 Soil samples: Five grams of soil is removed from the 2-oz jar and placed in a 40-mL vial with 10-mL of water for headspace screening.

10.2.4 1 mL of headspace gas is removed from the headspace of the sample vial (waters and soils) with a gas-tight syringe and injected directly into the injection port of the GC/FID. The headspace response is recorded on an integrator.

10.2.5 The headspace FID response is compared to the headspace FID response of a 100 ug/L water standard or 100 ug/kg soil sample prepared in 1 mL of reagent water or 1 g of standard soil.

10.2.6 Sample dilutions:

Samples requiring dilution based on the headspace screening response will be diluted as follows:

10.2.6.1 Appropriate aliquots of the water samples will be added to reagent water to a total volume of 5 mL.

10.2.6.2 Appropriate aliquots of the methanol from the vial containing 5 grams of soil sample in 10 mL of methanol will be added to 5 mL reagent water.

10.3 Preparation of Water Samples for Purge and Trap

10.3.1 Remove the plunger from a 10-mL gas-tight syringe with an open/shut valve and close the value.

10.3.2 Open the sample vial and pour the sample into the to the 10-mL mark on the syringe.

10.3.3 Reinsert the plunger into the syringe, turn it upright, and bring the plunger end to the 5- mL mark.

10.3.4 With a 10-uL microsyringe, add 5-uL of the surrogate/internal standard spiking solution to the 5-mL sample in the syringe by inserting the microsyringe needle through the 10-mL syringe valve. Close the syringe valve. The concentration of the surrogates and internal standards will be 20 ug/L.

10.3.5 If the sample is a matrix spike, add 5 uL of the calibration standard spiking solution to the sample. The concentration of the spiked compounds will be 20 ug/L.

10.3.6 If the sample is a standard or laboratory control spike follow the above procedure using laboratory reagent water and the appropriate amount of the calibration standard or laboratory control sample spiking solutions.

10.3.7 Open a clean 40-mL vial and slowly inject the water sample into the vial.

10.4 Preparation of Soil Samples for Purge and Trap

10.4.1 Soil sample vials will not be opened by the analyst prior to analysis (with the exception of the jar designated for percent moisture determination).

10.4.2 With a 10 uL microsyringe, add 10 uL of the surrogate/internal standard spiking solution to the sample by inserting the microsyringe needle through the sample vial septum. The concentration of the surrogates and internal standards will be 20 ug/L in the in the soil sample.

10.4.3 If the sample is a matrix spike, add 10 uL of the calibration standard spiking solution to the sample by inserting the microsyringe needle through the sample vial septum. The concentration of the spiked compounds will be 20 ug/L in the soil sample

10.4.4 Soil samples are attached to the purge and trap retrofit apparatus for analysis.

10.5 Concentrator Operating Conditions:

10.5.1 Adsorbent trap: Supelco K (10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001)

10.5.2 Delivery pressure: ~ 30 psi Helium

10.5.3 Valve temp: 150°C

10.5.4 Transfer line temp: 150°C

10.5.5 Purge temp set point: 40°C

10.5.6 Purge program:

10.5.6.1 Purge: 11 minutes

10.5.6.2 Dry purge: 2 minutes

10.5.6.3 Desorb preheat : 245°C

10.5.6.4 Desorb: 250°C/2 min

10.5.6.5 Bake : 260°C/4 min, bake gas bypass: 120 sec

10.6 Autosampler Operating Conditions:

10.6.1 Valve temp: 95°C

10.6.2 Transfer line temp: 110°C

10.6.3 Sample Preheat tem: 40^{o}C for 1 minute

10.7 GC/MS Operating Conditions:

10.7.1 GC Operating Conditions For Full 8260 Compound List:

10.7.1.1 Column: Rtx-624, 20m x 0.18m, 1.0mm film

10.7.1.2 Injector: 4mm ID low volume glass insert

10.7.1.3 Injector temp: 225°C

10.7.1.4 Detector temp: 280°C

10.7.1.5 Oven temperature program:

10.7.1.5.1 Initial temp: 45°C, hold for 2 minutes

10.7.1.5.2 Ramp temp: 10°C/min

10.7.1.5.3 Final temp: 180°C, hold for 1.0 minutes

10.7.1.6 Column flow : constant flow, 5.2 psi @ 45°C

10.7.1.7 Split flow: 40:1

10.7.2 MS Operating Conditions:

10.7.2.1 Mass range: 35-250 amu

10.7.2.2 Scan time: 2 sec/scan

10.7.2.3 Source Temp: 280 ^{o}C

10.8 Analytical Run Sequence:

10.8.1 BFB Tuning

10.8.2 Method blank

10.8.3 Initial calibration

10.8.3.1 1 ug/L calibration standard

10.8.3.2 5 ug/L calibration standard

10.8.3.3 10 ug/L calibration standard

10.8.3.4 20 ug/L calibration standard

10.8.3.5 50 ug/L calibration standard (use for daily calibration)

10.8.3.6 100 ug/L calibration standard

10.8.4 Method blank

10.8.5 Laboratory control sample (second source standard)

10.8.6 Method blank

10.8.7 Samples (including MS and MSD)

10.8.8 Method blank

10.8.9 Calibration verification standard (every 12 hours)

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative analysis:

Qualitative identification of each target compound is based on retention time and comparison of the sample mass spectrum with the characteristic ions in the reference mass spectrum – the three ions of greatest intensity or any ions over 30% relative intensity. Compounds are identified as present in the sample when the following criteria are met:

11.1.1 The characteristic ions of a compounds is maximized in the same scan or within one scan of each other.

11.1.2 The retention time of the compound in the sample is with $\pm$ 6 seconds of the retention time of the compound in the calibration standard.

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

11.1.4 Structural isomers that produce similar mass spectra and are sufficiently resolved, should be identified as individual isomers.

11.2 Quantitative analysis

11.2.1 The quantitation of identified target analytes is based on the integrated abundance of the extracted ion current profile of the primary and secondary characteristic ion(s). These are listed in Table 5, p. 37-39, of Reference 14.1. The internal standard used for quantitation is the one nearest to the retention time of the analyte.

11.2.2 The average response factors from initial calibration are used to calculate the concentration of each compound in the sample using the following equation:

$$Cs = \frac{A_s \times C_{is} \times D}{A_{is} \times RRF \times V_s(M_s)}$$

A_s = peak area of analyte in sample
C_{is} = concentration of internal standard
D = dilution factor
A_{is} = peak area of internal standard
RRF = mean response factor
V_s (M_s) = volume of mass of sample

$$RRF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = peak area of analyte
A_{is} = peak area of internal standard
C_s = concentration of analyte
C_{is} = concentration of internal standard

12.0 DATA ASSESSMENT, QC CRITERIA, AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

12.1 Data is initially reviewed by the analyst for acceptability.

12.2 The table below lists the corrective actions that the analyst will follow if QC criteria are not initially met.

12.3 If the corrective action fail to correct the problem, the analyst must notify the client in the field and the KB Labs operations or QA officer for a decision on data usability.

Table 10.3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action(s)
Initial Demonstration of Capabililty (IDOC) – 4 replicate standard matrix spikes of all target analytes @ 10 - 50 times MDL	Prior to analysis of any samples	Historical lab acceptance limits, but not to exceed 70 – 130 percent.	Prepare and reanalyze new samples. Recalibrate, if necessary.
GC/MS Tuning – 4-bromofluorobenzene (BFB)	Prior to initial calibration and every 12 hours of analysis time	BFB ion abundance criteria must be met as listed in method.	Retune instrument. If necessary, clean source.
Initial Calibration (5 concentration levels) for all target analytes. Lowest conc. level at reporting limit.	Prior to sample analysis	The RSD of target analyte RFs must be $\leq$ 15%. Minimum mean RFs of SPCCs as listed in method must be met during initial calibration. The RSD	Rerun calibration standards. Clean purge and trap transfer lines. Rerun initial calibration. Check for system leaks, clip six inches

		of CCC RFs during initial calibration must be $\leq$ 30%.	off column, change column. If necessary, prepare new calibration standards.
Calibration Verification (– a midlevel standard run every 12 hrs) prepared from separate source from calibration standards	Daily before sample analysis and every 12 hrs of analysis time.	RF criteria for SPCCs the same as during initial calibration. RF of CCCs must be $\leq$20 percent difference from initial calibration. The IS retention times must be < 30 secs from those in the midpoint standard of most recent initial calibration. The IS responses must be within –50% to +100% of those in the midpoint standard of the most recent initial calibration.	Rerun CCS. Then rerun initial calibration, if necessary.
Method Blank	One per daily analysis batch.	No target analyte detected $\geq$ 5% or MRL	Bake out purge and trap system. Change adsorbent trap. Re-prep and reanalyzed method blank with associated samples.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) – all target analytes spiked at same conc. as LCS	One MS/MSD every 20 samples per matrix.	Should be within control limits established by lab.	Check LCS to determine if matrix effects apply.
Laboratory Control Sample (LCS) – all target analytes spiked at $\leq$ 50% of linear range calibrated. Prepared same as CVS.	One per daily analysis batch.	Must be within control limits established by lab.	Reprep and reanalyze LCS. Reanalyzed associated samples.
Surrogates – 4-Bromofluorobenzene, 1,2-Dichloroethane-d4, Toluene-d8, 1,4-Dichlorobenzene.	All samples, spikes, standards, and method blanks.	Must be within control limits established by lab or the method.	Reanalyze sample. If one or more still remain outside criteria, recalibrate and or remake

			surrogate solution.
Internal Standards - Fluorobenzene, Chlorobenzene-d5, 1,4-dichlorobenzene-d4.	All samples, spikes, standards, and method blanks.	Area must be –50 to +100% of last calibration check. RT must be ± 30 secs from last calibration check.	Reanalyze sample. If one or more still remain outside criteria, recalibrate and or remake IS solution.

13.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

13.1 Even though data that is out-of-control might be considered unusable, its usabililty will be decided after review and discussion between the client and KB Labs.

13.2 Out-of-control or unacceptable data will be provided with a definite qualifier and an explanation in the project narrative on the final report to the client.

14.0 METHOD PERFORMANCE

14.1 Method performance is established by determining the Method Detection Limits (MDLs) in the matrix of interest. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL that is achieved for a given analyte will vary depending on instrument sensitivity and matrix effects.

14.2 The MDL for both waters and soils is experimentally determined using procedures described in 40 CFR, Part 136, Appendix B and as per 91-04.

14.2.1 Seven replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest.

14.2.2 The concentrations for each analyte are then experimentally determined using the procedures described above for this method.

14.2.3 The standard deviation of the found concentrations for the seven replicates is then calculated.

14.2.4 The MDL for each analyte is then determined by multiplying the standard deviation by 3.

15.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Refer to procedures described in KB Labs' *Standard Operating Procedure SOP010 (Waste Disposal)* and KB Labs' *Health and Safety Manual.*

16.0 REFERENCES

14.1 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846*, Method 8260B, Revision 2, December 1996

14.2 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846*, Method 8000B, Revision 2, December 1996

14.3 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846*, Method 5030B, Revision 2, December 1996

14.4 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846*, Method 5035, Revision 0, December 1996

14.5 Code of Federal Regulations, Title 40, Part 40, Appendix B.

14.6 Hewlett-Packard 5973 MSD Hardware Manual Number.

14.7 Tekmar LSC 3000 Purge and Trap Concentrator User Manual.

14.8 KB Labs' Quality Assurance Manual, August, 2003.

14.9 KB Labs' Health and Safety Manual, 1998.

14.10 KB Labs Standard Operating Procedure (SOP) No. 007, *Sample Receipt and Acceptance*, July 2003, Revision 1.

C2: TestAmerica Nashville SOPs

Title: ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP SPECTROSCOPY SW-846 METHOD 3010A

Approvals (Signature/Date)

	7/15/13		7/15/13
Rodney Street Metals Department Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date
	7/3/13		
Michael H. Dunn Technical Director Quality Assurance Manager	Date		

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used for the preparation of aqueous samples, mobility-procedure (TCLP, SPLP) extracts, and wastes that contain suspended solids for analysis, by inductively coupled argon plasma spectroscopy (ICP or ICP/MS). It is not applicable to dissolved metals. The procedure is appropriate for the following total metals:

Analyte	CAS #
Aluminum, Al	7429-90-5
Antimony, Sb	7440-36-0
Arsenic, As	7440-38-2
Barium; Ba	7440-39-3
Beryllium; Be	7440-41-7
Bismuth, Bi	7440-69-9
Boron, B	7440-42-8
Cadmium; Cd	7440-43-9
Calcium; Ca	7440-70-2
Chromium; Cr	7440-47-3
Cobalt; Co	7440-48-4
Copper; Cu	7440-50-8
Iron; Fe	7439-89-6
Lead; Pb	7439-92-1
Lithium, Li	7439-93-2

Analyte	CAS #:
Magnesium, Mg	7439-95-4
Manganese, Mn	7439-96-5
Molybdenum, Mo	7439-98-7
Nickel, Ni	7440-02-0
Potassium, K	7440-09-7
Selenium, Se	7782-49-2
Silver, Ag	7440-22-4
Sodium, Na	7440-23-5
Strontium, Sr	7440-24-6
Sulfur, S	7704-34-9
Tin, Sn	7440-31-5
Titanium, Ti	7440-32-6
Thallium, Tl	7440-28-0
Vanadium, V	7440-62-2
Zinc, Zn	7440-66-6

1.2 Reporting Limits: See the determinative method (6010 / NV06-44 or 6020 / NV06-215) and the Laboratory Information Management System (LIMS).

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor/Manager or the Laboratory Technical Director. All abnormalities must be noted in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A mixture of nitric acid and the sample is refluxed in a covered hot block digestion vessel. This step is repeated with additional portions of nitric acid, if necessary, until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it **must** be discarded and the sample re-digested.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Interferences are discussed in the determinative analytical method.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health

practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors causes breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms include coughing, choking, and irritation of the nose, throat, and respiratory tract. Causes redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Hydrochlo-ric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Causes redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

None.

6.2 Supplies

Labware: All digestion vessels and volumetric ware must be carefully acid washed and rinsed with reagent water. Polymeric or glass volumetric ware and storage containers must be cleaned by leaching with more dilute acids (approximately 10% v/v) appropriate for the specific plastics used and then rinsed with reagent water and dried in a clean environment. To avoid precipitation of silver, ensure that all HCl has been rinsed from the vessels. Commercial, certified-clean containers are acceptable.

- Certified, plastic digestion vessel with caps.
- Graduated cylinder or equivalent, 50 or 100-mL, Class A.
- Funnel or equivalent.
- Hot block, or equivalent, adjustable and capable of maintaining a temperature of 90-95^{o}C.
- Volumetric flasks and pipets of suitable precision and accuracy (Class A)
- Watch glass, ribbed or non-ribbed (plain).

- Syringe filter, PTFE membrane. The filter diameter and pore size are not significant.

7.0 Reagents and Standards

7.1 **Reagent water**, analyte-free (< MDL).

7.2 Spectroscopic grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 **Hydrochloric acid**, 1:1: HCl. If the method blank is less than the MDL, the acid is acceptable.

7.4 **Nitric acid**, concentrated: HNO_3. If the method blank is less than the MDL, the acid is acceptable.

7.5 **Stock Element Solution for Method 6010**, commercial, certified, for LCS and Matrix Spikes. See the determinative method for the standard, its preparation, and its spike amount.

7.6 **Stock Element Solution for Method 6020**, commercial, certified, for LCS and Matrix Spikes. See the determinative method for the standard, its preparation, and its spike amount. ,

7.7 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE or Glass[1]	50 mL	HNO_3 to pH < 2	6 months	SW846 Section 2.0

[1]All sample containers must be pre-washed with detergents, acids and water. Plastic, certified containers are used if the containers are supplied by TestAmerica Nashville. Temperature preservation is not required.

If samples are preserved at the lab, wait 24 hours after preservation before digestion.

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 **Sample QC**

The following quality control samples are prepared with **each batch of no more than 20 samples.**		
Quality Controls	**Frequency**	**Acceptance Criteria**
Method Blank	1 per batch	See the determinative method.
Laboratory Control Sample (LCS)[1], second source		
Matrix Spike		
Matrix Spike Duplicate		

[1]For AZ, TX, WV samples, a LCS duplicate is required.

- **Method Blank:** The laboratory prepares and analyzes a method blank with each batch of the same matrix. Blank data are used to assess contamination from the laboratory environment.
- A **Laboratory Control Sample (LCS)** is analyzed with every batch; it is made from a standard **different** from the calibration standard. See the determinative method for the spike amount.
- **Matrix Spike/Matrix Spike Duplicate:** Sample homogeneity and the chemical nature of the

sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquots from the sample for replicate and fortified analyses can in some cases assess the effect. Unless otherwise specified by the data user, the matrix spike (MS) / matrix spike duplicate (MSD) procedure is required. Prepare a MS/MSD each batch. The MS/MSD aliquots must be duplicates of the aliquot used for sample analysis and spiked prior to sample preparation. The added analyte concentration must be the same as that used in the LCS.

9.2 Instrument QC: See the determinative method.

10.0 Procedure

10.1 Sample Digestion

Matrix	Sample Size
Water	50.0 mL

Refer to SOP Sample Homogenization, Sub-sampling, and Compositing / NV08-229.

Note: If running dissolved metals, use SOP 3005 / NV06-103.

CAUTION: The addition of hydrochloric acid must be in the form of concentrated hydrochloric acid and not from a premixed combination of acids as a buildup of chlorine gas, as well as other gases, will result from a premixed acid solution. These gases may be violently released upon heating. This is avoided by adding the acid in the described manner.

CAUTION: Toxic nitrogen oxide and chlorine fumes may be evolved; therefore all work must be performed in a properly operating ventilation system. Be aware of the potential for a vigorous reaction. If a vigorous reaction occurs, allow cooling before capping the vessel.

1	Transfer a 50.0-mL representative aliquot of the well-mixed sample to a certified digestion tube and add 1.5 mL of concentrated HNO_3. • For the method blank, use 50.0 mL of reagent water. • For LCS, use 50.0 mL reagent water, and spike with the appropriate amount of spike. • For MS and MSD, spike 50.0 mL of a sample with the appropriate amount of spike.
2	Cover the digestion tube with the watch glass. Place the vessel in hot block and cautiously evaporate to a low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the container is allowed to go dry.
3	Cool the container and add additional 1.5 mL portion of concentrated HNO_3. Cover the container with a watch glass and return to the hot block. Adjust the temperature of the hot plate so that a gentle reflux action occurs. Note: If a sample is allowed to go to dryness, low recoveries result. Should this occur, discard the sample and re-digest. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing)
4	Uncover the container, and evaporate to a low volume (about 5 mL), not allowing any portion of the bottom of the container to go dry. Cool.
5	Add 5.0 mL of 1:1 HCl, cover, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation. .

6	Wash down the container walls and adjust the final volume to 50.0 mL with reagent water in a certified centrifuge tube.
7	Allow any undissolved material to settle overnight, filter using a PTFE membrane, or centrifuge a portion of the prepared sample until clear. Record the filter lot number. The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted sample cannot be characterized, all analyses are performed as soon as possible after the completed preparation. If any sample is filtered, the Method Blank and LCS must also be filtered

11.0 Calculations / Data Reduction
Not applicable.

12.0 Method Performance
12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.
12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.
12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.
12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.
12.5 Control Charts: Laboratory method performance can be shown with the use of control charts, available from the QA department.

13.0 Pollution Control
It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management
14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.
14.2 Wastestreams Produced by the Method:

- Digestates are taken to the waste disposal area for neutralization and discharge to the sanitary sewer.

15.0 References / Cross-References

15.1 SW-846 Method 3010A, Rev. 1, July 1992.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.4 SOPs: Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, 6010 / NV06-44, 6020 / NV06-215, Standard Purchase, Preparation, Control, Documentation / NV08-214, Sample Homogenization, Sub-sampling, and Compositing / NV08-229.

15.5 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachments

None.

18.0 Revision History

- Revision 6, dated 10 September 2008
  - Integration for TestAmerica and STL operations.
  - Change ppm to µg/mL.
- Revision 7, dated 25 September 2009
  - Addition of OH VAP requirements.
  - Included new stock standard concentrations and final digestate concentrations.
- Revision 8, dated 29 July 2011
  - Organizational changes.
  - Addition of QAF-45 and Section 14.2.
  - Addition of Bismuth, Lithium, and Sulfur, standardization of element lists.
- Revision 9, dated 31 July 2012
  - Add SOP Sample Homogenization, Sub-sampling, and Compositing / NV08-229.
  - Specify that this method is not applicable to dissolved metals.
  - Update 6010 standards and preparations. Add 6020 standards and preparations.
- Revision 10, dated 31 July 2013
  - Organizational changes.
  - Addition of a PTFE filter. Removal of standard information with reference to the determinative method.
  - Re-order the last steps of the digestion process.

SOP Number/Revision No.: **3510 / NV03-24.13b** Effective Date: 9/30/2013

Last Mod. Date: 7/31/2013

SOP Title: **Method 3510C, Separatory Funnel Liquid-Liquid Extraction**

Affected SOP Section Number(s): Section 10.1, Sample Preparation

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 03P

Revision Number with Mod ID: **13c**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: **Add underlined text; delete crossed-out text.**

Section 10.1, Sample Preparation

1	Generally, the **entire** contents of the sample bottle are to be extracted. Mark the level of sample on the outside of the bottle and measure against a calibrated bottle of the same size and shape. (Bottles are calibrated quarterly using Class A volumetric flasks. See Section 17.3.) Alternatively, using a Class A, graduated cylinder, measure the required amount of sample. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1 L with organic-free reagent water, or samples may be collected in smaller sample bottles and the whole sample used. Shake the sample container well and transfer to a separatory funnel that has been pre-rinsed with about 10 mL of Methylene chloride.
2	For TCLP: 500 mL of the TCLP extract is used for semivolatile (BNA) extraction, 100 mL is used for pesticide extraction. ~~For herbicide extraction, use 5 mL of TCLP extract.~~ Reagent water is used to bring the volume to about 1 L. Add surrogates (Table 2) to all samples and QC. • For TCLP BNA, spike the LCS, MS/MSD with 1 mL of the TCLP BNA spike. • For TCLP Pesticides, spike with 0.5 mL TCLP Pesticides spike and surrogate and 1 mL of Toxaphene. • ~~For TCLP herbicides, spike with 1.0 mL of TCLP Herbicide spike.~~
3	Mixing by shaking is sometimes ineffective as solids settle during the time required to secure the sub-sample aliquot for analysis. Usually decantation prior to shaking or after initial settling following mixing (in order to preserve the suspended solids) is not an appropriate homogenization procedure. However, if decantation is used, documentation of the process must be made in the preparation or analysis logbook or benchsheet. If the inclusion of solid material adversely affects the extraction or analysis procedure, notify the project manager. The project manager must contact the client to verify if they would like the lab to extract only the aqueous portion. In this case, pour out only the liquid portion into a graduated cylinder and note the

	volume decanted and the approximate percentage of sediment present on the benchsheet.
4	Using a glass syringe, add the surrogate spiking solution into each sample in the **graduated cylinder or sample bottle** ~~separatory funnel~~ and mix well. (See Table 2 for details on the surrogate standard solution. This addition of surrogate must be made into both client and QC samples.)
5	Using a glass syringe, add the matrix spike/LCS spiking solution into the appropriately designated **graduated cylinder or sample bottle** ~~separatory funnels~~ and mix well. (See Table 2, the determinative method, and LIMS for details on the matrix spike/LCS solution.)
6	Check the pH of the sample by immersing a glass or Teflon™ rod tip or a pipet in the sample and touch to wide-range pH paper and adjust the pH, if necessary, to the pH indicated in **Table 1**, using 1:1 (v/v) Sulfuric acid or 10 N Sodium hydroxide. Rinse the glass or Teflon™ rod with Methylene chloride into the separatory funnel. Other strengths of acid or base solution may be employed, provided that they do not result in a significant change (< 1%) in the volume of sample extracted.
7	For Method 8270 BNA, always adjust to acid pH first. If only PAHs are being analyzed by Method 8270, then only a ~~the base-~~neutral extraction needs to be done. In order to extract only the base-neutral, all samples within the batch must be PAH only, and the QC **must** be treated the same as the samples.
~~8~~	~~QC samples are prepared using 1 liter organic-free reagent water.~~

Jacoby Peterson 9/26/13	Jay Do J. 9/26/13
Department Manager Approval Date	Operations Manager Approval Date
Michael A. Burns 9/26/13	
Technical Approval Date Quality Manager Approval	

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **3510 / NV03-24.13a** Effective Date: 7/31/2013

Last Mod. Date: 3/29/2013

SOP Title: **Method 3510C, Separatory Funnel Liquid-Liquid Extraction**

Affected SOP Section Number(s): Section 8.0, Sample Collection, Preservation, Shipment and Storage

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 03P

Revision Number with Mod ID: **13b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: **Add underlined.**

Section 8.0, Sample Collection, Preservation, Shipment and Storage: Add a note to the table.

For South Carolina, a 1 liter sample is required.

7/30/13	7/30/13
Department Manager Approval Date	Operations Manager Approval Date
7/30/13	
Technical Approval Date	
Quality Manager Approval	

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **3510 / NV03-24.13** Effective Date: 3/29/2013

Last Mod. Date: 12/31/2012

SOP Title: **Method 3510C, Separatory Funnel Liquid-Liquid Extraction**

Affected SOP Section Number(s): Section 10.3, Sample Extraction (non-fluff), and Section 10.4, Sample Concentration by Kuderna-Danish Technique.

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 03P

Revision Number with Mod ID: **13a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: **Delete crossed out, add underlined.**

Section 10.3, Sample Extraction (non-fluff), Step 7

Dry the extract by passing it through a Teflon™ funnel containing about 2/3 full of pre-rinsed, anhydrous Sodium sulfate. Collect the dried extract in a clean Erlenmeyer flask. Rinse the funnel ~~Erlenmeyer flask, which contained the solvent extract,~~ with 20-30 mL Methylene chloride and add it to the flask ~~funnel~~ to complete the quantitative transfer. Perform the concentration using the Kuderna-Danish technique.

Section 10.4, Sample Concentration by Kuderna-Danish technique, Steps 2, 6, and 8

2	Add one or two clean boiling chips to the KD flask. Quantitatively transfer extract to the KD. Rinse the flask with approximately 10-20 mL solvent. ~~and a~~Attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of Methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15 – 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the temperature and/or flask position to complete the concentration in 10 - 20 minutes. Record the temperature. At the proper rate of distillation, the balls of the column actively chatter, but the chambers do not flood. When the apparent volume of liquid reaches about 10 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

	Nitrogen blow-down technique
6	Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the just below final volume indicated in Table 1, using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). ~~Bring the extract to the required final volume using Class A volumetric flasks.~~
8	The sample is then transferred to a Class A volumetric flask and adjusted to the final volume by using the rinsate from the tube ~~using the last solvent used~~.

Jacoby Petersen 3/21/13	Jay DeJ. 3/21/13
Department Manager Approval Date	Operations Manager Approval Date
Michael A. Burns 3/21/13	
Technical Approval Date	
Quality Manager Approval	

SOP Number/Revision No.: **3510 / NV03-24.13** Effective Date: 3/29/2013

Revision Number with Mod ID: **13a**

Title: SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION SW-846 METHOD 3510C

Approvals (Signature/Date)

Jacolby Robinson Department Manager	Date 12/17/12	Johnny Davis Extractions Operations Manager Health & Safety Manager / Coordinator	Date 11/27/12
Michael H. Dunn Technical Director Quality Assurance Manager	Date 11/20/12		

1.0 Scope and Application

1.1 Analyte, Matrices: This method predominantly describes a procedure for extracting water-insoluble and slightly water-soluble organic compounds from aqueous samples and concentrating them for injection onto a gas chromatograph set up for an appropriate determinative method.

1.2 Reporting Limits: Results are dependent on the volume used, degree of contamination, ability to concentrate, and the sensitivity of the determinative method.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Manager. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A measured volume of sample, nominally 1000, 250, or 125 mL, at a specified pH, is serially extracted with Methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used.

3.0 Definitions

See Appendix 5 of TestAmerica Nashville's QA Manual for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Interferences are often due to contamination from solvents or extraction glassware.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Use the hoods to evacuate solvent vapors from the building and dispose of solvent wastes appropriately.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material causes burns in contact with the skin or eyes. Inhalation of Sodium Hydroxide dust causes irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material causes burns if comes into contact with the skin or eyes. Inhalation of vapors causes irritation of the nasal and respiratory system.
Acetonitrile	Flammable Poison	40 ppm TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Kuderna-Danish (K-D) apparatus.
  - Concentrator tube, 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts. \
  - Evaporation flask, 250 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.
  - Snyder column, Three-ball macro (Kontes K-503000-0121 or equivalent).
  - Snyder column, 3 chamber micro (Kontes K-569001-0219 or equivalent).
  - Clamps
- Nitrogen Evaporator: N-Evap Model #116 by Organomation, or equivalent.

6.2 Supplies

- Separatory funnel, 2 liter, 500 mL, or 250 mL, Teflon™ with polytetrafluoroethylene (PTFE) stopcock.
- Funnel, Teflon™. Put a plug of glass wool in a funnel and fill about 2/3 full with sodium sulfate. Rinse funnel and Sodium sulfate with 5-10 mL of Methylene chloride before use.
- Boiling chips, solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).
- Water bath, heated, capable of temperature control (± 5^{o}C). The bath is used in a hood.
- Vials, glass with PTFE-lined screw-caps.
- pH indicator paper, 0 – 14 pH range.
- Erlenmeyer flask, Teflon™250 mL or 500 mL.
- Graduated cylinder, glass, Class A, 1 liter, or equivalent.
- Volumetric flasks, Class A, at 1, 5, and 10 mL.
- Centrifuge, capable of approximately 2000 rpm.
- Nitrogen, compressed gas, high purity.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents must conform to the specifications of the Committee on Analytical Reagents of the America Chemical Society, where such specifications are available. Other grades may be used, however, provided it is first ascertained that the reagent is of sufficiently high purity to

permit its use without lessening the accuracy of the determination. Reagents are stored in glass or Teflon™ to prevent the leaching of contaminants from plastic containers.

7.2 **Reagent water**, analyte-free.

7.3 **Sodium hydroxide** solution (10 N), NaOH, commercial source.

7.4 **Sodium sulfate** (granular, anhydrous), Na_2SO_4. Purify by heating to about 400°C for about 4 hours in a shallow tray. Store in a sealed, glass container. A commercially baked product is acceptable.

7.5 **Sulfuric acid** solution, H_2SO_4, commercial source. Make a 1:1 dilution with reagent water.

7.6 **Extraction/exchange solvents**: All solvents are pesticide quality or equivalent and from commercial sources: **Methylene chloride,** CH_2Cl_2, **Hexane**, C_6H_{14}, **Acetonitrile**, CH_3CN.

7.7 **Sodium chloride** (NaCl), commercial source, granular.

7.8 **Acetone**, CH_3COCH_3, commercial source.

7.9 **Spiking Solutions**: See the determinative method and LIMS for information. These are purchased ready for use or prepared in the analysis department.

7.10 See SOP Reagent and Standard Purchase, Preparation, control, Documentation / NV08-214 for information on shelf-life and storage requirements for standards and reagents.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time from Collection	Reference
Water	Amber Glass, 1 L, 250 mL, or 125 mL Teflon™-lined cap	varies	0-6°C	7 days until extraction, 40 days until analysis	SW846 Chapter 2

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

The following quality control samples are prepared with each batch of no more than 20 samples.

Quality Controls	Frequency
Method Blank	1 in 20 or fewer samples
Laboratory Control Sample (LCS)[1], second source	1 in 20 or fewer samples
Matrix Spike	1 in 20 or fewer samples
Matrix Spike Duplicate	1 in 20 or fewer samples

[1]For AZ, TX, WV samples, a LCS duplicate is required.

- **Method blank:** The laboratory prepares and analyzes a method blank (reagent water) with each batch.
- A **Laboratory Control Sample (LCS)**, reagent water spiked with a source different from the calibration standard, is analyzed with every batch. Use the same sample preparations, analytical methods, and QA/QC procedures employed for the test samples.
- **Matrix Spike/Matrix Spike Duplicate:** Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquots from the sample for replicate and fortified analyses can in some cases assess the

effect. Unless otherwise specified by the data user, the MS/MSD procedure is required.

- The laboratory must add a known amount of each analyte to be reported to a minimum of 5% of the routine samples. In each case the MS/MSD aliquot must be a duplicate of the aliquot used for sample analysis and added **prior** to sample extraction.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	1000, 250 , or 125 mL

1	Generally, the **entire** contents of the sample bottle are to be extracted. Mark the level of sample on the outside of the bottle and measure against a calibrated bottle of the same size and shape. (Bottles are calibrated quarterly using Class A volumetric flasks. See Section 17.3.) Alternatively, using a Class A, graduated cylinder, measure the required amount of sample. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1 L with organic-free reagent water, or samples may be collected in smaller sample bottles and the whole sample used. Shake the sample container well and transfer to a separatory funnel that has been pre-rinsed with about 10 mL of Methylene chloride.
2	For TCLP: 500 mL of the TCLP extract is used for semivolatile (BNA) extraction, 100 mL is used for pesticide extraction. For herbicide extraction, use 5 mL of TCLP extract. Reagent water is used to bring the volume to about 1 L. Add surrogates (Table 2) to all samples and QC. • For TCLP BNA, spike the LCS, MS/MSD with 1 mL of the TCLP BNA spike. • For TCLP Pesticides, spike with 0.5 mL TCLP Pesticides spike and surrogate and 1 mL of Toxaphene. • For TCLP herbicides, spike with 1.0 mL of TCLP Herbicide spike.
3	Mixing by shaking is sometimes ineffective as solids settle during the time required to secure the sub-sample aliquot for analysis. Usually decantation prior to shaking or after initial settling following mixing (in order to preserve the suspended solids) is not an appropriate homogenization procedure. However, if decantation is used, documentation of the process must be made in the preparation or analysis logbook or benchsheet. If the inclusion of solid material adversely affects the extraction or analysis procedure, notify the project manager. The project manager must contact the client to verify if they would like the lab to extract only the aqueous portion. In this case, pour out only the liquid portion into a graduated cylinder and note the volume decanted and the approximate percentage of sediment present on the benchsheet.
4	Using a glass syringe, add the surrogate spiking solution into each sample in the separatory funnel and mix well. (See Table 2 for details on the surrogate standard solution. This addition of surrogate must be made into both client and QC samples.)
5	Using a glass syringe, add the matrix spike/LCS spiking solution into the appropriately designated separatory funnels and mix well. (See Table 2, the determinative method, and LIMS for details on the matrix spike/LCS solution.)
6	Check the pH of the sample by immersing a glass or Teflon™ rod tip or a pipet in the sample and touch to wide-range pH paper and adjust the pH, if necessary, to the pH indicated in **Table 1**, using 1:1 (v/v) Sulfuric acid or 10 N Sodium hydroxide. Rinse the glass or Teflon™ rod with Methylene chloride into the separatory funnel. Other strengths of acid or base solution may be employed, provided that they do not result in a significant change (< 1%) in

	the volume of sample extracted.
7	For Method 8270 BNA, always adjust to acid pH first. If only PAHs are being analyzed by Method 8270, then only the base-neutral extraction needs to be done. In order to extract only the base-neutral, all samples within the batch must be PAH only, and the QC **must** be treated the same as the samples.
8	QC samples are prepared using 1 liter organic-free reagent water.

10.2 Sample Extraction

1	Use Methylene chloride to rinse the graduated cylinder (or sample container) and transfer this rinse solvent to the separatory funnel containing the sample. **Record** the sample volume on the benchsheet. For 1 L sample volume, use 60 mL Methylene chloride. For 250 mL sample volume, use 15 mL Methylene chloride. For 125 mL sample volume use 8 mL Methylene chloride.
2	Seal and shake the separatory funnel vigorously for 1 - 2 minutes with periodic venting to release excess pressure. Methylene chloride creates excessive pressure very rapidly; therefore, initially vent **immediately** after the separatory funnel has been sealed and shaken once. Vent the separatory funnel into a hood to avoid exposure of the analyst to solvent vapors.
3	Allow the organic layer to clearly separate from the water phase. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation at approximately 2000 rpm for about 5 minutes, or other physical methods. Collect the solvent extract in an Erlenmeyer flask.
4	Repeat the extraction **two** more times using fresh portions of solvent. Combine the three solvent extracts.
5	If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH indicated in **Table 1 (Section 17)**. Serially extract three times with proportional volumes of Methylene chloride. Collect and combine the extracts and label the combined extract appropriately.
6	If performing GC/MS analysis (Method 8270), the acid/neutral and base extracts are combined prior to concentration. However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable (e. g., if for regulatory purposes the presence or absence of specific acid/neutral or base compounds at low concentrations must be determined, separate extract analyses may be warranted).
7	Dry the extract by passing it through a Teflon™ funnel containing about 2/3 full of pre-rinsed, anhydrous sodium sulfate. Collect the dried extract in a clean Erlenmeyer flask. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of Methylene chloride and add it to the funnel to complete the quantitative transfer. Perform the concentration using the Kuderna-Danish Technique.
8	Extracts may be held prior to concentration if stored covered; in addition, 8310, 8270, and 8081 extracts when held are stored at 0-6^{o}C and in the dark. Proceed to sample concentration.

10.3 Sample Concentration by Kuderna-Danish Technique

1	Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 250-mL, or smaller, evaporation flask.

2	Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of Methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15 – 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the temperature and/or flask position to complete the concentration in 10 - 20 minutes. Record the temperature. At the proper rate of distillation, the balls of the column actively chatter, but the chambers do not flood. When the apparent volume of liquid reaches about 10 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.
3	If a solvent exchange is required (as indicated in Table 1), remove the Snyder column and KD, and using nitrogen blow-down technique, reduce the volume to about 2 mL. To 4 mL of Hexane or 2 mL of Acetonitrile, whichever is the appropriate exchange solvent, add a new boiling chip, and attach the 3-chamber micro-Snyder column to the concentrator tube. Concentrate the extract and alter the temperature of the water bath, if necessary, to maintain proper distillation.
4	Remove the Snyder column and rinse it and its lower joints into the concentrator tube with 1 - 2 mL of Methylene chloride or exchange solvent. The extract is further concentrated by using the technique outlined in Section 10.3.6 or adjusted in a Class A volumetric to 1.0 - 10.0 mL with the solvent last used.
5	If further concentration is indicated in Table 1, use the nitrogen blow-down technique to adjust the extract to the final volume required.
Nitrogen blow-down technique	
6	Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the just below final volume indicated in Table 1, using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). Bring the extract to the required final volume using Class A volumetric flasks.
7	The internal wall of the tube must be rinsed several times with Methylene chloride or appropriate solvent during the operation. During evaporation, the tube must be positioned to avoid water condensation (i. e., the solvent level is below the level of the water bath). Under normal procedures, the extract must not be allowed to become dry.
8	The sample is then transferred to a Class A volumetric flask and adjusted to the final volume using the last solvent used.
9	Transfer the sample to a vial with a PTFE-lined cap and label appropriately. Individual states may require silica gel clean-up. Refer to Table 1, state-specific SOPs and/or 8015 / NV05-31 for details. The extract may now be analyzed for the target analyses using the appropriate determinative technique(s). Store refrigerated.

10.4 Example Analysis Queue / Sequence

See the determinative method.

11.0 Calculations / Data Reduction

Enter sample volume and final extract volume in LIMS.

12.0 Method Performance

12.1 Method Detection Limits (MDLs): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses

performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.1 Wastestreams produced by this method:

- Extracted aqueous samples are collected, neutralized to a pH between 2.0 and 10.0, and discharged to the sanitary sewer.
- Used sodium sulfate and glass wool or filter paper contaminated with Methylene chloride from the extract drying step are placed in a hood overnight, then discarded in the trash.
- Assorted flammable solvent waste from various rinses is collected in the flammable waste drums.

15.0 References / Cross-References

15.1 SW-846 Method 3510C, Update III, Revision 3, December 1996.

15.2 CA LUFT Manual, Version 2.0, October 4, 2010.

TestAmerica Nashville's Quality Assurance Manual.

15.3 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.4 SOPs: Waste Disposal / NV10-83, 8270 / NV04-22, 8081 / NV04-16, 8082 / NV04-105, 8015 / NV05-31, 8310 / NV04-57, FL PRO / NV04-78, WI DRO / NV04-38, MADEP-EPH / NV04-168, NWTPH-Dx / NV04-190, NWTPH-EPH / NV04-191, OA-2 / NV04-188, OK DRO / NV04-74, TN EPH / NV04-187, CT ETPH / NV04-86, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Reagent and Standard Purchase, Preparation, control, Documentation / NV08-214.

15.5 Controlled Documents: PF-1, Prep Lab Summary Chart, QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	Use of reduced sample volumes.

17.0 Attachments

17.1

Table 1. Specific Extraction Conditions for Various Determinative Methods

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Exchange Solvent Req. for Analysis	Exchange Solvent Required for Cleanup	Volume of Extract Required for Cleanup (mL)	Final Extract Volume for Analysis (mL)[a]
8081	5-9	None	Hexane	Hexane	5.0	5.0
8082	5-9	None	Hexane	Hexane	5.0	5.0
8270[a]	<2	>11	None	-	-	1.0
8310	As rec'd	None	Acetonitrile	Acetonitrile	-	1.0
8015	None	None	None	-	-	1.0
CA TPH (LUFT)	<2	None	None			1.0[b]
CT-ETPH	<2	None	None	None		1.0
FL PRO	<2	None	None	-	2.0	2.0[b]
MA-EPH	<2	None	None	Hexane	1.0	1.0[b]
NWTPH-Dx	<2	None	None		1.0	1.0[b]
NWTPH-EPH	<2	None	None	Hexane	2.0	2.0[b]
OA-2	<2	None	None	-	-	1.0
TN-EPH	<2	None	None	-	-	1.0
WI/OK-DRO	<2	None	None	-	-	1.0

[a] If only PAHs are being analyzed by Method 8270, then only the base extraction needs to be done. In order to do this, all samples within the batch must be PAH only and the QC **must** be treated the same as the samples.

[b] Silica gel cleanup required.

17.2

Table 2. Surrogate and Matrix Spike/LCS Amounts

Method	Surrogate Spike Amount	MS/LCS Spike Amount	Sample Volume
8081	1.0 mL of Pest/PCB surrogate	1.0 mL Pest spike, 1.0 mL Toxaphene/Technical chlordane spike	1 L
8081	200 µL of Pest/PCB surrogate	200 µL Pest spike, 200 µL Toxaphene/Technical chlordane spike	125 mL
8082	1.0 mL of Pest/PCB surrogate	1.0 mL PCB spike	1 L
8082	200 µL of Pest/PCB surrogate	200 µL PCB spike	125 mL
8270	1.0 mL of BNA surrogate	500 µL of BNA spike	1 L
8270	200 µL of BNA surrogate	100 µL of BNA spike	250 mL

Method	Surrogate Spike Amount	MS/LCS Spike Amount	Sample Volume
8310	1.0 mL of HPLC surrogate	1.0 mL of HPLC spike	1
8015	1.0 mL of o-Terphenyl surrogate	1.0 mL DRO spike	1 L
8015	0.200 µL of o-Terphenyl surrogate	200 µL DRO spike	250 mL
CA TPH (LUFT)	1.0 mL of o-Terphenyl surrogate	1.0 mL DRO spike	1
CT ETPH	1.0 mL of o-Terphenyl surrogate	2.0 mL of FL/WI spike	1
FL PRO	2.0 mL of o-Terphenyl surrogate 2 mL of C_{35} surrogate	2.0 mL of FL/WI spike	1
MA-EPH	1 mL of MA surrogate 1 mL fractionation surrogate	1 mL of MA spike	1
NWTPH-Dx	1.0 mL of o-Terphenyl surrogate	1 mL of DRO spike	1
NWTPH-EPH	2 mL o-Terphenyl surrogate	2 mL MA spike	1
OA-2	1 mL o-Terphenyl surrogate	1 mL DRO spike	1
OK-DRO	1 mL of o-Terphenyl surrogate	2.0 mL of FL/WI spike	1
TN-EPH	1 mL o-Terphenyl surrogate	1 mL TN EPH spike	1
WI/OK-DRO	1.0 mL of C_{35} surrogate	2.0 mL of FL/WI spike	1
See individual SOPs for information on compounds, concentrations, and how to make the surrogate and spike solutions. Also, see the Prep Summary Chart for additional spike and surrogate information.			

17.3 Calibration of Bottles: Representative bottles, of varying size and shape, are calibrated using 100 mL, 50 mL, and 10 mL Class A volumetric flasks. Graduation marks are made accordingly, and bottles are then ready to be used as measuring guides for liquid samples. Date of calibration is noted on the bottle. The calibrated bottles are verified for accuracy quarterly and the verification recorded in a logbook.

18.0 Revision History

- Revision 8, dated 30 April 2008
  - Integration for TestAmerica and STL operations.
- Revision 9, dated 25 September 2009
  - Ohio VAP requirements
- Revision 10, dated 29 January 2010.
  - Addition of centrifuge to Section 6.2.
  - Define acronyms, abbreviations and/or refer to QAF-45.

  - Add Section 14.2 to document.
  - Change sample volume to 1000 mL.
- Revision 11, dated 30 June 2011.
  - Addition of Nitrogen to list of supplies (Amendment 10a).
  - Change sample volume for 8081/8082 to 1 Liter.
  - Clarify TCLP 8151 spike amounts.
  - Remove vertical adjustment of the apparatus in the water bath from sample concentration procedure description.
  - Distinguish Hexane and Acetonitrile volume additions when solvent exchange is required.
  - Organizational changes.
  - Change glass funnels to Teflon™ funnels.
- Revision 12, dated 30 April 2012
  - Organizational changes.
  - Addition of change form 11a, removing C35 surrogate from NWTPH-EPH.
  - Add considerations for samples with large amounts of solids, and revise sequence of steps for sample preparation.
  - Noted bottle calibration quarterly frequency in Section 10. Added date of calibration noted on bottle in section 17.3.
- Revision 13, dated 31 December 2012
  - Organizational changes.
  - Add information for Reduced Volume Extraction / Low Volume Injection.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **5030 / NV05-107.10a** Effective Date: 6/28/2013

Last Mod. Date: 5/31/2013

SOP Title: **Method 5030B/5030C - Purge-And-Trap For Aqueous Samples, Method 5030A – Purge And Trap For Soils And Sediments**

Affected SOP Section Number(s): Section 8.0, Sample Collection, Preservation, Shipment and Storage

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 05V

Revision Number with Mod ID: **10b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

Summary of Procedure Change: Add underlined text.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Glass	40-mL VOA vials, No headspace	Cool 0-6°C, pH < 2 with HCl*	14 days from time of collection if preserved; 7 days if not preserved	SW-846 Section 2.0
High-Concen-tration Soil		2-oz. container, Teflon-lined cap, or EnCore™	Cool 0-6°C, Cover with Methanol.		
Low-Concen-tration, Bulk Soil		5 g / VOA	Cool 0-6°C, Sodium bisulfate*		

Additional information on soil samples is found in SOP 5035 / NV05-108.

*Trisodium phosphate is optional, as needed. If TSP is used, the pH must be greater than 11.

6/26/13	6/26/13
Supervisor Approval Date	Supervisor Approval Date

Glen L. Norton 6/26/13	Michael A. Burns 6/26/13
Department Manager Approval Date	Technical Director Approval Date Quality Assurance Approval

SOP Number/Revision No.: **5030 / NV05-107.10b** Effective Date: 6/28/2013

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8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Glass	40-mL VOA vials, No headspace	Cool 0-6°C, pH < 2 with HCl*	14 days from time of collection if preserved; 7 days if not preserved	SW-846 Section 2.0
High-Concen-tration Soil		2-oz. container, Teflon-lined cap, or EnCore™	Cool 0-6°C, Cover with Methanol.		
Low-Concen-tration, Bulk Soil		5 g / VOA	Cool 0-6°C, Sodium bisulfate*		

Additional information on soil samples is found in SOP 5035 / NV05-108.

*Trisodium phosphate is optional, as needed. If TSP is used, the pH must be greater than 11.

6/26/13	6/26/13
Supervisor Approval Date	Supervisor Approval Date

Glen L. Norton 6/26/13	Michael A. Burns 6/26/13
Department Manager Approval Date	Technical Director Approval Date Quality Assurance Approval

SOP Number/Revision No.: **5030 / NV05-107.10b** Effective Date: 6/28/2013

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **5030 / NV05-107.10** Effective Date: 5/31/2013

Last Mod. Date: 3/29/2013

SOP Title: **Method 5030B/5030C - Purge-And-Trap For Aqueous Samples, Method 5030A – Purge And Trap For Soils And Sediments**

Affected SOP Section Number(s): Section 10.4**,** Sample pH Determination and Residual Chlorine Check

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 05V

Revision Number with Mod ID: **10a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

Summary of Procedure Change: Add underlined text; delete crossed-out text.

10.4 Sample pH Determination and Residual Chlorine Check: After the sample has been analyzed, check the pH of the sample using narrow-range, pH paper. Compare the test strip to the chart on the test strip container. **Record in LIMS ~~the pH on the runlog or benchsheet~~**. If the pH is > 2 or < 11, then a NCM ~~comment~~ must be added ~~placed on the benchsheet and~~ in LIMS. For non-preserved samples and samples from North Carolina, check for residual chlorine using test strips. Record if present and note or generate a NCM, if needed, in LIMS.

5/17/13	5/17/13
Supervisor Approval Date Glen L. Norton 5/20/13	Supervisor Approval Date Michael A. Brown 5/17/13
Department Manager Approval Date	Technical Director Approval Date Quality Assurance Approval

Title: PURGE-AND-TRAP FOR AQUEOUS SAMPLES METHOD 5030B/5030C PURGE AND TRAP FOR SOILS AND SEDIMENTS METHOD 5030A

Approvals (Signature/Date)

John Haley	2/12/13		3/4/13
John Haley Department Manager	Date	Daniel Otero Department Manager	Date
Glenn L. Norton	02/21/13	Johnny Davis	3/4/13
Glenn Norton Volatiles Operations Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date
Michael H. Dunn	1/28/13		
Michael H. Dunn Technical Director Quality Assurance Manager	Date		

1.0 Scope and Application

1.1 Analyte, Matrices: This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples, bulk soil (low-level), and water miscible liquid samples. It also describes the analysis of high concentration soil and waste sample Methanol extracts prepared in Method 5035 / NV05-108. The gas chromatographic determinative steps are found in Methods 8015 / NV05-31 (GRO) and 8021 / NV05-90. The method is also applicable to GC/MS Method 8260 / NV05-77.

1.2 This method is used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits may be higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitrites, acetates, acrylates, ethers, and sulfides.

1.3 This method, in conjunction with Method 8015 (GC/FID), is used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons (TPH), e. g., gasoline. A total determinative analysis of gasoline fractions is obtained using GC/FID) with Method 8015. The analysis of gasoline, TPH and VPH are covered in the SOP for 8015 / NV05-31 or the state-specific method SOP.

1.4 Water samples are analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water are determined by dilution of the sample prior to the purge-and-trap process. Compositing is allowed.

1.5 Reporting Limits: See the determinative method SOP and LIMS.

1.6 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor/Manager or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 Aqueous Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, where the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatograph.

2.2 High Concentration Extracts from Method 5035: An aliquot of the extract prepared in Method 5035 / NV05-108 is combined with organic-free reagent water in a VOA vial. It is then analyzed by purge-and-trap following the normal aqueous method.

2.3 Low-concentration, Bulk Soil. Transfer to a preserved VOA vial. See SOP 5035 / NV05-108.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

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

determinative step.

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

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, follow with an analysis of organic-free reagent water to check for and minimize the potential for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the entire system is required. Repeat any samples where contamination is suspected.

4.4 The laboratory where volatiles analysis is performed must be completely free of solvents. Special precautions are taken to determine methylene chloride. The analytical and sample storage areas are isolated from all atmospheric sources of methylene chloride. Otherwise random background levels are possible. Since methylene chloride permeates through PTFE tubing, all GC carrier gas lines and purge gas plumbing are constructed of copper, nickel or Silcosteel® tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed also leads to random background levels and the same precautions are taken.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Employees must wear Kevlar gloves when opening and closing VOA vials.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and causes skin to become dry and cracked. Skin absorption can occur; symptoms parallel inhalation exposure. Irritant to the eyes.
Hydrochlo-ric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Causes redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure causes burn if not flushed with water. Causes mild irritation to skin. Prolonged exposure causes burn if not flushed with water.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Trisodium phosphate		None listed	Keep in closed container; avoid high temperatures and strong acids.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Purge-and-trap system - The purge-and-trap device consists of two separate pieces of equipment: the autosampler and purger/trap.
  - The purging chamber is designed to accept 5, 10 or 25 mL samples. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.
  - The desorber is capable of rapidly heating the trap to 260°C for desorption, Tekmar or Encon.
  - Autosampler, Archon or Centurion.
  - Trap Packing Materials
    - 2, 6-Diphenylene oxide polymer- 60/80 mesh, chromatographic grade (Tenax GC or equivalent).
    - VOCARB 3000 (trap K), Carbopack B/ Carboxen 1000 & 1001. Trap for 8260 / NV05-77.
    - BTEX TRAP, Carbopack C & B. Trap for 8015/ NV05-31 (GRO) and 8021 / NV05-90.

      Note: It is possible for even newly constructed traps to have become contaminated prior to their first use from airborne vapors. These highly adsorptive materials must be kept tightly sealed in an area of minimum organic vapor contamination.

    - Condition the trap at the manufacturer's recommended temperature in the purge mode with an inert gas flow of about 20 mL/minute.
  - Capillary GC Columns - Any GC capillary column that meets the performance specifications of the determinative method may be used. See the specific determinative method (8015 / NV05-31, 8021 / NV05-90, or 8260 / NV05-77) for recommended columns, conditions and retention times.
  - Purge and Trap Operating Conditions:

	Analysis Method		
	8015[1]	8260	8021 & 8015[2]
Purge gas	He or N_2	He or N_2	He or N_2
Purge gas flow rate (mL/minute)	40	40	40
Purge time (minute)	15.0 ± 0.1	11.0 ± 0.1	12.0 ± 0.1
Purge temperature (°C)	Ambient	Ambient	Ambient
Desorb temperature (°C)	240	240	240
Backflush inert gas flow (mL/minute)	20-60	20-60	20-60
Desorb time (minute)	4	4	4
Dry Purge time (minute)	1-3	1-3	3
[1] For Method 8015 (gasoline or TPH only), refer to state-specific method if applicable. If a state specific method is not applicable, then use the conditions in this table.			

	Analysis Method		
	8015[1]	8260	8021 & 8015[2]
[2] When Method 8015 and 8021 are analyzed in combination and a state-specific method is not applicable, use the conditions in this table.			
These conditions are recommended and may be changed to optimize the instrument. See instrument maintenance log for instrument conditions. See the department supervisor or the laboratory technical director before altering the purge time, purge temperature or desorb time.			

- For GC/MS, optimize the flowrate to provide the best response for chloromethane and bromoform if these compounds are analytes. Excessive flowrate reduces chloromethane response, whereas insufficient flow reduces Bromoform response.

6.2 Supplies

- Microsyringes - 10 uL, 25 uL, 100 uL, 250 uL, 500 uL, and 1,000 uL. These syringes are equipped with a needle having a length sufficient to extend from the sample inlet to approximately 1" below liquid level.
- 5-mL glass syringe with Luer-lock top.
- Volumetric flasks, Class A - 10 mL, 50 mL, and 100 mL, with ground-glass stoppers.
- Vials – VOA vials, 40 mL, PTFE septa.
- Narrow-range pH paper
- Free Chlorine Check paper (Industrial Test Systems or equivalent)

7.0 Reagents and Standards

7.1 **Reagent water**, analyte-free.
7.2 **Methanol**, purge-and-trap grade.
7.3 **Sodium bisulfate.**
7.4 See the determinative method (8015 / NV05-31, 8021 / NV05-90, 8260 / NV05-77) for other reagents and standards.
7.5 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-224 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Glass	40-mL VOA vials, No headspace	Cool 0-6°C, pH < 2 with HCl*	14 days from time of collection if preserved ; 7 days if not preserved	SW-846 Section 2.0
High-Concen-tration Soil		2-oz. container, Teflon-lined cap, or EnCore™	Cool 0-6°C, Cover with Methanol.		
Low-Concen-tration, Bulk Soil		5 g / VOA	Cool 0-6°C, Sodium bisulfate*		

Additional information on soil samples is found in SOP 5035 / NV05-108.
*Trisodium phosphate is optional, as needed.

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records

to document the quality of the data generated.

9.1 Sample QC: Quality control samples are prepared with **each batch of no more than 20 samples**. See the determinative methods for specifics on how these are made up. QC samples are prepared with reagent water (an exception is the Methanol method blank for the high concentration samples, Section 10.1.4).

9.2 Instrument QC: See the determinative methods.

10.0 Procedure

10.1 Sample Preparation

- All samples and standard solutions are allowed to warm to ambient temperature before analysis.
- This procedure is suitable for aqueous samples and samples that are water miscible.
- Compositing must be done in a closed system via a syringe. Withdraw equal amounts of each sample through the septa into a 50 mL syringe. Transfer to a VOA vial for analysis. Do not exceed 5 samples per composite,

Matrix	Sample Size
Water	5-25 mL
Soil	5 g

10.1.1 **Waters received in VOAs**

- Analyze the VOA sample as received by placing it in the autosampler and starting the instrument.
- If the sample vial contains sediment that could potentially be pulled to the autosampler, use another vial of the sample that does not have sediment. If no secondary vial is available or the secondary vial also has sediment, transfer the liquid contents of the sample vial into a clean, unused VOA vial. To eliminate headspace, dilute the sample or use the second sample vial to obtain a full VOA. If combining vials, flag the data indicating actions taken.

10.1.2 **Waters received in VOAs requiring dilution**

- If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the upper calibration standard, the sample must be reanalyzed at a higher dilution.
- When a sample is analyzed that has saturated response from a compound, follow with the analysis of a blank containing organic-free reagent water. If this subsequent blank analysis is not free of interferences, the system must be decontaminated. Sample analysis for that target analyte must not resume until a blank is less than the reporting limits (RLs) for all target compounds. Repeat any samples suspected to have been affected by the contamination.
- Keep dilution responses of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to the specific determinative method for details on calculating analyte response.
- Dilutions must be made in Class A volumetric flasks (10 mL to 100 mL). Select the volumetric flask that allows for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.
- Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

- Inject the proper aliquot of samples into the flask. Avoid using aliquots of less than 1 mL. Dilute the sample to the mark with organic-free reagent water. Cap the flask; invert three times. Repeat the above procedure for additional dilutions. Immediately transfer to a clean VOA vial.

10.1.3 **Water-miscible liquids** are analyzed as water samples after first diluting them at least 50-fold with organic-free reagent water.

- Initial and serial dilutions are prepared by pipetting 2.0 mL of the sample into a 100-mL, Class A, volumetric flask and diluting to volume with organic-free reagent water. Invert 3 times. Transfer immediately to a clean, unused VOA vial. All steps must be performed without delays until the diluted sample is in a clean, unused VOA vial. (Intermediate dilutions may be necessary for extremely large dilutions.)

10.1.4 For the analysis of solvent **extracts from High Concentration Samples** prepared by Method 5035 / NV05-108.

- Estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume.
- If the sample was submitted as a high-concentration sample, examples of the quantity of Methanol extract required are shown in the table in this section. Start with 100.0 µL Methanol extract per 5 mL reagent water, i. e., 1.0 mL to 50 mL reagent water.
- Do not exceed 100 µL Methanol per 5 mL of water.
- All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.
- Run a method blank using 1.0 mL Methanol to 50 mL reagent water in a Class A volumetric flask. Cap and carefully invert three times before pouring into a VOA vial.
- Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks also contain the same volume of Methanol to simulate the sample conditions.

10.1.5 Low-concentration Bulk Soils: The low-concentration method is based on purging a heated sediment/soil sample mixed with Sodium bisulfate-preserved or Trisodium phosphate-preserved, organic-free reagent water containing the surrogate and, if applicable, internal and matrix spiking standards.

- Analyze all reagent blanks and standards under the same conditions as the samples.
- Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow spatula. Weigh the amount determined. See SOP 5035 / NV05-108.
- Use a 5 g sample.
- Please the VOA vial in the autosampler and start the program. The sample is heated to 40 ± 1°C as described in the determinative method.

10.2 Calibration and Sample Analysis

10.2.1 **Initial Calibration**

- Prior to using this introduction technique for any determinative method, the system must be calibrated. See the determinative method for details on calibration procedures.
- GC/MS methods require instrument tuning prior to proceeding with calibration and analysis.

- Place the final solutions (in VOA vials) containing the required concentrations of calibration standards, including surrogate standards, in the autosampling device.
- Set the autosampler to transfer 5.0 to 25 mL of organic-free water or calibration standard to the purging device.
- The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL, 10 mL, or 25 mL).
- Internal standards, if applicable, and surrogates are automatically added by the autosampler. The same amount of internal standard/surrogate must be added to each standard and sample. The surrogate and internal standards are typically mixed and added as a single spiking solution (see determinative method for details).
- Process the calibration and calculate the response factors (RF) or calibration factors (CF) for each analyte of interest. See the determinative method for specific initial calibration requirements.

10.2.2 **Calibration Verification:** To prepare a calibration verification standard, inject an appropriate volume of a primary dilution standard in an aliquot of organic-free reagent water in a 50 mL volumetric flask. Invert three times. Transfer to a clean VOA vial by slowly pouring down the side of the VOA. Cap and place in the autosampler.

- Place the sample VOA vial in the autosampler and start the method, i.e., “water” or “soil”.
- All samples and QC must be purged under the same conditions.
- Refer to the determinative method for details on ongoing calibration verification acceptance criteria. These criteria must be met prior to analyzing samples.

10.2.3 **Sample Desorption (for non-cryogenic interfaces)**

- The autosampler program automatically performs these steps. After the 11-, 12-, or 15-minute purge, the purge-and-trap system goes to desorb mode and preheats the trap to the temperature recommended for the specific trap packing material without a flow of carrier gas passing through the trap.

  Note: Some purge-and-trap systems are capable of performing a moisture removal step (e. g., dry purge) which can eliminate excess moisture from the trap and gas lines by purging the trap just prior to the desorption step. However, the utility of a moisture removal step depends on the nature of the trap packing material. In general, when using a carbon-based, hydrophobic trap packing, this step prevents moisture from entering the GC system and affecting chromatography. Optimum results are achieved through the proper choices of: the moisture control device, the trap packing material, trap temperature during moisture removal, and carrier gas flow. The use of trap back pressure control is also necessary. Consult instructions from both the manufacturer of the purge-and-trap system and the supplier of the trap packing material before employing a moisture removal step.

- The GC temperature program begins, and data acquisition is started. The carrier gas flowrate depends on the trap employed. Desorption time is 4 minutes.
- After desorbing the sample, the trap is reconditioned by reforming the purge-and-trap device to the purge mode. The trap temperature is maintained per the manufacturer’s recommendations.
- While the trap is being desorbed into the gas chromatograph, the purging chamber is drained and washed with a **minimum** of two 5 mL flushes of organic-free reagent water to minimize carryover of volatile organics into subsequent analyses.

10.2.4 **Sample Analysis**

- The samples prepared by this method are analyzed by Methods 8015, 8021, or 8260. Refer to these methods for appropriate analysis conditions. For the analysis of gasoline,

use Method 8021 with GC/PID for BTEX in series with Method 8015 with the GC/FID detector for hydrocarbons.

10.3 Example Analysis Queue / Sequence: See the determinative method.
10.4 Sample pH Determination and Residual Chlorine check: After the sample has been analyzed, check the pH of the sample using narrow-range, pH paper. Compare the test strip to the chart on the test strip container. **Record the pH on the runlog or benchsheet**. If the pH is > 2 or < 11, then a comment must be placed on the benchsheet and in LIMS. For non-preserved samples and samples from North Carolina, check for residual chlorine using test strips. Record if present and note in LIMS.

11.0 Calculations / Data Reduction

11.1 Accuracy: Not applicable.
11.2 Precision (RPD): Not applicable.
11.3 For final concentrations, see the determinative method.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.
12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.
12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required for each matrix.
12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are managed, stored, and disposed of in accordance with all federal and state laws and regulations. Waste description rules

and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- The samples are taken to the waste disposal area for neutralization and discharge to the sanitary sewer.

15.0 References / Cross-References

15.1 Method 5030A, SW-846 Update I, Revision 1, July 1992, **5030B**, SW-846 Update III, Revision 2, December 1999, and **5030C**, SW-846, May 2003.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.5 SOPs: 8015 / NV05-31, 8260 / NV05-77, 8021 / NV05-90, 5035 / NV05-108, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachment

None.

18.0 Revision History

- Revision 7, dated 10 September 2008
  - Integration for TestAmerica and STL operations.
- Revision 8, 9 October 2009
  - Update for OH VAP requirements.
- Revision 9, 28 February 2011
  - Addition of QAF-45 and Section 14.2.
  - Addition of free chlorine check in Section 10.4.
  - Removed WY batch of 10 samples requirement.
- Revision 10, 29 March 2013
  - Organizational changes.
  - Add 5030A and 5030C; low-concentration, bulk soil; compositing instructions.
  - OK no longer limits batch size to 10 samples.
  - Remove reference to the Control Limits Manual.
  - Add reference to Trisodium phosphate.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **6010 / NV06-44.14a** Effective Date: **6/28/2013**

Last Mod. Date: 5/31/13

SOP Title**: Method 6010B/C: Inductively Coupled Plasma-Atomic Emission Spectrometry**

Affected SOP Section Number(s): Sections 6.2, 10.1

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 06

Revision Number with Mod ID: **14b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change. Add underlined text; delete crossed-out text.

Section 6.2, Supplies

- Syringe filter, PTFE membrane.

Section 10.1, Sample Preparation, add the following in a paragraph below the sample size table:

Allow any undissolved material to settle overnight, filter using a PTFE membrane, or centrifuge a portion of the prepared sample until clear. Record the filter ID and lot number The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted sample cannot be characterized, all analyses should be performed as soon as possible after the completed preparation. If any sample is filtered, the Method Blank and LCS must also be filtered.

	6/24/13		6/21/13
Supervisor Approval	Date	Technical Manager Approval Quality Assurance Approval	Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **6010 / NV06-44.14** Effective Date: **5/31/2013**

Last Mod. Date: 9/14/12

SOP Title**: Method 6010B/C: Inductively Coupled Plasma-Atomic Emission Spectrometry**

Affected SOP Section Number(s): Sections 1.1, 6.1, 9.2, 10.3, 15.3

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ISSUED TO: QA Server, 06

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The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change. Add underlined text; delete crossed-out text.

- Section 1.1, Analyte, Matrices, Table 1: Remove references to ICP2.
- Section 6.1, Instrumentation, Table 3, Remove ICP2 column.
- Section 9.2, Instrument QC, Linear Range bullet, Lower Limit of Quantitation Check Sample (LLQC) bullet:
  - **Linear Range Standard (LRS):** ~~For single-point calibration, run at the beginning of the analytical sequence.~~ ~~Run~~ Use the highest standard level, to show linearity to that concentration.
  - **Lower Limit of Quantitation Check Sample (LLQC):** The lower limit of quantitation check (LLQC) sample is analyzed once daily. This check sample and the low-level calibration verification standard are prepared at the same concentrations ~~(Table 8)~~ with the only difference being the LLQC sample is carried through the digestion procedure.
- Section 10.3, Calibration Table, edit steps 1 and 5:

1	See Table 5 ~~8~~ for calibration standard preparation.
5	Run LRS standards and evaluate ~~by the criteria in Section 9~~.

- Section 15.3, ~~**TestAmerica Nashville's Control Limits Manual.**~~

5/22/13		5/22/13	
Supervisor Approval	Date	Technical Manager Approval Quality Assurance Approval	Date

Title: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY SW-846 METHOD 6010B/C

Approvals (Signature/Date)	
Rodney Street Department Supervisor	8-3-12 Date
Johnny Davis Health & Safety Manager / Coordinator	8-3-12 Date
Michael H. Dunn Quality Assurance Manager	9-7-12 Date
Michael H. Dunn Technical Director	8-3-12 Date

1.0 Scope and Application

1.1 Analyte, Matrices: The method is applicable to the elements listed below. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and SPLP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not need acid digestion. Refer to SOPs Method 3010 / NV06-18, 3015 / NV06-19, Method 3050 / NV06-93, 3051 / NV06-94, and 3005 / NV06-103 for the appropriate digestion procedures.

Table 1: Recommended Wavelengths and Typical Reporting Limits

Element	CAS #	Wavelength[a] (nm)	Typical RL (µg/L)	Typical RL (mg/kg)
Aluminum	7429-90-5	308.215	100	20
Antimony	7440-36-0	206.833	10	10
Arsenic	7440-38-2	189.0	10	1.0
Barium	7440-39-3	493.4 ICP2 233.5 ICP4,5	10	2.0
Beryllium	7440-41-7	313.042	4	1.0
Boron	7440-42-8	249.678x2	50	10
Cadmium	7440-43-9	226.502	1	1.0
Calcium	7440-70-2	373.6 ICP4, 5 317.933	1000	100
Chromium	7440-47-3	267.716	5	1.0
Cobalt	7440-48-4	228.616	20	3.0
Copper	7440-50-8	324.754	10	2.0
Iron	7439-89-6	271.4 ICP2,4,5 259.9 ICP4,5	100	20
Lead	7439-92-1	220.353	5	1.0
Lithium	7439-93-2	670.784	50	10
Magnesium	7439-95-4	279.079	1000	100
Manganese	7439-96-5	257.610	15	3.0
Molybdenum	7439-98-7	202.030	50	3.0
Nickel	7440-02-0	231.604x2	10	1.0
Potassium	7440-09-7	766.491	1000	100
Selenium	7782-49-2	196.026	10	2.0
Silver	7440-22-4	328.068	5	1.0
Sodium	7440-23-5	589.5[b] ICP ICP4,5 330.2 ICP2,4,5.	1000	200
Strontium	7440-24-6	421.5	50	10
Sulfur	7704-34-9	182.0	500	2.0
Thallium	7440-28-0	190.864	10	10
Tin	7440-31-5	189.980x2	50	10
Titanium	7440-32-6	334.941	50	10
Vanadium	7440-62-2	292.402	20	10
Zinc	7440-66-6	213.856x2 ICP4,5 206.2 ICP2	50	IS
Yttrium	7440-65-5	224.3 ICP4,5 360.0 ICP4,5	IS	IS
Scandium	7440-20-2	361.384 ICP2	IS	IS
Indium	7440-74-6	230.6	IS	

[a]The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e. g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. In time, other elements may be added as more information becomes available and as required.
[b]Highly dependent on operating conditions and plasma position.

1.2 Reporting Limits: Detection limits and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the analytical wavelengths and typical reporting limits in clean matrices.

1.3 Use of this method is restricted to analysts who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 Prior to analysis, samples are solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multi-elemental determinations by ICP-AES using simultaneous and sequential systems. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.0 Definitions

3.1 Field Reagent Blank: An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose is to determine if method analytes or other interferences are present in the field environment.

3.2 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit.

3.3 Internal Standard: Pure analyte added to a sample, in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

3.4 Linear Range (LR): The concentration range over which the instrument response to an analyte is linear.

3.5 Spectral Interference Check (SIC) Solution: Used to prepare ICSA and ICSAB. A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known inter-element spectral interferences with respect to a defined set of method criteria. See Thermo Jarrell Ash method 136972-00 revision 0 (7/28/95) for

procedure.

3.6 Inter-element Correction (IEC): Single element solutions are used to determine the appropriate location for background correction and to establish the inter-element correction routine.

3.7 Toxicity Characteristics Leaching Procedure (TCLP): An extraction process which attempts to simulate the leaching of samples into the ground/soil at a municipal landfill.

3.8 TCLP Blank Matrix: An aliquot of TCLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the TCLP extraction fluid used in the preparation of TCLP samples.

3.9 Synthetic Precipitation Leaching Procedure (SPLP): An extraction process which attempts to simulate the leaching of samples in to the soil from a rain event.

3.10 SPLP Blank Matrix: An aliquot of SPLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the SPLP extraction fluid used in the preparation of SPLP samples.

3.11 See TestAmerica Nashville's QA Manual Appendix 5 for other laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. The locations selected for the measurement of background intensity are determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

4.1.2 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences produce false positive determinations and are reported as analyte concentrations. Analysts may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. The interferences listed are only those that occur between method analyses. Only interferences of a direct overlap nature are listed.

Table 2: Potential Interferences by Analyte

Analyte	Wavelength (ηm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	XX	--	--	XX
Antimony	206.833	XX	--	XX	--	XX	--	--	--	XX	XX
Arsenic	189.0	XX	--	XX	--	--	--	--	--	XX	
Barium	493.4	--	--	--	--	--	--	--	--	--	--

Analyte	Wavelength (ηm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Beryllium	313.042	--	--	--	--	--	--	--	--	XX	XX
Cadmium	226.502	--	--	--	--	XX	--	--	XX	--	--
Calcium	317.933	--	--	XX	--	XX	XX	XX	--	XX	XX
Chromium	267.716	--	--	--	--	XX	--	XX	--	--	XX
Cobalt	228.616	--	--	XX	--	XX	--	--	XX	XX	--
Copper	324.754	--	--	--	--	XX	--	--	--	XX	XX
Iron	271.4	--	--	--	--	--	--	XX	--	--	--
Lead	220.353	XX	--	--	--	--	--	--	--	--	--
Lithium	670.784										
Magnesium	279.079	--	XX	XX	--	XX	--	XX	--	XX	XX
Manganese	257.610	XX	--	XX	--	XX	XX	--	--	--	--
Molybdenum	202.030	XX	--	--	--	XX	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	XX	--	--	--	XX	--	--	--	--	--
Sodium	588.995 / 330.2	--	--	--	--	--	--	--	--	XX	--
Sulfur	182.034										
Thallium	190.864	XX	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	XX	--	XX	--	--	--	XX	--
Zinc	213.856 / 206.2	--	--	--	XX	--	--	--	XX	--	--
Dashes indicate that no interference was observed even when interferants were introduced.											
XX - Interference may be possible.											

4.1.3 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i. e., false analyte concentrations) arising from the linear range of the interference element. Instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects **must** be evaluated for each individual instrument since the intensities vary. This evaluation is filed in the method of the instrument's computer.

4.1.4 Inter-element correction accuracy must be verified daily by analyzing spectral interference check solutions. Inter-element correction factors must be verified every **six** months or when ICSA or ICSAB fail(s) criteria.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Physical interferences are reduced by diluting the sample and by using an internal standard.

4.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by matrix matching. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory interferences result when analyses in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. This method requires a rinse period of at least 45 seconds between samples and standards.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements:

- The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- The inductively coupled plasma should only be viewed with proper eye protection from the ultraviolet emissions.
- The ICP uses high voltage.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Causes redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors causes breathing difficulties and leads to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Causes redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Inductively coupled plasma emission spectrometer, Thermo Scientific ICAP 6000 Series, or equivalent.
  - Computer-controlled emission spectrometer with background- correction capability. The spectrometer must be capable of meeting and complying with the requirements described and referenced in Section 4.0.
  - Radio-frequency generator compliant with FCC regulations.
  - Argon gas supply - High purity grade (99.99%).
  - A peristaltic pump is required to deliver both internal standard and sample solutions to the nebulizer.
  - Mass flow controller to regulate the argon flow rate, especially the aerosol transport gas.

- See Table 3 for instrument operating conditions selected as being optimal to provide the lowest reliable instrument detection limits and method detection limits.

Table 3: Inductively Coupled Plasma Instrument Operating Conditions

	ICP2	ICP4 & ICP5
Incident rf power	1100 watts	1150 watts
Reflected rf power	< 5 watts	--
Viewing height above work coil	15 mm	--
Injector tube orifice i.d.	1 mm	2 mm center tube
Argon supply	liquid Argon	liquid Argon
Argon pressure	40 psi	80+ psi
Coolant argon flow rate	19 L/min	12 L/min
Aerosol carrier argon flow rate	620 mL/min	0.5 L/min
Auxiliary (plasma) argon flow rate	300 mL/min	0.5 L/min
Sample uptake rate controlled to	1.2 mL/min	50 rpm

- Specific wavelengths are listed in Table 1.
- **Optimization of the plasma operating conditions:** This function may be performed at any time and is a routine which ensures that the wavelengths are correctly located on the detector. During this routine, the pump stops, and the nebulizer gas is turned off because the routine uses plasma wavelength positions, so no sample is required. The routine runs automatically when the plasma is ignited.

- Analytical balance, with capability to measure to 1 mg.

6.2 Supplies

For determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) must be sufficiently clean for the task objectives.

- Volumetric flasks, 25 mL, 100 mL, 200 mL, Class A.
- Adjustable Eppendorf pipettors, 10 µL - 100 µL, 100 µL - 1000 µL, with disposable plastic tips.
- Disposable serological pipettes, 1 mL, 5 mL, 10 mL.
- Graduated Cylinders, 50 mL, 250 mL, 500 mL, Class A.
- Beakers, 150 mL, with ribbed watch glass.
- Centrifuge tubes, plastic, 50 mL, certified, graduated, with screw caps.
- Watch glass, plastic, ribbed (for use with the centrifuge tubes).
- Plastic centrifuge tube racks.
- Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125 L to 1-L capacities.
- One-piece stem FEP wash bottle with screw closure, 125 L capacity.
- pH test strips.
- Teflon™ boiling chips for solid matrix blank (Chemware P/N D1069103, or equivalent).

7.0 Reagents and Standards

7.1 Reagent water, analyte-free.

7.2 Reagents may contain elemental impurities, which might affect analytical data. Only high-purity reagents that conform to the American Chemical Society specifications are used. If the purity of a reagent is in question, analyze for contamination. All acids used for this method **must be** of ultra high-purity grade or equivalent.

7.3 **Hydrochloric acid**, concentrated (sp.gr. 1.19), HCl. To prepare a 5% solution, add 10 mL concentrated HCl to 190 mL reagent water.

7.4 **Nitric acid**, concentrated (sp.gr. 1.41), HNO_3. To prepare a 5% solution, add 10 mL concentrated HNO_3 to 190 mL reagent water.

7.5 **Standard Stock Solutions**: Stock standards are purchased as certified commercial solutions at 100, 500 or 1000 µg/mL (recommended). Stock solutions are stored in FEP bottles. Replace stock standards yearly when succeeding dilutions for preparation of calibration standards cannot be verified. Store standards containing silver in the dark.

7.6 **Spectral Interference Check solutions** are prepared in the same acid mixture as the calibration standards and stored in FEP bottles. See Table 6 for SIC Preparation (ICSA and ICSAB).

7.7 **Internal Standards**: Yttrium and Indium for ICP4. Purchase Ultra Scientific, or equivalent, commercial standards at 1000 µg/mL in 2% HNO_3: IAA-249-5 for Indium; IAA-239-5 for Yttrium. Dilute 5 mL to 1 liter with 5% HNO_3 for ICP2. For ICP4 and ICP5, dilute 5 mL Yttrium and 30 mL Indium to 1 liter with 5% HNO_3.

7.8 **Inter-element Correction (IEC) Single-element Standards**: Purchase the following single-element standards at 1000 µg/mL from Ultra Scientific, or equivalent:

Element	Catalog #	Element	Catalog #	Element	Catalog #
Aluminum	IAA-213	Copper	IAA-229	Silicon	IAA-214
Antimony	IAA-251	Iron	IAA-226	Silver	IAA-247
Arsenic	IAA-233	Lead	IAA-282	Sodium	IAA-211
Barium	IAA-256	Lithium	IAA-203	Strontium	IAA-238
Beryllium	IAA-204	Magnesium	IAA-212	Sulfur	IAA-016
Boron	IAA-205	Manganese	IAA-225	Thallium	IAA-281
Cadmium	IAA-248	Molybdenum	IAA-242	Tin	IAA-250
Calcium	IAA-220	Nickel	IAA-228	Titanium	IAA-222
Chromium	IAA-224	Potassium	IAA-219	Vanadium	IAA-223
Cobalt	IAA-227	Selenium	IAA-234	Zinc	IAA-230
				Zirconium	ICP-040

7.9 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water, TCLP Extract	HDPE or Glass	125 mL	HNO_3 to pH ≤ 2[1]	6 months	SW-846 Chapter 2
Soil	HDPE or Glass	50 grams	No requirement	6 months	SW-846 Chapter 2

[1]If water samples are preserved in the lab, they should be held for at least 24 hours before analysis; record acidification start/stop time and pH. Temperature preservation is not required.

9.0 Quality Control

Refer to the quality control section of TestAmerica-Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples must be prepared with each batch of no more than 20 samples:			
Quality Controls	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Blank	1 each batch	< ½ RL or MDL whichever is greater	Re-analyze. If contamination persists, correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. If target > 10X blank, acceptable to report.
Laboratory Control Sample (LCS)[1], second source	1 each batch	80-120%[3] recovery	Correct problem then re-prep and analyze the LCS and all affected targets in the affected analytical batch. If high and ND, OK to report. For 6010C, LCS may be re-analyzed once.
Matrix Spike	1 each batch	75-125%[3] recovery	Perform post-digestion spike.
Matrix Spike Duplicate	1 each batch	75-125%[3] recovery <20[2]% RPD	Perform post-digestion spike.
Post digestion spike addition	When MS/MSD fail.	Recovery within 25% for 6010B and within 20% for 6010C of the expected results.	Perform dilution test.
Dilution test	If MS/MSD fail.	1:4 fold dilution (5X) must agree within 10% of the original determination.	Qualify results.

[1]AZ, TX, WV require an LCS duplicate in each batch.
[3]If historical limits are calculated, they cannot exceed these limits.

- **Method Blank:** The laboratory prepares and analyzes one blank (reagent water or Teflon™ boiling chips) with each batch of up to 20 samples of the same matrix.
- A **Laboratory Control Sample (LCS)** must be analyzed with every batch. See Table 4 for LCS preparation using 50 mL reagent water for water batches and 0.5 gram Teflon™ chips for soil batches.
- **Matrix Spike / Matrix Spike Duplicate:** Analyze a matrix spike and matrix spike duplicate at a frequency of one per matrix batch up to 20 samples. In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determinations added prior to sample preparation. See SOP Sample Homogenization, Subsampling, and Compositing / NV08-229. The added analyte concentration and standard source must be the same as that used in the LCS (Table 4).
  - For each batch, check for matrix effects as follows:
    - If MS/MSD is outside the QC limits, the same sample from which the MS/MSD aliquots were prepared is also spiked with a **post-digestion spike** (i. e., nominally add 250 µL of the LCS/MS/MSD solution to 25 mL digested aliquot). Otherwise, another sample from the same preparation is used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and is recovered as described in the above table. The spike addition produces a minimum level of 10 times and a

maximum of 100 times the lower limit of quantitation. If this spike fails, then the dilution test is run on this sample. If both the MS/MSD and the post-digestion spike fail, then matrix effects are confirmed.

- **Dilution test**: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (5X) should agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

9.2 Instrument QC

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Inter-element Correction, single element standards	6 months	± RL	Alter wavelength or background correction.
Spectral Interference Check Solutions, A and AB	Beginning and end of each day or every 8 hours	Target ± 2 times RL or ± 20% true.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples.
Instrument Detection Limits (IDL)	Quarterly	±3 standard deviations of the average response.	Re-run IDL. If > MDL, adjust MDL to equal IDL.
Independent Calibration Verification Sample (ICV), second source	Immediately after calibration	90-110 % recovery	Correct problem then repeat initial calibration.
Independent Calibration Blank (ICB)	Immediately after ICV	No target analytes above lower limit of quantitation	Correct problem, re-calibrate.
Linear Range Standard	Daily	90-110%	Repeat calibration (ICP2).
Continuing Calibration Verification Sample (CCV)	Every 10 samples and at the end of the run	90-110% true	Re-analyze once. If fails again, then repeat calibration and re-analyze all samples since last successful CCV.
Undigested Low Level Continuing Calibration Verification (LLCCV)	Beginning and end of each batch.	70-130% true	Re-calibrate.
Continuing Calibration Blank	Following the CCV	No target analytes above lower limit of quantitation	Correct problem then analyze calibration blank and previous 10 samples.
MDL Verification (digested)	Yearly	Detected	Re-evaluate MDL standard used and MDL; see Technical Director
Digested Lower Limit of Quantitation Check (LLQC) or Report Limit Verification (RLV)	Once daily	70-130% true	Re-calibrate.
Internal Standards	All samples, standards, QC	60-140% true	Dilute and re-run. For blank and LCS, correct problem and re-run batch.

- **Inter-element Correction (IEC):** When correction is appropriate, the concentration of all targets must be within ± the RL, i. e., RL = 0.01, acceptance is -0.01 to +0.01. Once established, the entire routine must be initially and periodically verified every six months, or whenever there is a change in instrument operating conditions or when ICSA or ICSAB fail criteria.

Single element standard concentrations that must be analyzed every six months are shown as follows:

Element	Required Final Concentration (mg/L)	Stock Standard Solution (µg/mL)	Volume of Stock Standard Solution (mL)	Final Volume (mL)
Aluminum	50	1000	25	50
Antimony	10	1000	0.5	50
Arsenic	50	1000	2.5	50
Barium	50	1000	2.5	50
Beryllium	10	1000	0.5	50
Boron	50	1000	2.5	50
Cadmium	10	1000	0.5	50
Calcium	100	1000	5.0	50
Chromium	50	1000	2.5	50
Cobalt	50	1000	2.5	50
Copper	50	1000	2.5	50
Iron	100	1000	5.0	50
Lead	50	1000	2.5	50
Lithium	25	1000	1.25	50
Magnesium	100	1000	5.0	50
Manganese	10	1000	0.5	50
Molybdenum	10	1000	0.5	50
Nickel	50	1000	2.5	50
Potassium	100	1000	5.0	50
Selenium	20	1000	1.0	50
Silicon	50	1000	2.5	50
Silver	10	1000	0.5	50
Sodium	100	1000	5.0	50
Strontium	50	1000	2.5	50
Sulfur	100	1000	5	50
Thallium	20	1000	1.0	50
Tin	50	1000	2.5	50
Titanium	50	1000	2.5	50
Vanadium	50	1000	2.5	50
Zinc	10	1000	0.5	50
Zirconium	50	1000	2.5	50

If interferences are observed, they must be mitigated by the use of interference correction equations or by changing to a different wavelength. The objective is to reduce interferences.

Required ICSA and ICSB solutions are described in Table 6 for daily verification. Initial and periodic verification data of the routine are filed in the maintenance of the instrument's computer.

- **Spectral interference check solution (ICSA and ICSAB):** The laboratory must periodically verify the inter-element correction routine by analyzing SIC solutions. The spectral interference check solution is run at the beginning and end of the day's sequence or every 8 hours, whichever is more frequent. If the SIC does not meet criteria, then the SICs are re-

analyzed. These solutions are used to periodically verify a partial list of the on-line (and possible off-line) inter-element spectral correction factors for the recommended wavelengths given in Table 1.

NOTE: The SIC solution must be analyzed more than once to confirm a change has occurred with adequate rinse time between solutions and before subsequent analysis of the calibration blank.

- Ensure that the analytical results of ICS Solution A (ICSA) fall within the control limit of ± 2 times the RL of the analyte's true value or ± 20% of the analyte's true value, whichever is greater (the true value is zero unless otherwise stated) in the ICSA. For example, if the analysis result(s) for Arsenic (RL = 10 µg/L, ICSA true value = 0 µg/L) in the ICSA analysis during the run is 19 µg/L, then the analytical result for Arsenic falls within the ± 2 times the RL window for Arsenic in the ICSA. If the analytical results of the ICSA do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the analytical samples analyzed since the last compliant ICSA was performed.
- Ensure that the results for the ICS Solution AB (ICSAB) during the analytical runs fall within the control limit of ± 2 times the RL of the true value or ± 20% of the true value, whichever is greater, for the analytes included in the ICSAB. If the analytical results of the ICSAB do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the analytical samples analyzed since the last compliant ICSAB was performed.

- **Instrument Detection Limits (IDL):** IDLs in µg/L are estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Perform each measurement as though it were a separate analytical sample (i. e., follow each measurement by a rinse and/or any other procedure normally performed between the analysis of separate samples). Determine IDLs at least every three months and keep with the instrument logbook. Compare the calculated IDLs to the MDLs. MDLs are equal to or greater than the IDL.
- **Independent Calibration Verification (ICV and ICB):** The laboratory analyzes the ICV and a calibration blank (ICB) immediately following daily calibration. See Table 7 for ICV preparation using a second-source standard. The ICB must not contain target analytes above the lower limit of quantitation.
- **Linear Range Standard (LRS):** For single-point calibration, run at the beginning of the analytical sequence. Run the highest standard level, to show linearity to that concentration. All samples exceeding **90%** this concentration are diluted. The LRS concentrations are Al, Fe, Ca and Mg at 200 µg/mL, Na, K, at 100 µg/mL, Ba at 50 µg/mL, Ag at 5 µg/mL, and all other elements at 10 µg/mL. The standard must be within 10% of the true values to continue.
- **Undigested Low-Level Continuing Calibration Verification (LLCCV):** Analyze an undigested LLCCV at the beginning and end of each batch. Prepare it from the primary calibration standard at the RL.
- **Continuing Calibration Verification (CCV and CCB):** Analyze after every 10th sample and at the end of the analytical sequence.
  - See Table 5 for CCV preparation using the primary source standard at the mid-point of the calibration curve. All samples **must be bracketed** by acceptable CCVs and CCBs.
  - The CCB (prepared by adding 25 mL concentrated HNO_3 to 500 mL reagent water) must not contain target analytes above the MDL or ½ the RL, whichever is greater. If the criterion is not met, terminate the analysis, correct the problem, and re-analyze the previous 10 samples.

- **MDL Verification:** A solution containing all target analytes at 2-3 times the MDL must be **digested** and analyzed after the completion of the MDL study and on an annual basis. Detection limits are verified when all analytes in the MDL check solution are detected.
- **Lower Limit of Quantitation Check Sample (LLQC):** The lower limit of quantitation check (LLQC) sample is analyzed once daily. This check sample and the low-level calibration verification standard are prepared at the same concentrations (Table 8) with the only difference being the LLQC sample is carried through the digestion procedure.
- **Internal Standards:** Use the internal standard technique by adding one or more elements (not in the samples and verified not to cause an uncorrected inter-element spectral interference) at the same concentration (which is sufficient for optimum precision) to the prepared samples (blanks and standards) that are affected the same as the analytes by the sample matrix. Use the ratio of analyte signal to the internal standard signal for calibration and quantitation. Internal standards (Yttrium and Indium) are automatically added to all calibration standards, samples, and QC, by the instrument.

10.0 Procedure

10.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been pre-filtered and acidified do not need acid digestion. Samples which are not digested must use an internal standard. See Section 1.1 for digestion SOPs.

Matrix	Sample Size
Water	50 mL of sample
TCLP Extract	10 mL of extract
Soil	0.5 gram of sample

10.2 Instrument Setup

1	Inspect the sample introduction system including the nebulizer, torch, center tube, and uptake tubing for salt deposits, dirt and debris that would restrict solution flow and affect instrument performance. Clean the system when needed or on a daily basis.
2	Power up all accessories and the unit. Allow the instrument to become thermally stable before beginning (usually requiring about 30 minutes of operation prior to calibration).
3	Click on the plasma icon in the lower right of the Analyst screen. Then click the instrument status button. Make sure all interlocks have a green light. Then push the plasma on button. Confirm flow to the plasma.
4	To shutdown, click the plasma icon and push the plasma off button.
5	Instrument is automatically optimized when the program is opened.
6	Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The analyst must follow the instructions provided by the instrument manufacturer and this SOP. For the 6500 series, the conditions usually vary from 750-1350 watts power, 10-20 liters/minute coolant gas flow, 0.25-1.5 liters/minute nebulizer gas flow, 0.25-2 liters/minute auxiliary gas flow, 20-125 rpm pump rate with a 40 second flush time and 30 seconds maximum integration time. Perform two replicate integrations; report the average.
7	Establish sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

8	All samples that exceed the upper calibration standard must be diluted and re-analyzed or a linear range standard must be run with 10% of the true value.

10.3 Calibration: Refer to SOPs Calibration Curves (General) / CA-Q-S-005 and Selection of Calibration Points / CA-T-P-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1	See Table 8 for calibration standard preparation. For multi-point calibration, use first-order linear regression (**$r \geq 0.998$, $r^2 \geq 0.996$**). The lowest non-zero standard concentration is considered the lower limit of quantitation, i. e., RL. Higher order fits are not allowed.
2	The absolute value of the results of the calibration blank is less than the value of the MDL.
3	After initial calibration, the calibration curve must be verified by use of an initial calibration verification (ICV) standard. The calibration curve must be verified at the end of each analysis batch and after every 10 samples by use of a continuing calibration verification (CCV) standard and a continuing calibration blank (CCB).
4	The calibration curve must also be verified prior to the analysis of any samples and at the end of the batch by use of a low-level continuing calibration verification (LLCCV) standard.
5	Run LRS standards and evaluate by the criteria in Section 9.
6	Verify the inter-element correction factors at the beginning and end of the daily sequence or every 8 hours.

10.4 Sample Analysis

- Follow the analysis sequence in Table 9.
- Samples exceeding the 90% of the LRS are diluted and rerun.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{\% Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Response of the analyte.
A_{is} = Response of the internal standard.
C_s = Concentration of the analyte.
C_{is} = Concentration of the internal standard

11.4 % Drift

% Drift = (Result - True Value) x 100
True Value

11.5 **Linear Calibration Using a Least Squares Regression**: This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$y = ax + b$

y = instrument response (peak area)
a = slope of the line
x = concentration of the calibration standard
b = the intercept

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

11.6 **Coefficient of Determination** **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.7 **Concentration:** Sample data are reported in units of mg/L for aqueous samples, mg/kg for solid samples. LIMS calculates the concentration from the raw data provided by the analyst.

Concentration (mg/L or mg/kg) = (µg/mL* from instrument)(digest volume, mL)(Dilution factor)
Sample Volume (mL) or Mass (g)

µg/mL from instrument = y = mx + b

***average of two replicates**

11.8 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the MDL.

11.9 Account for any additional dilution of the prepared sample solution needed to complete the determination of analytes exceeding 90% or more of the LRS upper limit. Do not report data below the determined analyte MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required to complete the analysis.

12.0 **Method Performance**

12.1 **Method Detection Limit Study (MDL):** The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99%

confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

Quarterly, run 7 IDLs on three non-consecutive days; calculate the standard deviation per day. Compare three times the average of the standard deviations to the MDL. If 3xIDL > MDL, rerun the IDLs. If the finding persists, select the higher of the two as the "working MDL" for that quarter.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

12.5 Control Charts: Laboratory method performance can be shown with the use of control charts, available from LIMS or the QA department.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Acidic aqueous wastes are taken to the waste disposal area, neutralized, and discharge to the sanitary sewer.

15.0 References / Cross-References

15.1 Method 6010B, SW-846 Update III, Revision 2, December 1996, and **Method 6010C**, SW-846 Update IV, Revision 3, February 2007.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 TestAmerica Nashville's Control Limits Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Calibration Curves (General) / CA-Q-S-005, Selection of Calibration Points / CA-T-P-002, Method 3005 / NV06-103; Method 3010 / NV3010; Method 3050 / NV06-93; Method 3051

/ NV06-94; Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Sample Homogenization, Subsampling, and Compositing / NV08-229, Reagent and Standard Purchase / NV08-214.

15.5 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	If 3030C digestion is specified, see the attachment in SOP 3005 / NV06-103 for that procedure.
2	For OK, NV, SC, and WY samples, reference 6010C. For all other states, reference 6010B.
3	Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.

17.0 Attachments

Table 4: LCS and MS/MSD Spiking Solution
Environmental Express HP3948
Use 0.5 mL as received

Analyte	Stock Std Concentration (µg/mL)	Final Concentration of Digestate (µg/mL)
Aluminum	200	2.0
Antimony	10	0.1
Arsenic	5	0.05
Barium	200	2.0
Beryllium	5	0.05
Boron	100	1.0
Cadmium	5	0.05
Calcium	500	5.0
Chromium	20	0.2
Cobalt	50	0.5
Copper	25	0.25
Iron	100	1.0
Lead	5	0.05
Lithium	100	1.0
Magnesium	500	5.0
Manganese	50	0.5
Molybdenum	50	0.5
Nickel	50	0.5
Potassium	500	5.0
Silver	5	0.05
Sodium	500	5.0
Sulfur	100	1.0
Thallium	5	0.05
Titanium	100	1.0
Vanadium	50	0.5
Zinc	50	0.5

Table 5: Calibration Solutions

Analyte	ICAL 1	ICAL 2	ICAL 3	ICAL 4	ICAL 5
Al, Sb, Ar, Ba, Be, B, Cd, Co, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, NI, Ag, Tl, S, Sn, Ti, V, Zn	ICUS 2648: 2.0 µg/mL	Dilute 20 mL ICUS 2648/ 40 mL: 1.0 µg/mL	Dilute 10 mL ICUS 2648/ 40 mL: 0.5 µg/mL		
Ba	ICUS 3033: 10.0 µg/mL				
Al	ICUS 3033: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
K	ICUS 3033: 10.0 µg/mL	ICUS 2614: 100 µg/mL			
Mg,	ICUS 3033: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
Ca	ICUS 3033: 10.0 µg/mL		5 mL 1000 µg Ca /mL stock/50 mL: 100 µg/mL	15 mL 1000 µg Ca /mL stock/50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL
Fe	ICUS 3033: 10.0 µg/mL	2.5 mL 1000 µg Fe/mL stock/50 mL: 50 µg/mL	5 mL 1000 µg Fe /mL stock/ 50 mL: 100 µg/mL	ICUS 2614: 200 µg/mL	
Na	ICUS 3033: 10.0 µg/mL		5 mL 1000 µg Na /mL stock/ 50 mL: 100 µg/mL	15 mL 1000 µg Na /mL stock/ 50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL
Sulfur	0.02 mL/40 mL 0.5 µg/mL	0.1 mL/50 mL 2.0 µg/mL	ICUS 3033 10.0 µg/mL	2.5 mL/50 mL 50 µg/mL	5 mL/50 mL 100 µg/mL
Silicon	0.5 mL 1000 µg/mL stock/ 10.0 µg/mL				

CCV and RLV Sources and Concentrations

Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Aluminum	10.0	0.1
Antimony	1.0	0.01
Arsenic	1.0	0.01
Barium	2.0	0.01
Beryllium	1.0	0.004
Boron	1.0	0.05

Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Cadmium	1.0	0.001
Calcium	10.0	1.0
Chromium	1.0	0.005
Cobalt	1.0	0.02
Copper	1.0	0.01
Iron	10.0	0.05
Lead	1.0	0.005
Lithium	1.0	0.05
Magnesium	10.0	1.0
Manganese	1.0	0.015
Molybdenum	1.0	0.05
Nickel	1.0	0.01
Potassium	10.0	1.0
Selenium	1.0	0.01
Silver	1.0	0.005
Sodium	10.0	1.0
Strontium	1.0	0.05
Sulfur	1.0	0.5
Thallium	1.0	0.01
Tin	1.0	0.05
Titanium	1.0	0.05
Vanadium	1.0	0.02
Zinc	1.0	0.05

Table 6: Spectral Interference Check Solutions

ICSA Standard

ICSA Stock, SPEX # INT-A1

ICSA Solution: Dilute 25 mL of ICSA Stock + 12.5 mL HNO_3 to 250 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSA Conc. (µg/mL)
Aluminum	5000	500
Calcium	5000	500
Magnesium	5000	500
Iron	2000	200

ICSAB Standard

ICSA Stock, SPEX # INT-A1
ICSB Stock, SPEX # INT-B1
ICSB2 Stock, SPEX # INT-B2

ICSAB Solution*: Dilute 25 mL of ICSA Stock + 2.5 mL of ICSB Stock + 2.5 mL of ICSB2* Stock + 12.5 mL HNO_3 to 250 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSB Stock Conc. (µg/mL)	ICSB2 Stock Conc. (µg/mL)	ICSAB Conc. (µg/mL)
Aluminum	5000		100	501
Calcium	5000		10	500.1
Magnesium	5000		10	500.1
Iron	2000		10	200.1
Barium		50		0.5
Beryllium		50		0.5
Cadmium		100		1.0
Chromium		50		0.5
Cobalt		50		0.5
Copper		50		0.5
Lead		100		1.0
Manganese		50		0.5
Nickel		100		1.0
Silver		100		1.0
Vanadium		50		0.5
Zinc		100		1.0
Antimony			100	1.0
Arsenic			100	1.0
Boron			100	1.0
Molybdenum			100	1.0
Selenium			100	1.0
Silica			10	0.1
Sodium			100	1.0
Thallium			100	1.0

Table 7: ICV Standard
Source: Inorganic Ventures STLTN-CAL-3 for all except Tin
Ultra Scientific 1AA-250, 1000 µg/mL for Tin
ICV Preparation: 0.05 mL Tin standard to 50 mL STLTN-CAL-3
Environmental Express HP100054-5, 1000 µg/mL for Sulfur
ICV Preparation: 0.05 mL Sulfur standard to 50 mL STLTN-CAL-3

Analyte	STLTN-CAL-3 (µg/mL)	Ultra 1AA-250 Sn (µg/mL)	Environmental Express HP100054 (µg/mL)	ICV Conc. (µg/mL)
Aluminum	10			10
Antimony	1			1
Arsenic	1			1
Barium	2			2
Beryllium	1			1
Boron	1			1
Cadmium	1			1
Calcium	10			10
Chromium	1			1
Cobalt	1			1
Copper	1			1
Iron	10			10

Analyte	STLTN-CAL-3 (µg/mL)	Ultra 1AA-250 Sn (µg/mL)	Environmental Express HP100054 (µg/mL)	ICV Conc. (µg/mL)
Lead	1			1
Lithium	1			1
Magnesium	10			1
Manganese	1			1
Molybdenum	1			1
Nickel	1			1
Potassium	10			10
Selenium	1			1
Silver	1			1
Sodium	10			10
Strontium	1			1
Sulfur		1000		1
Thallium	1			1
Tin			1000	1
Titanium	1			1
Vanadium	1			1
Zinc	1			1

Table 8: Linear Range Standard/High Calibration Standards

Analyte	ICUS 2614 Conc. (µg/mL)
Al, Ca, Mg, Na	500
Fe	200
K	100

Table 9: Typical Analytical Sequence

	Definitions
0.0 mg/L standard(or blank)	Calibration Standard
0.5 mg/L	Calibration Standard
1.0 mg/L	Calibration Standard
2.0 mg/L	Calibration Standard
10.0 mg/L	Calibration Standard
LRS	High Range Calibration Verification
100 mg/L	Calibration Standard
300 mg/L	Calibration Standard
50 mg/L	Calibration Standard
ICV	Independent (initial) Calibration Verification
ICB	Initial Calibration Blank
RLV	Report Limit Verification
ICSA	Initial Spectral Interference Check
ICSAB	Initial Spectral Interference Check
Up to 10 samples	
MS	Matrix Spike

MSD	Matrix Spike Duplicate
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
LCS	Laboratory Control Sample
RLV (as needed)	Report Limit Verification
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
ICSA	Spectral Interference Check
ICSAB	Spectral Interference Check
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
CCV	Final Continuing Calibration Verification
CCB	Final Continuing Calibration Blank
RLV	Final Report Limit Verification
ICSA	Final Spectral Interference Check
ICSAB	Final Spectral Interference Check

18.0 Revision History

- Revision 10, dated 29 February 2008
  - Integration for TestAmerica and STL operations.
  - Addition of control limits for ICSA and ICSAB.
  - Addition of text on Spectral Interference Check Solutions
  - Change from CRDL/CRQL to CRL, current terminology
  - Addition of current catalog numbers for Table 7 individual elements.
  - Correction of order of Table 9
- Revision 11, dated 17 October 2008
  - Updated to SW-846 Update IV
- Revision 12, dated 30 October 2009
  - Inserted information on new ICV standard, new standards for ICP4.
  - Modified analytical sequence and added ICP4 sequence.
  - Reformatting and condensing.
  - Addition of requirements of Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.
  - Corporate review.
- Revision 13, dated 21 February 2011
  - Addition of QAF-45 and Section 14.2
  - Addition of Sulfur information, updated wavelengths in Table 1.
  - Addition of operating conditions optimization for ICP4 and 5.
  - New vendor IDs for elements.
  - Delete ICP1, taken out of service.
  - ICP2 new calibration standard preparation; changed SIC solution preparation.

- Revision 14, dated 14 September 2012
  - Organizational changes.
  - Amendments 13a,b,c.
  - Iron RL change from 50 to 100 µg/L; from 10 mg/kg to 20 mg/kg.
  - Removal of ICP2.
  - Addition of Sulfur throughout.
  - Remove the requirement for 10-sample batches if samples are from OK or WY.
  - Addition of SOPs Calibration Curves (General) / CA-Q-S-005, and Selection of Calibration Points / CA-T-P-002. Remove 3015 / NV06-19 (archived).

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8260 / SA/NV05-77.18** Effective Date: **9/30/2013**

Last Mod. Date: 8/30/13

SOP Title: **Method 8260B: Volatile Organic compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 16.0, Method Modifications: TPH-GRO by Method 8260B

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 05V

Revision Number with Mod ID: **18a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add underlined, delete crossed-out. *(Italics are for 8260C.*

- Section 1.2, Reporting Limits

Compound	Retention Time (minutes)	Typical Reporting Limits: Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
tert-Amyl-alcohol (TAA)[3]	2.914	20	20	20 ~~2~~
Isopropyl alcohol	3.836	50 ~~20~~	50	50
Allyl chloride (3-Chloro-1-propene)	4.026	2	2 ~~10~~	10
3,3-Dimethyl-1-butanol	5.021	10	10	NA ~~0~~

- Section 9.2: Instrument QC Table, ICAL acceptance criteria

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Minimal five-point initial calibration for all target analytes. Single-point surrogate calibration	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8260B: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound, ~~and~~ %RSD ~~for RFs~~ for CCCs ≤ 30% and all other target analytes %RSD ~~for RF~~ ≤ 15%~~.~~ or correlation coefficient r^2 ≥ 0.990 or r ≥ 0.995. Re-calculate low point; must be within 30% true. *8260C: Minimum RF for initial and continuing calibration varies by analyte (see Calibration standards below). RSD ≤ 20% each target or correlation coefficient r^2 ≥ 0.990 r ≥ 0.995. Up to 10% of targets may exceed these*	Correct problem then repeat initial calibration.

		criteria. If using linear regression, re-fit lowest calibration point. It must be <u>within</u> ± 30% <u>of true</u> ~~or re-calculate~~.	

- Section 9.2: Instrument QC, **Initial Calibration Check Compounds (CCCs):**

  - *For 8260C, the minimum RF for initial and continuing calibration <u>must be</u> ~~is~~:*

RF	For These Compounds
<u>0.05</u>	*<u>1,2-Dichloro-3-propane</u>*
0.1	*<u>RF may be lower if data is used for screening: Acetone, Bromoform, Bromomethane, 2-Butanone, Carbon disulfide, Carbon tetrachloride, Chloroethane, Chloromethane, Cyclohexane, Dibromochloromethane, Dichlorodifluoromethane, 1,2-Dichloroethane, 1,1-Dichloroethene, cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, trans-1,3-Dichloropropene, Ethylbenzene, Ethylene dibromide, 2-Hexanone, Isopropylbenzene, Methyl acetate, 4-Methyl-2-butanone, Methylene chloride, Methyl cyclohexane, Methyl tert-butyl ether, m & p-Xylene, 1,1,1-trichloroethane, 1,1,2-Trichloroethane, 1,1,2-Trichloro-1,2,2-trifluoroethane</u>*
0.2	*1,1-Dichloroethane, Chloroform, Trichlorethene, Bromodichloromethane, cis-1,3-Dichloropropene, Tetrachloroethane, and 1,2,4-Trichlorobenzene*
0.3	*o-Xylene, Styrene, 1,1,2,2-Trichloroethane*
0.4	*Toluene, 1,2-Dichlorobenzene*
0.5	*Benzene, Chlorobenzene, 1,4-Dichlorobenzene*
0.6	*1,3-Dichlorobenzene*

    - ~~*For 8260C, the must be less than or equal to 20% for each target analyte with up to 10% of compounds meeting the 40% criterion.*~~

- Section 9.2: Instrument QC, **Initial Calibration Verification (ICV),** second bullet for 8260B:

  - The ICV of each target must be within 30% of the expected value, with the exception of the following poor purge efficiency analytes that may be within 40% of the expected value ~~for up to 20% of targets:~~. <u>No more than 20% of analytes are allowed to fail this criterion.</u>

Acrolein	Ethanol	2-Methylnaphthalene
t-Amyl alcohol (TAA)	t-Butyl formate (TBF)	Vinyl acetate
t-Butyl alcohol (TBA)	1-Methylnaphthalene	

- Section 9.2: Instrument QC, **Continuing Calibration Verification (CCV),** 8260C bullet:
  - *For 8260C, ~~see ICAL and ICV information.~~<u>the CCV % difference for each target must be ≤ 20% with only up to 20% of the targets of interest allowed to exceed 20% difference. The minimum RF must also be achieved.</u>*

John Kelley 9/17/13	Glen L. Norton 9/26/13
Department Manager Approval Date	Operations Manager Approval Date
Michael A. Burns 9/16/13	
Technical Director, Quality Manager Approval Date	

Title: VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) SW-846 METHOD 8260B/C

Approvals (Signature/Date)

John Haley	8/7/13	Glenn L. Norton	8/5/13
John Haley Department Supervisor	Date	Glenn Norton Volatiles Operations Manager	Date
Michael H. Dunn	8/30/13	Johnny Davis	8/15/13
Michael H. Dunn\ Technical Director Quality Assurance Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine volatile organic compounds in a variety of matrices; it is applicable to nearly all types of samples, regardless of water content, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

CAS No. (a)	Compound	CAS No. (a)	Compound
630-20-6	1,1,1,2-Tetrachloroethane[1, 2, 6, 7]	156-59-4	cis-1,2-Dichloroethene[1, 2, 5, 6, 7]
71-55-6	1,1,1-Trichloroethane[1, 2, 5, 6]	10061-01-5	cis-1,3-Dichloropropene[1, 2, 5, 6, 7]
79-34-5	1,1,2,2-Tetrachloroethane[1, 2, 5, 6, 7]	110-82-7	Cyclohexane[4, 5]
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane[5,6,7]	108-94-1	Cyclohexanone[4]
79-00-5	1,1,2-Trichloroethane[1, 2, 5, 6]	74-95-3	Dibromomethane[1, 2, 6, 7]
75-34-3	1,1-Dichloroethane[1, 2, 5, 6, 7]	75-27-4	Dichlorobromomethane[1,2, 6, 7]
75-35-4	1,1-Dichloroethene[1, 2, 5, 6, 7]	75-71-8	Dichlorodifluoromethane[1, 2, 5, 7]
563-58-6	1,1-Dichloropropene[1, 7]	75-43-4	Dichlorofluoromethane[4]
87-61-6	1,2,3-Trichlorobenzene[1]	64-17-5	Ethanol[3]
96-18-4	1,2,3-Trichloropropane[1, 2, 6, 7]	141-78-6	Ethyl acetate [4]
526-73-8	1,2,3-Trimethylbenzene	140-88-5	Ethyl acrylate
120-82-1	1,2,4-Trichlorobenzene[1, 2, 5]	60-29-7	Ethyl ether (Diethyl ether)[4]
95-63-6	1,2,4-Trimethylbenzene[1, 9]	97-63-2	Ethyl methacrylate[2, 7]
96-12-8	1,2-Dibromo-3-chloropropane[1, 2, 5, 7]	100-41-4	Ethylbenzene[1, 2, 5, 6, 7, 8, 9]
95-50-1	1,2-Dichlorobenzene[1, 2, 5, 6, 7]	106-93-4	Ethylene dibromide (EDB, 1,2-Dibromoethane)[2]
107-06-2	1,2-Dichloroethane[1, 2, 5, 6, 7, 8]	87-68-3	Hexachlorobutadiene[1, 2]
78-87-5	1,2-Dichloropropane[1, 2, 5, 6, 7]	110-54-3	Hexane[4]
176-02-8	1,3,5-Trichlorobenzene[4]	74-88-4	Iodomethane[2, 6, 7]
108-67-8	1,3,5-Trimethylbenzene[1, 9]	78-83-1	Isobutyl alcohol [2, 7]
541-73-1	1,3-Dichlorobenzene[1, 2, 5, 7]	67-63-0	Isopropy alcohol[4]
142-28-9	1,3-Dichloropropane[1, 7]	180-20-3	Isopropyl ether (IPE, Di-isopropyl ether)[3]
106-46-7	1,4-Dichlorobenzene[1, 2, 5, 6, 7]	98-82-8	Isopropylbenzene (Cumene)[1, 5, 9]
123-91-1	1,4-Dioxane[2, 8]	126-98-7	Methacrylonitrile[2, 7]
590-20-7	2,2-Dichloropropane[1, 7]	79-20-9	Methyl acetate[5]
78-93-3	2-Butanone (MEK)[1, 2, 5, 6, 7, 8]	80-62-6	Methyl methacrylate[2, 7]
126-99-8	2-Chloro-1,3-butadiene (Chloroprene)[2, 7]	1634-04-4	Methyl-t-butyl ether[1, 3, 4, 5, 9]
110-75-8	2-Chloroethyl vinylether[4]	108-87-2	Methylcyclohexane[5]
95-49-8	2-Chlorotoluene[1]	75-09-2	Methylene chloride[1, 2, 5, 6, 7]
591-78-6	2-Hexanone[1, 2, 5, 6, 7]	108-38-3	m-Xylene [9]
75-65-0	2-Methyl-2-propanol (tert-Butyl Alcohol)[3]	91-20-3	Naphthalene[1, 2, 9]

CAS No. (a)	Compound	CAS No. (a)	Compound
91-57-6	2-Methylnapthalene[4]	71-36-3	n-Butanol (n-Butyl Alcohol)[4]
79-46-9	2-Nitropropane[4]	123-86-4	n-Butyl acetate[4]
624-95-3	3,3-Dimethyl-1-butanol[3]	104-51-8	n-Butylbenzene[1, 9]
107-05-1	3-Chloro-1-propene (Allyl chloride)[2,7]	142-82-5	n-Heptane[4]
106-43-4	4-Chlorotoluene[1]	103-65-1	n-Propylbenzene[1, 9]
99-87-6	4-Isopropyltoluene (p-Isopropyltoluene)[1, 9]	95-47-6	o-Xylene[9]
108-10-1	4-Methyl-2-pentanone (MIBK)[1, 2, 5, 6, 7]	76-01-7	Pentachloroethane
67-64-1	Acetone[1, 2, 5, 6, 7]	107-12-0	Propionitrile[2, 7]
75-05-8	Acetonitrile[2, 7]	135-98-8	sec-Butylbenzene[1, 9]
107-02-8	Acrolein (Propenal)[2, 7]	100-42-5	Styrene[1, 2, 5, 6, 7]
107-13-1	Acrylonitrile[2, 6, 7]	75-85-4	tert-Amyl-alcohol (TAA)[3]
71-43-2	Benzene[1, 2, 5, 6, 7, 8, 9]	994-05-8	tert-Amyl-methyl-ether (TAME)[3]
100-44-7	Benzyl chloride[4]	637-92-3	tert-Butyl ethyl ether (Ethyl-tert-butyl-ether, ETBE)[3]
108-86-1	Bromobenzene[1]	762-75-4	tert-Butyl-formate (TBF)[3]
75-25-2	Bromoform[1, 2, 5, 6, 7]	98-06-6	tert-Butylbenzene[1, 9]
74-83-9	Bromomethane[1, 2, 5, 6, 7]	127-18-4	Tetrachloroethene[1, 2, 5, 6, 7]
106-99-0	Butadiene	109-99-9	Tetrahydrofuran[4]
STL00350	C4-C12	108-88-3	Toluene[1, 2, 5, 6, 7, 8, 9]
80006-61-9	C6-C10	156-60-5	trans-1,2-Dichloroethene[1, 2, 5, 6, 7]
75-15-0	Carbon disulfide[1, 2, 5, 6, 7, 8]	10061-02-6	trans-1,3-Dichloropropene[1, 2, 5, 6, 7]
56-23-5	Carbon tetrachloride[1, 2, 5, 6, 7]	110-57-6	trans-1,4-Dichloro-2-butene[2, 6, 7]
108-90-7	Chlorobenzene[1, 2, 5, 6, 7, 8]	79-01-6	Trichloroethene[1, 2, 5, 6]
74-97-5	Chlorobromomethane[1, 6, 7]	75-69-4	Trichlorofluoromethane[1, 2, 5, 6, 7]
124-48-1	Chlorodibromomethane[1, 2, 5, 6, 7]	108-05-4	Vinyl acetate[2, 6, 7]
75-00-3	Chloroethane[1, 2, 5, 6, 7]	75-01-4	Vinyl chloride[1, 2, 5, 6, 7]
67-66-3	Chloroform[1, 2, 5, 6, 7, 8]	1330-20-7	Xylene (total)[1, 2, .5, 6, 7, 8, 9]
74-87-3	Chloromethane[1, 2, 5, 6, 7]		

[1] - Laboratory normal 8260 compound
[2] - Appendix IX compound
[3] - Oxygenate
[4] - Additional compounds by request only
[5] TCL list (OLM 04.2 list)
[6] - Appendix I compound
[7] -Appendix II compound
[8] - Skinner list
[9] - NY Stars List
a = Chemical Abstract Service Registry Number0

1.2 Reporting Limits: The RL for an individual compound is instrument-dependent and also dependent on the choice of sample preparation/introduction method. The RL analyte concentration is defined by the lowest non-zero standard in the calibration curve. Using standard quadrapole instrumentation and the purge-and-trap technique, RLs, though highly matrix-dependent, are provided in the table below for guidance and may not be achievable. RLs listed

for soil are based on wet weight. When reported on a dry weight basis, RLs are higher, based on the percent dry weight in each sample. For the most current RL, refer to LIMS.

Chromatographic Retention Times and Typical Reporting Limits

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Dichlorodifluoromethane	1.884	1	0.5	2
Chloromethane	2.095	1	0.5	2
Vinyl chloride	2.222	1	0.5	2
Butadiene	2.264	1	0.5	2
Bromomethane	2.601	1	0.5	2
tert-Butyl-formate (TBF)[3]	2.609	20	20	20
Chloroethane	2.718	1	0.5	2
tert-Amyl-alcohol (TAA)[3]	2.914	20	20	2
Dichlorofluoromethane	2.950	1	0.5	2
Trichlorofluoromethane	3.013	1	0.5	2
Ethanol	3.235	100	100	200
Ethyl ether (Diethyl ether)	3.351	5	5	10
1,1-Dichloroethene	3.364	1	0.5	2
Acrolein	3.488	50	50	20
1,1,2-Trichloro-1,2,2-trifluoroethane	3.615	1	0.5	2
Acetone	3.667	5	5	50
Iodomethane	3.783	10	10	20
Isopropyl alcohol	3.836	20	50	50
Carbon disulfide	3.857	1	0.5	2
Acetonitrile	4.016	20	20	20
Allyl chloride (3-Chloro-1-propene)	4.026	2	10	10
Methyl acetate	4.047	10	10	10
Methylene chloride	4.163	5	5	10
2-Methyl-2-propanol	4.311	10	10	50
Acrylonitrile	4.448	10	10	10
trans-1,2-Dichloroethene	4.480	1	0.5	2
MTBE	4.490	1	0.5	2
Hexane	4.807	2	0.5	10
1,1-Dichloroethane	4.976	1	0.5	2
3,3-Dimethyl-1-butanol	5.021	10	10	0
Vinyl acetate	5.050	10	10	20
Isopropyl ether (IPE, Di-isopropyl ether)	5.071	2	0.5	2
2-Chloro-1,3-butadiene (Chloropopene)	5.092	5	5	5
tert-Butyl ethyl ether (Ethyl-tert-butyl-ether, ETBE)[3]	5.514	1	0.5	5
2,2-Dichloropropane	5.683	1	0.5	2

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
cis-1,2-Dichloroethene	5.683	1	0.5	2
2-Butanone	5.715	50	50	50
Ethyl acetate	5.788	5	5	50
Propionitrile	5.788	10	10	50
Methacrylonitrile	5.968	20	20	50
Chlorobromomethane	5.978	1	0.5	2
Tetrahydrofuran	6.063	5	5	20
Chloroform	6.084	1	0.5	2
1,1,1-Trichloroethane	6.327	1	0.5	2
Cyclohexane	6.390	5	1	10
1,1-Dichloropropene	6.527	1	0.5	2
Carbon tetrachloride	6.538	1	0.5	2
Isobutyl alcohol	6.696	50	50	100
Benzene	6.801	1	0.5	2
1,2-Dichloroethane	6.823	1	0.5	2
tert-Amyl-methyl-ether (TAME)	6.960	1	0.5	2
n-Heptane	7.150	2	2	4
n-Butanol	7.582	100	20	100
Trichloroethene	7.656	1	0.5	2
Ethyl acrylate	7.815	5	5	10
1,2-Dichloropropane	7.973	1	0.5	2
Methylcyclohexane	7.973	5	0.5	10
Dibromomethane	8.131	1	0.5	2
Methyl methacrylate	8.142	5	5	10
1,4-Dioxane	8.184	200	200	200
Dichlorobromomethane	8.353	1	0.5	2
2-Nitropropane	8.680	5	5	10
2-Chloroethylvinyl ether	8.796	10	10	20
cis-1,3-Dichloropropene	9.007	1	0.5	2
4-Methyl-2-pentanone	9.239	5	5	50
Toluene	9.503	1	0.5	2
trans-1,3-Dichloropropene	9.830	1	0.5	2
Ethyl methacrylate	9.988	10	10	10
1,1,2-Trichloroethane	10.115	1	0.5	5
Tetrachloroethene	10.337	1	0.5	2
1,3-Dichloropropane	10.368	1	0.5	2
2-Hexanone	10.516	5	5	50
Chlorodibromomethane	10.727	1	0.5	2
n-Butyl acetate	10.738	10	10	40

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Ethylene dibromide (EDB, 1,2-Dibromo-ethane)	10.907	1	0.5	2
Chlorobenzene	11.698	1	0.5	2
1,1,1,2-Tetrachloroethane	11.835	1	0.5	2
Ethylbenzene	11.888	1	0.5	2
m & p-Xylene	12.088	1	0.5	2
o-Xylene	12.743	1	0.5	2
Styrene	12.774	1	0.5	2
Bromoform	13.080	1	0.5	2
Isopropylbenzene (Cumene)	13.418	1	0.5	2
Cyclohexanone	13.555	50	50	50
Bromobenzene	13.914	1	0.5	2
1,1,2,2-Tetrachloroethane	13.925	1	0.5	2
1,2,3-Trichloropropane	13.988	1	0.5	2
trans-1,4-Dichloro-2-butene	14.030	5	5	10
n-Propylbenzene	14.115	1	0.5	2
2-Chlorotoluene	14.241	1	0.5	2
1,3,5-Trimethylbenzene	14.410	1	0.5	2
4-Chlorotoluene	14.421	1	0.5	2
tert-Butylbenzene	14.917	1	0.5	2
Pentachloroethane	14.927	5	5	10
1,2,4-Trimethylbenzene	14.990	1	0.5	2
1,3-Dichlorobenzene	15.391	1	0.5	2
4-Isopropyltoluene (p-Isopropyltoluene)	15.476	1	0.5	2
sec-Butylbenzene	15.524	1	0.5	2
1,4-Dichlorobenzene	15.529	1	0.5	2
1,2,3-Trimethylbenzene	15.613	1	0.5	2
Benzyl chloride	15.729	10	5	20
1,2-Dichlorobenzene	16.056	1	0.5	2
n-Butylbenzene	16.056	1	0.5	2
1,2-Dibromo-3-chloropropane	17.101	10	5	5
1,3,5-Trichlorobenzene	17.375	1	0.5	2
1,2,4-Trichlorobenzene	18.135	1	0.5	2
Hexachlorobutadiene	18.346	1	0.5	2
Naphthalene	18.431	5	5	5
1,2,3-Trichlorobenzene	18.716	1	0.5	2
2-Methylnaphthalene	19.729	10	5	5
INTERNAL STANDARDS/SURROGATES				
Dibromofluoromethane	6.284			
1,2-Dichloroethane-d_4	6.717			

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Fluorobenzene	7.160			
Toluene-d_8	9.408			
Chlorobenzene-d_5	11.656			
4-Bromofluorobenzene	13.671			
1,4-Dichlorobenzene-d_4	15.486			

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030 /NV05-107 (aqueous samples or Methanol extracts of bulk containers) and 5035 / NV05-108 (solid and waste oil samples), is used for volatile organic analytes.
1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor/Manager or the Technical Director. All abnormalities must be noted in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. The analytes are introduced directly to a capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) directly interfaced to the gas chromatograph (GC).
2.2 Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using at least a five-point calibration curve.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components are avoided, since such materials out-gas organic compounds which are concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, perform maintenance. **Subtracting blank values from sample results is not permitted**.
4.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. Re-analyze any suspect samples.
4.3 Special precautions are taken to analyze for Methylene chloride. The analytical and sample storage areas are isolated from all atmospheric sources of Methylene chloride. Otherwise, random background levels result. Since Methylene chloride permeates through PTFE tubing, all

gas chromatography carrier gas lines and purge gas plumbing is constructed from stainless steel or copper tubing.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly Methylene chloride and Fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank, prepared from organic-free reagent water and carried through the sampling, handling, and storage, serve as a check on such contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and should cool them to room temperature prior to working on them.
- The mass spectrometer is under vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- Kevlar gloves must be worn when opening and closing VOA vials.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure causes burn if not flushed with water. Causes mild irritation to skin. Prolonged exposure causes burn if not flushed with water.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Trisodium phosphate		None listed	Keep in closed container; avoid high temperatures and strong acids.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Purge-and-trap device for aqueous samples at ambient temperature, described in Method 5030 / NV05-107.
- Purge-and-trap device for solid samples at 40°C, described in Method 5035 / NV05-108.
- The trap is VOCARB 3000 – 10.0-cm Carbopack™ B/6.0-cm Carboxin™ 1000/1.0-cm Carboxin 1001. The amount of thermal decomposition products formed must be routinely tracked by daily monitoring of the formation of Chloromethane and Bromomethane.
- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP): Analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

Injector temperature:	250°C
MS interface temperature:	260°C
Carrier gas (He) flow rate:	Constant flow of 1.0 mL/minute.
Initial temperature:	45°C hold for 6 minutes.
Temperature program:	13°C/minute to 150°C; 18°C/minute to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted (2 minutes)
Split ratio (min.)	1:10

May vary by instrument; see maintenance log for current program.

- The capillary column is directly coupled to the source.
- Gas chromatographic column: DB-624, 20 m x 0.18 mm with 1.0 µm film thickness, or equivalent.
- Mass spectrometer: Capable of scanning from 35 to 300 amu every 1 second or less using 70 volts (nominal) electron energy in the electron impact ionization mode. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.
- Data system (HP Chem Station with Enviroquant and CHROM): A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching the GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is used that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is also available.

6.2 Supplies

- Microsyringes, 10, 25, 100, 250, 500, and 1,000 µL.
- Syringes, 5, 10, or 25 mL.
- Balance, analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.
- Glass scintillation vials, 20 mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.
- Disposable pipets, Pasteur.
- Volumetric flasks, Class A, 10 mL, 50 mL and 100 mL, with ground-glass stoppers.
- Spatula, stainless steel, or wooden tongue depressor.
- Helium for carrier gas.
- Nitrogen for purge-and-trap gas.
- Narrow-range pH paper.
- Residual chlorine test strips.
- Sea or Ottawa sand for blank and LCS soil matrix.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are generally used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. See the QA Manual and SOP Reagent and Standard Purchase / NV08-214 for more information on reagent chemicals, such as shelf-life and storage.

7.2 Reagent water, analyte-free.

7.3 Methanol, CH_3OH: Purge-and-trap grade or equivalent, demonstrated to be free of analytes at the MDL. Store apart from other solvents.

7.4 Hydrochloric acid (1:1 v/v), HCl: Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

7.5 Stock solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in Methanol, using assayed liquids. Any specific standards or procedure for making standards mentioned in this SOP may be substituted with equivalent standards or procedures. See standard log for specific standard information.

7.5.1 **Primary Standards**

		For Working Standard		
Name of standard	**Vendor[2]/Conc (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc. (µg/mL)**
Full List Non-gas Standard				
Custom 8260 VOC mega-mix[1] without gases	Restek 567641/2000, 4000, 10000,-20000, 40000	2.5	50	100 – 2000
Megamix additions	Restek 567647/2000, 4000,20000, 50000, 100000	2.5	50	100-5000
Ketones	Restek 567642/10000	2.5	50	500
Acrolein	Restek 567644 /5000	2.5	50	250
Cyclohexanone	567648/20000	2.5	50	1000
Vinyl acetate	Restek 567646/4000	2.5	50	200
2-Chloroethyl vinyl ether	Restek 567643 /2000	2.5	50	100
List 2: Pentachloroethane, 2-Methylnaphthalene	Restek 567719 / 2000	2.5	50	100

		For Working Standard		
Name of standard	**Vendor[2]/Conc (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc. (µg/mL)**
1-Methylnaphthalene	Restek 31283/1000	0.1	2	50
Full List Gas Standard				
Gas Mix	Restek 567645/2000	1.0	20	100
Short List				
Short List Mix	Ultra CUS-7011/100-1000	5	10	50-500
3,3-Dimethyl-1-butanol	Restek 563892/20000	0.25	10	500
1 Custom 8260 VOC mix has variable concentrations. See the standard log for exact compound concentrations.				
2 The vendors/catalog numbers are recommended; equivalent products are acceptable.				

7.5.1.1 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at ≤ 6°C or less or as recommended by the standard manufacturer. Return standards to storage as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of target compounds.

7.5.1.2 **Frequency of Standard Preparation**

7.5.1.2.1 Monitor standards for the permanent gases frequently by comparison to the initial calibration curve. Prepare fresh standards if this check exceeds a 20% drift. **Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented**. Dichlorodifluoromethane and Chloromethane are usually the first compounds to evaporate from the standard and, therefore, are to be monitored very closely when standards are held beyond one week.

7.5.1.2.2 Monitor standards for the non-gases frequently by comparison to the initial calibration. Prepare fresh standards if this check exceeds a 20% drift. Undiluted standards for non-gases usually need to be replaced after **one month for working standards and three months for opened stock standard** or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-Chloroethyl vinyl ether and Styrene may need to be prepared more frequently.

7.5.1.3 **Secondary dilution standards:** Using stock standard solutions, prepare secondary dilution standards in Methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards are stored with minimal headspace and, except for gases, are good for 2-4 weeks unless acceptability is demonstrated. Replace secondary standards for gases after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. Handle and store standards as stated above and return them to the refrigerator or freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

7.5.1.3.1 The working calibration standard for the Non-Gas mixture is made by adding 2.5 mL of each of the first six standards in the Primary Standard table above in 50.0 mL Methanol in a Class A volumetric. The Gas

Standard is added right before use as described in the calibration section.

7.5.2 **Internal Standard/Surrogate Standard Mix (IS/SS)**

7.5.2.1 The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. Prepare internal standard stock and secondary dilution standards in Methanol. Stock standard is 250 µg/mL, Restek 567649, or equivalent.

7.5.2.2 The surrogates are Toluene-d_8, 4-Bromofluorobenzene (the GC/MS Tuning Standard), 1, 2-Dichloroethane-d_4, and Dibromofluoromethane. Stock standard is 2500 µg/mL, Restek 567650, or equivalent.

7.5.2.3 Prepare a 250 µg/mL IS/SS standard by diluting 5.0 mL of stock internal standard (250 µg/mL) and 5.0 mL stock surrogate standard (2500 µg/mL) to a final volume of 50.0 mL of Methanol in a Class A volumetric.

7.5.3 **Bromoform Breakdown Check:** Purchase 50 g neat Bromoform from Sigma-Aldrich 241032-50G, or equivalent.

7.5.3.1 Prepare a 20 µg/L standard by adding 0.02 g of the neat Bromoform standard to 1000 mL reagent water.

7.5.4 **Second-Source Standards for Initial Calibration Standard (ICV):** The ICV is a **second-source** standard that contains all the 8260 compounds. Prepare as for the primary standard with the only difference being that the vendor numbers have a ".sec" on the end of the number.

7.6 Sodium bisulfate or Trisodium phosphate for soil sample preservation. See SOP 5035 / NV05-108.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time From Collection to Analysis	Reference
Water[2]	3 x 40-mL VOAs (Optional: TSP)	40 mL	pH < 2 with Hydrochloric acid. Cool 0-6°C, No headspace. Keep in dark. If Chlorine residual present, add 0.008% $Na_2S_2O_3$.	14 days, 7 days if not acidified.	SW846 Chapters 2 and 4
Low-concentration Soil	2 pre-weighed vials, stirring bar	5 g	0-6°C, 5 mL preservative[1]		
	2 EnCores™		0-6°C, Add 5 g sample and 5 mL preservative to pre-weighed vial with stirring bar within 48 hours of collection		
High concentration Soil	2-oz. glass[3] or 25 g Encore™	5g or 25 g	0-6°C, Add 1 mL Methanol/gram soil	Transfer to VOA within 48 hours, then 14 days	

[1]0.2 gram sodium bisulfate or Trisodium phosphate/mL reagent water

[2]2-Chloroethyl vinyl ether degrades in acid-preserved samples; its analysis requires a non-preserved vial. If analyzing a sample for combined purgeable halocarbons, aromatics, Acrolein, and Acrylonitrile, analyze the sample within 7 days. Alternatively, collect at least 2 separate vials for analysis: one vial preserved to pH 4-5 with HCl for Acrolein and Acrylonitrile, and a second vial for the other analytes preserved to pH <2 with HCl.

[3]See SOPs 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids, including the soil freezing option, with or without water.

Analysis Method	Sample Storage	Holding Times from Date and Time of Collection			
		MeOH Addition	Shipping	Extraction	Analysis
Wisconsin VOC Soils	VOC vial	Immediately	4 days	21 days	21 days
	Brass Tube	within 2 hours	4 days	21 days	21 days
	EnCore™	within 48 hours	40 hours	21 days	21 days

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC: The following QC is run **every batch of no more than 20 samples:**

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Method blank	One per analytical prep batch and after calibration (see Section 9.2)	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank. If target > 10x blank, report but qualify.
LCS[4] for all analytes using the primary standard.	One[6] per prep batch	See LIMS[5]	Re-prep[3] and analyze the LCS and all samples in the affected analytical batch. If high and samples are ND, report. If low, re-prep. If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the sample from that batch is less than the RL, the data is acceptable to report.
MS/MSD using the primary standard	One per batch per matrix, if insufficient sample for MS/MSD, then analyze a LCS/LCSD.	See LIMS	None (LCS is used to determine if data is acceptable).
Surrogate	Every sample, spike, standard, and blank.	See LIMS	Check system, re-analyze, re-prep[3], may qualify. If %recovery is high and the sample is ND, it is acceptable to report. If low, re-prep and rerun. If the surrogate % recovery exceeds the upper control limit AND a sample is greater than the RL, re-prepare and re-analyze the sample. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prepare the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed
pH check	All water samples.	pH ≤2or ≥ 11	If the pH is > 2 but less than 11, comment the data and LIMS.
Residual chlorine check (North Carolina samples only)	Each sample.	Residual chlorine must be negative.	If the residual chlorine is positive, then comment the data, and LIMS.

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted in LIMS.
[3]If unable to re-prep the samples because of insufficient sample volume or holding time has expired, place a comment in LIMS.

[4] All AZ, MA, TX, and WV samples require a LCS duplicate in each batch.
[5].See Section 16 for South Carolina LCS acceptance criteria and Minnesota Ethanol acceptance criteria.

- A **Method Blank** is run with each analytical batch. The blank is carried through all stages of the sample preparation and measurement using the appropriate blank matrix (reagent water or sand).
- A **Laboratory Control Sample (LCS)** is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix (reagent water or sand) similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes from the primary source.

Matrix	LCS Preparation	Final Concentration
Water	Add 50 µL of the primary source standard to 50.0 mL reagent water in a Class A volumetric flask.	50 – 5000 µg/L
Low-concentration Soil	Add 5 µL of the primary source standard to a VOA vial containing 5.0 g sand and 5 mL preservative and a stirring bar.	50 - 5000 µg/kg
High-concentration Soil (analyzed as waters)	Add 50 µL of the primary source standard to 50.0 mL reagent water in a Class A volumetric flask.	50 - 5000 µg/kg

- **Matrix Spike/Matrix Spike Duplicate:** Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair for each batch.

Matrix	MS/MSD Preparation	Final Concentration
Water Batch	Add 43 µL of the primary source standard to the client's sample in VOA vials.	50 – 5000 µg/L
Low-concentration Soil Batch	Add 5 µL of the primary source standard to a VOA vial containing 5 g preserved client sample (with stirring bars).	50 - 5000 µg/kg
High-concentration Soil Batch (analyzed as waters)	Add 1.0 mL of the Methanol-extract-of-client-sample and 50 µL of the primary source standard and dilute with reagent water in a 50-mL, Class A volumetric.	50 - 5000 µg/kg

- **Surrogate standards**: The analyst monitors both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, QA/QC standard, and blank with surrogate compounds which are not expected to be affected by method interferences. The surrogate and internal standards are prepared together as described in Section 7.
  - The IS/SS standard mix (250 µg/mL each) is added by the autosampler (nominally 1 µL) during all analyses with the exception of the calibration.

Purge Volume, mL	Concentration of IS/SS Standards in Sample, µg/L
5	50
10	25

- **pH Check:** The analyst must document that each sample has a pH ≤ 2 or ≥ 11 by checking with narrow-range pH paper. The pH check is performed after sample analysis to avoid

contamination and creation of a headspace in the sample vials. Record as pH <2 or >2 or > 11.

- **Residual Chlorine Check:** The analyst must document the presence/absence of residual chlorine in North Carolina samples by checking with residual chlorine test strips.

9.2 Instrument QC

Italicized information is unique to 8260C.

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
a. Check of mass spectral ion intensities, i. e., BFB Tune	Prior to initial calibra-tion or Continuing calibration verification, every 12 hours.	Refer to criteria for Tune criteria	Retune the instrument and verify (instrument maintenance may be needed).
b. Bromoform Break-down Check	At beginning of daily sequence.	≤ 0.5 µg/L Bromomethane; ≤ 0.5 µg/L Chloromethane	Re-condition or replace trap. Re-calibrate.
Minimal five-point initial calibration for all target analytes. Single-point surrogate calibration	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8260B: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%. $r^2 \geq 0.990$ or $r \geq 0.995$. Re-calculate low point; must be within 30% true. *8260C: Minimum RF for initial and continuing calibration varies by analyte (see Calibration standards below). RSD ≤ 20% each target.* $r^2 \geq 0.990$ or $r \geq 0.995$. *Up to 10% of targets may exceed these criteria. If using linear regression, re-fit lowest calibration point. It must be ± 30% or re-calculate.*	Correct problem then repeat initial calibration.
Initial calibration verification (ICV), must be from a second source	Immediately following each initial calibration.	All analytes within 30% of expected value. Problematic compounds may be within 40%.	Correct problem then repeat initial calibration. ICV must be run prior to reporting samples.
Continuing Calibration Verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8260B: CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). SPCCs: minimum RF. All other target compounds ≤ 30%, except for specific compounds which may have a % difference ≤ 40%. *8260C: All targets of interest ≤ 20%. Up to 20% of targets may exceed this criterion. Common targets meet minimum RF.*	Correct problem then repeat CCV (re-calibrate if necessary) and re-analyze any samples processed with that CCV. If the CCV is high and the sample is ND, it is acceptable to report.[3]
Continuing Calibration Blank	After each CCV.	< ½ RL or MDL, whichever is greater.	Correct problem, repeat.
Internal Standards[3]	Every sample, standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of most recent ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning.
Retention time window calculated for	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then re-analyze all samples analyzed since the last retention time

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
each analyte			check.
MDL verification (MDLV)	Minimum yearly.	Detectible.	Re-evaluate MDL standard used and MDL; see Technical Director.

[1] This is a summary of the acceptance criteria.
[2] All abnormalities must be noted in LIMS.
[3]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.

- **BFB Tuning and Breakdown Check**:
  - **BFB Tuning**: At the beginning of each 12-hour analytical shift and **prior** to the analysis of samples or calibration standards, inject 50 ηg or less of the 4-Bromofluorobenzene standard into the GC/MS system (1 µL of 250 µg/mL standard /50 mL reagent water, purged at a 1:10 split for a 25 ηg on the column). (BFB is one of the surrogate compounds.) The resultant mass spectra for the BFB **must** meet the tuning criteria below before sample analysis begins.

BFB (4-Bromofluorobenzene) Mass Intensity Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101 % of m/z 174
177	5 to 9% of m/z 176

  - Three options are available for acquiring the spectra for reference to meet the BFB tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ± 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct.

Option 2 The scan across the peak at one half peak height may be averaged and background-corrected.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is still required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance.**

- Note: All subsequent standards, samples, controls, and blanks associated with a BFB tune **must** use identical mass spectrometer instrument conditions.

- **Bromoform Breakdown Check**: The daily BFB Tune/Breakdown Check containing surrogates, internal standards, BFB, and 20 µg/L Bromoform must be analyzed prior to the analysis of the Continuing Calibration Verification (CCV). If levels of Chloromethane or Bromomethane exceed 0.5 µg/L, then the trap may be too contaminated with salts or tightly bound contamination for analysis to continue. The trap must be replaced, and the system re-calibrated.

- **Calibration standards**: See Section 10.2.

SPCCs and CCCs are unique to 8260B. ***Italicized text is unique to 8260C.***

- **Initial System Performance Check Compounds (SPCCs)**: A system performance check is made before the calibration curve is used. Five compounds (the System Performance Check Compounds) are checked for a minimum average response factor, compound instability, and degradation caused by contaminated lines or active sites in the system. These compounds are Chloromethane, 1,1-Dichloroethane, Bromoform, Chlorobenzene, And 1,1,2,2-Tetrachloroethane. The minimum mean response factors for the volatile SPCCs must be met and are as follows:

Chloromethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
1,1-Dichloroethane	0.10
Chlorobenzene	0.30

Example problems include:

- Chloromethane is the most likely compound to be lost if the purge flow is too fast.
- Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve Bromoform response.
- Tetrachloroethane and 1,1-Dichloroethane are degraded by contaminated transfer lines or active sites in trapping materials.

- **Initial Calibration check compounds (CCCs):** The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites in the system. Meeting the CCC criteria is **not** a substitute for successful calibration of the target analytes. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

- Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for **all** target analytes from the initial calibration with the equations in Section 11.
- The RSD must be less than or equal to **15% for each target analyte**; however, the

RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak or contamination and/or column reactive sites is necessary before re-attempting calibration. The CCCs may not be in the project target list. If that is the case, each target must have a RSD < 15% or a correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.990$) as calculated by the equations in Section 11. When using linear regression, re-calculate the low calibration point. It must be within 30% true.

- *For 8260C, the minimum RF for initial and continuing calibration is:*

RF	*For These Compounds*
0.1	*Critical compounds*
0.2	*1,1-Dichloroethane, Chloroform, Trichlorethene, Bromodichloromethane, cis-1,3-Dichloropropene, Tetrachloroethane, and 1,2,4-Trichlorobenzene*
0.3	*o-Xylene, Styrene, 1,1,2,2-Trichloroethane*
0.4	*Toluene, 1,2-Dichlorobenzene*
0.5	*Benzene, Chlorobenzene, 1,4-Dichlorobenzene*
0.6	*1,3-Dichlorobenzene*

  - *For 8260C, the must be less than or equal to 20% for each target analyte with up to 10% of compounds meeting the 40% criterion.*

- **Initial Calibration Verification (ICV)** is verified immediately after calibration using the introduction technique used for samples. Analyze a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS.
  - The ICV is made from the **second-source** standards, one at a time, as needed, to be run after an initial calibration: Add 25 µL of the second-source, non-gas, working standard to 50 mL reagent water in a 50-mL, Class A volumetric. Add 25 µL of the Gas Mix working standard to the Class A volumetric for a final concentration of 50-5000 µg/L.
  - The ICV of each target must be within 30% of the expected value, with the exception of the following poor purge efficiency analytes that may be within 40% of the expected value for up to 20% of targets:

Acrolein	Ethanol	2-Methylnaphthalene
t-Amyl alcohol (TAA)	t-Butyl formate (TBF)	Vinyl acetate
t-Butyl alcohol (TBA)	1-Methylnaphthalene	

  - If ICV criterion is not met, correct the problem and re-calibrate.
- **Continuing Calibration Verification (CCV):** Run every 12 hours of sample analysis, CCVs are often made each day for several instruments in the following proportions, always from the **primary** calibration standards:
  - Add 25 µL of the working calibration Non-Gas Standard and 25 µL of the primary Gas Mix to a 50-mL, Class A volumetric and dilute to the mark with reagent water. The final concentration is 50-5000 µg/L.
  - Area counts of the internal standards must be between 50 - 100% of the areas of the internal standards in the mid-point calibration standard. If not, inspect the GCMS for possible maintenance issues and then re-analyze. Contact the department supervisor for assistance in determining the appropriate course of action. **Do not report data from a failing internal standard associated with target compounds.**
  - **For 8260B Only: Continuing System Performance Check Compounds (SPCCs)**
    - A System Performance Check **must** be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard **must** meet its minimum

response factor. This is the same check that is applied during the initial calibration.
    - If the minimum response factors are not met, the system **must** be evaluated, and corrective action **must** be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.
- **For 8260B Only: Continuing Calibration Check Compounds (CCCs)**
    - After the system performance check is met, the CCCs are used to check the validity of the initial calibration, if present in the target list. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. See Section 11 for equations.
    - If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift), for any one CCC, then corrective action **must** be taken prior to the analysis of samples. **If the CCCs are not included in the list of analytes for a project and therefore not processed in the calibration standards, then all analytes must meet the 20% difference or drift criterion.**
    - Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC or target criteria **must** be met before sample analysis begins.
- **For 8260B Only: Continuing Evaluation of Non CCC/SPCC compounds** – The percent difference or drift for each of the non-CCC analytes is less than or equal to 30%. Recovery for some compounds with poor purge efficiency may exceed this 30% requirement and still be deemed acceptable provided all of the following criteria are met:
    - Poor performing analytes are one of the following: Acrolein, tert-Amyl alcohol (TAA), tert-Butyl alcohol (TBA), tert-Butyl formate (TBF), Ethanol, 2-Methylnaphthalene, Vinyl acetate.
    - The percent difference or drift is less than or equal to 40%.
- *For 8260C, see ICAL and ICV information.*
- **Continuing Calibration Blank (CCB):** The CCB is reagent water or sand.
- **Internal Standards** are used to evaluate the effect of the sample matrix. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected.
    - The retention times of the internal standards in the calibration verification standard are evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.
    - Internal standards permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Attachment 1). If interferences are noted, use the next most intense ion as the quantitation ion.
    - **Internal standard response** - If the EICP area for any of the internal standards in the calibration verification standard and samples changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made.

When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.

- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
  - If the internal standard is within criteria, report the second analysis.
  - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
  - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- See Attachment 2 for the analytes corresponding to each internal standard.

- **Retention time windows**: Target analytes are identified on the basis of retention time windows.
  - Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.
  - Establish the retention time windows for target analytes.
  - The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.
- **Method Detection Limit Verification (MDLV):** Annually verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	VOA vial
Low-concentration Soil	5 grams
High-concentration Soil	1 mL Methanol extract of soil

- All samples and standard solutions are allowed to warm to ambient temperature before analysis.
- Refer to SOP 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids.
- For Wisconsin VOC soils, the following procedure must be performed for Methanol extraction of Soil/Sediment:

1	Hand-shake the sample in its vial containing Methanol vigorously for 2 minutes. Sonicate for 20 minutes.
2	Allow sediment to settle until a layer of Methanol is apparent.
3	Withdraw an appropriate aliquot of the Methanol extract for sparging and add to a VOA vial.
4	Analyze all reagent blanks and QC samples on the same instrument as that used for the samples.
5	If the responses exceed the calibration or linear range of the systems, use a smaller aliquot of Methanol extract or dilute the aqueous sample.

10.2 Initial Calibration: Refer to SOP Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1 Evaluate the **BFB tuning criteria.**

2 Prepare the **Initial calibration standards** at a minimum of five different concentrations from the secondary dilution of stock standards or from a premixed certified solution in organic-free reagent water. **At least one of the calibration standards corresponds to a concentration at or below the laboratory reporting limit.** This low standard must have valid ion abundances for all monitored ions. The remaining standards define the working range of the system. Initial calibration standards are mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

Initial Calibration (5-point)

Primary Working Standard	Final Volume (mL)	Concentration (µg/L)
1	100	0.5-2.5
2	100	1 - 5
4	100	2 - 10
20	100	10 - 50
40	100	20 - 100
100	100	50 - 250
200	100	100 - 500
400	100	200 - 1000

1 µL of IS/SS Standard at 250 µg/mL is added by the autosampler to 5 mL for a 50-µg/L concentration in the standards and samples with a 5-mL purge; 1 µL/10 mL for a 25 µg/L concentration. See Section 9.1 for make-up and final concentrations for other purge volumes. The surrogate calibration is a single-point.

- All target analytes for a particular analysis must be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. **However, the laboratory must not report a quantitative result for a target analyte that was not included in the calibration standard(s).**
- **Internal Standards:** The calibration standards must also contain the internal standards chosen for the analysis. Calibration standards for soils must also contain the preservative Na_2SO_4. See Method 5035 / NV05-108 for how to accomplish the preservation.
- **Surrogates**: Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. With improvements in instrumentation and more reliance on the autosampler, an option is available allowing the autosampler to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option, the surrogate standards in the initial calibration can be averaged to develop a response factor and an effective one-point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option, the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout, and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.
- **Technique:** To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a Class A, volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection and stopper. Mix by inverting the flask three times. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and are prepared daily. Transfer each standard to separate VOA vials.

	• **Water or Soil Samples:** A different calibration curve is necessary for Methods 5030 / NV05-107 and 5035 / NV05-108. Calibration **must** be performed using the same sample introduction technique that is used for samples. For Method 5030, the purging efficiency varies with purge volume; therefore, develop the standard curve with whichever volume of sample that is to be analyzed.
3	**Calibration sequence**: 1 BFB Tuning Criteria 2 ICAL 3 ICV 4 ICB
4	**Tabulate the area response of the characteristic ions** (see Attachment 1) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards.
5	• Evaluate the RSD or linearity.
6	For 8260B: evaluate the SPCC and CCC compounds for the initial calibration criteria. For 8260C: evaluate each target.
7	Evaluate the retention times and minimum response factors.
8	Evaluate the success of the initial calibration by immediately running an Initial Calibration Verification (ICV).
9	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

10.3 Daily GC/MS Calibration Verification

1	Evaluate the BFB tuning and Breakdown Check criteria.
2	Evaluate the CCV and CCB.
3	For 8260B, evaluate the SPCC and CCC compounds for the continuing calibration criteria. For 8260C: evaluate each target.

10.4 Example Analysis Queue / Sequence (based on 12 hours)

1	Tune/Breakdown Check
2	CCV, for daily and ongoing calibration check
3	LCS
4	Blank
5	Samples
6	Matrix Spike
7	Matrix Spike Duplicate
When 12 hours have passed, run a 2nd tune and CCV before running more samples, no more than 20 samples in a 12-hour batch.	

- **Dilutions**: If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the upper calibration standard, the sample must be reanalyzed at a higher dilution. **Secondary ion quantitation is allowed only when there are sample interferences with the primary ion**.
  - When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free, reagent water blank or the repeating of suspected samples. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is

demonstrated to be free of interferences. Repeat all affected samples.

- Prepare dilutions such that the response of the major constituents (previously saturated peaks) is in the upper half of the linear range of the curve.
- The following procedure is used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays.

1	Dilutions are made in Class A, volumetric flasks (50 to 100 mL). Select the volumetric flask that allows for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.
2	Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.
3	Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, three times. Repeat above procedure for additional dilutions.
4	Fill a VOA vial with the diluted sample and cap.

- For high concentration samples, see SOP 5035 / NV05-108.

10.5 Qualitative analysis

The qualitative identification of each compound determined by this method is based on relative retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. See Attachment 1 for primary and secondary ions for each compound. Compounds are identified as present when the following criteria are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree with 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ± 30 % relative intensity, report the compound as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair.
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i. e., only one

chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum might contain extraneous ions contributed by the co-eluting compound. If all of the ions associate with the reference spectrum for the target analyte are present and within the ± 30% criteria, a positive result must be assumed even in the presence of extraneous ion fragments without presumptive evidence for a negative identification. (All ions associated with the target analyte are also present in the interfering peak.) The analyst must carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. Add appropriate qualifiers in Element (ID2).

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.
- For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the purpose of the analyses being conducted. Data system library search routines are not to use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
- For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:
  - Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample spectrum.
  - The relative intensities of the major ions must agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
  - Molecular ions present in the reference spectrum must be present in the sample spectrum.
  - Review ions present in the sample spectrum but not in the reference spectrum for possible background contamination or presence of co-eluting compounds.
  - Review ions present in the reference spectrum but not in the sample spectrum for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the **primary** characteristic ion. The internal standard used is the one nearest the retention time of that of a given analyte. See Attachment 1.
- If the RSD of a compound's response factors is 15% or less, then the concentration is determined using the average response factor (*RF*) from initial calibration data.
- Where applicable, the concentration of any non-target analyte identified in the sample may be estimated. The same formulae are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms, and the RF for the compound must be assumed to be 1.
- The resulting concentration is reported indicating:
  - that the value is an estimate, and

- in Level 4 data packages, which internal standard was used to determine the concentration. Use the nearest internal standard free of interferences.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\% \text{ Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard

11.4 Standard Deviation, Relative Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - RF_{mean}\right)^2}{n-1}} \qquad RSD = \frac{SD \times 100}{RF_{mean}}$$

RF_i = RF for each of the calibration standards
RF_{mean} = mean RF for each compound from the initial calibration
n = Number of calibration standards, e. g., 5

11.5 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF}) \times 100}{\text{(Avg. RF)}}$$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result - True Value} \times 100}{\text{True Value}}$$

11.6 Linear Calibration Using a Least Squares Regression: A linear calibration model based on a least squares regression may only be employed if RSD does not meet the acceptance criteria.

For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_I = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(x_i - x_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i(x_i - x_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).
x_i – observed instrument response (area) for the i^{th} calibration standard.
x_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used.

Examples of allowed weighting factors which place more emphasis on numbers of smaller value are:

$w_i - 1/x_i$ or $w_i = 1/x_i^2$

Do not include the origin (0, 0) as an extra calibration point. The use of a linear regression may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. If it is necessary to report results at lower concentrations, then the analyst must run a calibration that reaches those lower concentrations.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.990$.

11.7 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.8 Concentration Calculation

Concentration = (µg/L from instrument) (dilution factor)

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Aqueous waste generated from analysis may have a pH of less than 2.0. Transfer to waste disposal for neutralization and then dump into the sanitary sewer.
- Solid waste generated from analysis is placed in the trash.

15.0 References / Cross References

15.1 EPA Method 8260B, SW-846 Update III, Revision 2, December 1999, **Method 8260C**, Update.IV, Rev. 3, August 2006.

15.2 Method 8000B, SW-846, Revision 2, December 1996, **Method 8000C**, Revision 3, March 2003.

15.3 Method TPH-GRO by Method 8260B, MRBCA (Missouri) Guidance Document, Final Draft, February 24, 2004.

15.4 California GRO, CA LUFT 8015.

15.5 TestAmerica Nashville's Quality Assurance Manual.

15.6 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.7 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Calibration Curves (General) / CA-Q-S-005, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, Sample Homogenization, Sub-sampling & Compositing / NV08-229, 5030 / NV05-107, 5035 / NV05-108.

15.8 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

State	Modification
Ohio specific criteria	Only those compounds in EPA Method 8260B may be reported (superscript 1 in the table in Section 1.1). Some compounds in this SOP are not part of the original 8260B method. The method blank must be less than the RL for Ohio samples. See SOP 8260/NVOH05-77.
Missouri GRO	Prepare 1:1 mixture of unleaded gasoline and #2 diesel fuel in Methanol as GRO is defined by setting retention time window from 0.1 minutes before C_6 to 0.1 minutes after C_{10}. Verify RT window with the standard daily (every 24 hours).
California	California LUFT GRO uses gasoline and the retention time window of C_4 (t-Butanol) to

GRO	C_{12}.
Michigan GRO	See the Michigan GRO requirements below.
South Carolina	See the special LCS acceptance criteria and special state PQL requirements below.
Minnesota	See the special Ethanol analysis requirements below for water samples.

TPH-GRO by Method 8260B

Standards

- C4-C12 Standards
  - Primary: Restek 30096, 5000 µg/mL, or equivalent.
  - Secondary: O2SI 020246-S6, 10,000 µg/mL, or equivalent.
- C6-C10 Standards
  - Primary: Restek 31484, 20,000 µg/mL, or equivalent.
  - Secondary: Ultra CUS-8324, 10,000 µg/mL, or equivalent.

Sample Introduction

- Samples are purged onto the GC/MS system using all protocols specified in SW-846 Method 5030 and 5035.
- Surrogates and internal standards specified by Method 8260B are added to water and soil samples prior to purging.

Sample Analysis

- The BC/MS system is tuned to BFB tune criteria listed in Method 8260B at the frequency specified in Method 8260B.
- A 5-point standard curve is used to quantitate TPH-GRO by the internal standard technique.
- For **Missouri** GRO, the stock standard solution is a mixture of unleaded gasoline and Number 2 diesel fuel.
- For **Californi**a GRO, the stock standard is unleaded gasoline.
- The lowest calibration standard should be at or below the reporting limit of the method.
- For **Missouri,** retention time windows are defined for TPH-GRO by analyzing a standard containing C_6 and C_{10}. The retention time window is defined as $>C_6$ to C_{10}. The standard containing C_6 and C_{10} must be analyzed every day samples are analyzed in order to verify that the retention time windows are constant.
- For **California**, the retention time window are defined as $>C_4$ to C_{12}.
- For **Michigan**,
  - Use unleaded gasoline for calibration.
  - The retention time window is defined as C_6 (hexane) to C_{10} (n-decane).
  - The holding time for water and soil is 14 days.
  - For soil preparation, shake the Methanol and sample for 2 minutes, then sonicate in a water bath for 20 minutes.
  - For oil samples, add 2 g sample to 40 mL Methanol; wait 24 hours before analysis.
  - Use the internal calibration technique, summing the range.
  - Use only linear regression; $r \geq 0.990$, $r^2 \geq 0.981$.
  - ICV and CCV must be ± 20% true.
- Because the retention time window is several minutes wide for TPH-GRO, the GC/MS data system may not accurately or appropriately establish the proper baseline for calibration or quantitation. The analyst **must** visually examine the computer-generated baseline for every analytical run and manually adjust the baseline when needed. A properly drawn baseline must extend over the entire retention time window and include

the area under the entire TPH-GRO series of peaks. It is not appropriate to draw the baseline "peak to peak."

- The total ion chromatogram (TIC) **must** be used to calculate the area under the peak for TPH-GRO calibration and quantitation determinations over the entire retention time window.
- Area counts for the internal standards and surrogates added during sample preparation **must** be subtracted from the total area count for TPH-GRO. This is accomplished by subtracting the area count of the method blank from all subsequent calibration and analytical runs.
- The %RSD for the calibration curve for TPH-GRO must be less than or equal to 20%, so that linearity through the origin can be assumed and an average calibration factor used for calculations.
- A continuing calibration verification standard (CCV) must be analyzed at the beginning of each batch. The standard concentration should be at the mid-point of the calibration curve. If the %RSD exceeds 20%, a new curve must be generated.
- A method blank must be analyzed once per day to insure the analytical system is free of background contamination.

South Carolina LCS Acceptance Criteria and Special State PQL Requirements

- All routinely reported analytes require 70-130% LCS recovery. The following exceptions of poor-performing analytes require 60-140% LCS recovery:

Acetonitrile	Dichlorodifluoromethane
Acrolein	2,2-Dichloropropane
Acrylonitrile	3,3-Dimethyl-1-butanol
t-Amyl alcohol	1,4-Dioxane
Bromomethane	Ethanol
t-Butyl alcohol	2-Hexanone
t-Butyl formate	Isopropyl alcohol
Chloroethane	4-Methyl-2-pentanone
Chloromethane	Vinyl chloride
1,2-Dibromo-3-chloropropane	

- Instrumentation used for South Carolina samples must be able to achieve and report the following South Carolina PQLs when those compounds are requested:
  - Acrolein 5 ug/L
  - Acrylonitrile 5 ug/L
  - 2-Chloroethyl vinyl ether 5 ug/L
  - Methylene chloride 2 ug/L

Minnesota Ethanol Analysis Requirements for Water Samples

- The calibration standard used for Ethanol must be a water-based standard and not a Methanol-based standard. Ethanol water-based standards must be stored at <4°C.
- Initial calibration: The recovery (accuracy) for each point in the curve must be 70-130% except for the lowest point in the curve which must be 60-140%.
- Continuing calibration verification: Analyze one low-level Ethanol standard at the report level (RL) and one mid-level Ethanol calibration verification standard at approximately 500 µg/L prior to the samples. %R for Ethanol in the low-level standard must be 60-140% of the true

value. %R for Ethanol in the mid-level standards must be 70-130% of the true value and a % difference of ≤ 30%.

- For samples, absolute areas of the quantitation ions for the internal standard and surrogate must not decrease by more than 50% from the initial calibration.
- %R for Ethanol for the MS/MSD must be 70-130% with a relative percent difference (RPD) of ≤30%. %R for the LCS/LCSD must be 70-130% with a RPD ≤ 30%.
- The quantitation ion for Ethanol is 45 atomic mass units (AMU). Confirmation ions are 46 and 47 AMU. Ethanol standards must be analyzed separately from the normal VOC list due to the interference from Ethyl ether.

17.0 Attachments

17.1 Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds.

17.2 Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation.

Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds

Compound	Primary Characteristic Ion	Secondary Characteristic Ion
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,1-Trichloroethane	97	99, 61
1,1,2,2-Tetrachloroethane	83	131, 85
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151, 103, 153
1,1,2-Trichloroethane	97	83, 85
1,1-Dichloroethane	63	65, 83
1,1-Dichloroethene	96	61, 63
1,1-Dichloropropene	75	110, 77
1,2,3-Trichlorobenzene	180	182, 145
1,2,3-Trichloropropane	110	75, 77
1,2,3-Trimethylbenzene	0	105
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-chloropropane (DBCP)	157	155, 75
1,2-Dichlorobenzene	146	111.148
1,2-Dichloroethane	62	98
1,2-Dichloropropane	63	112
1,3,5-Trichlorobenzene	180	145, 182
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	111, 148
1,3-Dichloropropane	76	78
1,4-Dichlorobenzene	146	111, 148
1,4-Dioxane	88	58, 43, 57
2,2-Dichloropropane	77	97
2-Butanone (MEK)	72	43
2-Chloro-1,3-butadiene (Chloroprene)	53	88, 90, 51
2-Chloroethyl vinyl ether	63	65, 106
2-Chlorotoluene	91	126
2-Hexanone	58	43, 57, 100
2-Methyl-2-propanol (t-butyl alcohol)	59	41, 43
2-Methylnaphthalene	142	141, 115
2-Nitropropane	43	41, 39
3,3-Dimethyl-1-butanol	57	69, 41
3-Chloro-1-propene (Allyl chloride)	76	78

4-Chlorotoluene	91	126
4-Isopropyltoluene (p-Isopropyltoluene)	119	134, 91
4-Methyl-2-pentanone (MIBK)	58	43, 100, 85
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55
Acrylonitrile	53	52, 51
Benzene	78	--
Benzyl chloride	91	126, 65, 128
Bromobenzene	77	156, 158
Bromoform	173	175, 254
Bromomethane	96	94
Butadiene	0	54
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chlorobromomethane	130	49, 128
Chlorodibromomethane	127	129
Chloroethane	64	66 (51*)
Chloroform	83	85
Chloromethane	50	52 (51*)
cis-1,2-Dichloroethene	61	96, 98
cis-1,3-Dichloropropene	75	77, 39
Cyclohexane	56	84, 41, 69
Cyclohexanone	55	42, 98
Dibromomethane	93	95, 174
Dichlorobromomethane	83	85, 127
Dichlorodifluoromethane	85	87
Dichlorofluoromethane	67	69
Ethanol	45	46
Ethyl acetate	43	45, 61, 88
Ethyl acrylate	0	55
Ethyl ether (Diethyl ether)	59	45, 74
Ethyl methacrylate	69	41, 99, 86
Ethylbenzene	91	106
Ethylene dibromide (EDB, 1,2-Dibromoethane)	107	109, 188
Hexachlorobutadiene	225	223, 227
Hexane	57	41, 43, 56
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropyl alcohol	45	59
Isopropylbenzene	105	120
Isopropylether (IPE, Diisopropyl ether))	45	87, 59
m, p-Xylene	91	106
Methacrylonitrile	41	67, 39, 52
Methyl acetate	43	74, 59
Methyl methacrylate	41	69, 100, 39
Methylcyclohexane	83	55, 98, 41
Methylene chloride	84	86, 49
Methyl-t-butyl ether (MTBE)	73	57, 43
Naphthalene	128	-
n-Butanol (n-Butyl alcohol)	56	41, 43

n-Butyl acetate	43	56, 73, 61
n-Butylbenzene	91	92, 134
n-Propylbenzene	91	120
o-Xylene	91	106
Pentachloroethane	167	165, 169, 117, 83
Propionitrile (ethyl cyanide)	54	52, 55
sec-Butylbenzene	105	134
Styrene	104	78
tert-Amyl alcohol	59	55, 73, 43
tert-Amyl ethyl ether (TAME)	73	55, 87, 43
tert-Butyl ethyl ether (ETBE)	59	87, 41
tert-Butyl formate (TBF)	59	57, 41
tert-Butylbenzene	119	91, 134
Tetrachloroethene	166	129, 131, 164
Tetrahydrofuran	42	41, 71, 72
Toluene	91	92
trans-1,2-Dichloroethene	61	96, 98
trans-1,3-Dichloropropene	75	77, 39
trans-1,4-Dichloro-2-butene	53	88, 89
Trichloroethene	130	97, 95, 132
Trichlorofluoromethane	101	103, 105
Vinyl acetate	43	86
Vinyl chloride	62	84
Internal Standards/Surrogates:		
Fluorobenzene	96	70
Chlorobenzene-d_5	117	82
1,4-Dichlorobenzene-d_4	152	115, 78
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	111	113
1,2-Dichloroethane-d_4	65	67, 51
Toluene-d_8	98	100

Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Fluorobenzene	**Chlorobenzene-d_5**	**1,4-Dichlorobenzene-d_4**
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloro-1,2,2-trifluoroethane	1,1,2-Trichloroethane	1,2,3-Trichlorobenzene
1,1-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichloropropane
1,1-Dichloroethene	2-Chloroethylvinylether	1,2,3-Trimethylbenzene
1,1-Dichloropropene	2-Hexanone	1,2,4-Trichlorobenzene
1,2-Dichloroethane	3,3-Dimethyl-1-butanol	1,2,4-Trimethylbenzene
1,2-Dichloroethane-d_4 (s)	4-Methyl-2-pentanone (MIBK)	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dichloropropane	Benzyl chloride	1,2-Dichlorobenzene
1,4-Dioxane	Bromoform	1,3,5-Trichlorobenzene
2,2-Dichloropropane	Chlorobenzene	1,3,5-Trimethylbenzene
2-Butanone	Chlorodibromomethane	1,3-Dichlorobenzene

Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
2-Chloro-1,3-butadiene (Chloroprene)	cis-1,3-Dichloropropene	1,4-Dichlorobenzene
2-Methyl-2-propanol (tert-Butyl alcohol)	Ethyl methacrylate	2-Chlorotoluene
2-Nitropropane	Ethylbenzene	2-Methylnaphthalene
3-Chloro-1-propene (Allyl chloride)	Ethylene dibromide (1,2-Dibromoethane)	4-Chlorotoluene
Acetone	Isopropylbenzene	4-Isopropyltoluene (p-Isopropyltoluene)
Acetonitrile	m,p-Xylene	Bromobenzene
Acrolein	o-Xylene	Bromofluorobenzene (s)
Acrylonitrile	Styrene	Hexachlorobutadiene
Benzene	Tetrachoroethene	Naphthalene
Butadiene	Toluene	n-Butylbenzene
Chlorobromomethane	Toluene-d8 (s)	n-Propylbenzene
Bromomethane	trans-1,3-Dichloropropene	Pentachloroethane
Carbon disulfide		sec-Butylbenzene
Carbon tetrachloride		tert-Buylbenzene
Chloroethane		trans-1,4-Dichloro-2-butene
Chloroform		
Chloromethane		
cis-1,2-Dichloroethene		
Cyclohexane		
Cyclohexanone		
Dibromofluoromethane (s)		
Dibromomethane		
Dichlorobromomethane		
Dichlorodifluoromethane		
Dichlorofluoromethane		
Isopropyl ether (IPE, Diisopropyl ether)		
Ethanol		
Ethyl acetate		
Ethyl acrylate		
Ethyl ether (Diethyl ether)		
Hexane		
Iodomethane		
Isobutyl alcohol (Isobutanol)		
Isopropyl alcohol (Isopropanol)		
Methacrylonitrile		
Methyl acetate		
Methyl cyclohexane		
Methyl methacrylate		
Methylene chloride		
Methyl-tert-butyl ether (MTBE)		
n-Butanol (n-Butyl alcohol)		
n-Butyl acetate		
n-Heptane		
Propionitrile		

Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
t-Amyl alcohol		
tert-Amyl methyl ether (TAME)		
tert-Butyl ethyl ether (ETBE)		
tert-Butyl formate		
Tetrahydrofuran		
trans-1,2-Dichloroethene		
Trichloroethene		
Trichlorofluoromethane		
Vinyl acetate		
Vinyl chloride		

18.0 Revision History

- Revision 12, 10 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
- Revision 13, 25 September 2009
  - Move QC summary table and QC sample preparation instructions into Section 9.
  - Addition of new analytes: 3,3-Dimethyl-1-butanol (SC) and 1,3,5-Trichlorobenzene (NH).
  - Addition of Attachment 3 for South Carolina.
- Revision 14, 6 November 2009
  - Corporate review.
  - Addition of single-point surrogate calibration.
  - Incorporate Michigan GRO requirements.
- Revision 15, 30 October 2010
  - Addition of Amendments a (SC PQLs, Attachment 3), b (characteristic ions for 1,2,3-Trichloropropane), and c (WI soil extraction procedure).
  - Addition of new analytes: 1-Methylnaphthalene (New Mexico), Pentane, Octane, Nonane (Paraffin group).
  - Addition of QAF-45 and Section 14.2.
- Revision 16, 30 September 2011
  - Organizational changes.
  - Addition of requirement for non-preserved sample if 2-Chloroethyl vinyl ether is analyzed.
  - Addition of Minnesota Ethanol analysis requirements to Section 16.0.
  - Addition of reference to SOPs Calibration Curves (General) and Acceptable Manual Integration Practices / CA-Q-S-002.
  - Addition of 2-Chloroethyl vinyl ether, Acrolein, and Acrylonitrile preservation information.
- Revision 17, 29 February 2012
  - Organizational changes.
  - Addition of Bromoform Breakdown Check.
  - Remove paraffin standard reference.
  - TPH-GRO: change CCC frequency.
  - Corrected weighting equations.
- Revision 18, dated 30 August 2013
  - Organizational changes.
  - Specify that $r^2 \geq 0.990$.
  - OK no longer limits batch size to 10 samples.
  - Add Amendment a.

- Add 8260C.
- Add new standards and new analytes.
- Change the LCS, MS, MSD to use the primary standard.
- MA also requires LCSD.
- Addition of GRO standards

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8270 / NV/SA04-22.15a** Effective Date: **8/6/2013**

Last Mod. Date: **3/29/2013**

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 16.0, Modifications; Section 17.0, Attachments

CONTROLLED DISTRIBUTION
ISSUED TO: QA Server, 04B

Revision Number with Mod ID: **15b**

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1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add bold text, delete crossed-out text.

Section 16.0, Method Modification: Add a last sentence to item 4:

Item	Modification
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis. **SC has not approved RVE/LVI.**

Section 17.0, Attachments, Attachment 1, Characteristic Ions for Semivolatile Compounds: Modify the characteristic ions for the following compounds:

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
o,o,o-Triethylphosphorthioate	5.302	198	**121, 97** ~~80, 53, 54164, 63~~
1,4-Phenylenediamine	5.734	**108** ~~198~~	80, **107** ~~53, 54, 52~~
n-Octadecane	7.586	**57** ~~58~~	71~~, 85~~
Hexachlorophene	9.070	**196** ~~185~~	**198, 209** ~~209, 406~~

Michael A. Brum 8/6/13	8/6/13
Technical and Quality Assurance Approval Date	Operations Manager Approval Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8270 / NV/SA04-22.15** Effective Date: **3/29/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 3.0, Definitions; Section 7.0, Reagents and Standards, Section 9.1, Sample QC, Section 10.2, Calibration, Section 16.0, Method Modification

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ISSUED TO: QA Server, 04B

Revision Number with Mod ID: **15a**

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1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add bold text, delete crossed-out text.

Section 3.0, Definitions: Add a last sentence to 3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI)**: Generally, reduce all concentrations by a factor of RVE/LVI, i. e., 5.**

Section 7.0, Reagents and Standards

7.6 GC/MS Tuning Standard: Add to the first sentence: "A Methylene chloride solution containing 50 µg/mL **[RVE/LVI: 10 µg/L]** of Decafluorotriphenylphosphine (DFTPP) is prepared.

7.7 Surrogate Standards: To the bullet item, add a last sentence: **Dilute by five for RVE/LVI.**

Section 9.1, Sample QC, Surrogate recoveries: Delete the phrase: The limits for surrogate recoveries are updated biannually ~~(see TestAmerica Nashville's current Control Limits Manual (CLM))~~.

Section 10.2, Calibration, Initial Calibration, Steps 1 and 2: Add bold column.

- Prepare calibration standards at five (minimum) different concentrations.

Traditional Volume Concentration (µg/mL)	**RVE/LVI Concentration (µg/mL)**	µL of 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 200 µg/mL standard/500 µL (5 µL injection)
2	**0.4**	5	1

10	**2**	25	5
20	**4**	50	10
50	**10**	125	25
80	**16**	200	40
100	**20**	250	50

For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	**RVE/LVI Concentration (µg/mL)**	µL of 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 10 µg/mL standard/500 µL(5 µL injection)
0.05*	**0.01**	2.5	0.5
0.1	**0.02**	5	1
0.5	**0.1**	25	5
1	**0.2**	50	10
5	**1.0**	250	50
10	**2.0**	500	100

*The lowest calibration ~~0.05 µg/mL~~ standard must be used for low-level SIM analysis on samples from Wisconsin.

Section 16.0, Method Modification: Add item 5 to the table:

Item	Modification
5	**RVE/LVI**

UNCONTROLLED DOCUMENT

Michael H. Dunn 3/11/13	[signature] 3/11/13
Technical and Quality Assurance Approval Date	Operations Manager Approval Date

SOP Number/Revision No.: **8270 / NV/SA04-22.15** Effective Date: **3/29/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Revision Number with Mod ID: **15a**

Title: SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHOD 8270C/D

Approvals (Signature/Date)			
	12/28/12		12/28/12
Cory Spry Extractables Operations Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date
	12/28/12		
Michael H. Dunn Technical Director Quality Assurance Manager	Date		

Analyze and report by 8270D for Canadian, **NJ, NC, OK, SC, and WV** samples.

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of oily wastes, soils/sediments, concrete, and water samples. The following compounds can be determined by this method:

Analyte	CAS #	Analyte	CAS #
Acenaphthene[1, 2, 5]	83-32-9	Hexachlorocyclopentadiene[1, 2]	77-47-4
Acenaphthene-d_{10} (IS)		Hexachloroethane[1, 2]	67-72-1
Acenaphthylene[1, 2, 5]	208-96-8	Hexachlorophene[2]	70-30-4
Acetophenone[2]	98-86-2	Hexachloropropene[2]	1888-71-7
2-Acetylaminofluorene[2]	53-96-3	Indeno(1,2,3-cd)pyrene[1, 2, 5]	193-39-5
4-Aminobiphenyl[2]	92-67-1	*Indene*[4]	
Aniline[2]	62-53-3	Isodrin[2]	465-73-6
Anthracene[1, 2, 4, 5]	120-12-7	Isophorone[1, 2]	78-59-1
Aramite[2]	140-57-8	*cis-Isosafrole*[2]	17627-76-8
Azobenzene[3]	103-33-3	*trans-Isosafrole*[2]	4043-71-4
Benzidine[3]	92-87-5	Kepone[2]	143-50-0
Benzoic acid[3]	65-85-0	Methapyrilene[2]	91-80-5
Benz(a)anthracene[1, 2, 4, 5]	56-55-3	3-Methylcholanthrene[2]	56-49-5
Benzo(b)fluoranthene[1, 2, 4, 5]	205-99-2	*6-Methylchrysene*[4]	1705-85-7
Benzo(j)fluoranthene[4]		4,4'-Methylenebis(2-chloroaniline) (MOCA)[3]	101-14-4
Benzo(k)fluoranthene[1, 2, 4, 5]	207-08-9	Methyl methanesulfonate[2]	66-27-3
Benzo(g,h,i)perylene[1, 2, 5]	191-24-2	*2-Methylnaphthalene*[3, 4, 5]	90-12-0
Benzo(a)pyrene[1, 2, 4, 5]	50-32-8	2-Methylnaphthalene[1, 2, 5]	91-57-6
Benzyl alcohol[2]	100-51-6	Methyl parathion[2]	298-00-0
Bis(2-chloroethoxy)methane[1, 2]	111-91-1	2-Methylphenol[1, 2, 4]	95-48-7
Bis(2-chloroethyl)ether[1, 2]	111-44-4	3-Methylphenol[1, 2, 4]	108-39-4
Bis(2-chloroisopropyl)ether[1, 2]	108-60-1	4-Methylphenol[1, 2, 4]	106-44-5
Bis(2-ethylhexyl)adipate[3]	103-23-1	Naphthalene[1, 2, 4, 5]	91-20-3
Bis(2-ethylhexyl)phthalate[1, 2, 4]	117-81-7	Naphthalene-d_8 (IS)	
Bisphenol A[3]	80-05-7	1,4-Naphthoquinone[2]	130-15-4
4-Bromophenyl phenylether[1, 2]	101-55-3	1-Naphthylamine[2]	134-32-7
Butyl benzyl phthalate[1, 2, 4]	85-68-7	2-Naphthylamine[2]	91-59-8
Carbazole[1]	86-74-8	2-Nitroaniline[1, 2]	88-74-4
4-Chloroaniline[1, 2]	106-47-8	3-Nitroaniline[1, 2]	99-09-2
Chlorobenzilate[2]	510-15-6	4-Nitroaniline[1, 2]	100-01-6
4-Chloro-3-methylphenol[1, 2]	59-50-7	Nitrobenzene[1, 2]	98-95-3
1-Chloronaphthalene[3]	90-13-1	Nitrobenzene-d_5 (surr)	
2-Chloronaphthalene[1, 2]	91-58-7	2-Nitrophenol[1]	88-75-5
2-Chlorophenol[1, 2]	95-57-8	4-Nitrophenol[1, 2]	100-02-7
2-Chlorophenol-d_4 (surr)		5-Nitro-o-toluidine[2]	99-55-8
4-Chlorophenyl phenylether[2]	7005-72-3	Nitroquinoline-1-oxide[2]	56-57-5
Chrysene[1, 2, 4, 5]	218-01-9	n-Nitrosodi-n-butylamine[2]	924-16-3
Chrysene-d_{12} (IS)		n-Nitrosodiethylamine[2]	55-18-5
n-Decane[3]	124-18-5	n-Nitrosodimethylamine[2]	62-75-9
Diallate (cis and trans)[2]	2303-16-4	n-Nitrosomethylethylamine[2]	10595-95-6
Dibenz(a,h)acridine[4]	226-36-8	n-Nitrosodiphenylamine[1, 2] and Diphenylamine	86-30-6 and 122-39-4
Dibenz(a,j)acridine[3]	224-42-0	n-Nitrosodi-n-propylamine[1, 2]	621-64-7
Dibenz(a,h)anthracene[1, 2, 4, 5]	53-70-3	n-Nitrosomorpholine[2]	59-89-2
Dibenzofuran[1, 2]	132-64-9	n-Nitrosopiperidine[2]	100-75-4
2,3-Dichloroaniline[3]	608-27-5	n-Nitrosopyrrolidine[2]	930-55-2

1,2-Dichlorobenzene[1, 2, 4]	955-50-1	*n-Octadecane*[3]	593-45-3
1,2-Dichlorobenzene-d_4(surr)		Parathion[2]	56-38-2
1,3-Dichlorobenzene[1, 2, 4]	541-73-1	Pentachlorobenzene[2]	608-93-5
1,4-Dichlorobenzene[1, 2, 4]	106-46-7	*Pentachloroethane*[2]	76-01-7
1,4-Dichlorobenzene-d_4(IS)		Pentachloronitrobenzene[2]	82-68-8
3,3'-Dichlorobenzidine[1, 2]	91-94-1	Pentachlorophenol[1, 2]	87-86-5
2,4-Dichlorophenol[1, 2]	120-83-2	Perylene-d_{12} (IS)	
2,6-Dichlorophenol[2]	87-65-0	Phenacetin[2]	62-44-2
Diethyl phthalate[1, 2, 4]	84-66-2	Phenanthrene[1, 2, 4, 5]	85-01-8
Dimethoate[2]	60-51-5	Phenanthrene-d_{10} (IS)	
Dimethylaminoazobenzene[2]	60-11-7	Phenol[1, 2, 4]	108-95-2
7,12-Dimethylbenz(a)anthracene[2, 4]	57-97-6	Phenol-d_5 (surr)	
3,3'-Dimethylbenzidine[2]	119-93-7	1,4-Phenylenediamine[3]	106-50-3
2,4-Dimethylphenol[1, 2, 4]	105-67-9	Phorate[2]	298-02-2
a,a- Dimethylphenethylamine[2]	122-09-8	2-Picoline (2-Methylpyridine)[2]	109-06-8
Dimethyl phthalate[1, 2, 4]	131-11-3	Pronamide[2]	23950-58-5
Di-n-butyl phthalate[1, 2, 4]	84-74-2	Pyrene[1, 2, 4, 5]	129-00-0
1,3-Dinitrobenzene[2]	99-65-0	Pyridine[2, 4]	110-86-1
4,6-Dinitro-2-methylphenol[1, 2]	534-52-1	*Quinoline*[3]	91-22-5
2,4-Dinitrophenol[1, 2, 4]	51-28-5	Safrol[3]	94-59-7
2,4-Dinitrotoluene[1, 2, 5]	121-14-2	Terphenyl-d_{14}(surr)	1718-51-0
2,6-Dinitrotoluene[1, 2, 5]	606-20-2	*Alpha-Terpineol*[3]	7785-53-7
Dinoseb[2]	88-85-7	1,2,4,5-Tetrachlorobenzene[2]	95-94-3
1,4-Dioxane	123-91-9	2,3,4,6-Tetrachlorophenol[2]	58-90-2
1,2-Diphenylhydrazine[3]	122-66-7	*Tetraethyl dithiopyrophosphate (Sulfotepp)*[2]	3689-24-5
Di-n-octyl phthalate[1, 2, 4]	117-84-0	Tetraethylpyrophosphate[3]	107-49-3
Disulfoton[2]	298-04-4	Thionazine[2]	297-97-2
Ethyl methanesulfonate[2]	62-50-0	Thiophenol[4]	108-98-5
Famphur[3]	52-85-7	o-Toluidine[2]	95-53-4
Fluoranthene[1, 2, 4, 5]	206-44-0	2,4,6-Tribromophenol (surr)	118-79-6
Fluorene[1, 2, 5]	86-73-7	1,2,4-Trichlorobenzene[1, 2]	120-82-1
2-Fluorobiphenyl(surr)	321-60-8	2,4,5-Trichlorophenol[1, 2]	95-95-4
2-Fluorophenol (surr)	367-12-4	2,4,6-Trichlorophenol[1, 2]	88-06-2
Hexachlorobenzene[1, 2]	118-74-1	o,o,o-Triethylphosphorothioate[2]	126-68-1
Hexachlorobutadiene[1]	87-68-3	1,3,5-Trinitrobenzene[2]	99-35-4
Compounds in italics are not present in the EPA method.			
[1] – Normal laboratory 8270 compounds. ***For OH VAP projects, report only compounds designated by a 1 superscript; see Attachment 5.***			
[2] - Appendix IX compounds (by request only)			
[3] - additional compounds available by this method (by request only)			
[4] - Skinner List for Refinery Waste compounds (by request only)			
[5] - Compounds that are available by GC/MS-SIM (by request only)			
IS = These compounds are used as internal standards.			
surr = These compounds are used as surrogates.			

This method is used to quantitate neutral, acidic, and basic organic compounds that are soluble in Methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica capillary column coated with a slightly polar methyl silicone phase. This method is not appropriate for the quantitation of multi-component analytes, e. g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analyses. This method is appropriate for confirmation of the presence of these analytes when concentration in the extract

permits. However, it is appropriate for the multi-component analyte, Diesel Range Organics (DRO), as requested by Missouri and California; see Attachment 6.

1.2 Reporting Limits: The laboratory typical report limit (RL) is approximately 2 - 100 µg/L for water samples, 67 - 670 µg/kg (wet weight) for soil/sediment samples, and 10 - 1000 mg/kg for wastes (dependent on matrix and method of preparation). See the following table for typical RLs for each compound. For the most current analyte RLs, refer to LIMS.

Typical Reporting Limits for 8270 Compounds

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**Acenaphthene**	10	0.333	♦*Isosafrole*	50	1.67
♦**Acenaphthylene**	10	0.333	♦*Kepone*	10	0.333
♦*Acetophenone*	10	0.333	♦*Methapyrilene*	50	0.333
♦*2-Acetylaminofluorene*	10	0.333	♦*3-Methylcholanthrene*	10	0.333
♦*4-Aminobiphenyl*	10	0.333	♣*6-Methylchrysene*	10	0.333
♦*Aniline*	10	0.333	♦*Methyl methanesulfonate*	10	0.333
♦♣**Anthracene**	10	0.333	♣*1-Methylnaphthalene*	10	0.333
♦*Aramite*	50	1.67	♦**2-Methylnaphthalene**	10	0.333
Atrazine	10	0.333	♦*Methyl parathion*	10	1.67
Azobenzene	10	0.333	♦♣**2-Methylphenol**	10	0.333
Benzaldehyde	10	1.67	♦♣**3,4-Methylphenol**	10	0.333
Benzidine	100	1.67	♦♣**Naphthalene**	10	0.333
Benzoic acid	50	1.67	♦*1,4-Naphthoquinone*	10	1.67
♦♣**Benzo(a)anthracene**	10	0.333	♦*1-Naphthylamine*	10	0.333
♦♣**Benzo(a)pyrene**	10	0.333	♦*2-Naphthylamine*	10	0.333
♦♣**Benzo(b)fluoranthene**	10	0.333	♦**2-Nitroaniline**	25	0.833
♦**Benzo(g,h,i)perylene**	10	0.333	♦**3-Nitroaniline**	25	0.833
♣*Benzo(j)fluoranthene*	10	0.333	♦**4-Nitroaniline**	25	0.833
♦♣**Benzo(k)fluoranthene**	10	0.333	♦**Nitrobenzene**	10	0.333
♦*Benzyl alcohol*	10	0.333	♦*5-Nitro-o-toluidine*	10	1.67
Biphenyl	10	0.333	♦**2-Nitrophenol**	10	0.333
♦**Bis(2-chloroethoxy) methane**	10	0.333	♦♣**4-Nitrophenol**	25	0.833
♦**Bis(2-chloroethyl) ether**	10	0.333	♦*Nitroquinoline-1-oxide*	10	0.333
♦**Bis(2-chloroisopropyl) ether**	10	0.333	♦*n-Nitrosodiethylamine*	10	0.333
♦♣**Bis(2-ethylhexyl) phthalate**	10	0.333	♦*n-Nitroso-dimethylamine*	10	0.333
♦**4-Bromophenylphenyl ether**	10	0.333	♦*n-Nitrosodi-n-butylamine*	10	1.67
♦♣**Butyl benzyl phthalate**	10	0.333	♦**n-Nitroso-di-n-propylamine**	10	0.333
Caprolactum	10	0.333	♦**n-Nitroso-diphenylamine and Diphenylamine**	10	0.333
Carbazole	10	0.333	♦*n-Nitrosomethylethylamine*	10	0.333
♦**4-Chloro-3-methylphenol**	10	0.333	♦*n-Nitrosomorpholine*	10	1.67
♦**4-Chloroaniline**	10	0.333	♦*n-Nitrosopiperdine*	10	1.67
♦*Chlorobenzilate*	10	0.333	♦*n-Nitrosopyrrolidine*	10	1.67
1-Chloronaphthalene	10	0.333	*Octadecane*	50	0.333

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**2-Chloronaphthalene**	10	0.333	♦*Parathion*	10	1.67
♦**2-Chlorophenol**	10	0.333	♦*Pentachlorobenzene*	10	1.67
♦**4-Chlorophenylphenyl ether**	10	0.333	♦*Pentachloroethane*	10	0.333
♦♣**Chrysene**	10	0.333	♦*Pentachloronitroben-zene*	10	1.67
♦*cis-Diallate*	10	0.333	♦**Pentachlorophenol**	25	0.833
♦*trans-Diallate*	10	0.333	♦*Phenacetin*	10	1.67
♦**Dibenzofuran**	10	0.333	♦♣**Phenanthrene**	10	0.333
♣*Dibenz(a,h)acridine*	10	0.333	♦♣**Phenol**	10	0.333
Dibenz(a,j)acridine	10	0.333	♦*1,4-Phenylenediamine*	50	0.333
♦♣**Dibenzo(a,h)anthracene**	10	0.333	♦*Phorate*	10	0.333
♦♣**1,2-Dichlorobenzene**	10	0.333	♦*2-Picoline*	10	0.333
♦♣**1,3-Dichlorobenzene**	10	0.333	♦*Pronamide*	10	1.67
♦♣**1,4-Dichlorobenzene**	10	0.333	♦♣**Pyrene**	10	0.333
♦**3,3'-Dichlorobenzidine**	10	0.333	♦♣**Pyridine**	10	0.67
♦**2,4-Dichlorophenol**	10	0.333	♣*Quinoline*	10	0.333
♦*2,6-Dichlorophenol*	20	0.333	♦*Safrole*	10	0.333
3,4-Dichlorophenol	10	0.333	*Terbufos*	50	167
♦♣**Diethyl phthalate**	10	0.333	♦*1,2,4,5-Tetrachloro-benzene*	10	1.67
♦*Dimethoate*	10	1.67	♦*2,3,4,6-Tetrachloro-phenol*	10	0.333
♦*p-Dimethylaminoazobenzene*	10	1.67	♦*Tetraethylpyrophos-phate, Sulfotep*	10	1.67
♦*3,3'-Dimethylbenzidine*	50	0.333	♦*Thionazine*	10	1.67
♦♣*7,12-Dimethylbenz(a)an-thracene*	10	0.333	♣*Thiophenol*	50	1.67
♦*a,a-Dimethylphenethylamine*	50	1.67	♦*o-Toluidine*	10	1.67
♦♣**2,4-Dimethylphenol**	10	0.333	♦**1,2,4-Trichloroben-zene**	10	0.333
♦♣**Dimethyl phthalate**	10	0.333	♦**2,4,5-Trichlorophenol**	10	0..667
♦♣**Di-n-butyl phthalate**	10	0.333	♦**2,4,6-Trichlorophenol**	10	0.333
♦*1,3-Dinitrobenzene*	10	1.67	♦*o,o,o-Triethylphospho-rothioate*	10	1.67
♦**4,6-Dinitro-2-methylphenol**	25	0.833	♦*1,3,5-Trinitrobenzene*	10	0.333
♦♣**2,4-Dinitrophenol**	25	0.833	Acenaphthene, SIM	0.10	0.00333
♦**2,4-Dinitrotoluene**	10	0.333	Acenaphthylene, SIM	0.10	0.00333
♦**2,6-Dinitrotoluene**	10	0.333	Anthracene, SIM	0.10	0.00333
♦♣**Di-n-octyl phthalate**	10	0.333	Benzo(a)anthracene, SIM	0.10	0.00333
♦*Dinoseb*	10	0.333	Benzo(a)pyrene, SIM	0.10	0.00333
1,4-Dioxane	10	0.333	Benzo(b)fluoranthene, SIM	0.10	0.00333
1,2-Diphenylhydrazine	10	0.333	Benzo(g,h,i)perylene, SIM	0.10	0.00333
♦*Disulfoton*	10	1.67	Benzo(k)fluoranthene, SIM	0.10	0.00333
♦*Ethyl methanesulfonate*	10	0.333	Chrysene, SIM	0.10	0.00333
♦*Famphur*	10	0.333	Dibenzo(a,h)anthracene, SIM	0.10	0.00333
♦♣**Fluoranthene**	10	0.333	2,4-Dinitrotoluene, SIM	0.2	0.0067

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**Fluorene**	10	0.333	2,6-Dinitrotoluene, SIM	0.2	0.0067
♦**Hexachlorobenzene**	10	0.333	Fluoranthene, SIM	0.10	0.00333
♦**Hexachlorobutadiene**	10	0.333	Fluorene, SIM	0.10	0.00333
♦**Hexachlorocyclopentadiene**	10	0.333	Indeno(1,2,3-cd)pyrene, SIM	0.10	0.00333
♦**Hexachloroethane**	10	0.333	1-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachlorophene*	50	3.33	2-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachloropropene*	50	3.33	Naphthalene, SIM	0.10	0.00333
♦**Indeno(1,2,3-c,d)pyrene**	10	0.333	Phenanthrene, SIM	0.10	0.00333
♣*Indene*	10	1.67	Pyrene, SIM	0.10	0.00333
♦*Isodrin*	10	0.333	California / Missouri DRO	500	20
♦**Isophorone**	10	0.333	Calilfornia / Missouri ORO	500	20

♦ indicates Appendix IX compound ♣Skinner List compound
Bold compounds are reported in a standard list.
Italicized compounds are only available upon special request by this method.
SIM = Selective Ion Monitoring

1.3 The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration, and its chromatographic behavior is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- n-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- n-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. Use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation. See SOPs 3510 / NV03-24 for waters, 3550 / NV03-23 and 3541 / NV03-231 for soils and concrete, and 3580 / NV03-106 for oils, and, if necessary, sample cleanup procedures.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass

spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a multi-point calibration curve.

3.0 Definitions

3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI): The option to use a reduced sample volume for extraction combined with a larger volume extract injection on the instrument. Volumes for this option are shown in this document as RVE/LVI in brackets.

3.2 See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This method may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. Be aware of the locations of those zones, and cool them to room temperature prior to working on them.
- The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP or Agilent): Analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.
  - Column: 30 m x 0.25 mm ID with a 0.25 µm film thickness silicone-coated fused-silica capillary column (Phenomenex ZB-5, or equivalent) [RVE/LVI: and a 5 m x 0.32 mm ID guard column (Phenomenex 7CG-G000-000-GZ0, or equivalent].
  - Mass spectrometer capable of scanning from 35 to 500 amu every 1 second less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 2 when 1µL of the GC/MS tuning standard is injected (50 ηg or less of DFTPP).
  - Data system (Chemstation with Enviroquant): A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available that allows integrating the abundances in any EICP between specified time or scan-number limits. The EPA/NIST Mass Spectral Library is also available.
  - Suggested operating conditions (may vary by instrument; see maintenance log for current program):

Mass range:	35-500 amu
Scan time:	1 second/scan
Initial temperature:	40°C hold for 2 minutes
Temperature program:	Rate 1: 15°C/minute to 160°C
	Rate 2: 10°C/minute to 320°C
Final temperature:	320°C hold for at least 1.5 minute.
Injector temperature:	240-250°C
Transfer line temperature:	280°C
Source temperature:	According to manufacturer's specifications (nominally 250 – 275°C)
Injector:	Grob-type, split-less
Injection volume:	1 µL [RVE/LVI: 5 µL]
Carrier gas:	Helium at 1 mL/minute

6.2 Supplies

- Microsyringe, 10 µL.
- Balance, analytical, capable of weighing 0.0001 g
- Glass vials, glass with PTFE (polytetrafluoroethylene)-lined screw-caps or crimp tops.
- Volumetric flasks, Class A, appropriate sizes with ground-glass stoppers.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise, indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water, analyte-free.

7.3 Stock Calibration Standards: Commercially prepared, certified stock standards are purchased:

- The primary standard for the typical 8270 compound list is from Ultra Scientific CUS-6150, or equivalent, with the required targets at 200 µg/mL.
- For PAHs by SIM, use Accustandard Z-014G-FL, or equivalent, with the target PAHs at 2000 µg/mL.
- For Appendix IX and miscellaneous compounds, primary source standards are purchased from NSI; equivalent substitutes are acceptable.

Analyte/Analyte Group	NSI Catalog Number	Concentration (µg/mL)
AIX Mix	C-426	2000
Acid Extractables II	C-415	2000
Amines	C-412	2000
Aramite	922-05-02	2000
a,a-Dimethylphenylamine	922-05-02	2000
Benzidines	C-411	2000
BNA II mix	C-413	2000
B/N III mix	C-414	2000
Hexachlorophene	323-03	5000
Sulfonates	C-416	2000
8270 OP Pest	C-417	2000

7.4 Matrix Spike and Laboratory Control Standard contains all targets to be reported on the samples. The same compounds mentioned in Section 7.3 are designated as the SPCCs and CCCs for 8270C.

- For both a long semivolatile list and the PAH list by SIM, purchase as the second source a 100 µg/mL standard, NSI Catalog # c-408-50x, or equivalent.
- For Appendix IX and miscellaneous compounds, these second source standards are acceptable, as well as equivalents:

Analyte/Analyte Group	RestekCatalog Number	Concentration (µg/mL)
AIX #1 Mix	31625	2000
AIX #2 Mix	31806	2000
Calibration Mix	31618	2000
OP Mix	32419	2000

7.5 Internal standard solutions: The internal standards are 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}.

- Purchase certified, internal standard at 4000 µg/mL, NSI C-394, or equivalent.

7.6 GC/MS Tuning Standard: A Methylene chloride solution containing 50 µg/mL of Decafluorotriphenylphosphine (DFTPP) is prepared. The standard also contains 50 µg/mL each of 4, 4'-DDT, Pentachlorophenol, and Benzidine to verify injection port inertness and GC column performance.

- Purchase the tuning standard at 1000 µg/mL from Ultra Scientific, Catalog GCM-150, or equivalent.

7.7 Surrogate standards: The surrogates are Phenol-d_5, 2-Fluorophenol, 2,4,6-Tribromophenol, Nitrobenzene-d_5, 2-Fluorobiphenyl, and p-Terphenyl-d_{14}.

- Purchase the acid/base/neutral and PAH SIM surrogates from NSI, C-LIS-7070, or equivalent, at 50 µg/mL.

7.8 Acetone, Hexane, Methylene chloride, Isooctane, Carbon disulfide, Toluene, and other appropriate solvents, commercial source.

7.9 Sodium sulfate for blank and LCS soil matrix.

7.10 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions must be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Replace after **one year or sooner** if comparison with quality control check samples indicates a problem, or if the vendor specifies an expiration date sooner than one year.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	3 L, amber glass with Teflon®-lined cap	1 L [RWE/LVI: 250 mL]	Cool 0-6°C. Keep in dark.	7 days from collection until extraction, 40 days after extraction	SW-846 Chapter 2
Soil, Oil, Concrete	4 oz. glass jar with Teflon®-lined cap	30 g	Cool 0-6°C.	14 days from collection until extraction, 40 days after extraction	

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

Certain quality control and reporting criteria may vary depending on whether SW-846 8000B or 8000C criteria are required. In these cases, both sets of criteria have been noted in this SOP. 8000C criteria are required to be applied ONLY to Arizona and Washington samples. All other samples must be processed against referenced 8000B criteria. Exceptions may be required on a project-specific basis.

9.1 Sample QC:

The following QC samples are run with each batch of no more than 20 samples.			
QC Check	Frequency	Acceptance Criteria[1]	**Corrective Action[2]**
Method blank	One per analytical prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank.
LCS[6] for all analytes (2nd source)[6]	One[6] per prep batch	See LIMS and footnote 4 below.	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch.[4] If high and target is ND, OK to report.
MS/MSD (2nd source)	One per batch per matrix, if insufficient sample for MS/MSD, qualify data[3]	See LIMS.	None (the LCS is used to evaluate to determine if the batch is acceptable).
Surrogate(s)	Every sample, spike, standard, and blank	See LIMS.[5]	Check system, re-analyze, re-prep[3, 5].

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[3]If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then place a comment on the benchsheet and in LIMS.
[4]If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the samples from that batch is less than the RL, the data is acceptable to report.
[5]If the surrogate % recovery exceeds the upper control limit AND a sample result is positive above the RL, re-prep and re-analyze the batch. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prep the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed.
[6]LCSD is required for AZ, MA, TX, WV.

- A **Method blank** is extracted with every batch of samples.
- A **Laboratory Control Sample** (LCS) is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume (reagent water for water batches, Sodium sulfate for soil batches). It is spiked with the same analytes at the same concentrations as the matrix spike. **All target analytes must meet the LCS QC criteria** (laboratory historical limits in LIMS)**.** However, if the LCS is high, and a target is ND, it is acceptable to report the result.
  - The LCS spike is from a **different source than the calibration standards**. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches:**
      - Water: add 500 µL [RVE/LVI: 100 µL] of the standard per liter reagent water before extraction by Method 3510C.
      - Soil: add 500 µL of the standard per 30 gram Sodium sulfate before extraction.
      - TCLP: add 1 mL [RVE/LVI: 200 µL] of the standard/500 mL TCLP extraction fluid before extraction by Method 3510C.
      - The final concentration is 50 µg/mL on column.
    - **For SIM batches:**
      - Water: add 1 mL [RVE/LVI: 200 µL] of a 100 X dilution of the NSI standard per liter reagent water.
      - Soil: add 1 mL of a 100 X dilution of the NSI standard per 30 g Sodium sulfate.
      - The final concentration in the extracts is 1.0 µg/mL.
- **Matrix Spike / Matrix Spike Duplicate**: Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair.
  - The MS/MSD spike is from a **different source** than the calibration standards. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches**:

- Water: add 500 µL [RVE/LVI: 100 µL] of the standard per liter client sample.
- Soil: add 500 µL of the standard to 30 g client sample.
- TCLP: add 1 mL [RVE/LVI: 200 µL] of the standard per 500 mL client TCLP extract.
- The final concentration is 50.0 µg/mL on column..

- **For SIM batches**:
  - Water: add 1 mL [RVE/LVI: 200 µL] of a 100 X dilution of the NSI standard per liter reagent water.
  - Soil: add 1 mL of 100 X dilution per 30 g client sample.
  - The final concentration is 1.0 µg/mL on column.

- **Surrogate recoveries:** The laboratory evaluates surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The limits for surrogate recoveries are updated biannually (see TestAmerica Nashville's current Control Limits Manual (CLM)). If any surrogate is outside QC limits, and there is no obvious matrix interference, then re-analyze and/or re-extract the sample. If surrogates are still outside limits, flag the data in LIMS. However, if high and all results are non-detect, results are reportable. If surrogate recoveries are low, re-prep the batch.
  - **For Non-SIM**, add 1000 µL [RVE/LVI: 200 µL] of the surrogate standard at a concentration of 50 µg/mL to each sample and batch QC samples prior to extraction for a 50 µg/mL concentration.
  - **For SIM,** prepare a 1 µg/mL standard (500 µL surrogate standard) to 500 mL in methanol. Add 1.0 mL [RVE/LVI: 200 µL] to samples and QC (blanks, MS/MSD and LCS) prior to extraction. The concentration is 1.0 µg/mL.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
GC/MS Tuning			
a. Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours.	See below in this section for GC/MS Tuning criteria.	Retune the instrument and verify (instrument maintenance may be needed).
b. Column Breakdown	Prior to initial calibration or Continuing calibration verification, every 12 hours.	Breakdown ratio ≤ 20% (30% for 8270C).	Injector or column maintenance and re-calibration.
c. Tailing Factor	Prior to initial calibration or Continuing calibration verification, every 12 hours.	8270C 8270D Benzidine 3 2 Pentachlorophenol 5 2	Injector or column maintenance and re-calibration.
Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8270C: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15% If %RSD is > 15%, linear regression $r^2 \geq 0.990$, $r \geq 0.995$. 8270D: The minimum RF for all compounds in Attachment 5 must be met[5]. All targets RSD ≤ 20% or use linear regression.	Correct problem then repeat initial calibration.

QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration.	All analytes within 30% of expected value.	Correct problem then repeat initial calibration.
Initial calibration blank	Immediately after ICV	All analytes < MDL	Correct problem, re-calibrate.
Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8270C: SPCCs average RF ≥ 0.050 and CCCs: ≤30% difference (when using RFs) or drift (when using least squares regression). Non-CCC < 20% true; up to 4 may be < 40%. 8270D: The minimum RF for all compounds listed in Attachment 4 must be met and the percent difference or drift for each target compound ≤ 20%.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
Internal Standards	Every sample/standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL for CCV. EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. See footnote 4 below.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical manager for advice).
Relative Retention Time Window	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check.
MDL verification (extracted)	Minimum yearly.	Detectible	Re-evaluate MDL standard used and MDL; see the technical manager.

[1]8270 requires DFTPP.
[2]This is a summary of the acceptance criteria.
[3]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[4]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.
[5]LLCV: If RF is not met at the low-level standard, the criterion for a passing LLCV is detection only and must be run following the CCV.

- **Tuning**

GC/MS Tuning (Full Scan)

- Prior to the analysis of samples or calibration standards, the GC/MS system is hardware-tuned using a 50 ng or less injection of DFTPP (in the GC/MS Tuning Standard).
- The 50 µg/mL standard is prepared by adding 2.8 mL of 1000 µg/mL stock standard to 56 mL Methylene chloride . [RVE/LVI: Use a 5X dilution of this solution.]
- Analyses **must** not begin until the tuning criteria are met, and these criteria must be demonstrated at the beginning of each 12-hour shift. Three options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ±1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does

not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct

Option 2 The entire peak may be averaged and background-corrected. Average scans from 0.1 minute before to 0.1 minute after peak.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass, it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance**.

- All subsequent standards, samples, controls, and blanks associated with a DFTPP tune must use the identical mass spectrometer instrument conditions.
- Use the DFTPP mass intensity criteria as follows as tuning acceptance criteria.

DFTPP Key Ions and Ion Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40-60 percent of mass 198.
197	Less than 1 percent of mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of mass 198.
275	10-30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than mass 443.
442	Greater than 40 percent of mass 198.
443	17-23 percent of mass 442.

- **Breakdown Standard**: The GC/MS Tuning Standard is also used to assess the injection port inertness by evaluating the degradation of DDT to DDE and DDD. This ratio must **not** exceed 20%; see Section 9.2 for **percent breakdown** calculation. Perform injector or column maintenance and recalibrate if the ratio maximum is exceeded for either compound. The breakdown of DDT is measured **before** verification standards and samples are analyzed and every 12 hours throughout the sequence.
- **Tailing Factor**: To evaluate the GC column, Benzidine and Pentachlorophenol (in the GC/MS Tuning Standard) must be present at their normal responses and evaluated for peak tailing. The Benzidine and Pentachlorophenol tailing factors are calculated by the following equation:

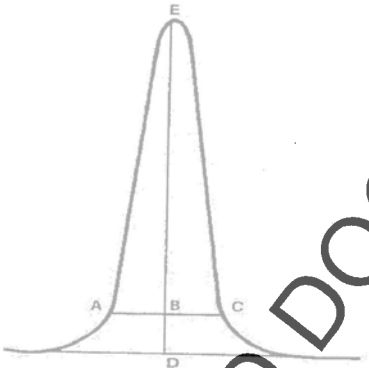
Tailing factor = BC/AB

Maximum Tailing Factor Ratios

Tailing Factor Compounds	8270C	8270D
Benzidine	3	2
Pentachlorophenol	5	2

where the peak is defined as follows: AC is the width at 10% height. DE is the height of peak and B is the height at 10% DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

FIGURE 1
TAILING FACTOR CALCULATION



TAILING FACTOR = $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm
Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

8270D - 72

Revision 4
February 2006

If all of the specified criteria are met, generate a hardcopy of the spectrum, the mass abundance data and the parameters under which the scans were acquired. This data is filed in the batch for documentation.

GC/MS Tuning (SIM)

- The objective of tuning for conventional full scan analysis is to produce a balanced mass spectrum over the range of interest. The DFTPP tune is, by necessity, done in the full scan mode. However, because the instrument is then immediately switched to the SIM mode, the DFTPP results have limited quality control value. In short, the DFTPP is not analyzed under the same conditions as the calibration, QC, and field samples. In the case of Selective Ion Monitoring (SIM) analysis, there are no comparisons between spectra; instead the instrument is optimized for the relative intensities of the pre-selected analyte ions of interest. For SIM analysis, the laboratory prints out a copy of the autotune (PFTBA) prior to analysis to demonstrate good mass assignment and peak width. No BFB tune is possible while in SIM mode. A printout of the instrument autotune (PFTBA) is included with the data for each day that SIM analyses are run in order to demonstrate good mass assignment and peak width.

- **Calibration**: See Section 10.2.
- **Initial Calibration System Performance Check Compounds (SPCCs)**: A system performance check is performed to ensure that minimum average RFs are met before the calibration curve is used.
  - **For 8270C**: The SPCCs are

System Performance Check Standards (SPCCs)	
Base/Neutral Fraction	**Acid Fraction**
n-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

The **minimum acceptable average RF for the SPCCs is 0.050**. They typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

  - **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
  - If the minimum response factors are not met, the system must be evaluated, and corrective action is taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. **This check must be met before sample analysis begins.** An option is to run a LLCCV to show sensitivity.
- **Initial Calibration Calibration Check Compounds (CCCs) for 8270C only**: The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes. The CCCs are:

Calibration Check Compounds (CCC)	
Base/Neutral Fraction	**Acid Fraction**
Acenaphthene*	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene*	2,4,6-Trichlorophenol
Benzo(a)pyrene*	

*For PAH SIM standard

  - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte.

Initial Calibration RSD Differences	
8270C	**8270D**
The RSD must be less than or equal to **15%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. However, the RSD for each individual CCC must be less than or equal to 30%.	The RSD must be less than or equal to **20%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. If not, check

If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.	errors in standard preparation, the possible presence of active sites in the GC system, poor chromatographic behaviors for analytes.

- The **Initial Calibration Verification (ICV)** is a second-source standard run immediately after the initial calibration. The acceptance limits are **70-130%** recovery.
  - Add 250 µL of the second-source standard to 250 µL Methylene chloride in an amber vial to prepare an ICV standard at 50 µg/mL.
  - For PAHs by SIM, use the second-source standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. The ICV at 1 µg/mL is made by taking 50 µL of intermediate into 450 µL of Methylene chloride in an amber vial.
  - If ICV acceptance criterion is not met, correct the problem and re-calibrate.
- **Initial Calibration Blank**: a reagent/solvent blank analyzed after the ICV to ensure the system is free of contaminants (< MDL). If not contaminant-free, re-run and/or perform system maintenance.
- The **Continuing Calibration Verification standard (CCV)** is evaluated each day (or every 12 hours) that analysis is performed to determine if the chromatographic system is operating properly.
  - Prepare a daily CCV at 50 µg/mL by adding 100uL of the primary stock solution to 300 µL Methylene chloride in an amber vial. [RVE/LVI: 20 µL to a final volume of 400uL Methylene chloride].
  - For PAHs by SIM, use the primary stock standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. A daily CCV at 1 µg/mL is made by taking 50 µL of intermediate into 450 µL of Methylene chloride in an amber vial. [RVE/LVI: 5 µL to a final volume of 500uL of Methylene Chloride].
  - The calibration verification standard is prepared at least weekly and stored at 4°C or less.
  - For 8270C, each **SPCC** in the calibration verification (CCV) standard must meet a **minimum response factor of 0.050**.. **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
  - After the system performance check is met, the **CCCs** are used for 8270C only to check the ongoing validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

CCC % Difference Evaluation Criteria	
8270C	**8270D**
CCCs ≤ 30% and all other target compounds require an RF ≤ 20%; however, up to 5 non-CCC target compounds may be ≤ 40%.	If the percent difference for each target compound is less than or equal to **20%**, then the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift) for any target, then corrective action is taken prior to the analysis of samples. All targets are considered as CCCs.

  - If the CCV criteria cannot be met, a new initial calibration must be generated.
- **Continuing Calibration Blank (CCB):** The CCB is run after each CCV. If the result is not ≤ MDL or ½ RL, correct the problem and re-run.
- **Internal standards** are added to every sample, standard, and QA/QC.

- **Retention time** - The retention times of the internal standards in the continuing calibration verification (CCV) standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than **30 seconds** from that in the mid-point standard level of the **most recent initial calibration** sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- **Response** - If the EICP area for any of the internal standards in the continuing calibration verification (CCV) standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the **most recent initial calibration sequence**, the mass spectrometer must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
  - If the internal standard is within criteria, report the second analysis.
  - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
  - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- The target analytes are quantitated with specific internal standards as shown in this table:

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Aniline	Benzoic acid	Acenaphthene
Benzyl alcohol	Bis(2-chloroethoxy) methane	Acenaphthylene
Bis(2-chloroethyl) ether	4-Chloroaniline	2-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,4-Dimethylphenol	Diethyl phthalate
1,4-Dichlorobenzene	Hexachlorobutadiene	Dimethyl phthalate
1,2-Dichlorobenzene	Isophorone	2,4-Dinitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene
Hexachloroethane	Naphthalene	2,6-Dinitrotoluene
2-Methylphenol	Nitrobenzene	Fluorene
3,4-Methylphenol	Nitrobenzene-d_8 (surr)	2-Fluorobiphenyl (surr)
n-Nitrosodimethylamine	2-Nitrophenol	Hexachlorocyclopentadiene
n-Nitroso-di-n-propyl- amine	1,2,4-Trichlorobenzene	2-Nitroaniline
Phenol	1-Methylnapthalene	3-Nitroaniline
Phenol-d_5 (surr)	Hexachloropropene	4-Nitroaniline
Pyridine	2,6-Dichlorophenol	4-Nitrophenol
2-Chlorophenol-d_4 (surr)	n-Nitrosodi-n-butylamine	2,4,6-Trichlorophenol
1,2-Dichlorobenzene-d_4 (surr)	1,4-Phenylenediamine	2,4,5-Trichlorophenol
1,4-Dioxane	trans-Isosafrole	1,2-Diphenylhydrazine
Pyridine	1,2,4,5-Tetrachlorobenzene	1,3-Dinitrobenzene
2-Picoline	cis-Isosafrole	Pentachlorobenzene
N-Nitrosomethylethylamine	Safrole	1-Naphthaleneamine
Methyl-methoanesulfonate	1-Chloronaphthalene	2-Naphthaleneamine
n-Nitrosodiethylamine	1,4-Naphthoquinone	2,3,4,6-Tetrachlorophenol
Ethylmethanesulfonate	**Quinoline**	Diphenylamine

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Pentachloroethane	**Chrysene-d_{12}**	5-Nitro-o-Toluidine
Acetonphenone	**6-Methylchrysene**	trans-Diallate
n-Nitrosopyrrolidine	**Dibenz(a,h)acridine**	cis-Diallate
2-Toluidine	**7,12-Dimethylbenz(a)anthracene**	1,3,5-Trinitrobenzene
n-Nitrosomorpholine		Phenacetin
n-Nitrosopiperidine		4-Aminobiphenyl
2-Butoxyethanol		
Indene		
Thiophenol		
Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
Anthracene	Benzidine	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Benzo(a)anthracene	Benzo(k)fluoranthene
Di-n-butyl phthalate	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol	Butyl benzyl phthalate	Benzo(a)pyrene
Diphenylamine	Chrysene	Dibenz(a, h)anthracene
Fluoranthene	3,3'-Dichlorobenzidine	Di-n-octyl phthalate
Hexachlorobenzene	Pyrene	Indeno(1,2,3-cd)pyrene
n-Nirosodiphenylamine	Terphenyl-d_{14} (surr)	Dibenz(a,j)acridine
Pentachlorophenol	4,4'Methylenebis(2-chloroaniline)	
Phenanthrene	Aramite	
Carbazole	3-Methylcholanthrene	
Bis (2-ethylhexyl)adipate		
Tribromophenol (surr)		
Thionazin		
Pronamide		
Pentachloronitrobenzene		
Dinoseb		
Sulfotepp		
Phorate		
Dimethoate		
Disulfoton		
4-Nitroquinoline-N-oxide		
Methapyrilene		
Isodrin		
Methyl Parathion		
Benzidine		
Parathion		
Hexachlorophene		
Kepone		
4-Dimethylaminoazobenzene		
Chlorobenzilate		
3,3'-Dimethylbenzidine		
2-Acetylaminofluorene		

(surr)= surrogate

- The internal standards selected permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion (i. e., for 1, 4-Dichlorobenzene-d_4, use 152 m/z for quantitation).
- Dilute the 4000 µg/mL internal standard by 2x with Methylene chloride. The resulting

solution contains each internal standard mixture at a concentration of 2000 µg/mL. Each 0.5 mL sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 2µL] of the internal standard solution, resulting in a concentration of 40 µg/mL of each internal standard.

- For SIM, dilute the 2000 µg/mL internal standard mix by 10x with Methylene chloride for a 200 µg/mL standard. Each 0.5 mL of sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 5 µL] of internal standard solution, resulting in a concentration of 2 µg/mL of each internal standard.

- **Evaluation of target analyte retention time:** The relative retention time (RRT) of each target analyte in each calibration standard must agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement. This criterion is met with the use of a ± 0.25 minute retention time window. Representative retention times are shown in Attachments 1 and 2.
- **Method Detection Limit Verification (MDLV)**: Annually, verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	1000 mL [RVE/LVI: 250 mL]
Soil, Concrete	30 grams
Oil	1 gram

- Samples are nominally prepared by one of the following methods prior to GC/MS analysis:

Matrix	Methods	SOP #
Water	3510	NV03-24
Soil/sediment/Concrete	3541, 3546, 3550	NV03-231, NV03-25
Oily Waste	3580	NV03-106

- QC samples and client samples must be extracted by the same preparation method.
- All calibration standards, QC samples, and client samples are introduced into the GC/MS using the same injection volume, IS and SS concentrations, and instrument conditions.

10.2 Calibration and Daily Continuing Calibration Verification: Refer to SOP Selection of Calibration Points / CA-P-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

- Initially and/or daily, evaluate the DFTPP tune criteria (Section 9.2).
- Evaluate the percent breakdown of DDT (Section 9.2).
- Evaluate the tailing factors for Benzidine and Pentachlorophenol (Section 9.2).

Initial calibration

1 Prepare calibration standards at five (minimum) different concentrations.

Concentration (µg/mL)	µL 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 200 µg/mL standard/500 µL (5 µL injection)
2	5	1
10	25	5

	20	50	10
	50	125	25
	80	200	40
	100	250	50

- At least one of the calibration standards corresponds to a sample concentration **at or below the laboratory reporting limit (RL)**. The remaining standards correspond to the working range of the GC/MS system.
- Each standard contains each analyte to be reported. These target analytes may not include the entire list of analytes for which the method has been demonstrated; however, the laboratory **must not** report a quantitative result for a target analyte that was not included in the calibration standard(s).
- Surrogates are included at the same concentrations.
- The internal standards are at a constant 40 µg/mL. Each 0.5 mL aliquot of calibration standard is spiked with 10 µL [RVE/LVI: 2 µL] of the internal standard solution prior to analysis.

2 For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	µL 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 10 µg/mL standard/500 µL(5 µL injection)
0.05*	2.5	0.5
0.1	5	1
0.5	25	5
1	50	10
5	250	50
10	500	100

*The 0.05 µg/mL standard must be used for low-level SIM analysis of samples from Wisconsin.

- Surrogates are included at the same concentrations.
- The internal standards are at a constant 2 µg/mL.
- See Attachments 2 and 3 regarding SIM Mass groups.

3 Analyze 1 µL [RVE/LVI: 5 µL] of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte. See Attachment 1. **Two characteristic ions must be valid for the low standard to be used.**

4 Calculate response factors (RFs) for each target analyte relative to one of the internal standards.

5 Evaluate the **system performance check compounds (SPCCs):** The minimum acceptable average RF for these compounds is 0.050 for 8270C. For 8270D, see Attachment 4. This check must be met before sample analysis begins.

6 Evaluate the **calibration check compounds (CCCs)**: If the RSD of any CCC is greater than 8270C criteria, then correct the chromatographic system reactivity before analysis begins. For 8270D, all compounds are treated as CCCs and must be within ± 20%.

7 Evaluate the **retention times.**

8 Evaluate the **linearity of target analytes** - If the RSD (8270C ± 15%; 8270D ± 20%) of any target analytes is within acceptance limits , then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor is used for quantitation. If the RSD of any target analyte is greater than the acceptance criteria , linear

	regression is used for calibration. The correlation coefficient r^2 must be at least 0.990 ($r \leq 0.995$). If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate. See Section 11 for equations and information on linear regression calibration.
9	Evaluate the intercept; it must be ≤ RL or re-calibrate.
10	Evaluate the success of the initial calibration by running an Initial Calibration Verification (ICV).
11	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

Initial Calibration Sequence Summary

1	DFTPP Tuning Criteria/DDT Breakdown/Tailing Factors
2	Calibration Standards
3	ICV
4	ICB

Daily continuing calibration verification - Calibration verification is performed at the beginning of **each** 12-hour analytical shift.	
1	The initial calibration for each compound of interest is verified once every 12 hours and prior to sample analysis by analyzing a continuing calibration verification (CCV) standard.
2	Evaluate the **system performance check compounds (SPCCs):** Each SPCC in the calibration verification (CCV) standard must meet the **minimum response factor criteria** for 8270C or 8270D in the initial calibration.
3	Evaluate the **minimum response factors** of each of the most common target analytes in the calibration verification standard (same as SPCCs).
4	Evaluate the **calibration check compounds (CCCs)** for method criteria. For 8270D or for shortened compound lists, all target analytes must meet ± 20% criteria. Use the initial calibration criteria.
5	Evaluate the **internal standard retention times** in the CCV.
6	Evaluate the **internal standard responses**.
7	Analyze an extraction blank after the continuing calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants.

10.3 Sample Analysis: Refer to Acceptable Manual Integration Practices / CA-Q-S-002.

1	Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL [RVE/LVI: 2 µL] of the internal standard solution to the 0.5 mL concentrated sample extract.
2	Inject a 1 µL [RVE/LVI: 5 µL] aliquot of the sample extract into the GC/MS system. The volume to be injected contains 50 ng of base/neutral and 50 ng of acid surrogates (assuming 100% recovery).
3	The recommended sequence for a 20-sample batch is as follows:

1	DFTPP Tuning Criteria /DDT Breakdown/Tailing Factors*
2	CCV
3	Method Blank
4	LCS
5	Matrix Spike
6	Matrix Spike
7	Samples 1-20

*Not used for SIM.

4	If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed in the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 µg/mL, unless a more sensitive GC/MS system is being used, e. g., 2 µg/mL for SIM).
5	Evaluate the specific internal standard response. Dilutions may be required to meet this criterion. Notes: Specific analytes associated with an internal standard within -50 to +100% from the last calibration verification (CCV) may be reported with approval from the supervisor or manager even if other internal standards in that analysis are outside limits. Only analytes associated with the internal standard(s) within limits may be reported from that analysis.
6	The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. Multiple ions are used for compound identification; see Attachment 2. Secondary ions may drop below 30% relative intensity at concentrations less than 1 µg/mL.

10.5 Qualitative analysis

- The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Attachments 1 and 2 list the primary and secondary ions for each analyte. Compounds are identified when the following criteria are met.
- The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. **Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.** When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ±30% relative intensity, the compound is reported as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair. (See Attachment 1.)
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analyses co-elute (i. e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound. The analyst must

carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. If all of the ions associate with the reference spectrum for the target analyte are present and within the ±30% criteria, a positive result is assumed even in the presence of extraneous ion fragments without presumptive evidence (all ions associated with the target analyte are also present in the interfering peak) for a negative identification.

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights for 8270C and 50% of the average of the two peak heights for 8270D samples. Mathematically, the two equations used are equivalent. Verification is performed on a mid-level control each day of use. Otherwise, structural isomers are identified as isomeric pairs. (See Attachment 1.)
- For samples containing components not associated with the calibration standards or the requested target list, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
  - For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:
    1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) are present in the sample spectrum.
    2) The relative intensities of the major ions agree within ±20%. **Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.**
    3) Molecular ions present in the reference spectrum are present in the sample spectrum.
    4) Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.
    5) Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance of the primary characteristic ion from the EICP.
- If the RSD of a compound's response factor is 15% for 8270C and 20% for 8270D, or less, then the concentration in the extract is determined using the average response factor (RF) from initial calibration data. If greater than the criteria , use linear regression.
- Where applicable, the concentration of any non-target compounds identified in the sample is estimated. The same formulae are used with the following modifications: The areas A_x and A_t are from the total ion chromatograms, and the RF for the compound is assumed to be 1.
- The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
-

10.7 Instrument Maintenance

Careful examination of the standard chromatogram indicates whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. Re-calibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require re-calibration. These significant maintenance activities include, changing, replacing, or reversing the column; cleaning the MS source; changing the electron multiplier; or injector port.

11.0 Calculations / Data Reduction

11.1 Accuracy

% Recovery = Measured concentration x 100
Known concentration

11.2 Precision (RPD)

RPD = Absolute value (orig. sample value - dup. sample value) x 100
(Orig. sample value + dup. sample value)/2

11.3 Breakdown Calculation:

% Breakdown of DDT = Sum of degradation peak areas (DDD + DDE) x 100
Sum of all peak areas (DDT + DDE + DDD)

1.4 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

11.5 Mean Response Factor, Standard Deviation, Relative Standard Deviation

$$RF_{mean} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \frac{\sum_{i=1}^{n} \left(RF_i - RF_{mean}\right)^2}{n-1}$$

$$RSD = \frac{SD \times 100}{RF_{mean}}$$

11.6 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF}) \times 100}{(\text{Avg. RF})}$$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result - True Value} \times 100}{\text{True Value}}$$

11.7 Linear Calibration Using a Least Squares Regression: This approach is not used for analytes that meet the RSD limits. For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_I = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(y_i - y_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i (y_i - y_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).

y_i – observed instrument response (area) for the i^{th} calibration standard.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used. Examples of allowed weighting factors which can place more emphasis on numbers of smaller value are:

$w_i - 1/x_i$ or $w_i = 1/x_i^2$

Do not include the origin (0, 0) as an extra calibration point. Reprocess each calibration standard as an unknown to determine the best fit model. Each calibration point above the RL must be ± 15% true (8000B) or ±20% true (8000C); the RL-level standard must be ± 30% true.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.990$.

11.8 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response or Response ratio
x = Concentration

11.9 Calculation

- For **aqueous** samples:

$$\textbf{Concentration (µg/L)} = \frac{A_x V_t D}{RF_{mean} V_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{mL \text{ extracted}}$$

A_x = Area of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (mL).
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
RF_{mean} = Mean response factor from the initial calibration (area/concentration).
V_s = Volume of the aqueous sample extracted in mL.

- For **non-aqueous** samples:

$$\textbf{Concentration (µg/kg)} = \frac{A_x V_t D}{RF_{mean} W_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{g \text{ extracted}}$$

A_x, V_t, D, RF_{mean} are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Dispose of waste extracts in the waste solvent drum.

15.0 References / Cross References

15.1 Method 8270C, SW-846 Update III Revision 3, December 1996 and **Method 8270D**, Update IV, Revision 4, February 2007.

15.2 Method 8000B, SW-846, Revision 2, December 1996, **Method 8000C**, Revision 3, March 2003.

15.3 TestAmerica Nashville's Quality Assurance Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Calibration Curves (General) / CA-Q-S-005, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method

Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, 3550 / NV03-23, and 3510 / NV03-24, 3541 / NV03-231, 3580 / NV03-106,8270/NVOH04-22.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

15.7 Corporate Quality Memorandum No. CA-Q-QM-005, May 19, 2010.

16.0 Method Modifications

Item	Modification
1	See Attachment 5 for the State of Ohio specific criteria.
2	See Attachment 6 for the State of Missouri DRO, CA LUFT DRO by GC/MS.
3	Verify with state certifications the correct version of this method to report. **Analyze and report by 8270D for Canadian, NJ, NC, OK, SC, and WV samples.**
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis.

17.0 Attachments

Attachment 1, Characteristic Ions for Semivolatile Compounds[a]

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
1,4-Dioxane	2.568	88	58
n-Nitrosodimethylamine	2.700	74	42, 44
Pyridine	2.714	79	52
2-Picoline	3.464	93	66, 92
n-Nitrosomethylethylamine	3.558	88	42, 43, 56
2-Fluorophenol (surr)	3.687	112	64
Methyl methanesulfonate	3.764	80	79, 65, 95
n-Nitrosodiethylamine	4.009	102	42, 57, 44, 56
Ethyl methanesulfonate	4.197	79	109, 97, 45, 65
Hexachloropropene	4.261	213	211,215,117,106,141
Phenol-d_5 (surr)	4.266	99	42, 71
Aniline	4.270	93	66, 65
Bis(2-chloroethyl) ether	4.294	93	63, 95
Phenol	4.275	94	65, 66
2-Chlorophenol	4.345	128	64, 130
1,3-Dichlorobenzene	4.425	146	148, 113
1,4-Dichlorobenzene-d_4 (IS)	4.444	152	150, 115
1,4-Dichlorobenzene	4.454	146	148, 113
Pentachloroethane	4.474	117	165, 167, 119
Benzyl alcohol	4.543	79	108, 77
n-Decane	4.550	57	
1,2-Dichlorobenzene	4.571	146	148, 113
2-Methylphenol	4.628	108	107, 77, 79, 90
Bis(2-chloroisopropyl) ether	4.632	45	77, 79
N-Nitrosodi-n-propylamine	4.717	130	42, 101, 70
3, 4-Methylphenol	4.717	107	108, 77, 79, 90
Hexachloroethane	4.764	117	201, 199
Nitrobenzene-d_5 (surr)	4.806	82	128, 54
Nitrobenzene	4.816	77	123, 65
n-Nitrosopyrrolidine	4.907	102	41, 42, 68, 69
Acetophenone	4.912	105	71, 51, 120
n-Nitrosomorpholine	4.916	108	116, 86
o-Toluidine	4.940	106	107, 77, 51, 79

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Isophorone	4.957	82	95, 138
2-Nitrophenol	5.018	139	109, 65
2,4-Dimethylphenol	5.037	122	107, 121
Bis(2-chloroethoxy)methane	5.088	93	95, 123
n-Nitrosopiperidine	5.114	114	42, 55, 56, 41
Benzoic acid	5.116	105	122, 77
2,4-Dichlorophenol	5.168	162	164, 98
1,2,4-Trichlorobenzene	5.215	180	182, 145
Naphthalene-d_8 (IS)	5.248	136	68
Naphthalene	5.257	128	129, 127
o,o,o-Triethylphosphorthioate	5.302	198	80, 53, 54164, 63
4-Chloroaniline	5.304	127	129, 65, 92
Hexachlorobutadiene	5.370	225	223, 227
a,a-Dimethylphenethylamine	5.372	58	91, 65, 134, 42
2,6-Dichlorophenol	5.523	162	
Hexachloropropene	5.556	213	211, 215, 117, 106, 141
4-Chloro-3-methylphenol	5.615	142	107, 144
2-Methylnaphthalene	5.704	142	141
n-Nitrosodi-n-butylamine	5.729	84	57, 41, 116, 158
1,4-Phenylenediamine	5.734	198	80, 53, 54, 52
1-Methylnaphthalene	5.779	142	141, 115
Hexachlorocyclopentadiene	5.854	237	235, 272
Isosafrole (trans)	5.861	162	131, 104, 77
2,4,6-Trichlorophenol	5.911	196	198, 200
2,4,5-Trichlorophenol	5.914	196	198, 97, 132, 99
2-Fluorobiphenyl (surr)	5.953	172	171
2-Chloronaphthalene	6.003	162	127, 164
Isosafrole (cis)	6.054	162	131, 104, 77
1,2,4,5-Tetrachlorobenzene	6.063	216	214,179,108,143,218
2-Nitroaniline	6.118	138	92, 65
2,3-Dichloroaniline	6.134	161	90, 63
Safrole	6.204	162	104, 77, 103, 135
Dimethyl phthalate	6.245	163	194, 164
1-Chloronaphthalene	6.284	162	127, 164
2,6-Dinitrotoluene	6.296	165	63,89, 121
Acenaphthylene	6.320	152	151, 153
1,4-Naphthoquinone	6.374	158	104, 102, 76, 50, 130
3-Nitroaniline	6.404	138	108, 92
Acenaphthene	6.447	154	153, 152
2,4-Dinitrophenol	6.470	184	63, 154
1,3-Dinitrobenzene	6.486	168	76, 50, 75, 92, 122
4-Nitrophenol	6.527	65	109, 139
Dibenzofuran	6.560	168	139
2,4-Dinitrotoluene	6.574	165	63, 89, 182
Acenaphthene-d_{10} (IS)	6.656	164	162, 160
Diethyl phthalate	6.738	149	177, 150
4-Chlorophenyl phenyl ether	6.790	204	206, 141
Fluorene	6.799	166	165,167
Pentachlorobenzene	6.806	250	252,108,248,215,254
4-Nitroaniline	6.837	138	65, 108, 92, 80, 39
1-Naphthylamine	6.844	143	115, 89, 63

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
4,6-Dinitro-2-methylphenol	6.865	198	51, 105, 182, 77
n-Nitrosodiphenylamine	6.879	169	168, 167
2-Naphthylamine	6.895	143	115, 116
2,3,4,6-Tetrachlorophenol	6.900	232	131,230,166,234,168
1,2-Diphenylhydrazine	6.903	77	105, 182
2,4,6-Tribromophenol (surr)	6.987	330	332, 141
Thionazine	7.027	107	96, 97,143, 79, 68
5-Nitro-o-toluidine	7.051	152	77, 79, 106, 94
Diphenylamine	7.107	168	169, 167
4-Bromophenyl phenyl ether	7.138	248	250, 141
Hexachlorobenzene	7.255	284	142, 249
Sulfotepp	7.276	322	97, 202
1,3,5-Trinitrobenzene	7.314	213	74, 120, 91, 63
Diallate (trans)	7.337	86	234, 43, 70
Phenacetin	7.337	108	180,179,109,137,80
Phorate	7.347	75	121, 97, 93, 260
Pentachlorophenol	7.387	266	264, 268
Diallate (cis)	7.403	86	234, 43, 70
Dimethoate	7.474	87	93, 125, 143, 229
Phenanthrene-d_{10} (IS)	7.476	188	94, 80
Phenanthrene	7.495	178	179, 176
Anthracene	7.528	178	176, 179
4-Aminobiphenyl	7.568	169	168, 170, 115
n-Octadecane	7.586	58	71, 85
Pronamide	7.619	173	175, 145, 109, 147
Carbazole	7.641	167	139, 84
Pentachloronitrobenzene	7.676	237	142,214,249,295,265
Disulfoton	7.723	88	97, 89, 142, 186
Dinoseb	7.737	211	163, 147, 117, 240
Di-n-butyl phthalate	7.914	149	150, 104
Methyl parathion	8.000	109	125, 263, 79, 93
Parathion	8.292	109	97, 291, 139, 155
4-Nitroquinoline-1-oxide	8.310	190	101, 128, 75, 116
Methapyrilene	8.371	58	50, 191, 71
Fluoranthene	8.374	202	100, 101, 203
Benzidine	8.464	184	92, 185
Isodrin	8.522	193	66, 195, 263,265,147
Pyrene	8.543	202	100, 101, 200, 203
Terphenyl-d_4 (surr)	8.652	244	122, 212
Aramite	8.870	191	319, 334, 197, 321
Dimethylaminoazobenzene	9.001	120	77, 105, 148, 42
Butyl benzyl phthalate	9.028	149	91, 206
Chlorobenzilate	9.034	139	253, 111, 141
Hexachlorophene	9.070	185	209,406
3,3'-Dimethylbenzidine	9.251	212	106, 196, 180
Bis (2-ethylhexyl) adipate	9.298	129	57, 112, 147
4,4'-Methylenebis (2-chloroaniline)	9.301	231	266, 140, 77
Kepone	9.316	272	274,237,178,143,270
3,3'-Dichlorobenzidine	9.423	252	254, 126
Benz(a)anthracene	9.446	228	229, 226
2-Acetylaminofluorene	9.453	181	180, 223, 152

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Chrysene-d_{12} (IS)	9.456	240	120, 36
Chrysene	9.474	228	226, 229
Bis(2-ethylhexyl) phthalate	9.474	149	167, 279
Di-n-octyl phthalate	9.926	149	167, 43
Benzo(b)fluoranthene	10.292	252	253, 125
3-Methylcholanthrene	11.305	268	252,253,126,134,113
Benzo(k)fluoranthene	10.311	252	253, 125
Benzo(a)pyrene	10.579	252	253, 125
7,12-Dimethylbenz(a)anthracene	10.600	256	241, 239, 120
Perylene-d_{12} (IS)	10.631	264	260, 265
Indeno)1,2,3-c,d)pyrene	11.778	276	138, 277
Dibenz(a,h)anthracene	11.783	278	139, 279
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Benzo(g,h,i)perylene	12.107	276	138, 277
IS = internal standard			
surr = surrogate			
[a]See Attachment 2 for Retention Times and Ions used with SIM			

Attachment 2, Characteristic Ions for PAH Compounds Using SIM

Compounds	RT	Primary	Secondary*
1,4-Dichlorobenzene-d_4	6.66	152	
2-Fluorophenol	5.471	112	64
Phenol-d_5	6.251	99	71.1
Naphthalene-d_8	8.24	136	
Nitrobenzene-d_5	7.32	82.1	128.1
Naphthalene	8.27	128.1	129.1
2-Methylnaphthalene	9.13	142.1	141.1
1-Methylnaphthalene	9.42	142.1	141.1
Acenaphthene-d_{10}	10.91	164.1	
2-Fluorobiphenyl	9.82	172.1	
Acenaphthylene	10.67	153	151.1
Acenaphthene	10.958	15.1	154.1
Fluorene	11.7	166.1	167.1
Phenanthrene-d_{10}	13.3	188	
2,4,6-Tribromophenol	12.167	329.8	331.8
Phenanthrene	13.33	178.2	176.2
Anthracene	13.4	178.2	176.2
Fluoranthene	15.28	202.2	101.1
Chrysene-d_{12}	17.64	240.1	
Pyrene	15.66	202.2	101.1
Terphenyl-d_{14}	15.89	244.2	
Benzo(a)anthracene	17.61	228.2	229.2
Chrysene	17.68	228.2	229.2
Perylene-d_{12}	20.2	264.2	
Benzo(b)fluoranthene	19.45	252.2	126.1
Benzo(k)fluoranthene	19.49	252.2	126.1
Benzo(a)pyrene	20.08	252.2	126.1
Indeno(1,2,3-cd)pyrene	22.69	276.2	277.2
Dibenzo(ah)anthracene	22.7	278.2	279.2

Compounds	RT	Primary	Secondary*
Benzo(ghi)perylene	23.43	276.2	277.2
Internal standards are in **bold.**			

Attachment 3, SIM Mass Groups

Mass Group	Compound	RT	Primary	Secondary	Dwell Time
1	2-Fluorophenol	5.42	112	64	25 ms
	Phenol-d_5	6.2	99	71	
	1,4-Dichlorobenzene-d_4	6.6	152		
	Nitrobenzene-d_5	7.26	82.1	128.1	
4.20 min.					
2	Naphthalene-d_8	8.17	136		50 ms
	Naphthalene	8.2	128.1	129.1	
	2-Methylnaphthalene	9.19	142	141.1	
	1-Methylnaphthalene	9.35	142.1	141.1	
	2-Fluorobyphenyl	9.75	172.1		
5.35 min.					
3	Acenaphthylene	10.6	152.1	151.1	25 ms
	Acenaphthalene-d_{10}	10.83	164.1		
	Acenaphthene	10.88	153.1	154.1	
	Fluorene	11.69	166.1	167.1	
	2,4,6-Tribromophenol	12.11	329.8	331.8	
6.55 min.					
4	Phenanthrene-d_{10}	13.21	188		50 ms
	Phenanthrene	13.25	178.2	176.2	
	Anthracene	13.32	178.2	176.2	
	Fluoranthene	15.21	202.2	101.1	
	Pyrene	15.58	202.2	101.1	
	Terphenyl-d_{14}	15.81	244.2		
7.75 min.					
5	Benzo(a)anthracene	17.52	228.2	229.2	100 ms
	Chrysene-d_{12}	17.55	240.1		
	Chrysene	17.59	228.2	229.2	
9.85 min.					
6	Benzo(b)fluoranthene	19.34	252.2	126.1	100 ms
	Benzo(k)fluoranthene	19.38	252.2	126.1	
	Benzo(a)pyrene	19.96	252.2	126.1	
	Perylene-d_{12}	20.07	264.2		
10.65 min.					
7	Indeno(1,2,3-cd)pyrene	22.5	276.2	277.2	100 ms
	Dibenzo(a,h)anthracene	22.52	278.2	279.2	
	Benzo(g,h,i)perylene	23.21	276.2	277.2	
12.20 min.					

Attachment 4, 8270D Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Attachments 1 and 2.

Compound	Minimum RF	Compound	Minimum RF
Benzaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800

Compound	Minimum RF	Compound	Minimum RF
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-tetrachlorobenzene	0.010
2,2'-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2, 4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3'-Dichlorobenzidine	0.010
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis-(2-ethylhexyl)phthalate	0.010
1,1'-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,2,3-cd)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

Attachment 5, State of Ohio Specific Criteria.
Only those compounds in the original EPA Method 8270C may be reported. Any compounds in this SOP in italics in Section 1 are not part of the original 8270C method. Run Ohio VAP samples according to SOP 8270/NVOH04-22.

Attachment 6, Missouri Department of Natural Resources (and CA LUFT) require(s) that **DRO** be analyzed by GC/MS.

- Tuning and frequency requirements are the same as in 8270, omitting DDT, Pentachlorophenol, and Benzidine.
- Extract water samples per SOP 3510 / SA03-24 and solid samples per SOP 3550 / SA03-23.
- Only base/neutral surrogates are needed.
- GC/MS mass range is 35-550 ηmu.
- Use a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 1,000 µg/mL each in Methylene chloride.

- Retention time windows set using C_{10}, C_{21}, and C_{35}. For DRO, set RT 0.1 minutes after C10 to 0.1 minutes after C21. For ORO, set RT 0.1 minutes after C_{21} to 0.1 minutes after C_{35}. Verify RT daily (24 hours) by running component standard.
- Quantitative using baseline-to-baseline, not valley-to-valley. The Total Ion Chromatogram must be used to quantitate.
- The Response Factor determined for DRO (C_{10}-C_{21}) must be used for C_{21}-C_{35}.
- Subtract area from any Internal Standard and surrogates.
- % RSD ≤ 20.
- Run a CCV at the beginning and end of each batch; it must contain all targets reported, at mid-point of calibration, % D ≤ 20.
- Run a Method Blank every extraction batch, and LCS and MS/MSD per batch.
- May reprocess file to quantitate PAH if needed. For individual targets, % RSD ≤ 15.
- Quantitation of DRO must be by external standard.

18.0 Revision History

- Revision 12, 22 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
  - To incorporate Update IV criteria.
- Revision 13, 9 October 2009
  - Consolidation of text, general editing.
  - Add Appendix IX and miscellaneous compound details.
  - Distinguish 8270C versus 8270D.
- Revision 14, 30 September 2011
  - Organizational changes.
  - Add amendments 13a and 13b.
  - Add reference to SOP 3541 for concrete and SOP Calibration Curves (General).
  - Add QAF-45 and Section 14.2.
  - Remove WY as a state requiring QC every 10 samples.
  - Change Attachment 5 to refer analysts to OH8270 SOP.
  - Add Attachment 7
  - Add option to run LLCCV to show sensitivity.
  - Add note about low-level calibration standard for SIM WI samples.
  - Lower several report limits.
  - Specify GC resolution between two isomer peaks for 8270C versus 8270D.
- Revision 15, 31 December 2012
  - Organizational changes.
  - Incorporation of amendments 14a, b, c.
  - OK no longer limits batch size to 10 samples.
  - Specify that $r^2 \geq 0.990$.
  - Substitute LIMS for the Control Limits Manual.
  - Distinguish between the RSD maximum for 8270C and 8270D. For 8270D, all targets are treated as CCCs.
  - Add re-fitting text to the linear calibration section.
  - Add Reduced Volume Extraction / Large Volume Injection (RVE / LVI).

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **300.0 &SM4110B / NV12-40.9 & 9056 / NV12-119.9** Effective Date: **2/28/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 300.0 & SM 4110B, 9056/9056A: Determination of Inorganic Anions by Ion Chromatography**

Affected SOP Section Number(s): Section 9.1, Sample QC

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 12

Revision Number with Mod ID: **9b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change

- Section 9.1, Sample QC table,
  - Method Blank, Acceptance Criteria: Add bold; delete crossed-out. These changes were requested by the North Carolina certification officer.

    For EPA 300.0: **<MDL** ~~<RL.~~

    For 9056/9056A: **<10% of the RL or MDL, whichever is greater** ~~<MDL~~

  - LCS, Acceptance Criteria for 9056./9056A only: **80-120%** ~~90-110%~~ recovery

Michael A. Brumm 2/11/13	2/12/13
Technical and Quality Assurance Approval Date	Operations Manager Approval Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville Standard Operating Procedure (SOP) Change Form

SOP Number/Revision No.: **300.0 &SM4110B / NV12-40.9 & 9056 / NV12-119.9** Effective Date: **12/31/12**

Last Mod. Date: 10/31/12 & 9/14/12

SOP Title: **Method 300.0 & SM 4110B, 9056/9056A: Determination of Inorganic Anions by Ion Chromatography**

Affected SOP Section Number(s): Section 7.0, Reagents and Standards

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 12

Revision Number with Mod ID: **9a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change

Section 7.2: add the bold language:

7.2 Eluent Stock Solutions

- Metrosep A Supp 4: Dissolve 19.1 grams anhydrous Sodium carbonate and 14.3 grams Sodium bicarbonate in one liter of anion-free, reagent water: 1.8 mM Na_2CO_3 and 1.7 mM $NaHCO_3$. **Prior to sequence initiation or daily,** prepare a dilution of 10 mL to 1 liter reagent water. Degas by purging with Helium or sonicating for about 2 minutes.
- An equivalent commercial product is acceptable (Inorganic Ventures Eluent 1817-5; add 20 mL/2 L reagent water for a 0.18M Na_2CO_3 and a 0.17M $NaHCO_3$).

12/14/12	12/14/12
Supervisor Approval Date	Operations Manager Approval Date
12/11/12	
Technical and Quality Assurance Approval Date	

Title: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY SW-846 METHOD 9056 / 9056A

Approvals (Signature/Date)

Annette Cobbs Department Supervisor	Date 8/20/12
Cory Spry Extractables Operations Manager	Date 8/20/12
Johnny Davis Health & Safety Manager / Coordinator	Date 9-10-12
Michael H. Dunn Quality Assurance Manager	Date 9-12-12
Michael H. Dunn Technical Director	Date 9-13-12

1.0 Scope and Application

1.1 Analyte, Matrices: This method addresses the determination in water, agricultural wastes, soils, the bomb combustion of solid waste, and oil samples of the following inorganic anions: Bromide, Chloride, Fluoride, Nitrate, Nitrite, and Sulfate.

1.2 Reporting Limits: The RL for each anion is:

Analyte	Water Samples, mg/L	Solid Samples, mg/kg
Bromide	0.1	1.0
Chloride	1.0	10
Fluoride	0.1	1.0
Nitrate-N	0.1 (0.02 SC only)	1.0
Nitrite-N	0.1 (0.02 SC only)	1.0
Sulfate	1.0	10

For agricultural applications, the water samples may be weighed.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard disk or cartridge, analytical column, suppressor, and conductivity detector.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 The Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution or retention time of an adjacent of other anion. Sample dilution and/or spiking is used to solve interference problems associated with retention times.

4.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.3 Samples that contain particles larger than 0.45 microns require filtration to prevent damage to instrument columns and flow systems; therefore, filter all samples with a 0.45 micron, IC-free filter (VWR 28/45-503, 25 mm with 0.45 µm polyester sulfone membrane, or equivalent).

4.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. High levels of interfering organic acids may be present in industrial wastes. Known co-elution is caused by small organic anions (i.e., formate and acetate). The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not allowed for leachates of solid samples when acetic acid is used for pH adjustments.

4.5 The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate etc.) which are conductive and elute near fluoride and could bias the fluoride quantitation in some drinking and many wastewaters.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This method may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: None.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Ion chromatograph (Metrohm): Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, and detectors.
  - Guard column: A protector of the separator column.
  - Anion separator column: Anion analytical column (Method A): Metrosep A Supp 4. An optional column may be used if comparable resolution of peaks is obtained, and the requirements of Section 9.2 can be met.
  - Anion suppressor device: micro-membrane, commercial.
  - Detector: conductivity cell: approximately 1.25 µL internal volume, capable of providing data as required in Sect. 9.2.
  - EZChrom data acquisition system, IC Net control system, Version 2.3SR5; Chrom.
  - Autosampler: Metrohm 838
  - 25-µL sample loop injection
  - Suppressor: ASRS Ultra II, self-regenerating, or equivalent.

Analyte	Peak	Retention Time, minutes						
		IC#1	IC#2	IC #5	IC#6	IC#7	IC#8	IC#9
Fluoride	1	3.39	3.72	3.62	3.75	3.82	3.72	3.84
Chloride	2	4.51	4.97	4.80	5.00	5.08	5.03	5.12
Nitrite-N	3	5.13	5.67	5.45	5.69	5.77	5.75	5.78
Bromide	4	6.11	6.76	6.48	6.79	6.87	6.88	6.86
Nitrate-N	5	6.76	7.48	7.16	7.52	7.61	7.71	7.50
Sulfate	7	11.38	13.3	12.25	12.91	12.92	13.46	14.05

Standard Conditions	
Columns:	Metrosep A Supp 4

Standard Conditions	
Pump Rate:	1.0 mL/min
Sample Loop:	20 µL

- Balance, analytical, capable of accurately weighing to the nearest 0.0001 g.
- Centrifuge, benchtop.

6.2 Supplies

- VWR 28/45-503, 25mm with 0.45 µm polyester sulfone membrane, or equivalent.
- Sample bottles, glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest
- Analyte-free sand for blank soil matrix.
- Volumetric flasks, Class A, various volumes.
- Pipets, Class A.
- Pipettor
- Syringe, disposable, Luer-lok.

7.0 Reagents and Standards

7.1 Reagent water, free of the anions of interest, i. e., <MDL.

7.2 Eluent Stock Solutions, Metrosep A Supp 4:

- Dissolve 19.1 grams anhydrous Sodium carbonate and 14.3 grams Sodium bicarbonate in one liter of anion-free, reagent water. 1.8 mM Na_2CO_3 and 1.7 mM $NaHCO_3$. Prepare a dilution of 10 mL to 1 liter reagent water. Degas by purging with Helium or sonicating for about 2 minutes.
- An equivalent commercial product is acceptable (Inorganic Ventures Eluent 1817-5; add 20 mL/2 L reagent water for a 0.18M Na_2CO_3 and a 0.17M $NaHCO_3$).

7.3 Regeneration Solution (micro-membrane suppressor): Prepare 2 L Sulfuric acid by adding 22.4 mL of conc. H2SO4 and 25.2 g Oxalic acid to a 2 L volumetric and diluting to the mark with reagent water.

7.4 Oxalic acid, reagent grade.

7.5 Stock Standard solutions, 1000 µg/mL: Stock standard solutions are purchased as certified solutions.

	Ultra Scientific ICUS-3113 Primary Standard (µg/mL)	Inorganic Ventures TANASH-1 Second-source Standard (µg/mL)
Fluoride	100	100
Chloride	1000	1000
Nitrite	100	100
Bromide	100	100
Nitrate	100	100
Sulfate	1000	1000

The primary standard is used for calibration and continuing calibration verification. The second-source standard is used for the initial calibration verification, laboratory control sample, and matrix and matrix spike duplicate.

NOTE: See the Certificate of Analysis for the standard's expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE	100 mL	Cool 0-6°C	48 hours for nitrate and nitrite; 28 days for bromide, chloride, fluoride, sulfate	SW-846 Chapter 3
Soil	HDPE	20 g	Cool 0-6°C.	After extraction: 48 hours for nitrate and nitrite; 28 days for bromide, chloride, fluoride, sulfate	SW-846 Chapter 3

Note: No specified holding time from sampling to extraction.

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) procedures. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated. The following quality control samples are prepared with each batch of samples.

9.1 Sample QC

The following QC is run **every batch of no more than 20 samples.**			
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Blank	One per Batch	< RL	Correct problem then re-prep and analyze blank and all samples processed with the contaminated blank. If target >10x blank, report but qualify.
LCS[1] for all analytes must be from a 2nd source.	One[1] per prep batch	90-110% recovery	Re-prep and analyze the LCS and all samples in the affected analytical batch. If high and samples are ND, report.
MS/MSD must be from a 2nd source	One pair per batch	80-120% recovery, < 15% RPD	None (LCS is used to determine if data is acceptable.)
Retention time window calculated for each analyte.	System set-up, with major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the established RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.

1 - All AZ, TX, and WV samples require a LCS duplicate in each batch.

- A **Method blank**, reagent water or analyte-free boiling chips, is run with each analytical batch.
- A **Laboratory Control Sample (LCS)** is included with each analytical batch. The LCS consists of an aliquot of a clean (control) reagent water of the same volume or boiling chips of the same mass as sample aliquots.
  - Water batches: Add 5 mL of the second-source standard to 100 mL reagent water (5 or 50 µg/mL).
  - Soil batches: Add 3 mL MS Spiking solution (next paragraph) to 27 mL reagent water and 3 grams boiling chips (50 or 500 µg/mg).
- **Matrix Spike Matrix Spike Duplicate:** The MS/MSD aliquots duplicate the aliquot used for sample analysis. Add the same concentration and solution as the LCS. Always spike a non-diluted sample. The MS must be in the first group of 10 samples in the batch.
  - MS Spiking Solution: Add 50 mL second-source standard to 100 mL reagent water (50 or 500 µg/mL).

  - Water batches: Add 1 mL MS Spiking Solution to 9 mL client sample (10 or 100 µg/mL).
  - Soil batches: Add 3 mL MS Spiking Solution to 27 mL reagent water and 3 grams client sample (100 or 1000 µg/mg).
- **Retention time windows:** Refer to the policies described in the retention time section of the QA Manual.
  - Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the target analytes in the sample matrix to be analyzed.
  - Make three injections of the standards over a minimum 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are tight.
  - Calculate the standard deviation of the three absolute retention times for each analyte.
  - The retention time range is defined as plus or minus three times the standard deviation of the absolute retention time. The analyst also relies primarily on pattern recognition.
  - In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ± 0.05 minute as the retention time window.
  - The laboratory must calculate retention time windows for each standard on each column and whenever a new column phase is installed. The data must be retained by the laboratory.
  - Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although anions are affected to some degree. In some cases, this peak migration may produce poor resolution or identification.

9.2 Instrument QC: For each batch of 20 samples.

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Minimal five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD ≤ 10%. Use CF. If not, use first-order linear – Least squares regression $r^2 \geq 0.995$, r ≥ 0.995. LCR criteria met.	Correct problem then repeat initial calibration.
Initial Calibration Verification (ICV), must be from a 2nd source	Immediately following each initial calibration	All analytes within 10% of expected value.	Correct problem then repeat initial calibration.
Initial Calibration Blank (ICB)	After the ICV	< RL	Correct problem, repeat.
Continuing Calibration Verification (CCV)	At the beginning, every 10 samples, and end of the analysis sequence	All analytes within 10% of expected value and within the RTW for 8000B, 20% for 8000C	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
Continuing Calibration Blank	After each CCV	< RL	Correct problem, repeat
MDL Verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - This is a summary of the acceptance criteria
2 - All abnormalities must be noted on the data, the benchsheet, and in LIMS.
3 - All AZ, TX, and WV samples require a LCS duplicate in each batch.

- **Calibration:** See calibration in Section 10.
- **Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): The ICV is the second-source standard** run immediately after the calibration curve standards at a mid-point concentration. **The CCV is the primary source standard** to verify the calibration on each working day and brackets the samples in a batch, every 10 samples and at the end of the batch.
  - ICV/CCV: Add 5 mL of the appropriate standard to 100 mL reagent water (5 or 50 µg/mL).
- **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB):** Use reagent water. The ICB is run after the ICV. The CCB is run after each CCV.
- **Method Detection Limit Verification (MDLV):** Annually at a minimum, or with each low standard, verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	10 mL
Soil	1.0 g

For soils, add an amount of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed for 10 minutes using a magnetic stirring device or mechanical shaker. If needed, centrifuge at >2000 rpm for about 10 minutes prior to filtration. Filter the resulting slurry before injecting onto the instrument by using a 0.45 µm membrane type filter, which can be the type that attaches directly to the end of the syringe. Take care to show that good recovery and identification of peaks is obtained with user's matrix through the use of fortified samples.

10.2 Calibration: Refer to SOP Selection of Calibration Points / CA-T-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

- For each analyte of interest, prepare a calibration standard at a minimum of five concentration levels using the primary standard. Add accurately measured volumes of on or more stock standards to a volumetric flask and dilute to volume with reagent water.

	Standard (µg/mL)									
Anion	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Fluoride (F^-)	0.02	0.1	0.2	0.5	1	1.5	2.5	5	7.5	10
Chloride (Cl^-)	0.2	1	2	5	10	15	25	50	75	100
Nitrite (NO_2^-)	0.02	0.1	0.2	0.5	1	1.5	2.5	5	7.5	10
Bromide (Br^-)	0.02	0.1	0.2	0.5	1	1.5	2.5	5	7.5	10
Nitrate (NO_3^-)	0.02	0.1	0.2	0.5	1	1.5	2.5	5	7.5	10
Sulfate (SO_4^{-2})	0.2	1	2	5	10	15	25	50	75	100

Note: Level 1, 0.02/0.2 µg/mL, applies to South Carolina samples.

- Using a constant injection volume for each calibration standard, tabulate peak area responses against the concentration. These results are used to prepare a calibration curve for each analyte. Record retention times.
- The %RSD must be less than 10%, or the correlation coefficient (r) must be ≥ 0.995 (r^2 ≥ 0.990). See the first-order linear calibration discussion in Section 11.

- Linear Calibration Range (LCR): The LCR must be determined at the time of each calibration and verified at least every 6 months or whenever a significant change in instrument response is observed. The initial demonstration of linearity must use sufficient standards to insure that they resulting curve is linear. The verification of linearity must use a minimum of a blank and five standards. Each standard response above the report limit must be within ±10% of its known value, and the standard at the report limit must be within 50% of its known value, or linearity range must be reestablished.
- The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples, with the analysis of a CCV. If the response or retention time for any analyte varies from the expected values by more than ± 10%, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, a new calibration curve must be prepared for that analyte, and all samples analyzed since the last valid CCV must be re-analyzed.

10.3 Sample Analysis: Refer to SOP Acceptable Manual Integration Practices / CA-Q-S-002.

1	Power all modules on.
2	Press "EXT" on eluent pump (indicator light will come on). Confirm pressure is approximately 6.0.
3	Start software using AnionX method. X = IC ID. Tighten the pump tubing against the rollers. The remote should be in the off position.
4	Filter and place the sample in a test tube, and place the test tube in the autosampler. Perform a visual comparison on each of the test tube sample IDs on the autosampler against the analytical sequence in the acquisition software, once at the start of the sequence run (after the autosampler is loaded) and a second time at the end of the sequence run (before the autosampler is unloaded).

- Compare the width of the retention time window with actual retention time variations of standards over the course of several days. See sample QC section.
- If the response for the peak exceeds the upper calibration standard, dilute the sample with an appropriate amount of reagent water and reanalyze. Record the dilution factor.
- If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

10.4 Example Analysis Queue / Sequence*

1	Initial calibration, if needed
2	ICV
3	Prime
4	CCV
5	CCB (water blank)
6	Method Blank
7	LCS
8	Sample 1

9	Matrix Spike
10	Matrix Spike Duplicate
11	Samples 2-10
12	CCV
13	CCB
14	Samples 11-20
15	CCV
16	CCB

*May be up to 20 samples.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{Recovery} = \frac{\text{Measured concentration x 100}}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value) x 100}}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 % Difference

$$\%\ \text{Difference} = \frac{\text{(Result - True Value) X 100}}{\text{True Value}}$$

11.4 Calibration Factor

$$\textbf{Calibration Factor} = \frac{\text{Peak areas of standard}}{\text{Concentration of the standard}}$$

11.5 Linear Calibration Using a Least Squares Regression: A linear calibration model based on a least squares regression is employed for analytes that do not meet the RSD limits. For external standard calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves generated tend to fit points at the upper calibration levels better than points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency is used. Examples of acceptable weighting factors which place more emphasis on numbers of smaller value are:

$w = 1/x$ or $w = 1/x^2$

Do not include the origin (0, 0) as an extra calibration point. However, most data systems and many commercial software packages allow the analyst to "force" the regression through zero. Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 ***before*** the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations. Re-process each calibration standard as an unknown to determine the best fit model. Each calibration point above the RL must be ± 15% true (8000B) or ±20% true (8000C); the RL-level standard must be ± 50% true.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.995$.

11.6 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.7 The acquisition software determines the anion sample concentration from the calibration curve by plotting instrument area response against standard concentration. Multiply by any dilution. Report results in mg/L or mg/kg. Report NO_3^- and NO_2^- as N.

Concentration (µg/mL) = (µg/mL from instrument) x (Dilution factor)

$$\text{Concentration (µg/g)} = \frac{\text{(µg/g from instrument)x(Dilution factor)x(Extract volume, mL)}}{\text{Sample weight, g}}$$

11.8 Sample values exceeding the high point of the calibration curve must be diluted and reanalyzed.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four pairs of sample duplicates with identical results for each pair are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an acceptable manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Samples, rinses, and waste reagents are discharged into the sanitary sewer.

15.0 References / Cross References

15.1 SW-846 Method 9056, Update II, Revision 0, September 1994, and **9056A**, Update IV, Rev. 1, February 2007.

15.2 YSI Model 5100 Probe Operation Manual.

15.3 TestAmerica Nashville's Quality Assurance Manual.

15.4 TestAmerica Nashville's Control Limits Manual.

15.5 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.6 SOPs: Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Selection of Calibration Points / CA-T-P-002, Acceptable Manual Integration Practices / CA-Q-S-002, Calibration Curves (General) / CA-Q-S-005, Sample Homogenization, Sub-sampling, and Compositing / NV08-229.

15.7 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachment

None.

18.0 Revision History

- Revision 5, dated 31 October 2008
  - Integration for TestAmerica and STL operations.
- Revision 6, dated 23 October 2009
  - Addition of SC requirements.
  - Addition of centrifuge.
- Revision 7, dated 10 December 2010
  - Addition of Section 14.2.
  - Removal of definitions and replacement with referral to the QA Manual and QAF-45.
  - Addition of IC #5, IC #6, IC #7.
  - Removal of instructions to program the autosampler for IC#1, IC#2, and IC#5 (replaced with new control system, IC Net.
  - Change to Liquid Chromatography department.

  - Addition of a 9th calibration standard.
  - Updated supplies list.
- Revision 8, dated 5 August 2011
  - Organizational changes.
  - Addition of reference to corporate SOP Calibration Curves (General) / CA-Q-S-005.
  - Remove requirement for maximum 10-sample batches for WY samples.
  - Remove references to Dionex ion chromatograph.
  - Addition of retention times for IC#8.
  - Change concentrations of calibration standards (adding a 10th one).
  - Addition of QC preparations.
- Revision 9, dated 14 September 2012
  - Organizational changes.
  - Reduce bromide RL for soil.
  - Add amendment 8a, 8b, 8c. Add agricultural wastes to matrices, and the potential to report water samples by weight (mg/kg).
  - Add Chrom software.
  - Add commercial eluent product and update the volume of Sulfuric acid for the regeneration solution.
  - OK no longer limits batch size to 10 samples; Added: All AZ, TX, and WV samples require a LCS duplicate in each batch.
  - Remove need for a sample duplicate. MS RPD < 15%. %Recovery for RL concentration calibration standard ≤ 50%.
  - Specify that $r^2 \geq 0.995$.
  - No exception for report level calibration standard accuracy.
  - Add SOP Sample Homogenization, Sub-sampling, and Compositing / NV08-229.

Title: RESIDUE, NON-FILTERABLE (GRAVIMETRIC, DRIED AT 103°-105°C) (TOTAL SUSPENDED SOLIDS, TSS) METHOD SM2540 D

Approvals (Signature/Date)

Sessily Overton-Gray	12/7/12		11/26/12
Sessily Overton-Gray Department Manager	Date	Matt Ricke Inorganics Operations Manager	Date
Michael H. Dunn	11/26/12	Johnny Davis	12/28/12
Michael H. Dunn Technical Director Quality Assurance Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date

1.0 Scope and Application

1.1 Analyte, Matrices: This method determines the concentration of total suspended residue (solids) in drinking, surface, and saline waters, domestic and industrial wastes.

1.2 Reporting Limits: The reporting limit is nominally 1.0 mg/L.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A well mixed sample is filtered through a standard glass fiber filter. The residue retained on the filter is dried to a constant weight at 103-105°C. The filtrate from this method may be used for Residue, Filterable (SM2540C & 160.1 / NV07-64).

3.0 Definitions

3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to a constant weight at 103-105°C.

3.2 See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for other laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

4.2 Samples high in dissolved solids, such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: None.

5.2 Primary Materials Used: Not applicable.

6.0 Equipment and Supplies

6.1 Instrumentation

- Analytical balance with sensitivity to 0.0001 grams.
- Drying oven for use at 103-105°C.
- Filter support: filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support.
- Suction flask, 300, 500 or 1,000 mL.
- Dessicator
- Filter holder, membrane filter funnel.
- Magnetic stirrer

6.2 Supplies

- Class A graduated cylinder, 10 - 1000 mL, or equivalent.
- Glass fiber filter discs, 47 mm, without organic binder, such as, Gelman type A/E, Environmental Express P/N F93447 MM, or equivalent. **Use with rough side up.**
- 50 mL wide-bore pipet. 1mL, 5mL, & 10 mL pipets.

7.0 Reagents and Standards

7.1 **Reagent water**, analyte-free.

7.2 **Standard Solution**, 100 mg/L as TSS: Dissolve 0.1000 g of Celite, Fisher Cat. No. C211-500 or equivalent in reagent water and bring to 1 L with reagent water. It is acceptable to prepare larger volumes.

7.3 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation for information on shelf-lives and storage requirements.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HPDE	1000 mL	Cool 0-6°C	As soon as possible but no more than 7 days.	40 CFR Part 136.3

- Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result (written guidance from client is required to exclude items).
- Chemical preservation of the sample is not used. Analysis should begin as soon as possible. Refrigeration or icing to 0-6°C, to minimize microbiological decomposition of solids, is recommended. Holding time is seven days.

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples are prepared with each batch of samples.			
Quality Controls	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Blank (MB)	1 in 20 or fewer samples	< Report Limit	Reprep, rerun.
Laboratory Control Sample (LCS), second-source	1 in 20 or fewer samples	90-110% recovery	Repeat.
Sample Duplicate	1 in 10 or fewer samples	≤ 5% RPD	Report

1 All AZ, TX, and WV samples require a LCS duplicate in each batch.

- One **Method Blank** of 1000 mL of reagent water is processed each batch.
- The **Laboratory Control Sample** (LCS) is prepared to verify that the laboratory can perform the analysis in a clean matrix. See Section 7.2 for its preparation; use a 1000-mL aliquot. See Section 11 for the equation for accuracy.
- **Sample Duplicate**: Run two identical aliquots in each group of 10 samples. Demonstrate precision by calculating the RPD. See Section 11 for the equation.

9.2 Instrument QC: Not applicable.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	~1000 mL

10.2 Calibration

The balance is calibrated daily according to SOP Balance Calibration / NV08-213.

10.3 Sample Analysis

1	Place a glass fiber filter disc on the filter apparatus, rough side up.
2	Selection of Sample Volume: For a 47 mm diameter filter, filter a suitable volume of sample, nominally 1000 mL. NOTE: If during filtration of this initial volume the filtration rate drops rapidly a smaller volume of sample should be used. Do not use less than 1.0 mL. Limit sample size to yield no more than 200 mg residue on the filter. If filter residue is greater than 200 mg repeat using a smaller volume.
3	Assemble the filtering apparatus and turn "on" vacuum pump. Wet the filter with a small volume of reagent water to seat it against the fritted support.
4	If using 1000 mL, or the entire container volume shake the sample vigorously and quantitatively transfer the sample volume to the filter using a Class A graduated cylinder. If using smaller aliquots, stir with a magnetic bar on a stir plate and remove a portion, center distance between container wall and vortex, with a 1, 5, 10, or 50-mL pipet. Remove all traces of water by continuing to apply vacuum after sample has passed through.
5	With vacuum on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with **three** portions of reagent water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.
6	Carefully remove the filter from the filter support and place it in the aluminum planchet. Dry at least one-hour at 103°-105°C. Cool in a desiccator for about an hour and weigh. Repeat the drying cycle until a constant weight is obtained of 4% or less than 0.5 mg, whichever is less. Record. If weight on filter is <0.0010 grams and less than 1000 mL was chosen, repeat steps 1-6 with a greater sample volume than initially.

10.4 Example Analysis Queue / Sequence*

1	Method Blank
2	LCS
3	Sample 1
4	Sample Duplicate
5	Samples 2- 11
6	Sample Duplicate
7	Samples 12-20

*May be up to 20 samples

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Concentration calculation:

$$\textit{Non-filterable residue, mg/L} = \frac{(A-B)x(1{,}000{,}000)}{C}$$

A = weight of filter + dried residue in g
B = weight of filter in g
C = mL of sample filtered

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- The filtrate is discharged into the sanitary sewer.
- The filter with solids is disposed into a trash receptacle.

15.0 References / Cross References

15.1 Method SM2540 D - 1997, Standard Methods for the Analysis of Water and Wastewater, On-line edition, 2011 editorial revisions.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.4 SOPs: Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214.

15.5 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachment

None.

18.0 Revision History

- Revision 7, 31 July 2009
  - Integration for TestAmerica and STL operations.
- Revision 8, 30 December 2010
  - Removal of reference to retired method EPA 160.2 (Amendment 7a).
  - Addition of QAF 45 and Section 14.2.
  - Change sample volume to nominally 1000 mL.
- Revision 9, 31 December 2012
  - Organizational changes.
  - OK and WY no longer limit batch size to 10 samples.
  - Provide instruction for aliquots smaller than 1L. Modify example sequence.
  - Reference the year of the method source and the on-line edition.

C3: TestAmerica Burlington SOPs

Title: Specific Gravity of Soil Solids by Water Pycnometer (ASTM D854-06 – Method B)

Approval Signatures:

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Approval Date: March 22, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
BR-GT-004	6	03/22/10	Specific Gravity of Soil Solids by Water Pycnometer

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	**Reviewer**	**Job Title**	**Yes[1]**	**No**
04/03/12	Christopher Colleton	Dept Manager		X
04/03/12	Eric Gagne	Analyst		X

[1]: *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

_____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

_____ The SOP Revision will be released as a new version of the SOP.

_____ The SOP Revision requires method validation or demonstration of capability

_____ The SOP Revision does not reqire method validation or demonatration of capability.

_____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

Julia Daigle
QA Signature

4/3/2012
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedure for the measurement of specific gravity in soils.

2.0 Summary of Method

A representative portion of sample passing a # 4 (4.75mm) sieve is weighed and placed in a calibrated volumetric flask to which reagent water is added. Specific gravity is determined by comparison of the density of water to the density of water + sample.

This SOP is based on the following reference methods:

- Method B of ASTM Standard D 854-06 "Standard Test Methods for Specific Gravity of Soils by Water Pycnometer". ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Specific Gravity of Soil Solids: The ratio of the mass of a unit volume of soil solids to the mass of the same volume of gass-free distilled water at 20°C.

4.0 Interferences

- Soil Solids for this test method does not include solids which can be altered by this method, contaminated with a substance that preagenthibits the use of this method, or are highly organic soil solids, such as fibreagentus matter which floats in water.

- The term soil solids is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Water-soluble solids such as lime or sodium chloride, typically require special treatment. ASTM methods for special treatment are listed in ASTM D854-06 but are not currently offered by the laboratory. If laboratory analysis on such materials is desired, the laboratory recommends that procedures for treatment of samples and reporting specifications be specified by the customer prior to the start of analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Oven, 110°C (+/-5)
- Top loading balance
- Aluminum measuring pans
- Stainless steel spatulas and spoons
- Volumetric flask, 250 mL. (Pycnometer)
- Mortar and Rubber Tipped Pestle
- Thermometer (+/-0.5°C)
- Insulated container or large cooler
- No. 4 (4.75mm) sieve
- Funnel
- 400 mL glass beaker
- Hot Plate
- Disposable pipettes

7.0 Reagents and Standards

Reagent Water

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection. Sample collection procedures are provided in this SOP for guidance only. The laboratory recommends that all samples be collected in accordance with a client specified sampling plan.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solid	Glass	200g	4°C	NA	D854-06

Unless otherwise specified by client or regulatory program, after analysis samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Calibration and Standardization

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-012 *Thermometer Calibration Check*. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the pycnometer annually or when the dry, empty mass is outside of 0.06 g of the average calibrated mass. The calibration procedure is provided in Appendix A of this SOP.

10.2 Sample Preparation

Dry enough sample to ensure at least 60 g of dried sample will pass the #4 (4.75mm) sieve. Dry the sample for 16 hours in a oven set at a temperature of 110°C then break up any soil aggregates formed during the drying process with a rubber-tipped pestle and mortar.

10.3 Procedure (Method B)

Obtain a clean dry flask for each sample. Weigh the flask and upload the measurement to the LIMS worksheet. The mass of the flask must be within 0.06 g of its previously averaged calibrated mass. If the mass is outside this range, re-calibrate the flask using the procedure given in Appendix A.

Place a funnel into the flask and transfer at least 60 g of sample to the flask. Rinse any solids that adhere to the funnel with reagent water to ensure a complete transfer.

Fill the flask with reagent water until the water level is between 1/2 and 1/3 of the depth of the main body of the flask. Agitate the flask until slurry forms and rinse any soil that adheres to the side of the flask into the slurry.

Place a hot plate inside a fume hood and place the flask on the hot plate. Boil the sample slurry for at least 2 hours. Shake the flask vigorously several times throughout the boil to prevent the sample slurry from sticking. Use only enough heat to keep the slurry boiling. After 2 hours has elapsed, remove flask from the hot plate.

Fill the flask above the reference line with reagent water and let sample cool to room temperature. Place the flask into a cooler, along with a thermometer, a pipette and a bottle of reagent water. Close the cooler and allow the sample to thermally equilibrate for at least 16 hours. After thermal equilibrium, adjust the volume of the water in the flask to the calibration mark, taking care to avoid entrapment of air.

Tare the top loading balance and weight the flask. Upload the weight measurement into the LIMS

worksheet.

Using the thermally equilibrated thermometer, measure the temperature of the sample and water and enter the temperature reading in the LIMS worksheet.

Tare the balance and label and weigh a 400 mL glass beaker uploading the weight measurement into the LIMS worksheet. Transfer the soil and water from the flask to the beaker. To ensure a complete transfer rinse the flask with reagent water as needed.

Place the beaker in a drying oven and dry the soil and water for at least 16 hours at a temperature of 110°C +/- 5°. After this time period has passed, remove the 400 mL glass beaker from the oven and cool to room temperature.

Reweigh the beaker and upload the weight measurement into the LIMS worksheet.

LIMS calculates the specific gravity using the equations given in Section 11.0

11.0 Calculations / Data Reduction

11.1 Calculations

- Equation 1: Mass of pycnometer and water at the test temperature

  $M_{\rho w,t} = M_p + (V_p * \rho_{w,t})$

  Where:
  $M_{\rho w,t}$ = mass of the pycnometer and water at the test temperature (T_t), g.
  M_p = the average calibrated mass of the dry pycnometer, g.
  V_p = the average calibrated volume of the pycnometer, mL.
  $\rho_{w,t}$ = the density of water at the test temperature (T_t), g/mL from table 1.

- Equation 2: Specific gravity of soil solids at the test temperature, G_t

  $G_t = \rho_s/\rho_{w,t} = M_s/(M_{\rho w,t} - (M_{\rho ws,t} - M_s))$

  Where:
  ρ_s = the density of the soil solids Mg/m^3 or g/cm^3
  $\rho_{w,t}$ = the density of water at the test temperature (T_t), g/mL or g/cm^3 from table 1
  M_s = the mass of the oven dry soil solids, g.
  $M_{\rho ws,t}$ = the mass of the pycnometer, water, and soil solids at the test temperature (T_t), g.

- Equation 3: Specific gravity of soil solids at 20°C

  $G_{20°C} = K * G_t$

  Where:
  K = the temperature coefficient given in table 1.

11.2 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.3 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

12.1 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures

are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste- Satellite Container: 5 Gallon Plastic Bucket.

15.0 References / Cross-References

- Method B of ASTM Standard D 854-06 "Standard Test Methods for Specific Gravity of Soils by Water Pycnometer". ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- SOP BR-QA-012 Thermometer Calibration Check.

16.0 Method Modifications

None

17.0 Attachments

- Density of Water and Temperature Coefficient (K) for Various Temperatures.
- Appendix A: Pycnometer Calibration Procedure

18.0 Revision History

BR-GT-004. Revision 6:
- Title Page: Updated approval signatures
- All Sections: Changed DI water to RO water
- Section 2: Changed No. 10 sieve to a No. 4 sieve
- Section 4.0: Interference paragraphs added
- Section 6: Removed vacuum pump, heated water bath and Styrofoam block. Added hot plate
- Section 10.4: Replaced paragraphs referencing the vacuum procedure with the hot plate procedure.
- Section 10.4: Changed boiling time from 1 hour to 2 hours.
- Section 10.4: Changed aluminum pan to 400 mL glass beaker.

Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)
15.0	0.99910	1.00090	16.0	0.99895	1.00074	17.0	0.99878	1.00057	18.0	0.99860	1.00039
.1	0.99909	1.00088	.1	0.99893	1.00072	.1	0.99876	1.00055	.1	0.99858	1.00037
.2	0.99907	1.00087	.2	0.99891	1.00071	.2	0.99874	1.00054	.2	0.99856	1.00035
.3	0.99906	1.00085	.3	0.99890	1.00069	.3	0.99872	1.00052	.3	0.99854	1.00034
.4	0.99904	1.00084	.4	0.99888	1.00067	.4	0.99871	1.00050	.4	0.99852	1.00032
.5	0.99902	1.00082	.5	0.99886	1.00066	.5	0.99869	1.00048	.5	0.99850	1.00030
.6	0.99901	1.00080	.6	0.99885	1.00064	.6	0.99867	1.00047	.6	0.99848	1.00028
.7	0.99899	1.00079	.7	0.99883	1.00062	.7	0.99865	1.00045	.7	0.99847	1.00026
.8	0.99898	1.00077	.8	0.99881	1.00061	.8	0.99863	1.00043	.8	0.99845	1.00024
.9	0.99896	1.00076	.9	0.99879	1.00059	.9	0.99862	1.00041	.9	0.99843	1.00022
19.0	0.99841	1.00020	20.0	0.99821	1.00000	21.0	0.99799	0.99979	22.0	0.99777	0.99957
.1	0.99839	1.00018	.1	0.99819	0.99998	.1	0.99797	0.99977	.1	0.99775	0.99954
.2	0.99837	1.00016	.2	0.99816	0.99996	.2	0.99795	0.99974	.2	0.99773	0.99952
.3	0.99835	1.00014	.3	0.99814	0.99994	.3	0.99793	0.99972	.3	0.99770	0.99950
.4	0.99833	1.00012	.4	0.99812	0.99992	.4	0.99791	0.99970	.4	0.99768	0.99947
.5	0.99831	1.00010	.5	0.99810	0.99990	.5	0.99789	0.99968	.5	0.99766	0.99945
.6	0.99829	1.00008	.6	0.99808	0.99987	.6	0.99786	0.99966	.6	0.99764	0.99943
.7	0.99827	1.00006	.7	0.99806	0.99985	.7	0.99784	0.99963	.7	0.99761	0.99940
.8	0.99825	1.00004	.8	0.99804	0.99983	.8	0.99782	0.99961	.8	0.99759	0.99938
.9	0.99823	1.00002	.9	0.99802	0.99981	.9	0.99780	0.99959	.9	0.99756	0.99936
23.0	0.99754	0.99933	24.0	0.99730	0.99909	25.0	0.99705	0.99884	26.0	0.99679	0.99858
.1	0.99752	0.99931	.1	0.99727	0.99907	.1	0.99702	0.99881	.1	0.99676	0.99855
.2	0.99749	0.99929	.2	0.99725	0.99904	.2	0.99700	0.99879	.2	0.99673	0.99852
.3	0.99747	0.99926	.3	0.99723	0.99902	.3	0.99697	0.99876	.3	0.99671	0.99850
.4	0.99745	0.99924	.4	0.99720	0.99899	.4	0.99694	0.99874	.4	0.99668	0.99847
.5	0.99742	0.99921	.5	0.99717	0.99897	.5	0.99692	0.99871	.5	0.99665	0.99844
.6	0.99740	0.99919	.6	0.99715	0.99894	.6	0.99689	0.99868	.6	0.99663	0.99842
.7	0.99737	0.99917	.7	0.99712	0.99892	.7	0.99687	0.99866	.7	0.99660	0.99839
.8	0.99735	0.99914	.8	0.99710	0.99889	.8	0.99684	0.99863	.8	0.99657	0.99836
.9	0.99732	0.99912	.9	0.99707	0.99887	.9	0.99681	0.99860	.9	0.99654	0.99833
27.0	0.99652	0.99831	28.0	0.99624	0.99803	29.0	0.99595	0.99774	30.0	0.99565	0.99744
.1	0.99649	0.99828	.1	0.99621	0.99800	.1	0.99592	0.99771	.1	0.99562	0.99741
.2	0.99646	0.99825	.2	0.99618	0.99797	.2	0.99589	0.99768	.2	0.99559	0.99738
.3	0.99643	0.99822	.3	0.99615	0.99794	.3	0.99586	0.99765	.3	0.99556	0.99735
.4	0.99641	0.99820	.4	0.99612	0.99791	.4	0.99583	0.99762	.4	0.99553	0.99732
.5	0.99638	0.99817	.5	0.99609	0.99788	.5	0.99580	0.99759	.5	0.99550	0.99729
.6	0.99635	0.99814	.6	0.99607	0.99785	.6	0.99577	0.99756	.6	0.99547	0.99726
.7	0.99632	0.99811	.7	0.99604	0.99783	.7	0.99574	0.99753	.7	0.99544	0.99723
.8	0.99629	0.99808	.8	0.99601	0.99780	.8	0.99571	0.99750	.8	0.99541	0.99720
.9	0.99627	0.99806	.9	0.99598	0.99777	.9	0.99568	0.99747	.9	0.99538	0.99716

[A]Reference: CRC Handbook of Chemistry and Physics, David R. Lide, Editor-in-Chief, 74th Edition, 1993–1994.
[B]mL = cm^3.

Appendix A: Pycnometer Calibration Check Procedure

Check the average mass of the pycnometer annually or when the measured mass is outside of 0.06g of the previous calibrated average mass.

Equipment & Supplies

- 250 mL volumetric flask
- Top loading balance
- RO water
- Thermometer
- Large cooler with lid
- Disposable pipettes

Procedure

Obtain a clean, dry flask and record the flask number in the Excel worksheet

Weigh the flask five times, recording the each weight measurements in the Excel worksheet as "Flask (g)"

Determine and record the average and standard deviation of the weight measurements. The standard deviation shall be less than or equal to 0.02g. If it is greater, attempt additional measurements or use a different balance.

Fill the flask to just above the calibration line with de-aired/de-ionized water taking care to not entrap air bubbles in the water.

Place the flask into the cooler, along with a thermometer, disposable pipettes and a bottle of deaired/deionized water.

Cover the cooler, and allow the flask and water to sit for at least three hours in order to reach thermal equilibrium.

Set the cooler next to a balance, and remove the cover.

Handling the flask only by the rim, remove and place the flask onto a block of Styrofoam.

Using the thermally equilibrated pipette, adjust the volume of the water in the flask to the calibration mark, taking care to avoid entrapment of air.

Check for and remove any water beads on the pycnometer stem or on the exterior of the flask.

Measure and record the mass of the pycnometer and water to the nearest 0.01g.

Using the thermally equilibrated thermometer, measure and record the temperature of the flask to the nearest 0.1°C.

Repeat steps 3 through 11 to obtain five independent measurements on each pycnometer.

Calculate the average and standard deviation of the five volume determinations. If the standard

deviation is greater than 0.05mL, discard the results and repeat the procedure.

Determine the calibrated volume of the pycnometer using the following equation:

$$V_p = \frac{(M_{pw,c} - M_p)}{\rho_{w,c}}$$

Where:

V_p = the calibrated volume of the pycnometer, mL.

$M_{pw,c}$ = the mass of the pycnometer and water at the calibration temperature, g.

M_p = the average mass of the dry pycnometer at calibration, g.

$\rho_{w,c}$ = the mass density of water at the calibration temperature g/mL, (Table 1)

Title: Particle Size Analysis (ASTM D 2217 and D422-63)

Approval Signatures:

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Approval Date: March 17, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
GR-GT-006	5	03/17/10	Particle Size Analysis

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	Reviewer	Job Title	Yes[1]	No
04/03/12	Christopher Calloway	Dept Manager		X
04/03/12	Eric Lee	Analyst		X

[1] *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

____ The SOP Revision will be released as a new version of the SOP.

____ The SOP Revision requires method validation or demonstration of capability

____ The SOP Revision does not reqire method validation or demonatration of capability.

____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

Kristin Daigle
QA Signature

4/3/2012
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

Small Sieves

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample\ -\ pan) - (pan + non\ -\ soil\ material\ -\ pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

Not Applicable

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Title: Water (Moisture) Content of Soil and Rock by Mass (ASTM D2216- 05, Method B)

Approval Signatures

William S. Cicero
Laboratory Director

Christopher G. Callahan
Department Manager

Kirstin L. McCracken
Quality Assurance Manager

Bryce E. Stearns
Technical Director

Dan W. Helfrich
Health & Safety Coordinator

Approval Date: March 2, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
BR-GT-016	6	03/02/10	Water (Moisture) Content of Soil and Rock

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	Reviewer	Job Title	Yes[1]	No
04/03/12		Dept Manager		X
04/03/12		Analyst		X

[1]: *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

_____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

_____ The SOP Revision will be released as a new version of the SOP.

_____ The SOP Revision requires method validation or demonstration of capability

_____ The SOP Revision does not reqire method validation or demonatration of capability.

_____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

_____

_____

Kristi Daigle
QA Signature

4/3/2012
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of water (moisture) content of soil, rock, and similar materials where the reduction in mass by drying is attributed to loss of water.

The procedure is applicable to solid materials as the term is used to mean naturally occurring mineral particles of soil and read and that are not readily soluble in water. The procedure should not be used to determine water content in materials with substantial amounts of soluble solids or materials that contain extraneous matter or in marine sediments. For these types of materials, ASTM recommends special treatment or qualification of analytical results. ASTM methods for special treatment are listed in ASTM D2216-05 but are not currently offered by the laboratory. If laboratory analysis on such materials is desired, the laboratory recommends that procedures for treatment of samples and reporting specifications be specified by the customer prior to the start of analysis.

2.0 Summary of Method

A portion of sample is dried in an oven maintained at a temperature of 110 ± 5 ºC for 16 hours or until constant mass. The loss of mass due to drying is considered to be water. The water content is calculated using the difference between the mass of the wet sample and the mass of the dry sample.

This SOP is based on the following reference method:

- ASTM Standard D 2216-05, 2005, "Determination of Water (Moisture) Content of Soil and Rock by Mass", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

If the laboratory's procedure has been modified from the reference method, a list of modifications will be provided in Section 16.0.

3.0 Definitions

- **Water Content by Mass:** The ratio of the mass of water contained in the pore spaces of soil or rock material, to the solid mass of particles in that materials, expressed as a percentage. A standard temperature of 110 ± 5°C is used to determine these masses. (ASTM D2216-05)

- **Constant Mass:** The state that a water content specimen has attained when further heating causes or would cause less than 1% or 0.1% additional loss in mass. (ASTM D2216-05)

4.0 Interferences

This SOP determines moisture content in solid materials without the application of any specific treatment to account for significant amounts of either dissolved or volatile solids.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous

material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Drying Oven, capable of temperature measurements at 110°C (±5°C), Barnstead LC Oven Model# 3513 or equivalent.
- Top loading balance, Mettler Model# PB3002 or equivalent.
- Aluminum Pans, Fisher Scientific or equivalent.
- Stainless Steel Spatulas and Spoons, Fisher Scientific or equivalent.
- Heat shield gloves / Oven Tongs, Fisher Scientific or equivalent.

7.0 Reagents and Standards

Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection. The reference method specifies that soil samples should be collected and preserved in accordance with ASTM D 4220 Section 8, Groups B, C or D for soils and rock samples collected in accordance with D 5079, Section 7.5.2.

Listed below are the laboratory recommended container types, sample amount, storage conditions and required holding times for analysis:

Matrix	Sample Container	Sample Amount	Storage	Holding Time
Solid	Glass	100-200 g	0-30°C	NA

NOTE: The reference method recommends the following sample amounts for analysis based on maximum particle size.

Maximum Particle Size (mm)	Standard Sieve Size	Sample Mass for Analysis
2 or less	# 10	20 g
2 to 4.75	# 4	100 g
4.76 to 9.5	3/8 inch	500 g
9.6 to 19.0	3/4 inch	2.5 Kg
19.1 to 37.5	1 ½ inch	10 Kg
37.6 to 75.0	3 inch	50 Kg

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Calibration and Standardization

Check the support equipment monitoring log to ensure the daily check of the balance was performed. If the daily balance check was not performed, perform the check prior to use of the balance with at least 2 Class S weights that bracket the range of use and record the check in the logbook designated for this purpose.

10.2 Analysis

Visually inspect the sample to identify the sieve size for which 100% of material will pass. Use a sample amount that corresponds to the to the sieve size in the chart in Section 8.0. If less than the recommended amount of sample was provided use the amount of sample available and record the anomaly with a LIMS nonconformance memo (NCM)

Mix the sample thoroughly following the homogenization procedures specified in laboratory SOP LP-QA-020. Label a clean aluminum pan with the sample ID then measure and record the weight of the pan to the nearest 0.01 g. Weigh the pre-determined sample mass into the pan and record the combined weight of the pan and the wet sample. Repeat for each sample.

Check the temperature of the drying oven(s) to ensure that the oven is within 105-115°C; then place the pans in the drying oven. Dry the samples for 16 hours or until constant mass.

Remove the pans from the oven and allow the pans to cool to room temperature or a temperature comfortable enough to handle the pans with bare hands. Measure and record the weight of the pan and dried sample.

Calculate the moisture content using the equation given in Section 11.0.

11.0 Calculations / Data Reduction

11.1 Calculation

Moisture Content

$w = [(M_{cws} - M_{cs})/(M_{cs} - M_c)]*100$

Where:
w = water content, %
M_{cws} = mass of container and wet sample, g
M_{cs} = mass of container and oven dry sample, g
M_c = mass of container, g

11.2 Data Reduction

Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

12.1 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste- Satellite Container: 5 Gallon Plastic Bucket.

15.0 References / Cross-References

- ASTM Standard D 2216-05, 2005, "Determination of Water (Moisture) Content of Soil and Rock by Mass", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- TestAmerica Corporate Safety Manual, current version.

- Laboratory SOPs as referenced, current version.

16.0 Method Modifications

Analysis is not always performed using the recommended sample amounts specified in the reference method because lesser sample amounts are typically received by the laboratory.

17.0 Attachments

None

18.0 Revision History

BR-GT-016, Version 6:

- Updated Approval Signatures
- Converted format of SOP to company template.
- Updated the method reference

Title: Density in Soils by Drive Cylinder Method (ASTM D2937-04)

Approval Signatures:

William S. Cicero
Laboratory Director

Christopher G. Callahan
Department Manager

Kirstin L. McCracken
Quality Assurance Manager

Bryce E. Stearns
Technical Director

Dan W. Helfrich
Health & Safety Coordinator

Approval Date: June 21, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
BR-GT-018	6	06/21/10	Density in Soils by Drive Cylinder Method

Review Statement: 5 KLD 11-12-3-12

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

Date	Reviewer	Job Title	Revision Needed	
			Yes[1]	No
11/28/12	Chris Cullen	Dept Manager		X

[1]: *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

_____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

_____ The SOP Revision will be released as a new version of the SOP.

_____ The SOP Revision requires method validation or demonstration of capability

_____ The SOP Revision does not reqire method validation or demonatration of capability.

_____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

_____

_____

Kristie Daigle
QA Signature

11/28/12
Date

Comments:

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of in-place density of natural, inorganic soil by the drive cylinder method.

This procedure is not suitable for organic soils that can compress upon sampling, very hard natural soils, heavily compacted soils, soils with low plasticity or soils with large amounts of gravel. The procedure is not recommended for friable soils or soft, highly plastic, non-cohesive, saturated or other soils which are not easily deformed.

2.0 Summary of Method

A representative portion of sample is removed from the sample collection tube and dried in an oven maintained at a temperature of 110°C for 16 hours. The moisture content of the sample is determined and expressed as a percent of the oven-dried mass. Density is subsequently calculated by dividing the value obtained for oven-dried mass by the determined volume of the drive cylinder.

This procedure is based on the following reference method:

- ASTM Standard D D2937-04 "Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method. ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

This method is not appropriate for samples that can be "altered" during sampling, shipping and/or processing.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Oven, 110°C (+/-5°C), Barnstead LC Oven Model# 3513 or equivalent.
- Top loading balance, Mettler Model# PB3002 or equivalent.
- Aluminum measuring pans, Fisher Scientific or equivalent.
- Stainless steel spatulas and spoons, Fisher Scientific or equivalent.
- Ruler/meter stick with millimeter increments
- Heat shield gloves / Oven Tongs, Fisher Scientific or equivalent.

7.0 Reagents and Standards

Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection. Sample collection procedures are provided in this SOP for guidance only. The laboratory recommends that all samples be collected in accordance with a client field and sampling plan. Immediately after collection the sample(s) should be cooled and stabilized with packing material or wax to prevent shifting during transport.

Holding times are not applicable. Unless otherwise specified by client or regulatory program, after analysis samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Calibration and Standardization

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

10.2 Sample Preparation/Analysis

Clean the outside of the sample collection tube then remove the end caps, wax any other protective material from each end of the sample collection tube. Remove any loose material from the collection tube. If the sample extends beyond the tube, trim the end of the sample so that it is equal to the length of the tube.

Measure the diameter of the sample tube and enter this value into the LIMS worksheet.

If sample fills the entire tube, measure the length of the tube and enter this value into the LIMS worksheet. If sample does not fill the entire tube, measure the length of the tube and measure the length of empty space in the tube, then subtract the length of empty space. Enter this value into the LIMS worksheet.

Weigh the tube and sample and enter this value into the LIMS worksheet.

Empty the sample into a large pan then clean the tube with a spatula to ensure all sample is taken into account. Weigh the clean tube and enter the weight measurement into the LIMS worksheet.

Label a clean aluminum pan with the sample ID and weigh the pan and enter this weight measurement into the LIMS worksheet.

Thoroughly mix the sample to homogenize with a spatula or spoon. Measure at least 100 g of the sample into the pan, or if practical, use the entire sample. Enter this weight measurement into the LIMS worksheet.

Place the sample into an oven maintained at 110°C and allow the sample to dry for a minimum of 16 hours. After this time period has elapsed, remove the pan from the oven and re-weigh. Enter this weight measurement into the LIMS worksheet.

The LIMS method calculate the moisture content, sample length, sample volume and in-place density using the equations give in Section 11.0.

11.0 Calculations / Data Reduction

11.1 Calculations

Percent Moisture Content

Moisture Content, % = [(A-B) * 100]/B

Where:
A = pan + wet sample, g
B = pan + dry sample, g

Sample length:

$L=L_1-(L_2+L_3)$ mm

Where:
L = sample length
L_1 = length of sample tube
L_2 = average recovery, top
L_3 = average recovery, bottom

Sample volume:

$V = [(\pi(D/2)^2)L]$

Where:
V = sample volume (calculated)
D = sample tube diameter
L = sample length

In-place Density:
$= m_1/V$ g/cm^3 (dry)

$= m_2/V$ g/cm^3 (wet)

Where:
= calculated dry density
m_1 = mass of sample, dry
m_2 = mass of sample, wet
m_3 = mass of sample tube
V = sample volume

11.2 Data Review

Primary Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet for each extraction and cleanup batch performed. Initiate NCMs for any anomalies observed during the process. Set the status of each batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of each batch to second level review.

Run the QC Checker, review the deliverable and set the job to lab complete.

12.0 Method Performance

12.1 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste- Satellite Container: 5 Gallon Plastic Bucket.

15.0 References / Cross-References

- ASTM Standard D D2937-04 "Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method. ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- Corporate Environmental Health and Safety Manual (CW-E-M-001)

16.0 Method Modifications

None

17.0 Attachments

None

18.0 Revision History

Revision 5:
- Title Page: Updated approval signatures.
- Section 4.0: Inserted a statement saying this method is not appropriate for all soil samples.
- Section 10.2: changed minimum weight for water content from 50 g to 100 g.

Title: Moisture, Ash, Organic Matter of Peat & Other Organic Soils (ASTM D2974- 07a – Methods A & C)

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the measurement of moisture content, ash content, and organic matter in organic soils. This procedure may also be used to determine moisture content in organic clays, silts, and mucks.

2.0 Summary of Method

To determine moisture content, a portion of sample is dried in an oven maintained at a temperature of 105 ± 5 °C for 16 hours. The moisture content of the sample is calculated as the difference between the "wet weight" and the "dried weight" of the sample expressed as a percent of oven dry mass.

To determine ash content, the oven-dried sample is ignited in a muffle furnace at a temperature of 440 ± 22 °C for 16 hours. The substance that remains after ignition is ash. The ash content is determined by dividing the mass of the ash by the mass of the oven-dried sample.

The percent organic matter is calculated by subtracting the percent ash content from one hundred.

This procedure is based on the following reference method:

- ASTM Standard D 2974-07a "Standard test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils". ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The recommended weight for this method is 50 g, however, samples containing a large volume of organic material or are contaminated with hydrocarbon can ignite in the muffle furnace and create smoke. When there is a concern that the sample will ignite and produce smoke a smaller volume of 5 to 10 grams can be used for this test.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Oven, 105°C (± 5°C), Barnstead LC Oven Model# 3513 or equivalent.
- Top loading balance, Mettler Model# PB3002 or equivalent.
- Aluminum measuring pans not less than 100 mL capacity, Fisher Scientific or equivalent.
- Stainless steel spatulas and spoons, Fisher Scientific or equivalent.
- Muffle Furnace, 440ºC (± 22°C), Barnstead Model# 30400 or equivalent.
- Heat shield gloves / Oven Tongs, Fisher Scientific or equivalent.

7.0 Reagents and Standards

Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection. Sample collection procedures are provided in this SOP for guidance only. The laboratory recommends that all samples be collected in accordance with a client specified sampling plan.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solid	Glass	50g	4°C	NA	D2974-07A

Unless otherwise specified by client or regulatory program, after analysis samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Calibration and Standardization

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

10.2 Analysis

Percent Moisture Content

For each sample label a clean aluminum pan with the sample ID.

Measure the weight of an empty aluminum pan to the nearest 0.01g for each sample and enter the weight measurements in the LIMS worksheet.

Homogenize the sample by mixing thoroughly with a spatula.

Place a pre-weighed aluminum pan onto the balance and measure at least 50 g of sample into the aluminum pan. Record the weight measurement in the LIMS worksheet and repeat for each sample.

Place the aluminum pans in an oven maintained at 105 ± 5 °C and let the samples dry for a minimum of 16 hours or until constant mass. Remove the aluminum pans from the oven and re-weigh. Record the weight measurements in the LIMS worksheet.

The LIMS worksheet calculates percent moisture content using the calculations provided in Section 11.0.

Ash Content

Place the oven-dried specimen (from moisture content) into a muffle furnace set to gradually heat to 440 ± 22 °C. Allow the samples to ash for a minimum of 16 hours. Remove the aluminum pans from the oven and let them sit until they are cool to the touch. Reweigh each pan and record the weight measurement in the LIMS worksheet.

The LIMS worksheet calculates ash content using the calculations provided in Section 11.0.

11.0 Calculations / Data Reduction

11.1 Calculations

- Percent Moisture Content

  Moisture Content, % = [(A-B) * 100]/B

  Where:
  A = as received test specimen, g
  B = mass of the oven dried specimen, g

- Percent Ash Content

  Ash Content, % = (C * 100)/B

  Where:
  C = ash, g
  B = oven dried test specimen, g

- Percent Organic Matter

  Organic Matter, % = 100.0 - D

  Where:
  D = ash content, %

11.2 Data Review

Primary Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet for each extraction and cleanup batch performed. Initiate NCMs for any anomalies observed during the process. Set the status of each batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of each batch to second level review.

Run the QC Checker, review the deliverable and set the job to lab complete.

12.0 Method Performance

12.1 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste- Satellite Container: 5 Gallon Plastic Bucket.

15.0 References / Cross-References

- ASTM Standard D 2974-07a "Standard test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils". ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- Corporate Environmental Health and Safety Manual (CW-E-M-001)

16.0 Method Modifications

The laboratory does not perform the subsampling procedure described in Section 7 of the reference method.

17.0 Attachments

None

18.0 Revision History

BR-GT-019, Revision 6:

- Title Page: Updated approval signatures
- Equations: Fixed equation for percent moisture.

BR-GT-019, Revision 5:

- Title Page: Updated approval signatures.
- Section 2: Changed oven temperature from 110 to 105 ± 5 °C and added ± 22 °C for temperature range of muffle furnace.
- Section 5.1: Added a paragraph stating lesser volumes can be used if sample will ignite and create smoke.
- Section 6: Changed oven temperature from 110 to 105 ± 5 °C and added ± 22 °C for temperature range of muffle furnace.
- Section 8: Inserted sample size, preservation and holding time requirements table.

- Section 10: Changed oven temperature from 110 to 105 ± 5 °C and added ± 22 °C for temperature range of muffle furnace.
- Section 16.0: Added laboratory modification of procedure from reference method.

C4: Beaver Engineering, Inc. SOPs

Designation: D 2216 – 90[ε1]

Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock[1]

This standard is issued under the fixed designation D 2216: the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[ε1] NOTE—The title was corrected editorially in June 1991.

1. Scope

1.1 This test method covers the laboratory determination of the water (moisture) content of soil, rock, and similar materials by mass. For simplicity, the word "material" hereinafter also refers to either soil or rock, whichever is most applicable.

1.2 The water content of a material is defined by this standard as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material.

1.3 The term "solid particles" as used in geotechnical engineering is typically assumed to mean naturally occurring mineral particles of soil and rock that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, and the like) may require special treatment or a qualified definition of water content. In addition, some organic materials may be decomposed by oven drying at the standard drying temperature for this method (110°C). Materials containing gypsum (calcium sulfate dihydrate or other compounds having significant amounts of hydrated water) may present a special problem as this material slowly dehydrates at the standard drying temperature (110°C) and at very low relative humidities, forming a compound (calcium sulfate hemihydrate) which is not normally present in natural materials except in some desert soils. In order to reduce the degree of dehydration of gypsum in those materials containing gypsum, or to reduce decomposition in highly organic soils, it may be desirable to dry these materials at 60°C or in a desiccator at room temperature. Thus, when a drying temperature is used which is different from the standard drying temperature as defined by this test method, the resulting water content may be different from standard water content determined at the standard drying temperature.

NOTE 1—Test Method D 2974 provides an alternate procedure for determining water content of peat materials.

1.4 Materials containing water with substantial amounts of soluble solids (such as salt in the case of marine sediments) when tested by this method will give a mass of solids which includes the previously soluble solids. These materials require special treatment to remove or account for the presence of precipitated solids in the dry mass of the specimen, or a qualified definition of water content must be used.

1.5 This test method requires several hours for proper drying of the water content specimen. Test Method D 4643 provides for drying of the test specimen in a microwave oven which is a shorter process.

1.6 This standard requires the drying of material in an oven at high temperatures. If the material being dried is contaminated with certain chemicals, health and safety hazards can exist. Therefore, this standard should not be used in determining the water content of contaminated soils unless adequate health and safety precautions are taken.

1.7 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock and Contained Fluids[2]

D 2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils[2]

D 4220 Practice for Preserving and Transporting Soil Samples[2]

D 4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[2]

D 4643 Test Method for Determination of Water (Moisture) Content of Soil by the Microwave Oven Method[2]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Soil and Rock Testing[2]

E 145 Specification for Gravity—Convection and Forced—Ventilation Ovens[3]

3. Terminology

3.1 Refer to Terminology D 653 for standard definitions of terms.

3.2 *Description of Term Specific to This Standard:*

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved Nov. 30, 1990. Published January 1991. Originally published as D 2216 – 63 T. Last previous edition D 2216 – 80.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

3.2.1 *water content* (of a material)—the ratio of the mass of water contained in the pore spaces of soil or rock material, to the solid mass of particles in that material, expressed as a percentage.

4. Summary of Method

4.1 A test specimen is dried in an oven to a constant mass. The loss of mass due to drying is considered to be water. The water content is calculated using the mass of water and the mass of the dry specimen.

5. Significance and Use

5.1 For many materials, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and its properties.

5.2 The water content of a material is used in expressing the phase relationships of air, water, and solids in a given volume of material.

5.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limits as determined by Test Method D 4318, is used to express its relative consistency or liquidity index.

6. Apparatus

6.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E 145 and capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

6.2 *Balances*—All balances must meet the requirements of Specification D 4753 and this Section. A Class GP1 balance of 0.01g readability is required for specimens having a mass of up to 200 g (excluding mass of specimen container) and a Class GP2 balance of 0.1g readability is required for specimens having a mass over 200 g.

6.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and change in mass upon repeated heating, cooling, exposure to materials of varying pH, and cleaning. Containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used. One container is needed for each water content determination.

NOTE 2—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial mass determination and to prevent absorption of moisture from the atmosphere following drying and before final mass determination.

6.4 *Desiccator*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium phosphate. It is preferable to use a desiccant which changes color to indicate it needs reconstitution. See Section 10.5.

NOTE 3—Anhydrous calcium sulfate is sold under the trade name Drierite.

6.5 *Container Handling Apparatus*, gloves, tongs, or suitable holder for moving and handling hot containers after drying.

6.6 *Miscellaneous*, knives, spatulas, scoops, quartering cloth, sample splitters, etc, as required.

7. Samples

7.1 Samples shall be preserved and transported in accordance with Practice 4220 Groups B, C, or D soils. Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight. Disturbed samples in jars or other containers shall be stored in such a way as to prevent or minimize moisture condensation on the insides of the containers.

7.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as thin-walled steel tubes, paint cans, etc.) or plastic sample bags are used.

8. Test Specimen

8.1 For water contents being determined in conjunction with another ASTM method, the specimen mass requirement stated in that method shall be used if one is provided. If no minimum specimen mass is provided in that method then the values given before shall apply.

8.2 The minimum mass of moist material selected to be representative of the total sample, if the total sample is not tested by this method, shall be in accordance with the following:

Maximum particle size (100 % passing)	Standard Sieve Size	Recommended minimum mass of moist test specimen for water content reported to ±0.1 %	Recommended minimum mass of moist test specimen for water content reported to ±1 %
2 mm or less	No. 10	20 g	20 g*
4.75 mm	No. 4	100 g	20 g*
9.5 mm	⅜-in.	500 g	50 g
19.0 mm	¾-in.	2.5 kg	250 g
37.5 mm	1½ in.	10 kg	1 kg
75.0 mm	3-in.	50 kg	5 kg

NOTE—*To be representative not less than 20 g shall be used.

8.2.1 If the total sample is used it does not have to meet the minimum mass requirements provided in the table above. The report shall indicate that the entire sample was used.

8.3 Using a test specimen smaller than the minimum indicated in 8.2 requires discretion, though it may be adequate for the purposes of the test. Any specimen used not meeting these requirements shall be noted in the report of results.

8.4 When working with a small (less than 200g) specimen containing a relatively large gravel particle, it is appropriate not to include this particle in the test specimen. However, any discarded material shall be described and noted in the report of the results.

9. Test Specimen Selection

9.1 When the test specimen is a portion of a larger amount of material, the specimen must be selected to be representative of the water condition of the entire amount of material. The manner in which the test specimen is selected depends on the purpose and application of the test, type of material being tested, the water condition, and the type of sample (from another test, bag, block, and the likes.)

9.2 For disturbed samples such as trimmings, bag samples, and the like, obtain the test specimen by one of the

following methods (listed in order of preference):

9.2.1 If the material is such that it can be manipulated and handled without significant moisture loss, the material should be mixed and then reduced to the required size by quartering or splitting.

9.2.2 If the material is such that it cannot be thoroughly mixed and/or split, form a stockpile of the material, mixing as much as possible. Take at least five portions of material at random locations using a sampling tube, shovel, scoop, trowel, or similar device appropriate to the maximum particle size present in the material. Combine all the portions for the test specimen.

9.2.3 If the material or conditions are such that a stockpile cannot be formed, take as many portions of the material as possible at random locations that will best represent the moisture condition. Combine all the portions for the test specimen.

9.3 Intact samples such as block, tube, split barrel, and the like, obtain the test specimen by one of the following methods depending on the purpose and potential use of the sample.

9.3.1 Carefully trim at least 3 mm of material from the outer surface of the sample to see if material is layered and to remove material that is drier or wetter than the main portion of the sample. Then carefully trim at least 5 mm, or a thickness equal to the maximum particle size present, from the entire exposed surface or from the interval being tested.

9.3.2 Slice the sample in half. If material is layered see Section 9.3.3. Then carefully trim at least 5 mm, or a thickness equal to the maximum particle size present, from the exposed surface of one half, or from the interval being tested. Avoid any material on the edges that may be wetter or drier than the main portion of the sample.

NOTE 4—Migration of moisture in some cohesionless soils may require that the full section be sampled.

9.3.3 If a layered material (or more than one material type is encountered), select an average specimen, or individual specimens, or both. Specimens must be properly identified as to location, or what they represent, and appropriate remarks entered on data sheets.

10. Procedure

10.1 Determine and record the mass of the clean and dry specimen container (and its lid, if used).

10.2 Select representative test specimens in accordance with Section 9.

10.3 Place the moist test specimen in the container and, if used, set the lid securely in position. Determine the mass of the container and moist material using a balance (See 6.2) selected on the basis of the specimen mass. Record this value.

NOTE 5—To prevent mixing of specimens and yielding of incorrect results, all containers, and lids if used, should be numbered and the container numbers shall be recorded on the laboratory data sheets. The lid numbers should match the container numbers to eliminate confusion.

NOTE 6—To assist in the oven-drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

10.4 Remove the lid (if used) and place the container with moist material in the drying oven. Dry the material to a constant mass. Maintain the drying oven at 110 ± 5°C unless otherwise specified (see 1.3). The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used.

NOTE 7—In most cases, drying a test specimen overnight (about 12 to 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the change in mass after two successive periods (greater than 1 h) of drying is an insignificant amount (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

NOTE 8—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the same oven. However, this would not be applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

10.5 After the material has dried to constant mass remove the container from the oven (and replace the lid if used). Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents and/or its being heated. Determine the mass of the container and oven-dried material using the same balance as used in 10.3. Record this value. Tight fitting lids shall be used if it appears that the specimen is absorbing moisture from the air prior to determination of its dry mass.

NOTE 9—Cooling in a desiccator is acceptable in place of tight fitting lids since it greatly reduces absorption of moisture from the atmosphere during cooling especially for containers without tight fitting lids.

11. Calculation

11.1 Calculate the water content of the material as follows:

$$w = [(M_{cws} - M_{cs})/(M_{cs} - M_c)] \times 100 = \frac{M_w}{M_s} \times 100$$

where:

w = water content, %,
M_{cws} = mass of container and wet specimen, g,
M_{cs} = mass of container and oven dry specimen, g,
M_c = mass of container, g,
M_w = mass of water ($M_w = M_{cws} - M_{cds}$), g, and
M_s = mass of solid particles ($M_s = M_{cds} - M_c$), g.

12. Report

12.1 The report (data sheet) shall include the following:

12.1.1 Identification of the sample (material) being tested, such as boring number, sample number, test number, container number etc.

12.1.2 Water content of the specimen to the nearest 1 % or 0.1 %, as appropriate based on the minimum sample used. If this method is used in concert with another method, the water content of the specimen should be reported to the value required by the test method for which the water content is being determined.

12.1.3 Indicate if test specimen had a mass less than the minimum indicated in 8.2.

12.1.4 Indicate if test specimen contained more than one material type (layered, etc.).

12.1.5 Indicate the method of drying if different from oven-drying at 110 ± 5°C.

12.1.6 Indicate if any material (size and amount) was excluded from the test specimen.

13. Precision and Bias

13.1 *Statement on Bias*—There is no accepted reference value for this test method; therefore, bias cannot be determined.

13.2 *Statements on Precision:*

13.2.1 *Single-Operator Precision*—The single-operator coefficient of variation has been found to be 2.7 percent. Therefore, results of two properly conducted tests by the same operator with the same equipment should not be considered suspect unless they differ by more than 7.8 percent of their mean.

13.2.2 *Multilaboratory Precision*—The multilaboratory coefficient of variation has been found to be 5.0 percent. Therefore, results of two properly conducted tests by different operators using different equipment should not be considered suspect unless they differ by more than 14.0 percent of their mean.

14. Keywords

14.1 Consistency; index property; laboratory; moisture analysis; moisture content; soil aggregate; water content

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METHOD: ASTM D422
STANDARD OPERATING PROCEDURE
FOR PARTICLE SIZE ANALYSIS OF SOILS

1.0 SCOPE AND APPLICATION

1.1 This method determines the particle size distribution in soil. Particles greater than 75um (gravel to fine sands) are determined by sieving, and particles less than 75um (silts and clays) are determined by sedimentation using a hydrometer.

1.2 Minimum quantity of sample depends on subsequent analyses to be performed. Typical range is 150 to 350 grams of dry soil. Larger amounts (from 500 to 5,000 grams) are specified for particle size analysis of soils with appreciable gravel component.

1.3 This preparation is amenable to samples containing sand, silt, clay and gravel.

2.0 SUMMARY OF METHOD

2.1 Soils for particle size analysis are prepared according to ASTM D421 or D2217. These soils are sieved in two steps. The particles greater than 2.00mm (retained on the No. 10 sieve) are sieved after the soil has been prepared. A portion of the soil passing the No. 10 sieve is prepared for hydrometer measurements. Six hydrometer readings are made over a 24 hour time frame. The soil in the hydrometer is rinsed on a No. 40, No. 100, and No. 200 (75um) sieve and dried for sieve analysis of material less than 2.0 (No. 10 sieve). Calculations are made to determine the percent finer of soil for each sieve and hydrometer reading. These calculations are dependent on percent solid, which is determined during the drying process, and the specific gravity that is assumed to be 2.65 (unless separate analysis is requested for specific gravity).

3.0 SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

4.0 EQUIPMENT AND SUPPLIES

4.1 Balance sensitive to 0.01 grams.

4.2 Mixer and dispersion cup.

4.3 1000 ml sedimentation cylinder.

4.4 Soil test hydrometer meeting specification E 100.

4.5 Mortar and rubber tipped pestle for breaking up soil aggregates.

4.6 Sieves of the following size:
1.0 inch (25.00mm)
3/4 inch (19.00mm)
1/2 inch (12.70mm)
No. 4 (4.75mm)
No.10 (2.00 mm)
No. 40 (425um)
No. 100 (150.0um)
No. 200 (75.0um)

4.7 Oven with temperature range of 60° C to 110° C.

4.8 Thermometer accurate to 0.5° C.

4.9 Timer with second hand and capable of counting up to 25 hours.

4.10 Mixing utensils, metal and bristle brushes for sample recovery.

4.11 Rototap machine.

5.0 REAGENTS AND STANDARDS

5.1 Sodium Hexametaphosphate (dispersion reagent)

6.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

6.1 Typical sample of 150 to 350 grams is used for analysis. Larger amounts (from 500 to 5000 grams) are specified for particle size analysis of soils with appreciable gravel component. The sample container must remain sealed to maintain natural water content.

6.2 There are no holding time requirements.

7.0 QUALITY CONTROL

7.1 Check balance daily.

7.2 Oven temperature is checked daily prior to start of work.

7.3 Thermometer is checked against similar or more accurate temperature device.

7.4 Duplicate samples are recommended every 20 samples.

8.0 PROCEDURE

8.1 Separate the portion retained on the No. 10 sieve into a series of fractions using the 1-inch (25.0mm), 3/4-inch (19.0mm), 1/2-inch (12.7mm), No. 4 (4.75mm), and No. 10 (2.0mm) sieves. Other sieves may be used as specified by the client.

8.2 The sieving operation is conducted by stacking the sieves, largest on top, and using a lateral and vertical motion accompanied by a jarring action to move the sample of the surface of the sieve.

8.3 Weigh and record the contents of each sieve.

9.0 HYDROMETER TEST: The soil passing the No. 10 sieve is used in this step.

9.1 Sample Preparation:

- Tare a 250 ml beaker. Place and record approximately 50 grams for silt or clay particles or 100 grams for sand particles into the beaker. Add 125 ml of a 40g/l sodium Hexametaphosphate solution to sample and allow to soak overnight.

- Rinse the sample with DI water into a dispersion cup. Fill the cup to the halfway mark with DI water and place cup on the blender. Mix sample for approximately one minute. Pour content of cup into a 1000 ml sedimentation cylinder. Fill the cylinder to the 1000 ml line.

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9.2 After preparing up to a maximum of 12 flasks, begin setup for hydrometer readings. The following paperwork is needed: hydrometer data sheet. Initiate timer to indicate the elapsed time, counting up from zero. Check readings of hydrometer and temperature probe in a DI water rinse bath. Get the rubber stopper to shake flask and prepare staging and test areas.

9.3 Initiate timer to indicate the elapsed time.

A reading consists of inserting the hydrometer gently into the cylinder, after the cylinder has been shaken for one minute, about 20 seconds before the actual reading. Read the hydrometer to the nearest 0.0005 at the top of the meniscus. Remove the hydrometer and insert a temperature sensor into the cylinder to a depth to which the hydrometer reached. Read the temperature meter to the nearest 0.1° C and remove the temperature sensor. The hydrometers and temperature sensor are rinsed in a DI bath between each reading.

After each cylinder is read, the hydrometer reading, temperature, and time is entered on the hydrometer data sheet. The readings are taken at 5, 30, 60, 120, 240, and 1440 minutes.

9.4 Small Sieve: Soils from the hydrometer test are rinsed on the No. 40, No. 100, and No. 200 sieves. The soil retained on the sieves is placed in an oven and dried overnight.

Weigh and record the content of each sieve.

10.0 CALCULATIONS:

10.1 Diameter of soil particles.

The diameter of particles corresponding to the percentage indicated by a given hydrometer reading shall be calculated according the Stoke's Law on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension.

10.2 Sieve analysis values for portions finer than the No. 10 sieve.

Calculate the total percentages passing each sieve by dividing the total mass passing by the total mass of the sample and multiplying the result by 100.

11.0 GRAPH

11.1 When hydrometer analysis is performed, data is presented in a graph form.

12.0 REFERENCES

12.1 Annual Book of ASTM Standards, Volume 04.05 Soil and Rock. American Society for Testing and Materials.

Designation: D 698 – 91

Test Method for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft³ (600 kN-m/m³))[1]

This standard is issued under the fixed designation D 698; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers laboratory compaction procedures used to determine the relationship between water content and dry unit weight of soils (compaction curve) compacted in a 4 or 6-in. (101.6 or 152.4-mm) diameter mold with a 5.5-lbf (24.4-N) rammer dropped from a height of 12 in. (305 mm) producing a compactive effort of 12,400 ft-lbf/ft³ (600 kN-m/m³).

NOTE 1—The equipment and procedures are similar as those proposed by R. R. Proctor (*Engineering News Record*—September 7, 1933) with this one major exception: his rammer blows were applied as "12 inch firm strokes" instead of free fall, producing variable compactive effort depending on the operator, but probably in the range 15,000 to 25,000 ft-lbf/ft³ (700 to 1,200 kN-m/m³). The standard effort test (see 3.2.2) is sometimes referred to as the Proctor Test.

NOTE 2—Soils and soil-aggregate mixtures should be regarded as natural occurring fine- or coarse-grained soils or composites or mixtures of natural soils, or mixtures of natural and processed soils or aggregates such as silt, gravel, or crushed rock.

1.2 This test method applies only to soils that have 30 % or less by weight of particles retained on the ¾-inch (19.0-mm) sieve.

NOTE 3—For relationships between unit weights and water contents of soils with 30 % or less by weight of material retained on the ¾-in. (19.0-mm) sieve to unit weights and water contents of the fraction passing ¾-in. (19.0-mm) sieve, see Practice D 4718.

1.3 Three alternative procedures are provided. The procedure used shall be as indicated in the specification for the material being tested. If no procedure is specified, the choice should be based on the material gradation.

1.3.1 *Procedure A:*

1.3.1.1 *Mold*—4-in. (101.6-mm) diameter.

1.3.1.2 *Material*—Passing No. 4 (4.75-mm) sieve.

1.3.1.3 *Layers*—Three.

1.3.1.4 *Blows per layer*—25.

1.3.1.5 *Use*—May be used if 20 % or less by weight of the material is retained on the No. 4 (4.75-mm) sieve.

1.3.1.6 *Other Use*—If this procedure is not specified, materials that meet these gradation requirements may be tested using Procedures B or C.

1.3.2 *Procedure B:*

1.3.2.1 *Mold*—4-in. (101.6-mm) diameter.

1.3.2.2 *Material*—Passing ⅜-in. (9.5-mm) sieve.

1.3.2.3 *Layers*—Three.

1.3.2.4 *Blows per layer*—25.

1.3.2.5 *Use*—Shall be used if more than 20 % by weight of the material is retained on the No. 4 (4.75-mm) sieve and 20 % or less by weight of the material is retained on the ⅜-in. (9.5-mm) sieve.

1.3.2.6 *Other Use*—If this procedure is not specified, materials that meet these gradation requirements may be tested using Procedure C.

1.3.3 *Procedure C:*

1.3.3.1 *Mold*—6-in. (152.4-mm) diameter.

1.3.3.2 *Material*—Passing ¾-inch (19.0-mm) sieve.

1.3.3.3 *Layers*—Three.

1.3.3.4 *Blows per layer*—56.

1.3.3.5 *Use*—Shall be used if more than 20 % by weight of the material is retained on the ⅜-in. (9.5-mm) sieve and less than 30 % by weight of the material is retained on the ¾-in. (19.0-mm) sieve.

1.3.4 The 6-in. (152.4-mm) diameter mold shall not be used with Procedure A or B.

NOTE 4—Results have been found to vary slightly when a material is tested at the same compactive effort in different size molds.

1.4 If the test specimen contains more than 5 % by weight oversize fraction (coarse fraction) and the material will not be included in the test, corrections must be made to the unit weight and water content of the specimen or to the appropriate field in place density test specimen using Practice D 4718.

1.5 This test method will generally produce a well defined maximum dry unit weight for non-free draining soils. If this test method is used for free draining soils the maximum unit weight may not be well defined, and can be less than obtained using Test Methods D 4253.

1.6 The values in inch-pound units are to be regarded as the standard. The values stated in SI units are provided for information only.

1.6.1 In the engineering profession it is customary practice to use, interchangeably, units representing both mass and force, unless dynamic calculations ($F = Ma$) are involved. This implicitly combines two separate systems of units, that is, the absolute system and the gravimetric system. It is scientifically undesirable to combine the use of two separate systems within a single standard. This test method has been written using inch-pound units (gravimetric system) where the pound (lbf) represents a unit of force. The use of mass (lbm) is for convenience of units and is not intended to convey the use is scientifically correct. Conversions are given in the SI system in accordance with Practice E 380. The use of balances or scales recording pounds of mass (lbm), or the recording of density in lbm/ft³ should not be regarded as nonconformance with this standard.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

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1.7 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]

C 136 Method for Sieve Analysis of Fine and Coarse Aggregate[2]

D 422 Test Method for Particle Size Analysis of Soils[3]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[3]

D 854 Test Method for Specific Gravity of Soils[3]

D 1557 Test Methods for Moisture-Density Relations of Soils and Soil Aggregate Mixtures Using 10-lb (4.54-kg.) Rammer and 18-in. (457 mm) Drop[3]

D 2168 Test Methods for Calibration of Laboratory Mechanical-Rammer Soil Compactors[3]

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock and Soil-Aggregate Mixtures[3]

D 2487 Test Method for Classification of Soils for Engineering Purposes[3]

D 2488 Practice for Description of Soils (Visual-Manual Procedure)[3]

D 4220 Practices for Preserving and Transporting Soil Samples[3]

D 4253 Test Methods for Maximum Index Density of Soils Using a Vibratory Table[3]

D 4718 Practice for Correction of Unit Weight and Water Content for Soils Containing Oversize Particles[3]

D 4753 Specification for Evaluating, Selecting and Specifying Balances and Scales For Use in Soil and Rock Testing[3]

E 1 Specification for ASTM Thermometers[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 319 Practice for the Evaluation of Single-Pan Mechanical Balances[5]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[5]

3. Terminology

3.1 *Definitions*—See Terminology D 653 for general definitions.

3.2 *Description of Terms Specific to This Standard:*

3.2.1 *oversize fraction (coarse fraction), P_c* in %—the portion of total sample not used in performing the compaction test; it may be the portion of total sample retained on the No. 4 (4.75-mm), 3/8-in. (9.5-mm), or 3/4-in. (19.0-mm) sieve.

3.2.2 *standard effort*—the term for the 12,400 ft-lbf/ft^3 (600 kN-m/m^3) compactive effort applied by the equipment and procedures of this test.

3.2.3 *standard maximum dry unit weight, γ_{dmax}* in lbf/ft^3 (kN/m^3)—the maximum value defined by the compaction curve for a compaction test using standard effort.

3.2.4 *standard optimum water content, w_o* in %—the water content at which a soil can be compacted to the maximum dry unit weight using standard compactive effort.

3.2.5 *test fraction (finer fraction), P_F* in %—the portion of the total sample used in performing the compaction test; it is the fraction passing the No. 4 (4.75-mm) sieve in Procedure A, minus 3/8-in. (9.5-mm) sieve in Procedure B, or minus 3/4-in. (19.0-mm) sieve in Procedure C.

4. Summary of Test Method

4.1 A soil at a selected water content is placed in three layers into a mold of given dimensions, with each layer compacted by 25 or 56 blows of a 5.5-lbf (24.4-N) rammer dropped from a distance of 12-in. (305-mm), subjecting the soil to a total compactive effort of about 12,400 ft-lbf/ft^3 (600 kN-m/m^3). The resulting dry unit weight is determined. The procedure is repeated for a sufficient number of water contents to establish a relationship between the dry unit weight and the water content for the soil. This data, when plotted, represents a curvilinear relationship known as the compaction curve. The values of optimum water content and standard maximum dry unit weight are determined from the compaction curve.

5. Significance and Use

5.1 Soil placed as engineering fill (embankments, foundation pads, road bases) is compacted to a dense state to obtain satisfactory engineering properties such as, shear strength, compressibility, or permeability. Also, foundation soils are often compacted to improve their engineering properties. Laboratory compaction tests provide the basis for determining the percent compaction and water content needed to achieve the required engineering properties, and for controlling construction to assure that the required compaction and water contents are achieved.

5.2 During design of an engineered fill, shear, consolidation, permeability, or other tests require preparation of test specimens by compacting at some water content to some unit weight. It is common practice to first determine the optimum water content (w_o) and maximum dry unit weight (γ_{dmax}) by means of a compaction test. Test specimens are compacted at a selected water content (w), either wet or dry of optimum (w_o) or at optimum (w_o), and at a selected dry unit weight related to a percentage of maximum dry unit weight (γ_{dmax}). The selection of water content (w), either wet or dry of optimum (w_o) or at optimum (w_o) and the dry unit weight (γ_{dmax}) may be based on past experience, or a range of values may be investigated to determine the necessary percent of compaction.

6. Apparatus

6.1 *Mold Assembly*—The molds shall be cylindrical in shape, made of rigid metal and be within the capacity and dimensions indicated in 6.1.1 or 6.1.2 and Figs. 1 and 2. The walls of the mold may be solid, split, or tapered. The "split" type may consist of two half-round sections, or a section of

[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vol 04.08.
[4] *Annual Book of ASTM Standards*, Vol 14.03.
[5] *Annual Book of ASTM Standards*, Vol 14.02.

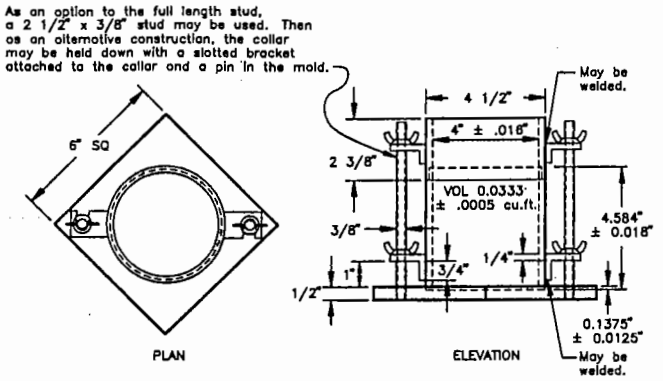

FIG. 1 4.0-in. Cylindrical Mold

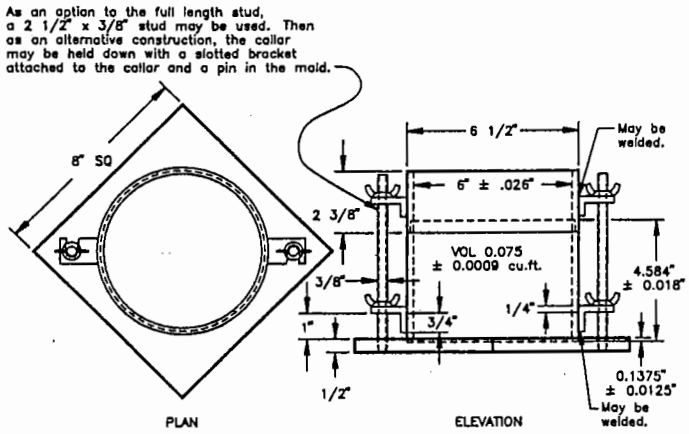

FIG. 2 6.0-in. Cylindrical Mold

pipe split along one element, which can be securely locked together to form a cylinder meeting the requirements of this section. The "tapered" type shall an internal diameter taper that is uniform and not more than 0.200 in./ft (16.7- mm/m) of mold height. Each mold shall have a base plate and an extension collar assembly, both made of rigid metal and constructed so they can be securely attached and easily detached from the mold. The extension collar assembly shall have a height extending above the top of the mold of at least 2.0 in. (50.8-mm) which may include an upper section that flares out to form a funnel provided there is at least a 0.75 in. (19.0-mm) straight cylindrical section beneath it. The extension collar shall align with the inside of the mold. The bottom of the base plate and bottom of the centrally recessed area that accepts the cylindrical mold shall be planar.

6.1.1 *Mold, 4 in.*—A mold having a 4.000 ± 0.016-in. (101.6 ± 0.4-mm) average inside diameter, a height of 4.584 ± 0.018-in. (116.4 ± 0.5-mm) and a volume of 0.0333 ± 0.0005 ft^3 (944 ± 14 cm^3). A mold assembly having the minimum required features is shown in Fig. 1.

6.1.2 *Mold, 6 in.*—A mold having a 6.000 ± 0.026-in. (152.4 ± 0.7-mm) average inside diameter, a height of 4.584 ± 0.018-in. (116.4 ± 0.5-mm), and a volume of 0.075 ± 0.0009 ft^3 (2124 ± 25 cm^3). A mold assembly having the minimum required features is shown in Fig. 2.

6.2 *Rammer*—A rammer, either manually operated as described further in 6.2.1 or mechanically operated as described in 6.2.2. The rammer shall fall freely through a distance of 12 ± 0.05-in. (304.8 ± 1.3-mm) from the surface of the specimen. The mass of the rammer shall be 5.5 ± 0.02-lbm (2.5 ± 0.01-kg), except that the mass of the mechanical rammers may be adjusted as described in Test Methods D 2168, see Note 5. The striking face of the rammer shall be planar and circular, except as noted in 6.2.2.3, with a diameter when new of 2.000 ± 0.005-in. (50.80 ± 0.13-mm). The rammer shall be replaced if the striking face becomes worn or bellied to the extent that the diameter exceeds 2.000 ± 0.01-in. (50.80 ± 0.25-mm).

NOTE 5—It is a common and acceptable practice in the inch-pound system to assume that the mass of the rammer is equal to its mass determined using either a kilogram or pound balance and 1 lbf is equal to 1 lbm or 0.4536 kg. or 1 N is equal to 0.2248 lbm or 0.1020 kg.

6.2.1 *Manual Rammer*—The rammer shall be equipped with a guide sleeve that has sufficient clearance that the free fall of the rammer shaft and head is not restricted. The guide sleeve shall have at least four vent holes at each end (eight holes total) located with centers ¾ ± 1/16-in. (19.0 ± 1.6-mm) from each end and spaced 90 degrees apart. The minimum diameter of the vent holes shall be ⅜-in. (9.5-mm). Additional holes or slots may be incorporated in the guide sleeve.

6.2.2 *Mechanical Rammer-Circular Face*—The rammer shall operate mechanically in such a manner as to provide uniform and complete coverage of the specimen surface. There shall be 0.10 ± 0.03-in. (2.5 ± 0.8-mm) clearance between the rammer and the inside surface of the mold at its smallest diameter. The mechanical rammer shall meet the calibration requirements of Test Methods D 2168. The mechanical rammer shall be equipped with a positive mechanical means to support the rammer when not in operation.

6.2.2.3 *Mechanical Rammer-Sector Face*—When used with the 6-in. (152.4-mm) mold, a sector face rammer may be used in place of the circular face rammer. The specimen contact face shall have the shape of a sector of a circle of radius equal to 2.90 ± 0.02-in. (73.7 ± 0.5-mm). The rammer shall operate in such a manner that the vertex of the sector is positioned at the center of the specimen.

6.3 *Sample Extruder (optional)*—A jack, frame or other device adapted for the purpose of extruding compacted specimens from the mold.

6.4 *Balance*—A class GP5 balance meeting the requirements of Specification D 4753 for a balance of 1-g readability.

6.5 *Drying Oven*—Thermostatically controlled, preferably of a forced-draft type and capable of maintaining a uniform temperature of 230 ± 9°F (110 ± 5°C) throughout the drying chamber.

6.6 *Straightedge*—A stiff metal straightedge of any convenient length but not less than 10-in. (254-mm). The total length of the straightedge shall be machined straight to a tolerance of ±0.005-in. (±0.1-mm). The scraping edge shall be beveled if it is thicker than ⅛-in. (3-mm).

6.7 *Sieves*—¾-in. (19.0-mm), ⅜-in. (9.5-mm), and No. 4 (4.75-mm), conforming to the requirements of Specification E 11.

6.8 *Mixing Tools*—Miscellaneous tools such as mixing pan, spoon, trowel, spatula, etc., or a suitable mechanical device for thoroughly mixing the sample of soil with increments of water.

7. Calibration

7.1 Perform calibrations before initial use, after repairs or other occurrences that might affect the test results, at intervals not exceeding 1,000 test specimens, or annually, whichever occurs first, for the following apparatus:

7.1.2 *Balance*—Evaluate in accordance with Specification D 4753.

7.1.3 *Molds*—Determine the volume as described in Annex 1.

7.1.4 *Manual Rammer*—Verify the free fall distance, rammer mass, and rammer face in accordance with Section 6.2. Verify the guide sleeve requirements in accordance with Section 6.2.1.

7.1.5 *Mechanical Rammer*—Calibrate and adjust the mechanical rammer in accordance with Test Methods D 2168. In addition, the clearance between the rammer and the inside surface of the mold shall be verified in accordance with 6.2.2.

8. Test Sample

8.1 The required sample mass for Procedures A and B is approximately 35-lbm (16-kg), and for Procedure C is approximately 65-lbm (29-kg) of dry soil. Therefore, the field sample should have a moist mass of at least 50-lbm (23-kg) and 100-lbm (45-kg), respectively.

8.2 Determine the percentage of material retained on the No. 4 (4.75-mm), 3/8-in. (9.5-mm), or 3/4-in. (19.0-mm) sieve as appropriate for choosing Procedure A, B, or C. Make this determination by separating out a representative portion from the total sample and determining the percentages passing the sieves of interest by Test Methods D 422 or Method C 136. It is only necessary to calculate percentages for the sieve or sieves for which information is desired.

9. Preparation of Apparatus

9.1 Select the proper compaction mold in accordance with the procedure (A, B, or C) being used. Determine and record its mass to the nearest gram. Assemble the mold, base and extension collar. Check the alignment of the inner wall of the mold and mold extension collar. Adjust if necessary.

9.2 Check that the rammer assembly is in good working condition and that parts are not loose or worn. Make any necessary adjustments or repairs. If adjustments or repairs are made, the rammer must be recalibrated.

10. Procedure

10.1 *Soils:*

10.1.1 Do not reuse soil that has been previously laboratory compacted.

10.1.2 When using this test method for soils containing hydrated halloysite, or where past experience with a particular soil indicates that results will be altered by air drying, use the moist preparation method (see 10.2).

10.1.3 Prepare the soil specimens for testing in accordance with 10.2 (preferred) or with 10.3.

10.2 *Moist Preparation Method (preferred)*—Without previously drying the sample, pass it through a No. 4 (4.75-mm), 3/8-in. (9.5-mm), or 3/4-in. (19.0-mm) sieve, depending on the procedure (A, B, or C) being used. Determine the water content of the processed soil.

10.2.1 Prepare at least four (preferably five) specimens having water contents such that they bracket the estimated optimum water content. A specimen having a water content close to optimum should be prepared first by trial additions of water and mixing (see Note 6). Select water contents for the rest of the specimens to provide at least two specimens wet and two specimens dry of optimum, and water contents varying by about 2 %. At least two water contents are necessary on the wet and dry side of optimum to accurately define the dry unit weight compaction curve (see 10.5). Some soils with very high optimum water content or a relatively flat compaction curve may require larger water content increments to obtain a well defined maximum dry unit weight. Water content increments should not exceed 4 %.

NOTE 6—With practice it is usually possible to visually judge a point near optimum water content. Typically, soil at optimum water content can be squeezed into a lump that sticks together when hand pressure is released, but will break cleanly into two sections when "bent". At water contents dry of optimum soils tend to crumble; wet of optimum soils tend to stick together in a sticky cohesive mass. Optimum water content is typically slightly less than the plastic limit.

10.2.2 Use approximately 5-lbm (2.3-kg) of the sieved soil for each specimen to be compacted using Procedure A or B, or 13-lbm (5.9-kg) using Procedure C. To obtain the specimen water contents selected in 10.2.1, add or remove the required amounts of water as follows: to add water, spray it into the soil during mixing; to remove water, allow the soil to dry in air at ambient temperature or in a drying apparatus such that the temperature of the sample does not exceed 140°F (60°C). Mix the soil frequently during drying to maintain an even water content distribution. Thoroughly mix each specimen to ensure even distribution of water throughout and then place in a separate covered container and allow to stand in accordance with Table 1 prior to compaction. For the purpose of selecting a standing time, the soil may be classified using Test Method D 2487, Practice D 2488 or data on other samples from the same material source. For referee testing, classification shall be by Test Method D 2487.

10.3 *Dry Preparation Method*—If the sample is too damp to be friable, reduce the water content by air drying until the material is friable. Drying may be in air or by the use of drying apparatus such that the temperature of the sample does not exceed 140°F (60°C). Thoroughly break up the aggregations in such a manner as to avoid breaking individual particles. Pass the material through the appropriate sieve: No. 4 (4.75-mm), 3/8-in. (9.5-mm), or 3/4-in. (19.0-mm). When preparing the material by passing over the 3/4-in. sieve for compaction in the 6-in. mold, break up aggregations sufficiently to at least pass the 3/8-in. sieve in order to facilitate the distribution of water throughout the soil in later mixing.

10.3.1 Prepare at least four (preferably five) specimens in accordance with 10.2.1.

10.3.2 Use approximately 5-lbm (2.3-kg) of the sieved soil for each specimen to be compacted using Procedure A or B,

TABLE 1 Required Standing Times of Moisturized Specimens

Classification	Minimum Standing Time, h
GW, GP, SW, SP	No Requirement
GM, SM	3
All other soils	16

TABLE 2 Metric Equivalents for Figs. 1 and 2

in.	mm
0.016	0.41
0.026	0.66
0.032	0.81
0.028	0.71
½	12.70
2½	63.50
2⅝	66.70
4	101.60
4½	114.30
4.584	116.43
4¾	120.60
6	152.40
6½	165.10
6⅝	168.30
6¾	171.40
8¼	209.60
ft³	**cm³**
1/30 (0.0333)	943
0.0005	14
1/13.333 (0.0750)	2,124
0.0011	31

or 13-lbm (5.9-kg) using Procedure C. Add the required amounts of water to bring the water contents of the specimens to the values selected in 10.3.1. Follow the specimen preparation procedure specified in 10.2.2 for drying the soil or adding water into the soil and curing each test specimen.

10.4 *Compaction*—After curing, if required, each specimen shall be compacted as follows:

10.4.1 Determine and record the mass of the mold or mold and base plate.

10.4.2 Assemble and secure the mold and collar to the base plate. The mold shall rest on a uniform rigid foundation, such as provided by a cylinder or cube of concrete with a mass of not less than 200-lbm (91-kg). Secure the base plate to the rigid foundation. The method of attachment to the rigid foundation shall allow easy removal of the assembled mold, collar and base plate after compaction is completed.

10.4.3 Compact the specimen in three layers. After compaction, each layer should be approximately equal in thickness. Prior to compaction, place the loose soil into the mold and spread into a layer of uniform thickness. Lightly tamp the soil prior to compaction until it is not in a fluffy or loose state, using either the manual compaction rammer or a 2-in. (5-mm) diameter cylinder. Following compaction of each of the first two layers, any soil adjacent to the mold walls that has not been compacted or extends above the compacted surface shall be trimmed. The trimmed soil may be included with the additional soil for the next layer. A knife or other suitable device may be used. The total amount of soil used shall be such that the third compacted layer slighly extends into the collar, but does not exceed ¼-in. (6-mm) above the top of the mold. If the third layer does extend above the top of the mold by more than ¼-in. (6-mm), the specimen shall be discarded. The specimen shall be discarded when the last blow on the rammer for the third layer results in the bottom of the rammer extending below the top of the compaction mold.

10.4.4 Compact each layer with 25 blows for the 4-in. (101.6-mm) mold or with 56 blows for the 6-in. (152.4-mm) mold.

NOTE 7—When compacting specimens wetter than optimum water content, uneven compacted surfaces can occur and operator judgement is required as to the average height of the specimen.

10.4.5 In operating the manual rammer, take care to avoid lifting the guide sleeve during the rammer upstroke. Hold the guide sleeve steady and within 5° of vertical. Apply the blows at a uniform rate of approximately 25 blows/min and in such a manner as to provide complete, uniform coverage of the specimen surface.

10.4.6 Following compaction of the last layer, remove the collar and base plate from the mold, except as noted in 10.4.7. A knife may be used to trim the soil adjacent to the collar to loosen the soil from the collar before removal to avoid disrupting the soil below the top of the mold.

10.4.7 Carefully trim the compacted specimen even with the top of the mold by means of the straightedge scraped across the top of the mold to form a plane surface even with the top of the mold. Initial trimming of the specimen above the top of the mold with a knife may prevent the soil from tearing below the top of the mold. Fill any holes in the top surface with unused or trimmed soil from the specimen, press in with the fingers, and again scrape the straightedge across the top of the mold. Repeat the appropriate preceding operations on the bottom of the specimen when the mold volume was determined without the base plate. For very wet or dry soils, soil or water may be lost if the base plate is removed. For these situations, leave the base plate attached to the mold. When the base plate is left attached, the volume of the mold must be calibrated with the base plate attached to the mold rather than a plastic or glass plate as noted in Annex 1, A1.4.

10.4.8 Determine and record the mass of the specimen and mold to the nearest gram. When the base plate is left attached, determine and record the mass of the specimen, mold and base plate to the nearest gram.

10.4.9 Remove the material from the mold. Obtain a specimen for water content by using either the whole specimen (preferred method) or a representative portion. When the entire specimen is used, break it up to facilitate drying. Otherwise, obtain a portion by slicing the compacted specimen axially through the center and removing about 500-g of material from the cut faces. Obtain the water content in accordance with Test Method D 2216.

10.5 Following compaction of the last specimen, compare the wet unit weights to ensure that a desired pattern of obtaining data on each side of the optimum water content will be attained for the dry unit weight compaction curve. Plotting the wet unit weight and water content of each compacted specimen can be an aid in making the above evaluation. If the desired pattern is not obtained, additional compacted specimens will be required. Generally, one water content value wet of the water content defining the maximum wet unit weight is sufficient to ensure data on the wet side of optimum water content for the maximum dry unit weight.

11. Calculation

11.1 Calculate the dry unit weight and water content of each compacted specimen as explained in 11.3 and 11.4. Plot the values and draw the compaction curve as a smooth curve through the points (see example, Fig. 3). Plot dry unit weight

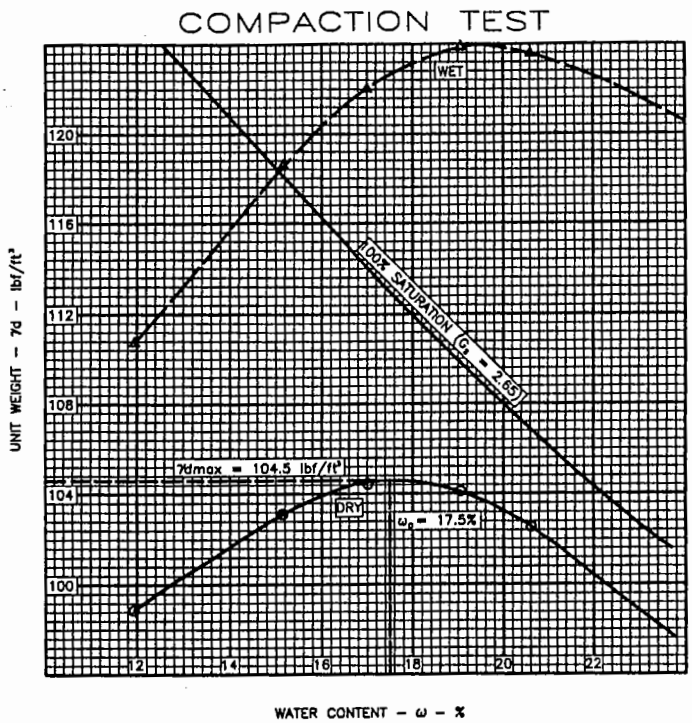

FIG. 3 Example Compaction Curve Plotting

to the nearest 0.1 lbf/ft³ (0.2 kN/m³) and water content to the nearest 0.1 %. From the compaction curve, determine the optimum water content and maximum dry unit weight. If more than 5 % by weight of oversize material was removed from the sample, calculate the corrected optimum water content and maximum dry unit weight of the total material using Practice D 4718. This correction may be made to the appropriate field in place density test specimen rather than to the laboratory test specimen.

11.2 Plot the 100 % saturation curve. Values of water content for the condition of 100 % saturation can be calculated as explained in 11.5 (see example, Fig. 3).

NOTE 8—The 100 % saturation curve is an aid in drawing the compaction curve. For soils containing more than approximately 10 % fines at water contents well above optimum, the two curves generally become roughly parallel with the wet side of the compaction curve between 92 % to 95 % saturation. Theoretically, the compaction curve cannot plot to the right of the 100 % saturation curve. If it does, there is an error in specific gravity, in measurements, in calculations, in test procedures, or in plotting.

NOTE 9—The 100 % saturation curve is sometimes referred to as the zero air voids curve or the complete saturation curve.

11.3 *Water Content, w*—Calculate in accordance with Test Method D 2216.

11.4 *Dry Unit Weights*—Calculate the moist density (Eq. 1), the dry density (Eq. 2), and then the dry unit weight (Eq. 3) as follows:

$$\rho_m = 1000(M_t - M_{md})/V \quad (1)$$

where:

ρ_m = moist density of compacted specimen, Mg/m³,
M_t = mass of moist specimen and mold, kg,
M_{md} = mass of compaction mold, kg, and
V = volume of compaction mold, m³ (see Annex 1)

$$\rho_d = \rho_m/(1 + w/100) \quad (2)$$

where:

ρ_d = dry density of compacted specimen, Mg/m³, and
w = water content, %.

$$\gamma_d = 62.43\ \rho_d \text{ in lbf/ft}^3 \quad (3)$$

or

$$\gamma_d = 9.807\ \rho_d \text{ in kN/m}^3$$

where:

γ_d = dry unit weight of compacted specimen.

11.5 To calculate points for plotting the 100 % saturation curve or zero air voids curve select values of dry unit weight, calculate corresponding values of water content corresponding to the condition of 100 % saturation as follows:

$$w_{sat} \frac{(\gamma_w)(G_s) - \gamma_d}{(\gamma_d)(G_s)} \times 100 \quad (4)$$

where:

w_{sat} = water content for complete saturation, %,
γ_w = unit weight of water, 62.43 lbf/ft³ (9.807 kn/m³),
γ_d = dry unit weight of soil, and
G_s = specific gravity of soil.

NOTE 10—Specific gravity may be estimated for the test specimen on the basis of test data from other samples of the same soil classification and source. Otherwise, a specific gravity test (Test Method C 127, Test Method D 854, or both) is necessary.

12. Report

12.1 The report shall contain the following information:
12.1.1 Procedure used (A, B, or C).
12.1.2 Preparation method used (moist or dry).
12.1.3 As received water content if determined.
12.1.4 Standard optimum water content, to the nearest 0.5 %.
12.1.5 Standard maximum dry unit weight, to the nearest 0.5 lbf/ft³.
12.1.6 Description of rammer (manual or mechanical).
12.1.7 Soil sieve data when applicable for determination of procedure (A, B, or C) used.
12.1.8 Description of material used in test, by Practice D 2488, or classification by Test Method D 2487.
12.1.9 Specific gravity and method of determination.
12.1.10 Origin of material used in test, for example, project, location, depth, and the like.
12.1.11 Compaction curve plot showing compaction points used to establish compaction curve, and 100 % saturation curve, point of maximum dry unit weight and optimum water content.
12.1.12 Oversize correction data if used, including the oversize fraction (coarse fraction), P_c in %.

13. Precision and Bias

13.1 *Precision*—Data are being evaluated to determine the precision of this test method. In addition, pertinent data is being solicited from users of the test method.

13.2 *Bias*—It is not possible to obtain information on bias because there is no other method of determining the values of standard maximum dry unit weight and optimum water content.

14. Keywords

14.1 NT—impact compaction using standard effort; RT—density; RT—moisture-density curves; RT—proctor test; UF—compaction characteristics; UF—soil compaction; USE—laboratory tests

ANNEX

(Mandatory Information)

A1. VOLUME OF COMPACTION MOLD

A1.1 Scope

A1.1.1 This annex describes the procedure for determining the volume of a compaction mold.

A1.1.2 The volume is determined by a water-filled method and checked by a linear-measurement method.

A1.2 Apparatus

A1.2.1 In addition to the apparatus listed in Section 6 the following items are required:

A1.2.1.1 *Vernier or Dial Caliper*—having a measuring range of at least 0 to 6 in. (0 to 150 mm) and readable to at least 0.001 in. (0.02 mm).

A1.2.1.2 *Inside Micrometer*—having a measuring range of at least 2 to 12 in. (50 to 300 mm) and readable to at least 0.001 in. (0.02 mm).

A1.2.1.3 *Plastic or Glass Plates*—Two plastic or glass plates approximately 8 in. square by ¼ in. thick (200 by 200 mm by 6 mm).

A1.2.1.4 *Thermometer*—0 to 50°C range, 0.5°C graduations, conforming to the requirements of Specification E 1.

A1.2.1.5 *Stopcock grease* or similar sealant.

A1.2.1.6 *Miscellaneous equipment*—Bulb syringe, towels, etc.

A1.3 Precautions

A1.3.1 Perform this procedure in an area isolated from drafts or extreme temperature fluctuations.

A1.4 Procedure

A1.4.1 *Water-Filling Method:*

A1.4.1.1 Lightly grease the bottom of the compaction mold and place it on one of the plastic or glass plates. Lightly grease the top of the mold. Be careful not to get grease on the inside of the mold. If it is necessary to use the base plate, as noted in 10.4.7, place the greased mold onto the base plate and secure with the locking studs.

A1.4.1.2 Determine the mass of the greased mold and both plastic or glass plates to the nearest 0.01-lbm (1-g) and record. When the base plate is being used in lieu of the bottom plastic or glass plate determine the mass of the mold, base plate and a single plastic or glass plate to be used on top of the mold to the nearest 0.01-lbm (1-g) and record.

A1.4.1.3 Place the mold and the bottom plastic or glass plate on a firm, level surface and fill the mold with water to slightly above its rim.

A1.4.1.4 Slide the second plate over the top surface of the mold so that the mold remains completely filled with water and air bubbles are not entrapped. Add or remove water as necessary with a bulb syringe.

A1.4.1.5 Completely dry any excess water from the outside of the mold and plates.

A1.4.1.6 Determine the mass of the mold, plates and water and record to the nearest 0.01-lbm (1-g).

A1.4.1.7 Determine the temperature of the water in the mold to the nearest 1°C and record. Determine and record the absolute density of water from Table A1.1.

A1.4.1.8 Calculate the mass of water in the mold by subtracting the mass determined in A1.4.1.2 from the mass determined in A1.4.1.6.

A1.4.1.9 Calculate the volume of water by dividing the mass of water by the density of water and record to the nearest 0.0001 ft^3 (1 cm^3).

A1.4.1.10 When the base plate is used for the calibration of the mold volume repeat A1.4.1.3 through A1.4.1.9.

A1.4.2 *Linear Measurement Method:*

A1.4.2.1 Using either the vernier caliper or the inside micrometer, measure the diameter of the mold 6 times at the top of the mold and 6 times at the bottom of the mold, spacing each of the six top and bottom measurements equally around the circumference of the mold. Record the values to the nearest 0.001-in. (0.02-mm).

A1.4.2.2 Using the vernier caliper, measure the inside height of the mold by making three measurements equally spaced around the circumference of the mold. Record values to the nearest 0.001-in. (0.02-mm).

A1.4.2.3 Calculate the average top diameter, average bottom diameter and average height.

A1.4.2.4 Calculate the volume of the mold and record to the nearest 0.0001 ft^3 (1 cm^3) as follows:

$$V = \frac{(\pi)(h)(d_t + d_b)^2}{(16)(1728)} \text{ (inch-pound)}$$

$$V = \frac{(\pi)(h)(d_t + d_b)^2}{(16)(10^3)} \text{ (SI)}$$

TABLE A1 Density of Water[A]

Temperature, °C (°F)	Density of Water, g/ml
18 (64.4)	0.99862
19 (66.2)	0.99843
20 (68.0)	0.99823
21 (69.8)	0.99802
22 (71.6)	0.99779
23 (73.4)	0.99756
24 (75.2)	0.99733
25 (77.0)	0.99707
26 (78.8)	0.99681

[A] Values other than shown may be obtained by referring to the Handbook of Chemistry and Physics, Chemical Rubber Publishing Co., Cleveland, Ohio.

where:
V = volume of mold, ft^3 (cm^3),
h = average height, in. (mm),
d_t = average top diameter, in. (mm),
d_b = average bottom diameter, in. (mm),
$1/1728$ = constant to convert in^3 to ft^3, and
$1/10^3$ = constant to convert mm^3 to cm^3.

A1.5 Comparison of Results

A1.5.1 The volume obtained by either method should be within the volume tolerance requirements of 6.1.1 and 6.1.2.

A1.5.2 The difference between the two methods should not exceed 0.5 % of the nominal volume of the mold.

A1.5.3 Repeat the determination of volume if these criteria are not met.

A1.5.4 Failure to obtain satisfactory agreement between the two methods, even after several trials, is an indication that the mold is badly deformed and should be replaced.

A1.5.5 Use the volume of the mold determined using the water-filling method as the assigned volume value for calculating the moist and dry density (see 11.4).

Designation: D 854 - 91

Standard Test Method for Specific Gravity of Soils[1]

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the specific gravity of soils that pass the 4.75-mm (No. 4) sieve, by means of a pycnometer. When the soil contains particles larger than the 4.75-mm sieve, Test Method C 127 shall be used for the material retained on the 4.75-mm sieve and this test method shall be used for the material passing the 4.75-mm sieve.

1.2 When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Test Method D 422, it is intended that the specific gravity test be made on that portion of the sample which passes the 2.00-mm (No. 10) sieve.

1.3 The values stated in acceptable metric units are to be regarded as standard.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods of Construction Materials[2]

D 422 Method for Particle-Size Analysis of Soils[3]

D 653 Terminology Relating to Soil, Rock, and Confining Fluids[3]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Soil and Rock Testing[3]

E 1 Specification for ASTM Thermometers[4]

E 11 Specifications for Wire-Cloth Sieves for Testing Purposes[5]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[6]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[5]

E 288 Specification for Volumetric Flaks[5]

3. Terminology

3.1 All definitions are in accordance with Terminology D 653 and E 12.

3.2 *Description of Terms Specific to This Standard:*

3.2.1 *specific gravity*—the ratio of the mass of a volume of solid soil particles at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature.

4. Significance and Use

4.1 The specific gravity of a soil is used in calculating the phase relationships of soils (that is, the relative volumes of solids to water and air in a given volume of soil).

4.2 The term solid particles, as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, and the like), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity less than one, typically require special treatment or a qualified definition of their specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL. Larger flasks are available and tend to produce better statistical values. If the stoppered bottle is used then the stopper shall be of the same material as the bottle, and shall have a small hole through its center to permit the emission of air and surplus water. The volume of the container filled to the mark shall be at least 50 % greater than the space required to accommodate the test specimen.

NOTE 1—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference but, in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

5.2 *Balance*—Class GP1 balances meeting the requirements of Specification D 4753 and this section are required. For use with the stoppered bottle, the balance must directly read to 0.001 g.

5.3 *Drying Oven*—Thermostatically-controlled oven, preferably of the forced-draft type, meeting the requirements of Specification E 145 and capable of maintaining a uniform temperature of 110 ± 5°C (230 ± 9°F) throughout the drying chamber.

5.4 *Thermometer*—A thermometer capable of measuring the temperature range within which the test is being performed, graduated in a 0.5°C (1.0°F) division scale and meeting the requirements of Specification E 1.

5.5 *Desiccator*—A desiccator cabinet or large desiccator

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] *Annual Book of ASTM Standards*, Vol 15.05.

jar of suitable size containing silica gel or anhydrous calcium sulfate.[7] It is preferable to use a desiccant which changes color to indicate when it needs reconstitution.

5.6 *Heating Apparatus*, hot plate or bunsen burner.

5.7 *Miscellaneous Equipment*, sample dishes, insulated gloves, rubber stoppers for vacuum system (optional), and the like.

6. Sampling

6.1 The soil to be used in the specific gravity test may contain its natural moisture or be oven-dried. The mass of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

6.2 *Samples Containing Natural Moisture*—When the sample contains its natural moisture, determine the mass of the sample on an oven-dry basis, M_o, at the end of the test by drying it to a constant mass in an oven maintained at 110 ± 5°C (230 ± 9°F) (See Note 2). Disperse samples of clay soils containing their natural water content in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422.

NOTE 2—Drying of certain soils at 110°C (230°F) may bring about loss of water of composition or hydration, and in such cases drying may be done in reduced air pressure or at a lower temperature.

NOTE 3—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500 mL flask is needed as the pycnometer.

6.3 *Oven-Dried Samples*—When an oven-dried sample is to be used, dry the sample to a constant mass in an oven maintained at 110 ± 5°C (230 ± 9°F) (see Note 2) and cool it in a desiccator. Determine and record the mass of the sample, M_o. For oven-dried specimens the soil shall be weighed after placement in the pycnometer after the true mass of the pycnometer is determined. The sample shall then be soaked for at least 12 h.

7. Calibration of Pycnometer

7.1 Determine and record the mass of a clean, dry pycnometer, M_f. Fill the pycnometer with distilled water at approximately room temperature, and determine and record the mass of the pycnometer and water, M_a. Insert a thermometer in the water, and determine and record its temperature, T_a, to the nearest 0.5°C (1.0°F).

NOTE 4—For some soils containing a significant fraction of organic matter, kerosene is a better wetting agent than water and may be used in place of distilled water for oven-dried samples. If kerosene is used, the entrapped air should only be removed by use of an aspirator. Kerosene is a flammable liquid and must be used with extreme caution.

7.2 From the mass, M_a, determined at the observed temperature, T_a, prepare a table of values of mass, M_a, for a series of temperatures that are likely to prevail when the mass of the pycnometer, soil, and water, M_b, is determined later. Calculate these values of M_a as follows:

$$M_a \text{ (at } T_x) = [(\text{density of water at } T_x/\text{density of water at } T_a) \times (M_a(\text{at } T_a) - M_f)] + M_f \quad (1)$$

where:

M_a = mass of pycnometer and water, g,
M_f = mass of pycnometer, g,
T_a = observed temperature of water, °C, and
T_x = any other desired temperature, °C.

NOTE 5—This test method provides a procedure that is more convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when masses M_a and M_b are taken, requires considerable time. It is important that masses M_a and M_b be based on water at the same temperature. Values for the density of water at temperatures from 18.0 to 30.0°C are given in Table 1.

8. Procedure

8.1 Determine the mass of pycnometer. Place the sample in the pycnometer and determine the mass of the pycnometer and soil. Add distilled water to fill the pycnometer to slightly above that required to cover the soil.

NOTE 6—Adding distilled water to just cover the soil makes it easier to control boil-over during removal of entrapped air.

8.2 Remove the entrapped air by either of the following methods:

8.2.1 Boil the specimen gently for at least 10 min while gently agitating the pycnometer occasionally to assist in the removal of air. Then cool the heated sample to room temperature.

8.2.2 Subject the contents to a vacuum (air pressure not exceeding 100 mm Hg) for at least 30 min (Note 7) either by connecting the pycnometer directly to an aspirator or vacuum pump or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be

TABLE 1 Density of Water and Correction Factor *K* for Various Temperatures

Temperature, °C	Density of Water (g/mL)	Correction Factor *K*
16.0	0.99897	1.0007
16.5	0.99889	1.0007
17.0	0.99880	1.0006
17.5	0.99871	1.0005
18.0	0.99862	1.0004
18.5	0.99853	1.0003
19.0	0.99843	1.0002
19.5	0.99833	1.0001
20.0	0.99823	1.0000
20.5	0.99812	0.9999
21.0	0.99802	0.9998
21.5	0.99791	0.9997
22.0	0.99780	0.9996
22.5	0.99768	0.9995
23.0	0.99757	0.9993
23.5	0.99745	0.9992
24.0	0.99732	0.9991
24.5	0.99720	0.9990
25.0	0.99707	0.9988
25.5	0.99694	0.9987
26.0	0.99681	0.9986
26.5	0.99668	0.9984
27.0	0.99654	0.9983
27.5	0.99640	0.9982
28.0	0.99626	0.9980
28.5	0.99612	0.9979
29.0	0.99597	0.9977
29.5	0.99582	0.9976
30.0	0.99567	0.9974

[7] Anhydrous calcium sulfate is sold under the trade name Drierite.

necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask.

NOTE 7—Samples with a high plasticity at the natural water content may require 6 to 8 h to remove entrapped air. Samples with a low plasticity at the natural water content may require 4 to 6 h to remove entrapped air. Oven-dried samples may require 2 to 4 h to remove entrapped air.

8.3 Fill the pycnometer with distilled water to near the calibration mark. Allow the pycnometer to obtain a uniform water temperature (Note 8). Fill the pycnometer with distilled water at the same temperature to the mark, clean the outside and dry with a clean, dry cloth. Determine the mass of the pycnometer and contents, M_b, and the temperature, T_b, of the contents as described in Section 7.

NOTE 8—To obtain a uniform water temperature the pycnometer may be allowed to sit overnight (12 to 16 h) or be placed in a constant temperature bath.

9. Calculation

9.1 Calculate the specific gravity of the soil, G, to the nearest 0.01, based on water at a temperature (T_b) as follows:

$$G \text{ at } T_b = M_o/[M_o + (M_a - M_b] \quad (2)$$

where:

M_o = mass of sample of oven-dry soil, g,
M_a = mass of pycnometer filled with water at temperature T_b (Note 9), g,
M_b = mass of pycnometer filled with water and soil at temperature T_x, g,
T_b = temperature of the contents of the pycnometer when mass M_b was determined, °C.

NOTE 9—This value can be obtained from the table of values of M_a, prepared in accordance with 7.2, for the temperatures prevailing when mass M_b was determined, °C.

NOTE 10—Equation 2 is for computing the specific gravity of the soil tested in water. When kerosene is used, the Eq must be adjusted by dividing the denominator by the specific gravity of kerosene at T_b and multiplying it by the density of water at T_b.

9.2 Calculate the weighted average specific gravity for soils containing particles both larger and smaller than the 4.75-mm sieve using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}} \quad (3)$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the 4.75-mm sieve,
R_1 = percent of soil particles retained on 4.75-mm sieve,
P_1 = percent of soil particles passing the 4.75-mm sieve,
G_1 = apparent specific gravity of soil particles retained on the 4.75-mm sieve as determined by Test Method C 127, and
G_2 = specific gravity of soil particles passing the 4.75-mm sieve as determined by this test method.

9.3 Unless otherwise required, report specific gravity (G) values based on water at 20°C. Calculate the value based on water at 20°C from the value based on water at the observed temperature T_b, as follows:

$$G \text{ at } 20°C = K \times (G \text{ at } T_b) \quad (4)$$

where:

K = a number found by dividing the density of water at temperature T_b by the density of water at 20°C. Values for the range of temperatures are given in Table 1.

NOTE 11—Different K values are required when kerosene or another fluid is used.

9.4 In some cases, it is desired to report the specific gravity value based on water at a different temperature. In these cases, the specific gravity value, based on any temperature T_x, may be calculated as follows:

$$G \text{ at } T_x = \frac{G \text{ at } 20°C}{K} \quad (5)$$

10. Report

10.1 The report shall include the following:

10.1.1 Identification of the sample (material) being tested, such as boring number, sample number, test number, etc.

10.1.2 Specific gravity at 20°C to the nearest 0.01.

10.1.3 Maximum particle size of the test specimen.

10.1.4 Specific gravity to the nearest 0.01 at a specified temperature other than 20°C, if applicable.

10.1.5 Type of fluid used.

10.1.6 When any portion of the original sample of soil is eliminated in the preparation of the test specimen, the portion on which the test has been made shall be reported.

11. Precision and Bias

11.1 *Precision*—Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the 4.75-mm sieve are given as follows:

Material and Type Index	Standard Deviation[A]	Acceptable Range of Two Results (percent of mean)[A]
Single-operator precision:		
Cohesive soils	0.021	0.06
Noncohesive soils	[B]	[B]
Multilaboratory precision:		
Cohesive soils	0.056	0.16
Noncohesive soils	[B]	[B]

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

[B] Criteria for assigning standard deviation values for non-cohesive soils are not available at the present time.

NOTE—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the two properly conducted tests.

11.2 *Bias*—There is no acceptable reference value for this test method; therefore, bias cannot be determined.

12. Keywords

12.1 soil; specific gravity

PROPERTIES OF SOIL

Note: Except when a symbol designates a specific unit, given dimensions are to designate their character and are not necessarily in the most convenient or common units. g indicates weight, cm indicates distance, sec indicates time. Special symbols using subscripts; such as w_L meaning water content at liquid limit, I_p plastic index, etc.; will be defined when first used in the text.

1.08 DEFINITIONS

In general, definitions will be presented as required. However, in order to establish basic relationships, it is necessary to become familiar with a few definitions in the beginning. Some of these common definitions are:

Void Ratio e = ratio of volume of voids to volume of solids in a given mass of soil. V_v/V_s.

Porosity n = ratio of volume of voids to the total volume of a given mass of soil. V_v/V. (Sometimes expressed as percentage).

Water Content w = ratio of the weight of the water to the weight of the solids in a given mass of soil. W_w/W_s. (Usually expressed as percentage).

Degree of Saturation S = ratio of the volume of water in a given mass of soil to the volume of voids. V_w/V_v. (Usually expressed as percentage).

Specific Gravity G = ratio of unit weight in air of a material to the unit weight in air of a reference material at a stated temperature. The most common reference material for determining specific gravity is distilled water at 4 degrees centigrade, γ_0. Hydrometers are commonly calibrated to read specific gravity referred to distilled water at the calibration temperature.

1.09 RELATIONSHIPS

In order to be able to determine the physical properties of soils from laboratory tests, it is necessary to know, or to be able to work out, the relationships which exist between the different properties. In working out these relationships, it is convenient to represent the solids and voids in the given volume by means of a sketch. If the area be

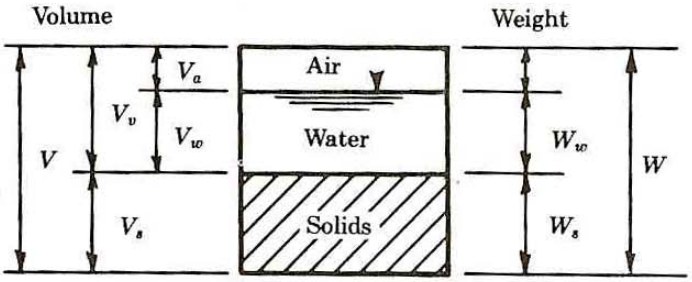

Figure 1.09a

assumed as unity, or other constant, the height will represent the volume. For convenience, the volumes may be shown on the left hand side of the sketch and the weights on the right hand side as shown in Figure 1.09*a*

$$e = \frac{V_v}{V_s} \qquad n = \frac{V_v}{V}$$

$$w = \frac{W_w}{W_s} \qquad S = \frac{W_w}{V_v \gamma_w}$$

$$W_s = V_s \cdot G_s \cdot \gamma_0$$

If the relationship between water content, void ratio, and specific gravity of solids is desired, the volume of the solids may be represented as 1, in which case the volume of voids is equal to the void ratio, e. If the voids are completely filled with water, the weight of the water is equal to the volume of water, e, times the unit weight of water, γ_w; and the weight of the solids is equal to the specific gravity of the solids, G_s, times the unit weight of water at reference temperature, γ_0, times the volume of solids, 1.

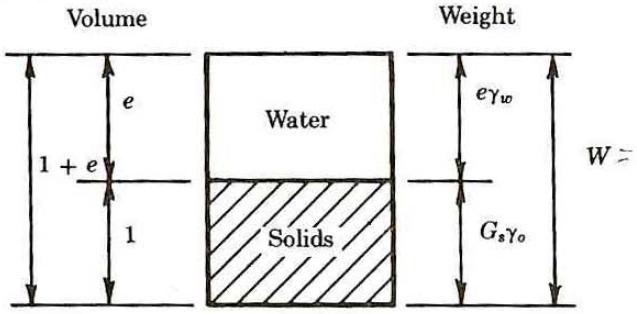

Figure 1.09b

These relationships are illustrated in Figure 1.09*b*.

$$w = \frac{e\gamma_w}{G_s\gamma_0} = \frac{e}{G_s} \text{ or } e = wG_s$$

γ_0 = unit weight of water at reference temperature, 4° C.
γ_w = unit weight of water at given temperature.
For most purposes the ratio γ_w/γ_0 may be considered as 1.

In relationships involving the use of porosity, n, a sketch may be used in which the total volume equals 1, in which case, by definition, the volume of voids is equal to the porosity, n, and the volume of solids is $1 - n$. From these sketches, Figure 1.09*b* and Figure 1.09*c*,

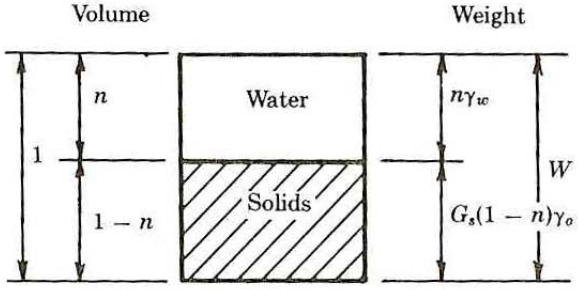

Figure 1.09c

one can readily arrive at the relationships between void ratio and porosity because the total volumes are proportional to the volumes of the voids and to the volumes of the solids in different volumes of the same soil, or they can be determined directly from the definitions.

$$\frac{1}{1+e} = \frac{n}{e} = \frac{1-n}{1}$$

$$n = \frac{e}{1+e} \qquad e = \frac{n}{1-n}$$

$$w = \frac{n\gamma_w}{G_s(1-n)\gamma_0} = \frac{n}{G_s(1-n)}$$

The unit weight of a given soil, dry, saturated, or submerged, can be determined from the void ratio and the specific gravity of the solids in the following manner:

As seen from Figure 1.09*d*, the dry weight of a volume of soil, $1 + e$, is equal to the weight of the solids, $G_s \cdot \gamma_0$. The unit weight of the dry soil is then equal to $\frac{G_s\gamma_0}{1+e}$. If the soil is completely saturated, the

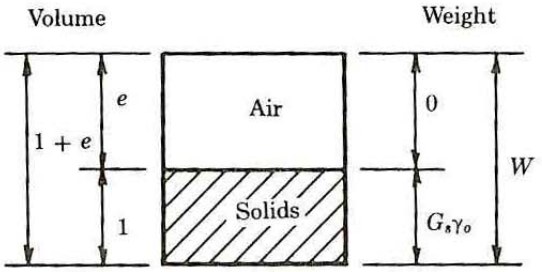

Figure 1.09d

total weight is $e\gamma_w + G_s\gamma_0$, and the unit weight is $\dfrac{e\gamma_w + G_s\gamma_0}{1 + e}$. If the soil is submerged, the unit weight of the submerged soil is equal to the unit weight of the saturated soil reduced by the unit weight of water; i.e., $\gamma_{sub} = \gamma_{sat} - \gamma_w$.

Similar relationships can be determined for partially saturated soils by imagining the solids, water, and gas separated as shown in Figure 1.09*e*. Water content can be determined by weighing a sample of the

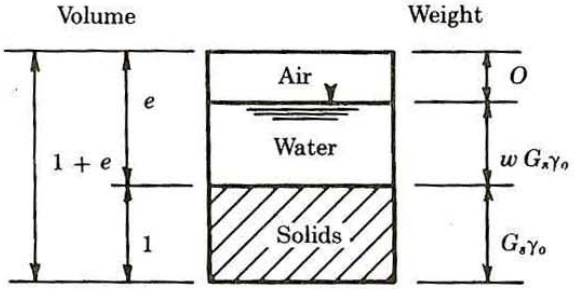

Figure 1.09e

soil with its entrapped water, then drying the soil in a drying oven until all the free water has been driven off, and then weighing the dry soil. The water content is equal to the weight of the water (difference in wet and dry weights) divided by the weight of the solids (dry weight). If the value of e is known, the degree of saturation S can be determined from the water content as shown in Figure 1.09*e*.

$$\text{Wt of water} = wG_s\gamma_0$$

If the voids were completely filled with water, the weight of the water would be $e\gamma_w$. From definition,

$$S = \frac{wG_s\gamma_0}{e\gamma_w} = \frac{wG_s}{e}.$$

It is sometimes desirable to express the ratio of the volume occupied by gas to the total volume of the soil, which is similar in definition to porosity, n. This ratio is usually referred to as air porosity and is designated n_a.

$$n_a = \frac{V_a}{V} = \frac{V_v - V_w}{V}$$

$$n_a = \frac{n - wG_s(1 - n)}{1}$$

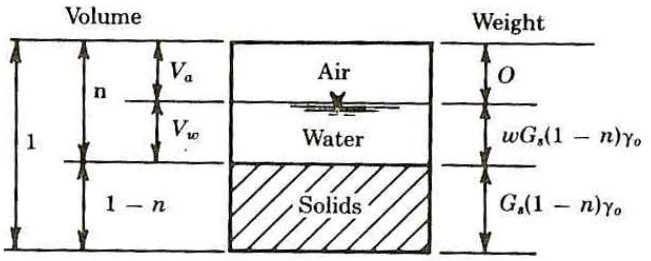

Figure 1.09f

Needed relationships should be worked out directly from basic definitions without the use of previously developed general formulas. The foregoing examples are illustrations of the use of phase diagrams as aids to an understanding and statement of specific relationships.

The following example illustrates the use of the phase diagram and basic definitions to the solution of a specific problem.

Example: In a compaction test the following data were obtained:

Volume of compaction cylinder	970 cm³
Weight of compacted soil	1986 g
Water content w	8.6%
Specific gravity of solids	2.71

a. Determine the dry weight of the soil per unit of volume.
b. Determine the porosity and the void ratio of the soil as compacted.
c. Determine the degree of saturation.

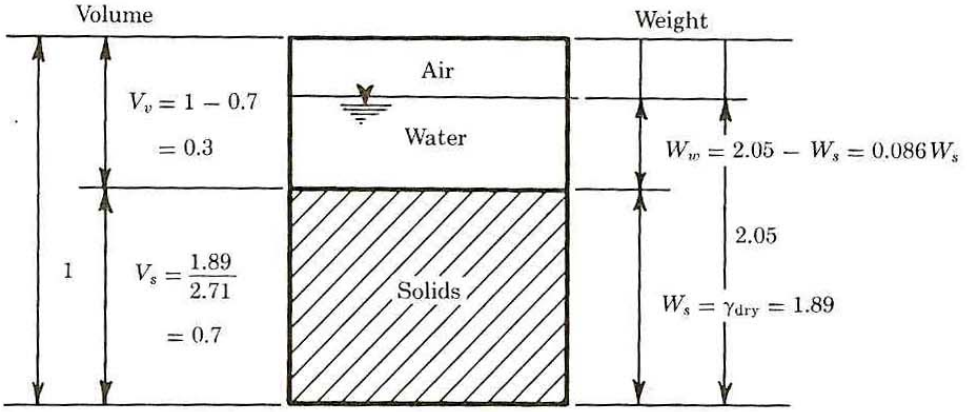

Figure 1.09g

Solution:

a. Unit weight, wet, $\gamma_{wet} = \frac{1986}{970} = 2.05$ g cm^{-3}

By definition, water content, $w = \frac{W_w}{W_s} = 0.086$

Therefore, $W_w = 0.086 W_s$

W_w is also equal to $W - W_s = 2.05 - W_s$

Then, $2.05 - W_s = 0.086 W_s$

From which $W_s = 1.89$ g and since the total volume is unity,

$W_s = \gamma_{dry} = 1.89$ g cm^{-3} $= 1.89 \times 62.4 = 118$ lb ft^{-3}.

A simple method of determining the unit weight of any substance in any other units than the known unit weight is by proportion with the known unit weights in the two units of a reference material. Water weighs 1 g cm^{-3} and 62.4 lb ft^{-3}, therefore n g cm^{-3} $= 62.4n$ lb ft^{-3}.

b. The porosity and void ratio can be determined directly from the diagram.

Volume of solids $V_s = \frac{W_s}{G_s \gamma_0} = \frac{1.89}{2.71} = 0.70$ cm^3.

Volume of voids $V_v = 1 - 0.70 = 0.30$ cm^3.

Porosity $n = \frac{0.3}{1} = 0.3$.

Void ratio $e = \frac{0.3}{0.7} = 0.43$.

c. Volume of water $V_w = \frac{2.05 - 1.89}{1} = 0.16$ cm^3.

Degree of saturation $S = \frac{V_w}{V_v} = \frac{0.16}{0.30} = 0.53$ or 53%.

Similar relationships can be readily worked out with the help of appropriate sketches.

1.10 CLASSIFICATION OF SOILS

In general, soils are classified as cohesionless and cohesive. By cohesion is meant shearing resistance inherent in the material which does not have to be developed by normal pressure or other outside influence.

Cohesionless soils possess no shearing resistance, except as developed by normal pressure between the grains. Sand, a cohesionless soil, has no shearing resistance on the surface, but at some depth beneath the surface where it is subjected to the pressure of the overburden, the

APPENDIX D

EXAMPLE CHAIN-OF-CUSTODY

URS

CHAIN OF CUSTODY/LABORATORY ANALYTICAL REQUEST FORM

URS Corporation ● 1600 Perimeter Park Drive, Morrisville, NC 27560 ● 919-461-1100 ● Fax 919-461-1415

PAGE ____ OF ____

Work Order #

Project Name and Location PSC PDI, Rock Hill, SC	Project Number 31827295.00008
Project Manager Brett Berra, URS Corporation	Report To: martha.meyers-lee@urs.com
Company/Address URS Corporation 1600 Perimeter Park Drive, Suite 400 Morrisville, NC 27560	
Phone # (919) 461-1100	FAX # (919) 461-1415
Sampler's Signature	Sampler's Printed Name

ANALYSIS REQUESTED ***(Include Method Number and Container Preservative)***

Total Number of Containers

: PRESERVATIVE

Preservative Key
0. NONE
1. HCl
2. HNO_3
3. H_2SO_4
4. NaOH
5. Zn. Acetate
6. MeOH
7. $NaHSO_4$
8. Other 4°C
9. Other _____

REMARKS

: LAB Name

: Bottle Codes

SAMPLE ID	Sample Type (Grab / Composite)	SAMPLING DATE	SAMPLING TIME	MATRIX

SPECIAL INSTRUCTIONS/COMMENTS

URS Contact: Martha Meyers-Lee, (919) 461-1519

See SOW ☐

See QAPP ☐

Matrix Key:
W = Water
S = Soil/Sediment
B = Biota
O = Other _____

Container Key:
P = Plastic
G = Glass
C = Clear
A = Amber
V = Vial
Z = Ziploc bag
M = Multiple types

TURNAROUND REQUIREMENTS
_____ RUSH (surcharges apply)
_____24 hr _____48 hr _____ 5 day
_____STANDARD
REQUESTED FAX DATE
_____
REQUESTED REPORT DATE
_____

REPORT REQUIREMENTS
_____ I. Results Only
_____ II. Results + QC Summaries (LCS, DUP, MS/MSD as required)
_____ III. Results + QC and Calibration Summaries
_____ IV. Data Validation Report with Raw Data
_____ Specialized Forms/Custom Report
Edata _____ Yes _____No

INVOICE INFORMATION
PO#
BILL TO:
SUBMISSION #:

RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY
Signature	Signature	Signature	Signature	Signature	Signature
Printed Name	Printed Name	Printed Name	Printed Name	Printed Name	Printed Name
Firm	Firm	Firm	Firm	Firm	Firm
Date/Time	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time

SAMPLE RECEIPT | Custody Seals In Place: Yes / No | Temp Blank: Yes / No | Cooler Temp on Receipt: _____°F/C | Samples Intact: Yes / No | Samples properly preserved: Yes / No

Cooler _____ of _____

AQUEOUS BOTTLE CODES						
Code	**Analyses**	**Bottle Size**	**Type**	**Chemical Preservatives**	**Storage Requirements**	**Number of Containers**
W-1	Total Metals	250 ml	HDPE bottle	HNO_3	None	1
W-2	VOCs	40 ml	Glass Vial	HCl	$\leq$6°C	3
W-3	Nitrate and Sulfate$^-$	250 ml	HDPE bottle	none	$\leq$6°C	1
W-4	TSS	1 - liter	HDPE bottle	none	$\leq$6°C	1
SOLID BOTTLE CODES						
Code	**Analyses**	**Bottle Size**	**Type**	**Chemical Preservatives**	**Storage Requirements**	**Number of Containers**
S-1	VOCs – Medium/High Concentrations and Percent Solids	2 oz	Wide-mouth glass jar with Teflon-lined screw cap	none	$\leq$6°C	1
S-2	VOCs – Low Concentration	40 ml	Glass VOA vial containing 5 ml of organic free water	none	Cooled to $\leq$6 °C and frozen within 48 hours of collection	1
S-3	Fractional Organic Carbon	125 ml	Wide-mouth glass jar with Teflon-lined screw cap	none	$\leq$6°C	1
S-4	Grain-size Distribution, Bulk Density, Wet Unit Weight, Specific Gravity, Air-filled Porosity, Percent Saturation	1 Liner	Capped, acetate liner placed on two layers of bubble wrap in cooler	none	Ship on dry ice	1

APPENDIX E

EXAMPLE DATA VALIDATION CHECKLIST

Data Validation Checklist
Organic and Inorganic Analyses

Project: _____ Project No: _____
Work Order: _____ Method: _____
Laboratory: _____ Associated Sample IDs: _____
Matrix: _____ Sample Date: _____
Reviewer: _____ Date: _____
Concurrence[1]: _____ Date: _____

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
1. Were holding times met?					
2. Were sample storage and preservation requirements met?					
3. Were measurement results for all project-specified target analytes reported?					
4. Were project-specified Reporting Limits Objectives achieved?					
5. Do sample prep dates occur before analytical dates?					
6. Was a method blank analyzed with each batch?					
7. Were target analytes reported in the method blanks?					
8. Were target analytes reported in the method and calibration blanks above the Detection Limit (DL)?					
9. Were target analytes reported in field blank analyses (e.g., trip, field, or equipment)?					
10. Were analytes detected in samples at concentrations similar to that observed in the blanks?					
11. Was a field duplicate analyzed?					
12. Was field duplicate results meet project specifications?					
13. Was a LCS prepared/analyzed with each batch?					
14. Were LCS' recoveries within lab/project specifications?					
15. Were LCS/LCSD RPD within lab /project specifications?					
16. Was a MS/MSD pair analyzed with each batch?					
17. Is the MS/MSD parent sample a project-specific sample?					
18. Were MS/MSD recoveries within lab and project specifications? *Only QC results for project samples are evaluated.*					

[1] Independent Technical Reviewer

WO # _____ **Data Validation Checklist (Continued)**

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
19. Were MS/MSD RPD within lab and project specifications? *Only QC results for project samples are evaluated.*					
20. Was a post digestion spike analyzed for inorganics?					
21. Was a project-specific sample selected by the laboratory for the post digestion spike analysis?					
22. Were post-digestion spike recoveries within lab/project specifications? *Only QC results for project samples are evaluated.*					
23. Was a serial dilution analysis for inorganics?					
24. Is the serial dilution parent sample a project-specific sample?					
25. Is the percent difference between the serially diluted result and undiluted result less 10% (for those analytes with native concentrations greater than 50x the DL)?					
26. Was a laboratory duplicate analyzed?					
27. Is the laboratory duplicate sample a project-specific sample?					
28. Does laboratory duplicate results meet lab specifications? *Only QC results for project samples are evaluated.*					
29. For organics, were initial and continuing calibration standards analyzed at the lab/project-specified frequency for each instrument?					
30. Were tuning and a calibration results within method and project specifications?					
31. Was an interference check sample (ICS) analyzed at the beginning and end of the ICP analytical run?					
32. Did ICS results meet lab/project specifications?					
33. Was a CRQL check standard (CRI) analyzed with each inorganic analytical batch?					
34. Did CRI results meet lab/project specifications?					
35. Were surrogate recoveries within lab specifications?					
36. Were internal standard results within lab specifications?			✓	Not evaluated; data not included in laboratory report.	
37. Were sample results, which were determined by GC/HPLC, confirmed using a second column of dissimilar stationary phase or detector, and the %D between primary and secondary results less than 40 for all detected analytes?					
38. Were TIC reported and were reported results qualified as estimated concentrations?					

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
39. Is the moisture content in any soil/sediment sample greater than 70%? If yes, then results should be reported on a wet-weight basis[2]?			✓		
40. Were laboratory-generated Corrective Action Reports issued? If yes, summarize contents or attach copy of the report.					
41. Were lab comments included in report? If yes, summarize contents or attach a copy of the narrative.					

Comments:

The data validation was conducted in accordance with the PDI QAPP (URS, May 2014). The data validation process was modeled after the *USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG) for Superfund Organic Methods Data Review* (EPA, June 2008) and *USEPA CLP NFG Inorganic Superfund Data Review* (USEPA, January 2010). Sample results have been qualified based on the results of the data review process (refer to **Attachment A**). In performing the data validation, the URS' data reviewer assumed that the data reported by the laboratory are complete, compliant, and an accurate representation of the raw data. Criteria for acceptability of data were based upon available site information, analytical method requirements, guidance documents, and professional judgment.

Flag Definitions:

J Estimated value
J+ Estimated high
J- Estimated low
UJ Not detected and the detection limit is estimated
U Not present above the associated level; blank contamination exists
R Unusable data

[2] Based on the USEPA Office of Water Regulations and Standard Industrial Technology Division definition that soil samples are "soils, sediments, and sludge samples that containing more than 30% solids" (reference USEPA Region I DV Guidance)