Geotechnical
Environmental
Water Resources
Ecological

Quality Assurance Project Plan (QAPP)

Gowanus Canal Superfund Site

Brooklyn, New York

USEPA ID# NYN000206222

Prepared By:
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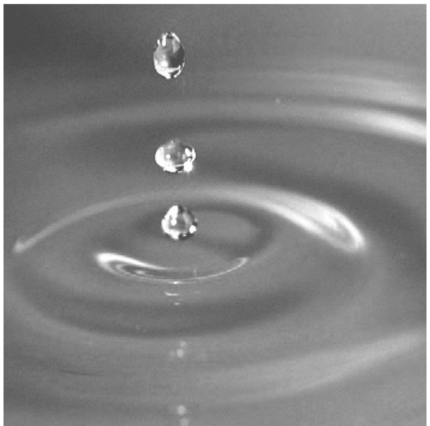

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INTRODUCTION

This Quality Assurance Project Plan (QAPP) is intended to integrate the technical and quality control aspects of a geotechnical investigation and in-situ solidification treatability study at properties where National Grid is responsible for remedial activities located along the Gowanus Canal Superfund Site. This QAPP is supplemented by detailed information in the Geotechnical Investigation and In-Situ Solidification Treatability Study Work Plan.

This QAPP details the planning processes for collecting data and describes the implementation of the quality assurance (QA) and quality control (QC) activities developed for this program. The purpose of this QAPP is to generate project data that are technically valid and legally defensible. The QAPP consists of four main components:

- Project Management
- Measurement and Data Acquisition
- Assessment and Oversight
- Data Validation and Usability

The above components will incorporate QA/QC requirements cited within the following United States Environmental Protection Agency (USEPA) documents:

- USEPA *Requirements for Quality Assurance Project Plans*, USEPA QA/R-5, February 2006
- USEPA *Guidance for the Data Quality Objectives Process*, QA/G-4, August 2000
- USEPA, US Department of Defense, and US Department of Energy *Uniform Federal Policy (UFP) for Quality Assurance Project Plans*, Final Version March 2005

It is United States Environmental Protection Agency (USEPA) Region 2 policy that all QAPPs be prepared in accordance with the Uniform Federal Policy (UFP) requirements, which are contained in the UFP QAPP Manual and its assorted Compendium and Worksheets. This document is arranged in accordance to USEPA Worksheets.

Project Background

The project location is shown in Figure 1. The Gowanus Canal (referenced as the Canal) is a 100-foot wide, 1.8-mile long brackish canal located in the New York City borough of Brooklyn, Kings County, New York. Connected to Gowanus Bay in Upper New York Bay, the Canal borders several residential neighborhoods including Gowanus, Park Slope, Cobble Hill, Carroll Gardens, and Red Hook.

The Gowanus Canal study area is situated in a dense urban area of mixed commercial, industrial, municipal, and residential land use. The historic land uses adjacent to the canal include oil and petroleum storage, asphalt and coal tar companies, manufacturing, chemical/fertilizer/plastics manufacturing and coal yards. The Gowanus Canal has also served for the conveyance of sewage and industrial wastes as part of the development and industrialization of the area. The canal also serves as the discharge point for CSO discharges, permitted industrial discharges and un-permitted discharges. These discharges have been degrading the quality of the Gowanus Canal.

The USEPA Region 2 is has recently completed a Feasibility Study (FS) Investigation of the Gowanus Canal Superfund Site prepared for the USEPA by CH2MHILL. Seven remedial alternatives were presented in the Feasibility Study. Alternative No. 7 was retained and is the focus of this QAPP.

Alternative 7 involves dredging the entire soft sediment column, solidifying the top 3 to 5 feet of the native sediment in targeted areas, and capping with a treatment layer, sand-and-gravel isolation layer, and armor layer.

National Grid has prepared two work plans, cited above, to address implementation of Alternative 7:

Geotechnical Investigation

Sediments below the proposed cap may be very soft and could impact the stability of the cap. The purpose of this work plan is to obtain geotechnical parameters for geotechnical strength parameters of materials beneath the cap for stability evaluation.

In-Situ Solidification Treatability Study

In situ solidification (ISS) has been chosen as a potential technology to address NAPL in the Gowanus Canal. An ISS treatability study is part of the planned pre-design activities necessary for implementing the ISS portion of the remedial action. The treatability study is being performed to identify appropriate additives to achieve the necessary permeability and compressive strength.

QAPP Worksheet #1 – Title and Approval Page

Site Name/Project Name: Gowanus Canal Superfund Site

Site Location: Gowanus Canal, Brooklyn, New York

Document Title: *QAPP for Gowanus Canal Superfund Site, Geotechnical Investigation, and In-Situ Solidification Treatability Study*

Lead Organization: National Grid

Preparer's Name and Organization: Brian Conte, MSPH, CHMM GEI Consultants, Inc.

Preparer's Address, Telephone Number and E-mail Address:

455 Winding Brook Drive, Suite 201, Glastonbury, CT 06033, bconte@geiconsultants.com

Preparation Date: February 17, 2012

Organization	Name	Signature
Investigative Organization's Project Manager	David Terry, PG LEP	
Investigative Organizations Project QC Officer	Jaimie Wargo	
Lead Organizations Project Manager	Tracey Bell	

Approval Signatures:____

Printed Name/Title:____

Approval Authority:____

QAPP Worksheet #2 – QAPP Identifying Information

Site Number/Code: EPA ID No. CERCLA -02-2010-2011

Operable Unit: Not Applicable

Contractor Name: GEI Consultants, Inc.

Contractor Number: Not Applicable

Contract Title: Not Applicable

Work Assignment Number: Not Applicable

1. **Identify guidance used to prepare QAPP:**
   Uniform Federal Policy for Quality Assurance Project Plans, March 2005

2. **Identify regulatory program:**
   Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)

3. **Identify approval entity:** USEPA Region 2

4. This QAPP is project specific.

5. Scoping Sessions occurred between October 2011 – February 2012.

6. **List dates and titles of QAPP documents written for previous site work, if applicable:**

Title	Received Date
Gowanus Canal Superfund Site, Surface Water and Sediment Sampling	Revision 1, December 2010
Gowanus Canal Superfund Site, Surface Water and Sediment Sampling	Revision 2, August 2011

7. **List organizational partners (stakeholders) and connection with lead organization:** The primary project organizational partners include representatives from National Grid and GEI Consultants, Inc. National Grid will provide project and contract management guidance.

8. **List data users:** USEPA Region 2, National Grid, and GEI Consultants, Inc.

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Project Management and Objectives		
2.1 Title and Approval Page	1	- Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	2	- Table of Contents - QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	3 4	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table

QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
2.5 Project Planning/Problem Definition		- Project Planning Session Documentation (including Data Needs tables)
2.5.1 Project Planning (Scoping)	9	- Project Scoping Session Participants Sheet
2.5.2 Problem Definition, Site History, and Background		- Problem Definition, Site History, and Background
	10	- Site Maps (historical and present)
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process	11	- Site-Specific PQOs
2.6.2 Measurement Performance Criteria	12	- Measurement Performance Criteria Table
2.7 Secondary Data Evaluation		- Sources of Secondary Data and Information
	13	- Secondary Data Criteria and Limitations Table
2.8 Project Overview and Schedule	14	- Summary of Project Tasks
2.8.1 Project Overview	15	- Reference Limits and Evaluation Table
2.8.2 Project Schedule		- Project Schedule/ Timeline Table
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QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Measurement/Data Acquisition		
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3.1.1 Sampling Process Design and Rationale		- Sample Location Map
3.1.2 Sampling Procedures and Requirements	18	- Sampling Locations and Methods/SOP Requirements Table
3.1.2.1 Sampling Collection Procedures		- Analytical Methods/SOP Requirements Table
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3.1.2.6 Field Documentation Procedures		
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QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
3.2 Analytical Tasks		- Analytical SOPs
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3.2.2 Analytical Instrument Calibration Procedures	24	- Analytical Instrument Calibration Table
3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures		- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
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3.3.2 Sample Handling and Tracking System		- Sample Handling Flow Diagram
3.3.3 Sample Custody		- Example Chain-of-Custody Form and Seal
3.4 Quality Control Samples	27	- QC Samples Table
3.4.1 Sampling Quality Control Samples		- Screening/Confirmatory Analysis Decision Tree
3.4.2 Analytical Quality Control Samples		

QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
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3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	30 32 31	
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	30 31	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table
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QAPP Worksheet #2 – QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
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QAPP Worksheet #3 – Distribution List

List those entities to which copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be distributed.

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Christos Tsiamis	USEPA Project Coordinator	USEPA NY Remediation Branch	212.637.4257		Tsiamis.christos@epa.gov	
Tracey Bell	Lead Organizations Project Manager	National Grid	718.963.5645		Tracey.Bell@us.ngrid.com	
David Terry	Investigative Organizations Project Manager	GEI Consultants	860.368.5300	860.368.5307	dterry@geiconsultants.com	
Tim Olean	Treatability Study Project Quality Control Officer	GEI Consultants	401.533.5151	860.368.5307	tolean@geiconsultant.com	
Sean DiBartolo	Geotechnical Investigation Project Quality Control Officer	GEI Consultants	973.873.7115	973.509.9625	sdibartolo@geiconsultant.com	
To Be Announced	Field Team Leader	GEI Consultants	860.368.5300	860.368.5307	kweber@geiconsultants.com	

Electronic copies of the final QAPP and related project documents will also be available in the project directory and the project database for personnel named in the organization chart provided as Figure 1 and other personnel who will be assigned to work on the project. Those names will be responsible for distributing the QAPP and related documents to others in their organization.

QAPP Worksheet #4 – Project Personnel Sign-Off Sheet

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: GEI Consultants, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
David Terry	Investigative Organizations Project Manager	860.368.5300		
Tim Olean	Treatability Study Project Quality Control Officer	401.533.5151		
Sean DiBartolo	Geotechnical Investigation Project Quality Control Officer	973.873.7115		
TBD	Field Team Leader/Project Safety Officer	860.368.5300		

APP Worksheet #4 – Project Personnel Sign-Off Sheet (cont.)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: United States Environmental Protection Agency

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt

APP Worksheet #4 – Project Personnel Sign-Off Sheet (cont.)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: National Grid

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt

QAPP Worksheet #5 – Project Organizational Chart

Project Organization

The Organization Chart, provided as Figure 1, the description of project organization, and the roles of the team members are summarized below:

Project/Task Organization Overview

The project management team will consist of representatives from National Grid, USEPA Region 2, and GEI Consultants, Inc. GEI will provide technical oversight to the project during the planning and investigation, serve as the primary contractor, bear responsibility for developing and implementing the investigation, and provide project management for the other subcontractors. Figure 2 presents a diagram of the project organization.

Investigation Team Members

This section contains a description of the project organizational structure. The National Grid Project Manager will have contract management with responsibility for the Gowanus Canal Investigation. GEI, as the primary contractor, will be responsible for developing and implementing the investigation, and conduct project management for other subcontractors. Additional project team members from other companies may serve as subcontractors to GEI.

<u>Project Manager</u> (PM) – The PM is accountable to the PO throughout the duration of the project. The PM will be the primary point of contact with National Grid. The PM may delegate authority to expedite and facilitate the implementation of the project plan.

The Technical/Administrative PM, is responsible for:

- Coordination with National Grid
- Budget control
- Subcontractor performance
- Project coordination to implement Work Plans
- Allocation of staffing and resources to implement the QA/QC program and the Health and Safety Plan (HASP)
- Review of investigation, engineering, and interim reports

[Note: David Terry is GEI's Administrative and Technical PM responsible for all administrative and technical aspects of the project.]

<u>Corporate Health and Safety Manager</u> (CHSM) – The Corporate Health and Safety Manager is responsible for development and implementation of GEI's Health and Safety program. The CHSM serves as the administrator of GEI's Corporate Health and Safety program. The CHSM bears responsibility for:

- Proper training for GEI field personnel
- Medical clearance of GEI field personnel

QAPP Worksheet #5 – Project Organizational Chart (cont.)

- Field personnel having adequate experience with personal protective equipment
- Providing guidance on data interpretation
- Determining levels of worker protection
- Directing and assisting the Project Safety Officer (PSO)

[Note: Robin DeHate is GEI's Corporate Health and Safety Manager, and will serve on this project.]

Project Quality Control Officer – The Project QC Officer is responsible for project-specific supervision and monitoring of the QA program and reports to the Project Manager. Additional responsibilities include:

- Ensuring that field personnel are familiar with and adhere to proper sample identification, and chain-of-custody procedures
- Coordinating with the analytical laboratory for the receipt of samples, the reporting of analytical results, and recommending corrective actions to correct deficiencies in the analytical protocol or sampling

  [Note: Timothy OLean will serve as Project Quality Control Officer for the In-Situ Solidification Treatability Study and Sean DiBartolo with serve as Project Quality Control Officer for the Geotechnical Investigation.]

Field Team Leader – The Field Team Leader will serve as the contact person GEI for field investigations and activities. The Field Team Leader will be responsible for the logistics of the field activities. The Field Team Leader will:

- Ensure that proper sampling procedures and field measurement techniques are performed
- Inspect and replace equipment
- Prepare daily activity reports
- Prepare samples (in coordination with the Sample Management Officer) for shipment
- Coordinate field activities
- Schedule sampling and other field activities

[Note: The Field Team Leader is to be determined.]

QAPP Worksheet #6 – QAPP Communication Pathways

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Approval of Amendments to the QAPP	GEI Consultants, Inc.	Project Quality Control Officer (Jaimie Wargo) and Lead Organization's Project Manager (Tracey Bell)	860.368.5300	Obtain initial approval from the Investigative Organization PM and submit documented amendments within 10 working days of initial approval
Document and Records Control	GEI Consultants, Inc.	Investigative Organization Project Manager (David Terry)	860.368.5300	Project Document Preparation and distribution. Document and records control posting procedure implemented within 5 working days of receipt by GEI Consultants, Inc.
Stop Work and Initiation of Corrective action	GEI Consultants, Inc.	Investigative Organization Project Manager (David Terry)	860.368.5300	The PM communicates within 24 hours of stop work to the project organization by phone, with confirming e-mail.
Real time modification, notifications and approval	GEI Consultants, Inc.	Field Team Leader (Kari Weber)	860.368.5300	Real time modification to the project will require the approval of the Project Quality Officer and PM (or designee) and will be documented using the Field Modifications Form in Attachment 5 within 5 working days.
Reporting of serious issues	GEI Consultants, Inc.	Project Managers	860.368.5300	Report any serious issues to National Grid and other concerned parties by e-mail or memorandum.
Meeting Minutes	GEI Consultants, Inc.	Investigative Organization Project Manager (David Terry)	860.368.5300	Post approved meeting minutes or distribute by email within 5 working days of meeting.
Corrective action, audit finding	GEI Consultants, Inc.	Project Quality Control Officer (Jaimie Wargo)	860.368.5300	Problems or negative audit findings are reported to the PM by e-mail within 3 days.

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Years of Professional Experience	Education and Experience Qualifications
Tracey Bell	Project Manager	National Grid	Lead Organization's Project Manager	20 +	
David Terry	Project Manager	GEI Consultants, Inc.	Investigative Organization's Project Manager	20	PG, MS Geology
Kari Weber	Project Engineer	GEI Consultants, Inc.	Field Team Leader	5	BS in Civil Engineering
Jaimie Wargo	Senior Data Technician	GEI Consultants, Inc.	Project Quality Officer	5	

QAPP Worksheet #8 – Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/ Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Team	Safety and OSHA training and medical monitoring as specified in the HASP	GEI Consultants, Inc.	Training dates kept in company/ project training records	All field team members working on Project.	All GEI Consultant and subcontractor personnel working on the Project.	GEI Consultant’s Project Files; available upon request

QAPP Worksheet #9 – Project Scoping Session Participants Sheet

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Project team meetings and conference calls have taken place to plan the project. During this period GEI Consultants Investigative team and National Grid developed project plans and work scope. Project participants have discussed the project objectives and status. Project team members have visited the Site to evaluate field conditions and gather information needed to develop investigation plans.

Project Team Participants

Project Name: Geotechnical Investigation and In-Situ Solidification Treatability Study			**Site Name:** Gowanus Canal Superfund Site
Projected Date(s) of Sampling: March 2012			**Site Location:** Brooklyn, New York
Project Managers:	T. Bell, National Grid	D. Terry, GEI Consultants	

Date of Session: December 2011 - February 2012
Scoping Session Purpose: Review the status of project plans

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Tracey Bell	Project Manager	National Grid	718-963-5645	Tracey.Bell@us.ngrid.com	Lead Organization Project Manager
Christos Tsiamis	Project Manager	USEPA Region 2	212-637-4257	tsiamis.christos@epa.gov	USEPA Project Manager
David Terry	Project Manager	GEI Consultants	860-368-5412	dterry@geiconsultants.com	Administrative Project Manager
Kari Weber	Project Engineer	GEI Consultants	860-368-5300	kweber@geiconsultultants.co	Quality Control Officer

Comments/Decisions: Documented in meeting minutes stored in GEI Consultants files.

QAPP Worksheet #10 – Problem Definition

The problem to be addressed by the project:

The United States Environmental Protection Agency (EPA) recently completed a Feasibility Study for the Gowanus Canal Superfund Site, prepared by CH2MHILL. Seven remedial alternatives were proposed. One retained alternative, Alternative No. 7, involved dredging the entire soft sediment column of the Canal, solidifying the top 3 to 5 feet of the native sediment, in targeted areas, and capping with a treatment layer, sand-and-gravel isolation layer and armor layer.

The proposed activities focus on the implementation of Alternative No. 7. A geotechnical investigation is proposed to evaluate the strength and characteristics of the soft sediment where the cap will be placed. An in-situ solidification treatability study is proposed to assess the effectiveness and feasibility of the solidification mix designs.

Non-aqueous phase liquid (NAPL) is present in the canal sediments. This NAPL has the potential to migrate upwards through the native sediment and soft sediment interface. This presents a risk for direct contact with human or ecological receptors. It also presents a potential continuing source of contamination to groundwater discharging from the canal.

Alternative No. 7 would include the installation of a cap to prevent migration of residual NAPL after the dredging has been completed. The geotechnical investigation would assess the strength of the soft and native sediments where the proposed cap may be placed. Detailed information is included in the associated Work plan: Geotechnical Investigation Work Plan.

The in-situ solidification treatability study would focus on the appropriate mixture of additives to achieve the permeability and compressive strength criteria required. Detailed information is included in the associated Work plan: In Situ Solidification Treatability Study Work Plan.

The environmental questions being asked:

The environmental question being asked is *can the sediments of the canal be stabilized and support a cap that prevents the migration of NAPL in the canal and prevent the NAPL from serving as a continuous source of contaminants to groundwater discharging to the canal.*

Observations from any site reconnaissance reports:

See synopsis of secondary data or information from site reports below.

A synopsis of secondary data or information from site reports:

An investigation of the canal was performed by U.S. Army Corps of Engineers, Site Investigation Gowanus Bay and Gowanus Canal, 2003. The data will provide additional soil strength information on the sediments within the Canal used in the geotechnical investigation.

The possible classes of contaminants and the affected matrices:

Possible classes of contaminants in the sediment are limited to components of Manufactured Gas Plants:

- Total benzene, toluene, ethyl benzene, and xylene (BTEX)
- Total polyaromatic hydrocarbons (PAHs)

The rationale for inclusion of chemical and nonchemical analyses:

The majority of analysis will focus on the physical and geotechnical characterization of the canal sediments. Chemical analysis is limited to characterizing the initial sediments and contaminants that may leach from the stabilized material following the in-situ remediation.

Project decision conditions:

No major variations in this proposed work are anticipated.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

National Grid, USEPA Region 2, NYSDEC, the City of New York, and GEI will use this data.

What will the data be used for?

Geotechnical Investigation

- Provide a continuous strength profile of soft and native sediment of selected areas of Gowanus Canal.
- Characterize strength of soft sediments to assess their compatibility with the proposed cap.

In-Situ Solidification Treatability Study

- Characterize untreated sediment from nine discrete canal locations.
- Develop six mixtures for solidification of the sediment.
- Perform treatability study in a phased approach:
    - Strength testing and volumetric expansion testing on all 54 samples (9 samples times 6 mixtures).
    - Hydraulic conductivity testing on best performing mixtures (approximately 13 samples)
    - Leaching tests on five of the best performing mixtures. Analyze for total BTEX and total PAHs during seven cycles for a total of 35 samples).

What type of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Geotechnical Investigation

- Cone penetration tests (CPT) will assess the geologic features of selected areas of the canal sediment.
- Samples will be collected (sonic or vibracore) of the soft sediment for further laboratory testing.
- Standard Penetration Tests (SPTs) will be performed at standard 5-foot intervals in borings to provide additional sediment strength information.
- Field screening includes Photo Ionization Detector (PID) readings and developing boring logs with visual observations.
- Laboratory testing includes
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Unit Weight (ASTM D7263)

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (cont.)

    - Moisture Content (ASTM D2216)
    - Organic Content (ASTM D2974)
    - Atterberg Limits (ASTM D4318)
    - Isotropically-Consolidated Undrained Compression (ASTM D4767)
- Differential global positioning of boring locations.

In Situ Solidification Treatability Study Work Plan

- Samples will be collected (sonic or vibracore) of the canal sediment for laboratory testing.
- Field screening includes Photoionization Detector (PID) readings and developing boring logs with visual observations.
- Laboratory testing of untreated materials samples:
    - Moisture Content (ASTM D2216)
    - Unit Weight (ASTM D7263)
    - pH (EPA Method 9045D)
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Total BTEX (EPA Method 8260B)
    - Total PAH (EPA Method 8270C)
- Laboratory testing for Stabilization Evaluation:
    - Unconfined Compressive Strength (ASTM D2166)
    - Penetrometer Evaluations
    - Hydraulic Conductivity (ASTM D5084)
    - Volumetric Expansion (Bulking)
    - Leachability (ANSI/ANS 16.1)
        - Total BTEX (EPA Method 8260B)
        - Total PAHs (EPA Method 8270C)

How "good" do the data need to be in order to support the environmental decision?

Worksheet 15, Reference and Evaluation Table, summarizes the analytical parameters and the associated quantitation limits. These parameters are for the environmental analysis, namely total BTEX and total PAHs. The remaining analyses are geotechnical parameters without set limits. In the case of the treatability study, the analysis performed on the stabilization mixtures are qualitative and measured against each other.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (cont.)

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

Geotechnical Investigation

- Five sediment samples analyzed for:
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Unit Weight (ASTM D7263)
    - Moisture Content (ASTM D2216)
    - Organic Content (ASTM D2974)
    - Atterberg Limits (ASTM D4318)
    - Isotropically-Consolidated Undrained Compression (ASTM D4767)
- Nineteen CPTs

In Situ Solidification Treatability Study Work Plan

- Nine untreated material characterization samples analyzed for:
    - Moisture Content (ASTM D2216)
    - Unit Weight (ASTM D7263)
    - pH (EPA Method 9045D)
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Total BTEX (EPA Method 8260B)
    - Total PAH (EPA Method 8270C)
- Fifty-four samples to evaluate stabilization mixtures analyzed for:
    - Unconfined Compressive Strength (ASTM D2166)
    - Penetrometer Evaluations
    - Volumetric Expansion (Bulking)
- Thirteen samples analyzed for:
    - Hydraulic Conductivity (ASTM D5084)
- Five samples leached using ANS 16.1 Leach Test performed over seven cycles (35 samples)
    - Total BTEX (EPA Method 8260B)
    - Total PAHs (EPA Method 8270C)

Where, when, and how should the data be collected/generated?

GEI will retain subcontractors perform drilling activities. GEI will perform sample collection and submission to the laboratories. Both the geotechnical investigation and solidification treatability study will be performed during the second quarter of 2012.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (cont.)

Who will collect and generate the data?

GEI will collect environmental monitoring data and samples and conduct field tests and tabulate and report field measurements. Laboratories will analyze samples for chemical analytical parameters and issue reports of analyses. GEI will conduct the data usability assessment.

How will the data be reported?

The laboratories will submit reports of analyses to GEI, according to the requirements in Worksheet 29, including electronic data deliverables (EDD) in a USEPA Region 2 compatible format found at:
http://www.epa.gov/Region2/superfund/medd.htm

How will the data be archived?

GEI will maintain electronic and hard copies of the data. The data will be submitted to National Grid, USEPA Region 2, and the NYSDEC.

QAPP Worksheet #12 – Measurement Performance Criteria Table

There are two environmental analysis performed in this investigation: total BTEX (8260B) and total PAHs (8270C). These two parameters will be performed under strict performance criteria cited below.

Matrix	Sediment
Analytical Group[1]	Total BTEX
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	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)
	SW846 8260B/ SOP 001	Sensitivity and Accuracy	Less than CRQLs	Triplicate analysis performed on one of the samples	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Triplicate analysis performed on one of the samples	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory	Matrix Spike	A
		Accuracy/Bias	Deuterated Monitoring Compound recoveries per requirements	Deuterated Monitoring Compounds	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, > 90% laboratory analysis	Data Completeness Check	S&A

QAPP Worksheet #12 – Measurement Performance Criteria Table (cont.)

Matrix	Sediment
Analytical Group[1]	Total PAHs
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	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)
	SW846 8270C/ SOP 002	Sensitivity and Accuracy	Less than CRQLs	Triplicate analysis performed on one of the samples	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Triplicate analysis performed on one of the samples	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory	Matrix Spike	A
		Accuracy/Bias	Deuterated Monitoring Compound recoveries per requirements	Deuterated Monitoring Compounds	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, > 90% laboratory analysis	Data Completeness Check	S&A

QAPP Worksheet #12 – Measurement Performance Criteria Table (cont.)

The remaining analysis will adhere to ASTM or ANS standards. These methods do not have equivalent performance standards. However, triplicate samples will be collected during the treatability study at significant stages of mixture development. Triplicate samples will be analyzed of the optimal mixture for the UCS (ASTM 2166) and hydraulic conductivity (ASTM 5084).

QAPP Worksheet #13 – Secondary Data Criteria and Limitations

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
An investigation of the canal was conducted during 2003 by the U.S. Army Corps of Engineers	U.S. Army Corps of Engineers, *Site Investigation Gowanus Bay and Gowanus Canal*, 2003	U.S. Army Corps of Engineers Advancement of 30 subsurface borings within the canal using the Standard Penetration Test (SPT) method during April through May 2003. Collection of sediment for Geotechnical testing: Atterberg Limits, Grain Size Analysis with Hydrometer, Specific Gravity, and Water Content	Data will provide additional soil strength information on the sediments within the Canal.	None

QAPP Worksheet #14 – Summary of Project Tasks

Sampling Tasks for Geotechnical Investigation:

Task 1: Collect 19 CPTs ranging from 25 to 30 feet deep at various locations throughout the canal.

Task 2: Install five test borings and collect samples using Shelby tubes. Perform SPTs at standard 5-foot intervals.

Analysis Tasks:

Laboratory testing of the soft sediments will be conducted to provide additional strength information on the sediments. Laboratory testing will include mechanical grain size distribution (ASTM D422), unit weight (ASTM D7263), moisture content (ASTM D2216), organic content (ASTM D2974), Atterberg Limits (ASTM D4318), and isotropically-consolidated undrained compression (ASTM D4767) tests.

Quality Control Tasks:

The analytical and testing laboratories will be required to analyze QC samples listed in the ASTM methods given in Worksheet 28.

Secondary Data:

Not applicable.

Other Data:

Boring logs will be created for all of the samples collected. The CPTs will generate strength profiles of the subsurface. Horizontal coordinates will be measured using Differential Global Positioning System (DGPS) survey methods. The depth to mudline will be measured at each exploration location with an accuracy of 0.1-feet.

Data Management Tasks:

All analytical data will be stored in a database on a server which will be maintained in the GEI office in Glastonbury, Connecticut. All electronic data will be backed up. Hard copies of data will also be stored in project files. See Worksheet 29 for discussion of data management.

Documentation and Records:

All hard copy data (field notebooks, photos, hard copies of Chain of Custody forms.) will be stored at the GEI office in Glastonbury, Connecticut, and stored in the project files.

Assessment/Audit Tasks:

The Geotechnical Work Plan and Field Sampling SOPs (Appendix B) will be reviewed prior to the performance of tasks.

Data Review Tasks:

GEI will review sampling and laboratory data (see Worksheets 23, 28, 35 and 36).

Sampling Tasks for Treatability Study:

Task 1: Nine samples will be collected from a barge using augers vibracore samplers to a depth of approximately 5 feet below the native sediment surface. Several cores may be required; sampler will be used to ensure that sufficient sample volume will be collected. Sample material will only be collected at depths where ISS is planned. Discreet samples will be collected from both the soft and native sediment layers. Samples will be placed in sealed 5-gallon plastic buckets and stored on ice prior to shipping to the laboratory.

Analysis Tasks:

- Laboratory testing of untreated materials samples:
  - Moisture Content (ASTM D2216)
  - Bulk Density (ASTM D7263)
  - pH (EPA Method 9045C)
  - Grain Size Distribution (ASTM D422)
  - Total BTEX (EPA Method 8260B)
  - Total PAH (EPA Method 8270C)

- Laboratory testing for Stabilization Evaluation:
  - Unconfined Compressive Strength (ASTM D2166)
  - Penetrometer Evaluations
  - Hydraulic Conductivity (ASTM D5084)

Analysis Tasks (cont):

Volumetric Expansion (Bulking)
Leachability (ANSI/ANS 16.1)
Total BTEX (EPA Method 8260B)
Total PAHs (EPA Method 8270C)

Quality Control Tasks:

The analytical and testing laboratories will be required to analyze QC samples listed in the EPA and ASTM methods given in Worksheet 28.

Secondary Data:

Not applicable.

Other Data:

Boring logs will be created for all of the samples collected. All samples locations will be analyzed using Differential Global Positioning System (DGPS) survey methods.

Data Management Tasks:

All analytical data will be stored in a database on a server which will be maintained in the GEI office in Glastonbury, Connecticut. All electronic data will be backed up. Hard copies of data will also be stored in project files. See Worksheet 29 for discussion of data management.

Documentation and Records:

All hard copy data (field notebooks, photos, hard copies of Chain of Custody forms) will be stored at the GEI office in Glastonbury, Connecticut, and stored in the project files.

Assessment/Audit Tasks:

The In-Situ Solidification Treatability Study Work Plan and Field Sampling SOPs (Appendix B) will be reviewed prior to the performance of tasks.

Data Review Tasks:

GEI will review sampling and laboratory data (see Worksheets 23, 28, 35 and 36).

QAPP Worksheet #15 – Reference Limits and Evaluation Tables

The following series of tables summarizes the project quantitation limits (PQL) laboratory method detection limits (MDLs) and quantitation limits (QLs) for the target compounds and analytes for soil. GEI also intends to engage the laboratory in an effort to achieve the lowest practical QL for these parameters.

Matrix: Sediment
Analytical Group: Total BTEX
Concentration Level: Low to moderate

Analyte (Sediment TCL Volatiles)	CAS	Project Quantitation Limit (ug/kg)	MDL (ug/kg)	QL (ug/kg)
Benzene	71-43-2	0.005	0.0002	0.005
Toluene	108-88-3	0.005	0.00008	0.005
Ethylbenzene	100-41-4	0.005	0.000055	0.005
Xylenes (total)	1330-20-7	0.005	0.00015	0.005

Matrix: Sediment
Analytical Group: Total PAHs
Concentration Level: Low to moderate

Analyte (Soil TCL SVOCs)	CAS	Project Quantitation Limit (ug/kg)	MDL (ug/kg)	QL (ug/kg)
Naphthalene	91-20-3	0.33	0.012	0.33
2-Methylnaphthalene	91-57-6	0.33	0.015	0.33
Acenaphthylene	208-96-8	0.33	0.015	0.33
Acenaphthene	83-32-9	0.33	0.013	0.33

Analyte (Soil TCL SVOCs)	CAS	Project Quantitation Limit (ug/kg)	MDL (ug/kg)	QL (ug/kg)
Fluorene	86-73-7	0.33	0.015	0.33
Phenanthrene	85-01-8	0.33	0.012	0.33
Anthracene	120-12-7	0.33	0.014	0.33
Fluoranthene	206-44-0	0.33	0.012	0.33
Pyrene	129-00-0	0.33	0.014	0.33
Benzo[a]anthracene	56-55-3	0.33	0.013	0.33
Chrysene	218-01-9	0.33	0.018	0.33
Benzo[b]fluoranthene	205-99-2	0.33	0.023	0.33
Benzo[k]fluoranthene	207-08-9	0.33	0.03	0.33
Benzo[a]pyrene	50-32-8	0.33	0.012	0.33
Indeno[1,2,3-cd]pyrene	193-39-5	0.33	0.014	0.33
Dibenz(a,h)anthracene	53-70-3	0.33	0.011	0.33
Benzo[g,h,i]perylene	191-24-2	0.33	0.013	0.33

QAPP Worksheet #16 – Project Schedule/Timeline Table

Activities	Organization	Time Frame		Deliverable	Deliverable Due Date
		Anticipated Time Frame of Initiation	Anticipated Time Frame of Completion		
Development of the Sampling Rationale, Geotechnical Investigations and In-Situ Solidification Treatment and QAPP	GEI	February 2012	February 2012	Work Plan, HASP, QAPP	15 days from USEPA comments
Sampling for Geotechnical Investigation and Treatability Study	GEI	March – April 2012	April 2012	NA	NA
Monthly Progress Report	National Grid/GEI	March 2012	September 2012	Verbal/Email	Each month
Draft Treatability Study Report	National Grid/GEI	May 2012	July 2012	Report Based on Preliminary Results	August 2012
Geotechnical Investigation Report	National Grid/GEI	May 2012	July 2012	Investigation Summary Report	July 2012
Final Treatability Study Report	National Grid/GEI	October 2012	October 2012	Report Based on Final Results	October 2012

QAPP Worksheet #17 – QAPP Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach:

The rationale for the sampling approach is two fold: 1) Geotechnical investigation and 2) In-Situ Stabilization Treatability Study. Both approaches were developed to evaluate the potential of creating a protective cap over the dredged subgrade in the Gowanus Canal.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

The sampling design and rationale was developed to evaluate the sediment in the Gowanus Canal. The geotechnical evaluation focuses on the strength of the sediments to accommodate a cap. The treatability study focuses on the optimization of mixtures to solidify the sediments.

Geotechnical Investigation

- Five sediment samples analyzed for:
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Unit Weight (ASTM D7263)
    - Moisture Content (ASTM D2216)
    - Organic Content (ASTM D2974)
    - Atterberg Limits (ASTM D4318)
    - Isotropically-Consolidated Undrained Compression (ASTM D4767)
- Nineteen CPTs

In Situ Solidification Treatability Study Work Plan

- Nine discrete samples analyzed for:
    - Moisture Content (ASTM D2216)

In Situ Solidification Treatability Study Work Plan (cont.)

    - Unit Weight (ASTM D 7263)
    - pH (EPA Method 9045D)
    - Grain Size Distribution with Hydrometer (ASTM D422)
    - Total BTEX (EPA Method 8260B)
    - Total PAH (EPA Method 8270C)
- Fifty four samples analyzed for:
    - Unconfined Compressive Strength (ASTM D2166)
    - Penetrometer Evaluations
    - Volumetric Expansion (Bulking)
- Thirteen samples analyzed for:
    - Hydraulic Conductivity (ASTM D5084)
- Five samples leached using ANS 16.1 Leach Test performed over seven cycles (35 samples)
        - Total BTEX (EPA Method 8260B)
        - Total PAHs (EPA Method 8270C)

Quality assurance/quality control samples will include field blanks of selected analysis (See Worksheet 20).

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth (ft)	Analytical Group	Concentration Level	Number of Samples[1]	Sampling SOP Reference[2]	Rationale for Sampling Location
GC-B-002	SED	0-25	Geotechnical Characterization	Unknown	1	003, 004, 006, 010, 011, 012	Sediment Characterization
GC-B-006	SED	0-25	Geotechnical Characterization	Unknown	1	003, 004, 006, 010, 011, 012	Sediment Characterization
GC-B-010	SED	0-25	Geotechnical Characterization	Unknown	1	003, 004, 006, 010, 011, 012	Sediment Characterization
GC-B-014	SED	0-25	Geotechnical Characterization	Unknown	1	003, 004, 006, 010, 011, 012	Sediment Characterization
GC-B-018	SED	0-30	Geotechnical Characterization	Unknown	1	003, 004, 006, 010, 011, 012	Sediment Characterization
RTA1N	SED	5 feet below the native sediment surface	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
RTA1S	SED	5 feet above the native sediment surface in soft sediment	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
RTA2AN	SED	5 feet below the native sediment surface	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization

Sampling Location/ID Number	Matrix	Depth (ft)	Analytical Group	Concentration Level	Number of Samples[1]	Sampling SOP Reference[2]	Rationale for Sampling Location
RTA2AS	SED	5 feet above the native sediment surface in soft sediment	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
RTA2BN	SED	5 feet below the native sediment surface	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
RTA2BS	SED	5 feet above the native sediment surface in soft sediment	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
TBN1N	SED	5 feet below the native sediment surface	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
TBN1S	SED	5 feet above the native sediment surface in soft sediment	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization
RTA3S	SED	5 feet above the native sediment surface in soft sediment	Treatability Parameters	Unknown	1	001, 002, 003, 004, 005, 006, 007, 008, 009	Sediment Characterization

Notes:

[1] Does not include quality assurance/quality control samples

[2] Appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #19 – Analytical SOP Requirements Table

Sediment Analytical Requirements

Parameter	Analytical Method	Containers (Number, size and type)	Preservation Requirements (temperature, light, chemical)	Maximum Holding Time (preparation/ analysis)
Total BTEX[1]	EPA Method 5035A/ 8260B	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	Ice samples in field and proceed with laboratory freezing within 48 of sample collection.	48 hours to freeze with 14 days after thaw for analysis.
Total PAH[2]	EPA Method 8270C	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	Ice samples in field and proceed with laboratory freezing within 48 of sample collection.	14 days for analysis
Moisture Content	ASTM D2216	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters) Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	3-30° C Keep out of direct sunlight	No hold time specified
Unit Weight	ASTM D7263	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters) Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	No preservation requirements specified	No hold time specified
pH	EPA Method 9045D	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	No preservation requirements specified	No hold time specified
Grain Size Distribution with Hydrometer	ASTM D422	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters) Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	No preservation requirements specified	No hold time specified
Unconfined Compressive Strength	ASTM D2166	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	No preservation requirements specified	No hold time specified
Hydraulic Conductivity	ASTM D5084	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	No preservation requirements specified	No hold time specified
Leachability	ANS 16.1	Taken from 1 – 5 gallon plastic container for all other tests (Treatability Parameters)	No preservation requirements specified	No hold time specified
Organic Content	ASTM D2974	Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	No preservation requirements specified	No hold time specified
Atterberg Limits	ASTM D4318	Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	No preservation requirements specified	No hold time specified
Isotropically-Consolidated Undrained Compression	ASTM D4767	Taken from 1 – 24”x3” Shelby tube for all other tests (Geotechnical Characterization)	No preservation requirements specified	No hold time specified

QAPP Worksheet #20 – Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Laboratory Duplicate Pairs[1, 2]	Total No. of Samples to Lab
Sediment	Total BTEX	Unknown	EPA Method 8260B	Approx. 9	1 (1 per 20)	10
Sediment	Total PAHs	Unknown	EPA Method 8270C	Approx. 9	1 (1 per 20)	10
Sediment	UCS testing during stabilization evaluation	Unknown	ASTM D2166	54	1 triplicate sample of optimized mixture to demonstrate adequate homogenization	55
Sediment	Hydraulic conductivity during stabilization evaluation	Unknown	ASTM D5084	13	1 triplicate sample of optimized mixture to demonstrate adequate homogenization	14

Notes:

Quality assurance/quality control samples will include laboratory duplicate samples.

QAPP Worksheet #21 – Project Sampling SOP Reference Table

List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, and testing. All project sampling SOPs are provided in Appendix B.

Reference Number	Title, Revision Date, and/or Number	Originating Organization	Modified for Work Plan
001	FD-001 Field Notebook (Rev. 2, June 2011)	GEI	No
002	FD-002 Field Observation Report (Rev. 2, June 2011)	GEI	No
003	FD-003 Sample Management and Chain of Custody (Rev. 3, June 2011)	GEI	No
004	FD-004 Photo Documentation (Rev. 2, June 2011)	GEI	No
005	FD-006 Handheld Global Positioning Receiver Operation (Rev. 2, June 2011)	GEI	No
006	DM-001 General Guidance on Determination of Appropriate Drilling Methods (Rev. 2, June 2011)	GEI	No
007	DM-004 Sonic Drilling (Rev. 2, June 2011)	GEI	No
008	SC-001 General Guidance on Sample Collection (Rev. 2, June 2011)	GEI	No
009	SC-002 Sample Handling (Rev. 3, June 2011)	GEI	No
010	SC-003 Investigation Derived Waste (Rev. 2, June 2011)	GEI	No
011	SC-004 Headspace VOC Screening (Rev. 2, June 2011)	GEI	No
012	SM-001 Soil Sampling Including Split-Spoon (Rev. 2, June 2011)	GEI	No
013	SM-003 Soil Classification (Rev. 2, June 2011)	GEI	No
014	QA-001 Equipment Decontamination (Rev. 2, June 2011)	GEI	No
015	QA-002 Field Quality Control Procedures (Rev. 3, June 2011)	GEI	No
016	Penetration Testing Services SOP	Conetec	No

QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing and Inspection Table

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Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference[1]
Portable Photoionization Detector (PID)	Calibration, Maintenance, Testing and Inspection requirements are specified in GEI's SOP SC-004 provided in Appendix B – Field Sampling SOPS							Field Team Leader	GEI's SOP SC-004 Appendix B – Field Sampling SOPS
Cone Penetrometer	Calibration, Maintenance, Testing and Inspection requirements are specified in D 5778 – 07 provided in Appendix B – Field Sampling SOPS and Conetec's Cone Penetration Testing Services SOP							Field Team Leader	Provided in Appendix B.

Notes:

Field Instrumentation: The Field Team Leader will be responsible for insuring that these instruments are calibrated before each field sampling event. Field equipment must be inspected and calibrated before use according to the criteria given in the SOP. If problems occur with field instruments or equipment which cannot be resolved by the field team personnel, they should contact the Field Team Leader. If field equipment fails inspection, it is the Field Team Leader's responsibility to investigate and resolve the problem. The GEI Field Team Leader can coordinate with equipment vendors, assist in resolution of problems with field equipment, and supply or obtain any spare or replacement parts or equipment.

QAPP Worksheet #23 – Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
001	Volatile Organic Compounds by GC/MS, SW846 8260B, Rev. 2, 1996	Definitive	Total BTEX	GCMS	Kemron Environmental Services, Inc.	No
002	Semivolatile Organic Compounds by GC/MS, SW846 8270C, Rev. 4, 2007	Definitive	Total PAH	GCMS	Kemron Environmental Services, Inc.	No
003	Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass, ASTM D2216, 2010	Definitive	Moisture Content	Drying Oven	Kemron Environmental Services, Inc. and Terra Sense, Inc.	No
004	Laboratory Determination of Density (Unit Weight) of Soil Specimens, ASTM D7263, 2009	Definitive	Unit Weight	Drying Oven	Kemron Environmental Services, Inc. and Terra Sense, Inc.	No
005	pH, EPA Method 9045D, Rev. 4, 2004	Definitive	pH	pH Meter	Kemron Environmental Services, Inc.	No
006	Particle-Size Analysis of Soils, ASTM D422 (Reapproved 2007)	Definitive	Grain Size Distribution with Hydrometer	Hydrometer	Kemron Environmental Services, Inc. and Terra Sense, Inc.	No

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
007	Unconfined Compressive Strength of Cohesive Soil, ASTM D2166, 2006	Definitive	Unconfined Compressive Strength	Compression Device	Kemron Environmental Services, Inc.	No
008	Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter ASTM D5084, 2010	Definitive	Hydraulic Conductivity	Falling Head Hydraulic System	Kemron Environmental Services, Inc.	No
009	Leach Test, ANS 16.1	Definitive	Leachability	NA	Kemron Environmental Services, Inc.	No
010	Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, ASTM D2974, 2007	Definitive	Organic Content	Drying Oven	TerraSense, LLC	No
011	Liquid Limit, Plastic Limit, and Plasticity Index of Soils, ASTM D4318, 2010	Definitive	Atterberg Limits	Liquid Limit Device	TerraSense, LLC	No
012	Consolidated Undrained Triaxial Compression Test for Cohesive Soils, ASTM D4767, 2004	Definitive	Isotropically-Consolidated Undrained Compression	Axial Loading Device	TerraSense, LLC	No

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	Initial RSD $\leq$15% Linear Regression r $\geq$ 0.995 ICV 75-125% Recovery CCV $\leq$20%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	001, 002

[1] Worksheet No. 23 specifies the number SOP.

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	Clean/bake sample lines and trap, clip/replace column, clean source	SW846 8260B SW846 8270C	Pass BFB Tune, Continuing Calibration	Daily or as needed	BFB Criteria CCV ≤ 20% Diff	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	001, 002
Drying Oven	See ASTM D2216	Moisture Content	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D2216 (Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass).				Assigned Lab personnel	003
Drying Oven	See ASTM D2937	Unit Weight	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D2937 (Density of Soil in Place by the Drive-Cylinder Method).				Assigned Lab personnel	004
pH Meter	Clean electrodes	pH	Two point minimum calibration	Each sample	0.05 pH units	Perform Maintenance, Recalibrate	Assigned Lab personnel	005
Hydrometer	See ASTM D422	Grain Size Distribution with Hydrometer	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D422 (Particle-Size Analysis of Soils).				Assigned Lab personnel	006
Compression Device	See ASTM D2166	Unconfined Compressive Strength	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D2166 (Unconfined Compressive Strength of Cohesive Soil).				Assigned Lab personnel	007

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Falling Head Hydraulic System	See ASTM D5084	Hydraulic Conductivity	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D5084 (Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter).				Assigned Lab personnel	008
Leach Test	See ANS 16.1	Leach Test	Measurement of the leachability of solidified low-level radioactive wastes by a short-term test procedure				Assigned Lab personnel	009
Drying Oven	See ASTM D2974	Organic Content	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D2974 (Moisture, Ash, and Organic Matter of Peat and Other Organic Soils).				Assigned Lab personnel	010
Liquid Limit Device	See ASTM D4318	Atterberg Limits	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D4318 (Liquid Limit, Plastic Limit, and Plasticity Index of Soils).				Assigned Lab personnel	011
Axial Loading Device	See ASTM D4767	Isotropically-Consolidated Undrained Compression	Equipment maintenance, testing and inspection will be performed in accordance with ASTM D4767 (Consolidated Undrained Triaxial Compression Test for Cohesive Soils).				Assigned Lab personnel	012

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[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #26 – Sample Handling System

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): GEI Consultants Field Team supervised by the Field Team Leader will
Sample Packaging (Personnel/Organization): GEI Consultants Field Team
Coordination of Shipment (Personnel/Organization): GEI Consultants Sample Management Officer
Type of Shipment/Carrier: Federal Express for Overnight Delivery or courier to the laboratory
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Assigned laboratory personnel
Sample Custody and Storage (Personnel/Organization): Assigned laboratory personnel
Sample Preparation (Personnel/Organization): Assigned laboratory personnel
Sample Determinative Analysis (Personnel/Organization): Assigned laboratory personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will not be stored in the field, but will be kept in cooler at 4 degree C and shipped within 24 hours of collection. If due to an emergency they are stored in the field, they will be kept in a cooler or transferred to a refrigerator kept at 4 degrees C.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion will be conducted according to the SOPs and the requirements given in Worksheet 19.
SAMPLE DISPOSAL
Personnel/Organization: Test America Laboratories Sample Custodians
Number of Days from Analysis: At least 60 days

QAPP Worksheet #26 – Sample Handling System (cont.)

Sample Handling System

Sample handling and custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample is considered to be in a person's custody if it is in:

- his/her possession;
- his/her view, after being in his/her possession;
- his/her possession and has been placed in a secure location; or
- a designated secure area

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample, including time of sample collection and any preservation techniques, and a Chain of Custody (COC) record which serves as physical evidence of sample custody. Custody procedures will be similar to the procedures outlined in the USEPA's Contract Laboratory Program Guidance for Field Samplers (USEPA, 2007). The COC documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. Sample custody procedures are developed in three areas: sample collection, laboratory analysis, and final evidence files, which are described below.

Field Sample Handling and Custody

Field records provide a means of recording information for each field activity performed at the Site. COC procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the COC intact. Worksheet 19 lists the specific sample preservation requirements for each test method.

Field Procedures

The general responsibilities of the field team are listed below:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred to the Sample Management Officer (SMO) or until they are properly dispatched. As few people as possible should handle the samples.

QAPP Worksheet #26 – Sample Handling System (cont.)

- The Field Team Leader, or designee, is responsible for entering the proper information in the field logbook, including all pertinent information such as sample identification number, date and time of sample collection, type of analysis, and description of sample location. The information entered into the field log book will be used to generate a COC.
- All sample containers will be labeled with the project identification, sample identification, matrix, type of analysis required, and preservation requirements.
- The samples will be properly preserved, bagged, and packed into coolers. The original COC form will be placed into the lead cooler and will be shipped to the laboratory.
- The SMO or designee will review all field activities to determine whether proper custody procedures were followed during the field work and if additional samples are required.

Field Records

The field log book will provide the means of recording data collection activities. Entries will be described in as much detail as possible, so that persons going to the Project Properties can reconstruct a particular situation without reliance on memory. At the beginning of each field day, the date, start time, weather, and names of all sampling team members present will be entered. The names of visitors to the Project Properties and the purpose of their visit will also be recorded. All field measurements, as well as the instrument(s), will be noted.

Samples will be collected following the sampling procedures documented in the SOPs (Appendix B). Observations such as sampling conditions or any problems will also be recorded. Sample identification numbers will be assigned at the time the data are entered in the logbook. Field duplicate samples, which will receive a unique sample identification number, are “blind” to the laboratory and will be identified under the sample description so that they can be associated with their respective samples by project staff.

Sample Identification System

All samples collected from the Project Properties must be identified with a sample label in addition to an entry on a COC record. Indelible ink will be used to complete sample labels and handwritten COC records.

QAPP Worksheet #26 – Sample Handling System (cont.)

Sample Labels/Tags

Sample labels will require the field team to complete the following information for each sample container:

1. Sample Number
2. Sample Matrix
3. Parameters to be analyzed
4. Date of Collection
5. Time of Collection
6. Preservation Method(s)
7. Sampler's Name

QAPP Worksheet #27 – Sample Custody Requirements

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Samples will be collected per the procedures described in the Appendix B – Field Sampling SOPs. The field sample custody procedures including sample packing, shipment, and delivery requirements are discussed in the text in Worksheets 17 and 26.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

Each laboratory will have a sample custodian who accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. The laboratory sample custody procedures were discussed further in the following text.

Sample Identification Procedures:

The sample identification scheme that will be employed is described in the Geotechnical Investigation Work Plan and In-Situ Solidification Treatability Study Work Plan. Sample labeling procedures are discussed in the text in Worksheet 26.

Chain-of-Custody Procedures:

A COC record will accompany the samples from the time of sampling through all transfers of custody. The COC procedures are detailed in the following text.

QAPP Worksheet #27 – Sample Custody Requirements (cont.)

Sample Custody Requirements:

Chain of Custody Procedure

The following information should be recorded on COC forms. All COC forms must be signed in ink:

- Project name and/or project number
- Signature of SMO or designee
- Sampling station number
- Date and time of collection
- Grab or composite sample designation
- Sample matrix
- Sampling location description
- Field identification number
- Analyses required
- Preservation technique
- Signatures and dates for transfers of custody
- (if applicable) Air express/shipper's bill of lading identification numbers

The COC form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The COC record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. Three copies of the COC form are created; one copy is retained by the Field Team Leader and two are sent to the laboratory. An electronic copy of each COC should be also made and kept in the project directory. The SMO or designee completes a COC record to accompany each shipment from the field to the laboratory.

The completed COC is put in a zip-lock bag and taped to the inside cover of the sample shipping container. If there is more than one container in a shipment, copies of the COC form will be placed in each container. Each container is then sealed with custody seals and custody is transferred to the laboratory.

QAPP Worksheet #27 – Sample Custody Requirements (cont.)

Transfer of Custody and Shipment

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below:

- All samples are accompanied by a COC. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This form documents sample custody transfer from the SMO or designee, through the shipper, to the analytical laboratory. Since a common carrier will usually not accept responsibility for handling COC forms, the name of the carrier is entered under "Received by," the bill-of-lading number is recorded in the comments section, and the COC form is placed in a zip-lock plastic bag and taped to the inside lid of the lead shipping cooler. Copies of the COC form will be placed in each additional cooler in a shipment.

- Samples will be packaged for shipment and either picked up at a pre-arranged location by the laboratory or dispatched to the appropriate laboratory via overnight delivery service. A separate COC record must accompany each shipment. Shipping containers will be sealed for shipment to the laboratory. Two custody seals will be applied to each cooler to document that the container was properly sealed and to determine if the container was tampered with during shipment. The custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened (*i.e.*, diagonally opposite corners of the cooler lid).

- The original COC will accompany the shipment. A copy will be retained by the Field Team Leader.

- If the samples are sent by common carrier or air freight, proper documentation must be maintained. For example, the bill of lading must be retained by the Field Team Leader.

Laboratory Custody Procedures

The laboratory custody procedures will be equivalent to those described in the latest edition of the SOW. The following will be addressed in the laboratory custody SOPs:

- A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory.

QAPP Worksheet #27 – Sample Custody Requirements (cont.)

- Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.
- To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers.
- The following stages of analysis must be documented by the laboratory:
  - Sample Extraction/Preparation.
  - Sample Analysis
  - Data Reduction
  - Data Reporting
- Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.
- When sample analyses and QA checks have been completed in the laboratory, the used portion of the sample must be stored or disposed of in accordance with the protocols specified in the SOW or the subcontract agreement. Identifying labels, data sheets, COCs, and laboratory records will be retained until analyses and QA checks are completed in accordance with the protocols specified in the subcontract agreement.

Final Evidence Files

This is the final phase of sample custody. The COC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, and correspondence, logs, pictures, and data review reports will be archived by GEI Consultants, Inc.

Sample Holding Times

Information on sample holding times and required preservation for each test method are provided in Worksheet 19.

Sample Packaging and Shipping Requirements

Custody of samples must be maintained through the shipment of samples to the selected laboratory. All samples will be packaged and shipped at the end of each day unless other arrangements are made with the laboratory.

QAPP Worksheet #28 – QC Samples Table

Matrix	Sediment
Analytical Group	Total BTEX
Concentration Level	Low
Sampling SOP	See worksheet 20
Analytical Method/ SOP Reference	SW846 8260B, SOP 001
Sampler's Name	GEI Sampling Crew
Field Sampling Organization	GEI Consultants, Inc.
Analytical Organization	Kemron Environmental Services, Inc.
No. of Sample Locations	See worksheet 18

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory triplicate	1 per 20 field samples	QAPP	If the limits exceed limits for the field duplicate, this will be addressed by the GEI Data Reviewer	GEI Field Team Leader	Precision	RPD < 30% for duplicate for values greater than or equal to five times the CRQL
Surrogates	Each sample, standard, blank	Per laboratory SOP	Reanalyze sample	TestAmerica Laboratory	Accuracy/Bias	Per laboratory SOP
Method Blank	Once every 12 hours	< CRQL	Reanalyze Batch	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Blank Spike	Once every 12 hours	Per laboratory SOP	Reanalyze Batch	TestAmerica Laboratory	Accuracy	Per laboratory SOP
Internal Standard	Each sample, standard, Blank	Per Laboratory SOP	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per laboratory SOP
Method Detection Limits	Annual	Per Laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #28 – QC Samples Table (cont.)

Matrix	Sediment
Analytical Group	Total PAHs
Concentration Level	Low
Sampling SOP	See worksheet 20
Analytical Method/ SOP Reference	SW846 8270C, SOP 002
Sampler's Name	GEI Sampling Crew
Field Sampling Organization	GEI Consultants, Inc.
Analytical Organization	Kemron Environmental Services, Inc.
No. of Sample Locations	See worksheet 18

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Triplicate	1 per 20 field samples	QAPP	If the limits exceed limits for the field duplicate, this will be addressed by the GEI Data Reviewer	GEI Field Team Leader	Precision	RPD < 30% for duplicate for values greater than or equal to five times the CRQL
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG or each SDG, whichever is most frequent	QAPP	None if blank spike passes	TestAmerica Laboratory	Accuracy/Bias and Precision	Per laboratory SOP
Surrogates	Each sample, standard, blank	Per laboratory SOP	Reanalyze sample	TestAmerica Laboratory	Accuracy/Bias	Per laboratory SOP
Method Blank	Once per batch	< CRQL	Re-extract Batch	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Blank Spike	Once per batch	Per laboratory SOP	Re-extract Batch	TestAmerica Laboratory	Accuracy	Per laboratory SOP
Internal Standard	Each sample, standard, Blank	Per laboratory SOP	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per laboratory SOP
Method Detection Limits	Annual	Per laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #29 – Project Documents and Records Table

This section identifies the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on- and off-site analysis, and data assessment.

Project Documents and Records Table

Sample Collection Documents and Records (as applicable):

- Field Notes and or data sheets
- Drilling logs and Well construction logs
- Chain of Custody Forms
- Air bills
- Analytical and Testing Sample Data Packages
- Data review discussion

On-Site Activities Documents and Records:

- Sample collection and processing record and custody records
- Sample custody records
- Air bills (if applicable)
- Custody records
- Copies of field notes

Off-Site Analysis Documents and Records

- Chain of Custody (COC) records will be made and stored in the project files
- Copies of air bills (if applicable) will be kept in project files
- Copies of all Analytical Data Deliverables stored in Lab and transferred to Project files, instrument calibration records, lab, raw data stored in electronically or in hardcopy. Laboratory electronic data deliverables (EDD) will be obtained in a USEPA Region 2 compliment format.

Data Assessment Documents and Records

- Project Records: Copies of all field notes must be sent to GEI Consultants, Inc.
- Project Records: Copies of COC must be kept by GEI Consultants, Inc.
- Field and/or lab inspection reports/checklists
- Corrective action documentation
- Data review discussion
- Final Report

QAPP Worksheet #29 – Project Documents and Records Table (cont.)

This section describes the project data management process, tracing the path of the data from their generation to their final use or storage. All project data and information must be documented in a format useable to the project personnel.

Project Document Control System

Project documents will be controlled by the GEI Consultants, Inc. The Project Manager will maintain and distribute project documents, including any amendments. Electronic copies of project information will be maintained in the project directory on the server at GEI Consultants, Inc., Glastonbury, Connecticut office, which is backed up at least once per day.

Data Recording

Data for this project will be collected by handwritten entries and will be recorded into field logbooks or on forms. Software may be the used to generate COC records and sample labels, or COCs and labels may be created manually. Computer-generated data associated with laboratory analyses will be managed under the control of the laboratory's laboratory information management system (LIMS).

Laboratory Data Transmittal

Laboratory data are managed by the laboratory's LIMS system, beginning with sample check-in on the sample receiving data terminal. Full laboratory data reports will be delivered to GEI Consultants, Inc. and will include electronic data deliverables (EDDs).

Data Storage and Retrieval

Paper copies of the forms, electronic copies of files, and the photographic log will be transmitted regularly to the GEI PM or designee. The completed forms and notebooks will be stored in the custody of the PM for the duration of the project. The full laboratory data reports submitted to GEI Consultants, Inc. will be stored in the custody of the Project Quality Officer.

The Laboratory will maintain copies of documents and backups of all data associated with the analyses of samples. Raw data and electronic media of all field samples, including QC samples and blanks, will be archived from the date of generation and will be kept by the laboratory. Hard copies of project files will be archived at a secure facility and retained until the end of the contract. Data will be transferred to National Grid upon completion of the project. Retrieval of data by others will be at the discretion of National Grid and the USEPA. The length of time that records will be archived will be at the discretion of the National Grid and USEPA.

Each laboratory will archive, electronically, the sample analyses and submit the electronic data files along with the data deliverable package. Laboratory electronic data deliverables (EDD) will be obtained in a USEPA Region 2 format (http://www.epa.gov/Region2/superfund/medd.htm).

QAPP Worksheet #30 – Analytical Services Table

Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. Group by matrix, analytical group, concentration, and sample location or ID number. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organization cannot be used.

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Sediment	Total BTEX; SW846 8260B	Low	See Worksheet 18	001	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Total PAHs; SW846 8270C	Low	See Worksheet 18	002	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass, ASTM D2216, 2010	NA	See Worksheet 18	003	Ten Business Days	Kemron Environmental Services, Inc. and Terra Sense, Inc.	A backup lab has not been assigned at this time
Sediment	Laboratory Determination of Density (Unit Weight) of Soil Specimens, ASTM D7263, 2009	NA	See Worksheet 18	004	Ten Business Days	Kemron Environmental Services, Inc. and Terra Sense, Inc.	A backup lab has not been assigned at this time
Sediment	pH, EPA Method 9045D, Rev. 4, 2004	NA	See Worksheet 18	005	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Particle-Size Analysis of Soils, ASTM D422 (Reapproved 2007)	NA	See Worksheet 18	006	Ten Business Days	Kemron Environmental Services, Inc. and Terra Sense, Inc.	A backup lab has not been assigned at this time

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Sediment	Unconfined Compressive Strength of Cohesive Soil, ASTM D2166, 2006	NA	See Worksheet 18	007	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter ASTM D5084, 2010	NA	See Worksheet 18	008	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Leach Test, ANS 16.1	NA	See Worksheet 18	009	Ten Business Days	Kemron Environmental Services, Inc.	A backup lab has not been assigned at this time
Sediment	Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, ASTM D2974, 2007	NA	See Worksheet 18	010	Ten Business Days	TerraSense, LLC	A backup lab has not been assigned at this time
Sediment	Liquid Limit, Plastic Limit, and Plasticity Index of Soils, ASTM D4318, 2010	NA	See Worksheet 18	011	Ten Business Days	TerraSense, LLC	A backup lab has not been assigned at this time
Sediment	Consolidated Undrained Triaxial Compression Test for Cohesive Soils, ASTM D4767, 2004	NA	See Worksheet 18	012	Ten Business Days	TerraSense, LLC	A backup lab has not been assigned at this time

QAPP Worksheet #31 – Planned Project Assessment Table

Identify the type, frequency, and responsible parties of planned assessment activities that will be performed for the project.

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Field Safety Audit	Discretionary	Internal	GEI	GEI Corporate Health and Safety Officer	GEI PMs	GEI PMs	GEI PMs
Contractor Performance Evaluation	Monthly or as warranted	External	National Grid	National Grid delegate	GEI PMs	GEI PMs	GEI PMs

QAPP Worksheet #32 – Assessment Findings and Response Actions

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

PROJECT ASSESSMENT TABLE

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Party responsible for performing assessment	Person(s) responsible for responding to assessment findings	Person(s) responsible for identifying and implementing corrective actions
Field Sampling Technical Systems Audit	As needed.	Internal	GEI	Project QA/QC Officer, GEI, Inc.	Project Manager, GEI	Project Manager, GEI
Fixed Laboratory Technical Systems Audit	As needed.	External	GEI	Project QA/QC Officer, GEI, Inc.	Project Manager, Project Laboratory	Project Manager, Project Laboratory

Field Oversight

Field oversight of the project will be conducted by the Task Manager/Field Leader on a daily basis. The Task Manager/Field Leader will oversee the field samplers and subcontractors to see that the work goes smoothly and according to the site-specific plans. Corrective actions will be addressed immediately in the field and any issues that might possibly impact the data will be documented in the field notes.

Field Sampling Technical Systems Audit

A field sampling technical systems audit would take place on-Site early in the field program so that necessary corrective action measures can be implemented, if required. The audit would consist of an evaluation of sampling techniques, field parameter measurements, record keeping including log books and COCs, sample collection and handling sample design, subcontractor oversight and health and safety.

QAPP Worksheet #32 – Assessment Findings and Response Actions (cont.)

Fixed Laboratory Technical Systems Audit

A laboratory technical systems audit would consist of a review of any, but not necessarily all, of the following: sample handling procedures, equipment condition and operation, analytical methods and procedures and overall conformance with SOPs provided in this QAPP. The audit may span a period of one or more days, so that the audit team can view various types of analytical procedures that will be used on the project.

Other Technical Systems Audits

Checks of project activities may be performed by qualified personnel throughout the duration of the project. Activities will be reviewed by GEI with ultimate approval to be provided by the project QA/QC officer.

Assessment Findings and Corrective Action Responses

Deficiencies that are found as a result of the audits will be communicated both verbally to the responsible party upon discovery and will also be documented in a written audit report. A formal corrective action response in writing will be requested from the responsible party. The response will document the reason for the deficiency and the actions that will be put in place to correct the deficiency. Corrective action responses will be filed in the project files.

Additional QAPP Non-Conformances

The corrective action procedures discussed in this section will also be applied to significant deviations from procedures outlined in this QAPP. Project personnel who determine that a deviation has occurred will document the deviation and notify the GEI project manager. The project manager will evaluate the severity of the deviation, document deviations, and implement corrective action procedures as appropriate.

QAPP Worksheet #33 – Planned Project Assessment Table

Identify the frequency and type of planned QA Management Reports, the project delivery dates, the personnel responsible for report preparation, and the report recipients.

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Progress Reports	Monthly	15th day of each following month	GEI Administrative/Technical PM, or designee	USEPA and National Grid PMs
Technical System Field Internal Audit	Initially, within the first two weeks of field work and with follow up audits if significant deficiencies are found	Month after field work begins	Project QC Officer or designee	National Grid PM
Data Review Report	After laboratory data are received	Within 30 days after receiving the full deliverable	Project QC Officer or designee	Project QC Officer and PM
Corrective Action Reports	When corrective action is required	When corrective action is implemented	Technical Project Manager or designee	Project Team and PM(s)

The National Grid PM will receive various types of management reports, such as the results of any corrective action reports and data validation reports. In addition, Progress Report may contain a section on quality control issues. Problems or issues that arise between regular reporting periods may be identified to program management at any time. Information included in the progress report will include the following:

- Results of Technical System field audits conducted during the period.
- An assessment of any problems with the measurement data, including accuracy, precision, completeness, representativeness, and comparability.
- A listing of the non-conformance reports, including Stop-Work Orders issued during the period, related corrective actions undertaken, and an assessment of the results of these actions.
- Identification of significant quality assurance problems and recommended solutions, as necessary.

QAPP Worksheet #34 – Sampling and Analysis Verification (Step 1) Process Table

Describe the processes that will be followed to verify project data. Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the person responsible. *Internal* or *external* is in relation to the data generator.

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of custody (COC)	Form will be internally reviewed upon completion and verified against field logs and laboratory reports. Review will occur with the competition of each report.	I	GEI Consultants, Inc.
Field report	Field reports will be verified with the field logbooks.	I	GEI Consultants, Inc.
Laboratory data packages	Laboratory data packages will be used to review the reported results in the project report and against QAPP criteria.	I	GEI Consultants, Inc.

Data Verification

- The Field Team Leader or designee is required to review the logbook entries for errors or omissions. This information is transmitted to the Project QC Officer or designee for correction.

- In addition, the Project QC Officer or designee is responsible for reviewing field data for completeness and to verify that the field crew followed the QC requirements detailed in this QAPP (*e.g.*, the collection of QC samples at the required frequency, response checking the field instruments). If any problems with the information are found, the Project QC Officer or designee will document the problems.

The Project QC Officer or designee reviews the field data.

QAPP Worksheet #35 – Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Methods	Records support implementation of SOP in QAPP.	GEI Consultants, Inc.
IIa	Chain of Custody	Examine traceability of data from sample collection to generation of project report	GEI Consultants, Inc.
IIb	Deviations from SOP and project documents.	Assess impacts of any deviation from methods and the project plan.	Project Manager by GEI Consultants, Inc.

QAPP Worksheet #36 – Sampling and Analysis Validation (Steps IIa and IIb) SummaryTable

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

Step IIa/IIb	Matrix	Analytical Group[1]	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Sediment	Chemical Parameters	Low to High	Laboratory SOP Criteria	Project QC Officer or designee
IIa/IIb	Sediment	Chemical/Geotechnical Parameters	Low to High	Laboratory SOP Criteria	Project QC Officer or designee

[1]. Analytical data on chemical parameters produced by subcontract laboratories will be reviewed by a qualified data reviewer assigned by GEI.

QAPP Worksheet #37 – Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

The GEI Consultants' quality control officer will review chemical data in accordance with the protocols outlined on Worksheet 35. Data review alone does not insure usability of the data. Other factors will be considered, including comparison of actual reporting limits achieved by the lab on the samples collected to the project action levels and data needs.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

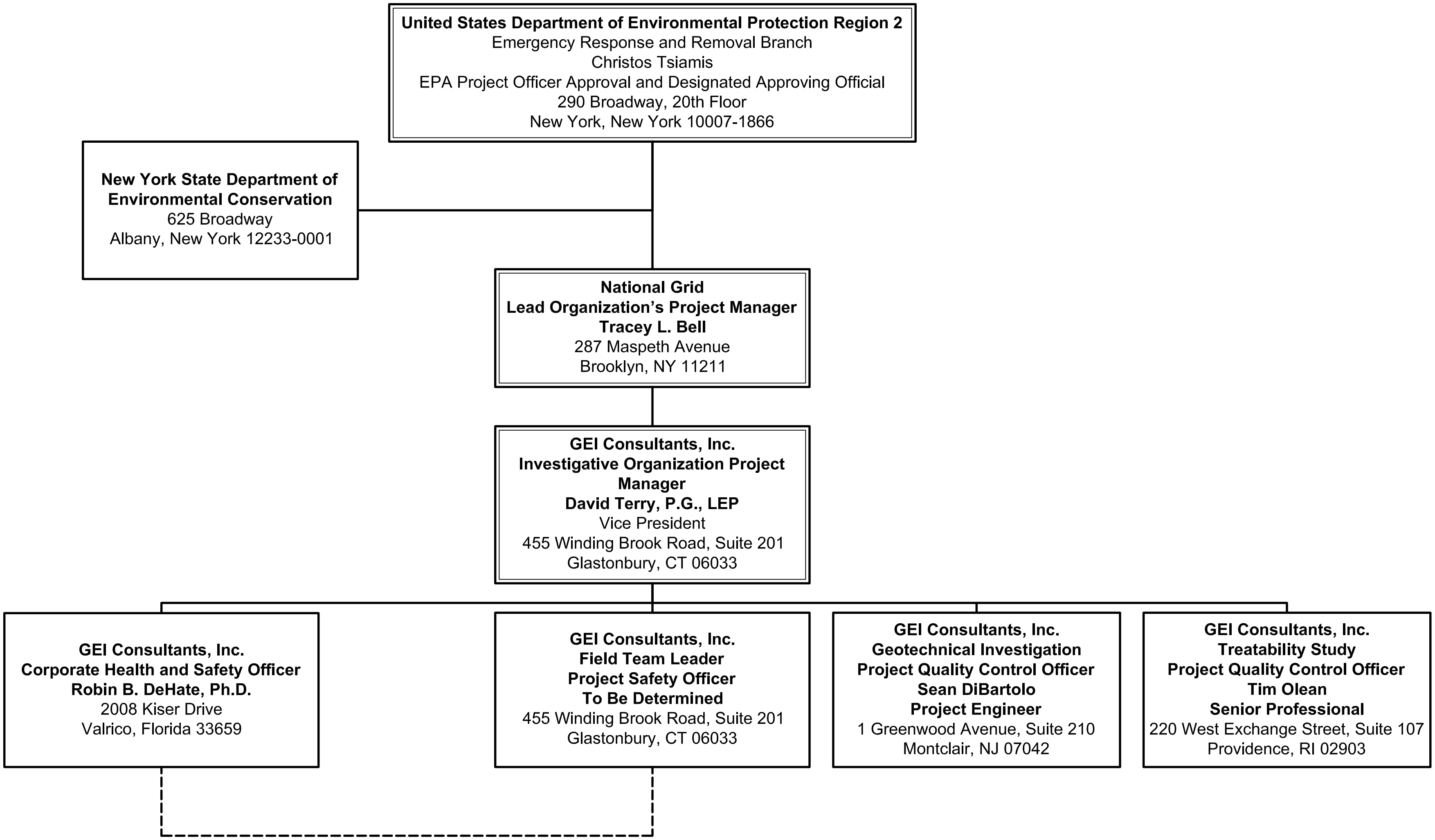
As part of the data review process, the quality control officer will identify quality control issues. These issues will be discussed and corrected by the laboratories.

Identify the personnel responsible for performing the usability assessment:

The usability of the data is the responsibility of the project team. The PMs will reconvene the project team after all data has been validated and reviewed. The data users performing the remediation design will participate in a usability assessment to determine if the data is sufficient to meet the data needs and the project DQOs, and will recommend if additional data is required. A usability assessment report will be issued by the PM or his designee documenting the results of the usability assessment review performed by the project team. The report will be submitted to the USEPA and National Grid for their approval and regulatory review.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The Usability Assessment Review will present the findings of the data evaluation processes. Resulting data quality and conformance with evaluation guidelines will be presented.



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PROJECT TEAM ORGANIZATION

Project 093010-5-1501

April 2012

Figure 1

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METHOD 8260B

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, 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:

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d_5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	I	nd	nd	nd	nd	pc

(continued)

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d_4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d_4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (IS)	540-36-3	nd	nd	nd	nd	c	nd
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	I	nd	nd	nd	nd	c
Ethanol	64-17-5	I	c	c	nd	nd	c
Ethyl acetate	141-78-6	I	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	I	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	I	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	I	nd	nd	nd	c
Methanol	67-56-1	I	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c

(continued)

Compound	CAS No.[b]	Appropriate Preparation Technique[a] 5030/ 5035	5031	5032	5021	5041	Direct Inject.
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	I	nd	nd	nd	c
β-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	pc
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	I	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-3	c	nd	c	c	c	c
Toluene-d_8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluidine	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethene	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
o-Xylene	95-47-6	c	nd	c	c	c	c
m-Xylene	108-38-3	c	nd	c	c	c	c
p-Xylene	106-42-3	c	nd	c	c	c	c

[a] See Sec. 1.2 for other appropriate sample preparation techniques
[b] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
nd = Not determined
I = Inappropriate technique for this analyte
pc = Poor chromatographic behavior
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocussed on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 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 should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 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. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 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 protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Automated static headspace device for solid samples - Described in Method 5021.

4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.

4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.

4.6 Desorption device for air trapping media for air samples - Described in Method 5041.

4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.

4.8 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An 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.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt_x-502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt_x-5, SPB-5, or equivalent), 1-µm film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8-µm film thickness, or equivalent.

4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. 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.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - 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 must be interfaced to the mass spectrometer. The computer must have software that allows searching any 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 must also be available 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 should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.15 Vials - 2-mL, for GC autosampler.

4.16 Disposable pipets - Pasteur.

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

4.18 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic 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.

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

5.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$ - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to

5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared 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 dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months 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.

5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/L
400 µL	200 µg/L

5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
10 µL	1 µg/L
20 µL	2 µg/L
50 µL	5 µg/L
100 µL	10 µg/L
250 µL	25 µg/L

5.8 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 must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced 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. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40^{o}C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40^{o}C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

7.2 Recommended chromatographic conditions

7.2.1 General conditions

Injector temperature:	200 - 225°C
Transfer line temperature:	250 - 300°C

7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 70°C, then 15°C/min to 145°C
Final temperature:	145°C, hold until all expected compounds have eluted.

7.2.5 Direct injection - Column 2

Carrier gas (He) flow rate:	4 mL/min
Column:	J&W DB-624, 70m x 0.53 mm
Initial temperature:	40°C, hold for 3 minutes
Temperature program:	8°C/min
Final temperature:	260°C, hold until all expected compounds have eluted.
Column Bake out:	75 minutes
Injector temperature:	200-225°C
Transfer line temperature:	250-300°C

7.2.6 Direct split interface - Column 4

Carrier gas (He) flow rate:	1.5 mL/min
Initial temperature:	35°C, hold for 2 minutes
Temperature program:	4°C/min to 50°C 10°C/min to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted
Split ratio:	100:1
Injector temperature:	125°C

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

Mass range:	35 - 260 amu
Scan time:	0.6 - 2 sec/scan
Source temperature:	According to manufacturer's specifications
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2-µL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of

BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 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 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. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should 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 Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) 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. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

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

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

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

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

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

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are 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 using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should 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 the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the 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. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 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 (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 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. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the 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. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 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 CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 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 criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification 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 the 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, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification 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, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 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.

7.5.6.3 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, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.

If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10 µL of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 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. 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.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound 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 generated by the laboratory 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. Compounds are identified as present when the following criteria are met.

7.6.1.1 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 will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 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%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be 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.

7.6.1.5 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.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 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 will be determined by the purpose of the analyses being conducted. Data system library search routines should 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. Use the following guidelines for making tentative identifications:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should 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 should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be 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.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be 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. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

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

9.0 METHOD PERFORMANCE

9.1 The method detection limit (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 actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

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TABLE 1

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2'[c]	(µg/L)
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2"[c]	(µg/L)
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03

TABLE 1 (cont.)

Compound	Retention Time (minutes) Column 1[a]	Column 2[b]	Column 2"[c]	MDL[d] (µg/L)
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d_5	23.10			
1,4-Dichlorobenzene-d_4	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d_4	32.30	19.08	27.25	
Dichloroethane-d_4	12.08			
Dibromofluoromethane	--			
Toluene-d_8	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

[b] Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

[c] Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10 °C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

[d] MDL based on a 25-mL sample volume.

TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

[a] Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

[b] MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment[b] µg/kg
5	1	5

[a] Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

[b] EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor[c]
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

[c] EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

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

[a] Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers" instructions), provided that method performance is not adversely affected.

TABLE 5

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d_4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
β-Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d_6	84	83
Bromobenzene-d_5	82	162
Bromochloromethane-d_2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d_5	117	
1,4-Dichlorobenzene-d_4	152	115, 150
1,1,2-Trichloroethane-d_3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d_1	84	
Dibromofluoromethane	113	

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d_4	102	
Toluene-d_8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

[a] Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

[b] Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene[a]	86-115	74-121
Dibromofluoromethane[a]	86-118	80-120
Toluene-d_8[a]	88-110	81-117
Dichloroethane-d_4[a]	80-120	80-120

[a] Single laboratory data, for guidance only.

TABLE 9

QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

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

Calculate appropriate dilution factor for concentrations exceeding this table.

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

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

TABLE 10

DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

* Alternate mass employed
** IS quantitation

Data are taken from Reference 9.

TABLE 11

SINGLE LABORATORY PERFORMANCE
DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene-d_6	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*,**	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride-^{13}C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene-d_5	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform-d_1	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene-d_4	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene-d_2	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*,**	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene-d_{10}	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane-^{13}C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene-d_5	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine-d_5	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl_3F_3ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene-d_8	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene-d_1	55	44	2.8	0.5

TABLE 11 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene-d_{10}	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene-d_{10}	67	44	3.7	10.0

* Alternate mass employed
** IS quantitation
ND = Not Detected

Data are based on seven measurements and are taken from Reference 9.

TABLE 12

METHOD DETECTION LIMITS (METHOD 5031)

Compound	MDL (µg/L)	Concentration Factor	
	Macro[a]	Macro	Micro
Acetone	31	25-500	-
Acetonitrile	57	25-500	200
Acrolein	-	-	100
Acrylonitrile	16	25-500	100
Allyl Alcohol	7	25-500	-
1-Butanol	-	-	250
Crotonaldehyde	12	25-500	-
1,4-Dioxane	12	25-500	150
Ethyl Acetate	-	-	100
Isobutyl alcohol	7	25-500	-
Methanol	38	25-500	140
Methyl Ethyl Ketone	16	25-500	-
2-Methyl-1-propanol	-	-	250
n-Nitroso-di-n-butylamine	14	25-500	-
Paraldehyde	10	25-500	-
2-Picoline	7	25-500	-
1-Propanol	-	-	240
Propionitrile	11	25-500	200
Pyridine	4	25-500	-
o-Toluidine	13	25-500	-

[a] Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

MDL = 3.134 x Std. Dev. of low concentration spike (ppb).

[b] When a 40-mL sample is used, and the first 100 µL of distillate are collected.

TABLE 13

TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

Target Compound	Surrogate	Internal Standard
Acetone	d_6-Acetone	d_8-Isopropyl alcohol
Acetonitrile	d_3-Acetonitrile	d_8-Isopropyl alcohol
Acrylonitrile	d_8-Isopropyl alcohol	
Allyl alcohol	d_7-Dimethyl formamide	
Crotonaldehyde	d_8-Isopropyl alcohol	
1,4-Dioxane	d_8-1,4-Dioxane	d_7-Dimethyl formamide
Isobutyl alcohol	d_7-Dimethyl formamide	
Methanol	d_3-Methanol	d_8-Isopropyl alcohol
Methyl ethyl ketone	d_8-Isopropyl alcohol	
N-Nitroso-di-n-butylamine	d_7-Dimethyl formamide	
Paraldehyde	d_7-Dimethyl formamide	
2-Picoline	d_7-Dimethyl formamide	
Propionitrile	d_8-Isopropyl alcohol	
Pyridine	d_5-Pyridine	d_7-Dimethyl formamide
o-Toluidine	d_7-Dimethyl formamide	

TABLE 14

RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/µL)
Internal Standards	
d_5-benzyl alcohol	10.0
d_{14}-Diglyme	10.0
d_7-Dimethyl formamide	10.0
d_8-Isopropyl alcohol	10.0
Surrogates	
d_6-Acetone	10.0
d_3-Acetonitrile	10.0
d_8-1,4-Dioxane	10.0
d_3-Methanol	10.0
d_5-Pyridine	10.0
Target Compounds	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 15

CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion[a]	Secondary Ions	Retention Time (min)[b]
Internal Standards			
d_8-Isopropyl alcohol	49		1.75
d_{14}-Diglyme	66	98,64	9.07
d_7-Dimethyl formamide	50	80	9.20
Surrogates			
d_6-Acetone	46	64,42	1.03
d_3-Methanol	33	35,30	1.75
d_3-Acetonitrile	44	42	2.63
d_8-1,4-Dioxane	96	64,34	3.97
d_5-Pyridine	84	56,79	6.73
d_5-Phenol[c]	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile[c]	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone[c]	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline[c]	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol[c]	94	66,65	15.43

[a] These ions were used for quantitation in selected ion monitoring.

[b] GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.

[c] Compound removed from target analyte list due to poor accuracy and precision.

TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION[a] (METHOD 5031 - MACRODISTILLATION TECHNIQUE)

(Single Laboratory and Single Operator)

Compound	25 ppb Spike Mean %R	%RSD	100 ppb Spike Mean %R	%RSD	500 ppb Spike Mean %R	%RSD
d_6-Acetone	66	24	69	14	65	16
d_3-Acetonitrile	89	18	80	18	70	10
d_8-1,4-Dioxane	56	34	58	11	61	18
d_3-Methanol	43	29	48	19	56	14
d_5-Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di-n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

[a] Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.

TABLE 17

RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 18
RECOVERIES IN C-HORIZON SOILS FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2

TABLE 19
RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 20

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Soil/H_2O[b] Recovery		Soil/Oil[c] Recovery		Soil/Oil/H_2O Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT[e]	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT[f]	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 20 (cont.)

Compound	Soil/H_2O[b] Recovery Mean	Soil/H_2O[b] Recovery RSD	Soil/Oil[c] Recovery Mean	Soil/Oil[c] Recovery RSD	Soil/Oil/H_2O Recovery Mean	Soil/Oil/H_2O Recovery RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d_8	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

[a] Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

[c] Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

[d] Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

[e] Interference by co-eluting compounds prevented accurate measurement of analyte.

[f] Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Efficiency Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	CONT[c]	
Acetone	CONT[c]	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT[d]	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT[d]	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT[d]	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT[c]	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 21 (cont.)

Compound	Efficiency Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d_8	88	24
Bromofluorobenzene	52	15

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

[b] No analyses.

[c] Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

[d] Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 22

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Method Detection Limit (ppb) External Standard Method	Method Detection Limit (ppb) Internal Standard Method
Chloromethane	7.8	7.3
Bromomethane	9.7	9.8
Vinyl chloride	9.5	9.4
Chloroethane	9.2	10.0
Methylene chloride	CONT[b]	CONT[b]
Acetone	CONT[b]	CONT[b]
Carbon disulfide	5.4	4.9
1,1-Dichloroethene	4.0	5.7
1,1-Dichloroethane	4.0	3.5
trans-1,2-Dichloroethene	4.4	4.0
cis-1,2-Dichloroethene	4.7	4.1
Chloroform	5.6	5.0
1,2-Dichloroethane	3.3	3.2
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	1.1	4.2
Carbon tetrachloride	3.2	3.5
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	3.2	2.8
1,1,2,2-Tetrachloroethane	4.4	3.8
1,2-Dichloropropane	3.8	3.7
trans-1,3-Dichloropropene	3.4	3.0
Trichloroethene	3.1	4.0
Dibromochloromethane	3.5	3.2
1,1,2-Trichloroethane	4.4	3.3
Benzene	3.6	3.2
cis-1,3-Dichloropropene	3.5	3.0
Bromoform	4.9	4.0
2-Hexanone	7.7	8.0
4-Methyl-2-pentanone	7.5	8.0
Tetrachloroethene	4.3	4.0
Toluene	3.0	2.5
Chlorobenzene	3.3	2.8
Ethylbenzene	3.6	3.5
Styrene	3.5	3.3
p-Xylene	3.7	3.5
o-Xylene	3.3	4.7

Footnotes are on the following page.

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[a] Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked a 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] Contamination of sample by analyte prevented determination.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	5 mL H_2O[b] Recovery Mean	5 mL H_2O[b] Recovery RSD	20 mL H_2O[c] Recovery Mean	20 mL H_2O[c] Recovery RSD	20 mL H_2O/Oil Recovery Mean	20 mL H_2O/Oil Recovery RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT[c]	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14

TABLE 23 (cont.)

Compound	5 mL H_2O[b] Recovery Mean	RSD	20 mL H_2O[c] Recovery Mean	RSD	20 mL H_2O/Oil Recovery Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d_8	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

[a] Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

[b] Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

[c] Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES USING VACUUM DISTILLATION (METHOD 5032) (INTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.2	8.0	7.3	N/A[f]
Bromomethane	2.8	4.9	9.8	N/A[f]
Vinyl chloride	3.5	6.0	9.4	N/A[f]
Chloroethane	5.9	6.0	10.0	N/A[f]
Methylene chloride	3.1	4.0	CONT[g]	0.05
Acetone	5.6	CONT[g]	CONT[g]	0.06
Carbon disulfide	2.5	2.0	4.9	0.18
1,1-Dichloroethene	2.9	3.2	5.7	0.18
1,1-Dichloroethane	2.2	2.0	3.5	0.14
trans-1,2-Dichloroethene	2.2	1.4	4.0	0.10
cis-1,2-Dichloroethene	2.0	2.3	4.1	0.07
Chloroform	2.4	1.8	5.0	0.07
1,2-Dichloroethane	1.7	1.5	3.2	0.06
2-Butanone	7.4	INT[h]	INT[h]	INT[h]
1,1,1-Trichloroethane	1.8	1.7	4.2	0.10
Carbon tetrachloride	1.4	1.5	3.5	0.13
Vinyl acetate	11.8	INT[h]	INT[h]	INT[h]
Bromodichloromethane	1.6	1.4	2.8	0.06
1,1,2,2-Tetrachloroethane	2.5	2.1	3.8	0.02
1,2-Dichloropropane	2.2	2.1	3.7	0.15
trans-1,3-Dichloropropene	1.5	1.7	3.0	0.05
Trichloroethene	1.6	1.7	4.0	0.04
Dibromochloromethane	1.7	1.5	3.2	0.07
1,1,2-Trichloroethane	2.1	1.7	3.3	0.05
Benzene	0.5	1.5	3.2	0.05
cis-1,3-Dichloropropene	1.4	1.7	3.0	0.04
Bromoform	1.8	1.5	4.0	0.05
2-Hexanone	4.6	3.6	8.0	INT[h]
4-Methyl-2-pentanone	3.5	4.6	8.0	INT[h]
Tetrachloroethene	1.4	1.6	4.0	0.10
Toluene	1.0	3.3	2.5	0.05
Chlorobenzene	1.4	1.4	2.8	0.06
Ethylbenzene	1.5	2.8	3.5	0.04
Styrene	1.4	1.4	3.3	0.18
p-Xylene	1.5	2.9	3.5	0.20
o-Xylene	1.7	3.4	4.7	0.07

Footnotes are found on the following page.

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[a] Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.

[b] Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.

[c] Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[d] Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.

[e] No analyses.

[f] Contamination of sample by analyte prevented determination.

[g] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 25

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES (METHOD 5032) (EXTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.1	8.6[f]	7.8	N/A[g]
Bromomethane	2.5	4.9[f]	9.7	N/A[g]
Vinyl chloride	4.0	7.1[f]	9.5	N/A[g]
Chloroethane	6.1	7.5[f]	9.2	N/A[g]
Methylene chloride	3.1	3.3	CONT[h]	0.08
Acetone	33.0[f]	CONT[h]	CONT[h]	0.12
Carbon disulfide	2.5	3.2	5.4	0.19
1,1-Dichloroethene	3.4	3.8	4.0	0.19
1,1-Dichloroethane	2.3	1.7	4.0	0.13
trans-1,2-Dichloroethene	3.0	3.2	4.4	0.09
cis-1,2-Dichloroethene	2.4	2.7	4.7	0.08
Chloroform	2.7	2.6	5.6	0.06
1,2-Dichloroethane	1.6	1.7	3.3	0.06
2-Butanone	57.0[f]	INT[i]	INT[i]	INT[i]
1,1,1-Trichloroethane	1.6	2.4	1.1	0.08
Carbon tetrachloride	1.5	1.7	3.2	0.15
Vinyl acetate	23.0[f]	INT[i]	INT[i]	INT[i]
Bromodichloromethane	2.0	2.3	3.2	0.05
1,1,2,2-Tetrachloroethane	3.6	3.2	4.4	0.09
1,2-Dichloropropane	2.9	3.7	3.8	0.12
trans-1,3-Dichloropropene	2.3	2.4	3.8	0.08
Trichloroethene	2.5	3.0	3.1	0.06
Dibromochloromethane	2.1	2.9	3.5	0.04
1,1,2-Trichloroethane	2.7	2.8	4.4	0.07
Benzene	1.7	2.9	3.6	0.03
cis-1,3-Dichloropropene	2.1	2.5	3.5	0.06
Bromoform	2.3	2.5	4.9	0.10
2-Hexanone	4.6	4.6	7.7	INT[i]
4-Methyl-2-pentanone	3.8	3.9	7.5	INT[i]
Tetrachloroethene	1.8	2.6	4.3	0.12
Toluene	1.8	4.4	3.0	0.09
Chlorobenzene	2.4	2.6	3.3	0.07
Ethylbenzene	2.4	4.1	3.6	0.09
Styrene	2.0	2.5	3.5	0.16
p-Xylene	2.3	3.9	3.7	0.18
o-Xylene	2.4	4.1	3.3	0.08

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[a] Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.

[b] Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.

[c] These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.

[d] These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.

[e] Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.

[f] Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.

[g] No analyses.

[h] Contamination of sample by analyte prevented determination.

[I] Interference by co-eluting compound prevented accurate quantitation.

TABLE 26

VOLATILE ORGANIC ANALYTE RECOVERY FROM OIL USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Recovery Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT[c]	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT[c]	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT[c]	
4-Methyl-2-pentanone	INT[c]	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 26 (cont.)

Compound	Recovery Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	137	30
Toluene-d_8	84	6
Bromofluorobenzene	48	2

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Not analyzed.

[c] Interference by co-evaluating compounds prevented accurate measurement of analyte.

TABLE 27

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN OIL (METHOD 5032)[a]

Compound	Method Detection Limit (ppb) External Standard Method	 Internal Standard Method
Chloromethane	N/A[b]	N/A[b]
Bromomethane	N/A[b]	N/A[b]
Vinyl chloride	N/A[b]	N/A[b]
Chloroethane	N/A[b]	N/A[b]
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT[c]	INT[c]
4-Methyl-2-pentanone	INT[c]	INT[c]
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70

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[a] Method detection limits are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] No analyses.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 28

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS (METHOD 5021)

Chloroform-d_1	1,1,2-TCA-d_3	Bromobenzene-d_5
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 29

PRECISION AND MDL DETERMINED FOR ANALYSIS OF FORTIFIED SAND[a] (METHOD 5021)

Compound	% RSD	MDL (µg/kg)
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5[b]
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62[c]
Naphthalene	16.8	3.4[c]
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2[c]
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 29 (cont.)

Compound	% RSD	MDL (µg/kg)
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

[a] Most compounds spiked at 2 ng/g (2 µg/kg)
[b] Incorrect ionization due to methanol
[c] Compound detected in unfortified sand at >1 ng

TABLE 30

RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)

Compound	Recovery per Replicate (ng) Sample 1	Sample 2	Sample 3	Mean (ng)	RSD	Recovery (%)
Benzene	37.6	35.2	38.4	37.1	3.7	185[a]
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104[a]
1,2-Dibromo-3-chloro-propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130[a]
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8[a]
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146[a]
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 30 (cont.)

Compound	Recovery per Replicate (ng)			Mean (ng)	RSD	Recovery (%)
	Sample 1	Sample 2	Sample 3			
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

[a] Compound found in unfortified garden soil matrix at >5 ng.

TABLE 31

METHOD DETECTION LIMITS AND BOILING POINTS FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)[a]

Compound	Detection Limit (ng)	Boiling Point (°C)
Chloromethane	58	-24
Bromomethane	26	4
Vinyl chloride	14	-13
Chloroethane	21	13
Methylene chloride	9	40
Acetone	35	56
Carbon disulfide	11	46
1,1-Dichloroethene	14	32
1,1-Dichloroethane	12	57
trans-1,2-Dichloroethene	11	48
Chloroform	11	62
1,2-Dichloroethane	13	83
1,1,1-Trichloroethane	8	74
Carbon tetrachloride	8	77
Bromodichloromethane	11	88
1,1,2,2-Tetrachloroethane**	23	146
1,2-Dichloropropane	12	95
trans-1,3-Dichloropropene	17	112
Trichloroethene	11	87
Dibromochloromethane	21	122
1,1,2-Trichloroethane	26	114
Benzene	26	80
cis-1,3-Dichloropropene	27	112
Bromoform**	26	150
Tetrachloroethene	11	121
Toluene	15	111
Chlorobenzene	15	132
Ethylbenzene**	21	136
Styrene**	46	145
Trichlorofluoromethane	17	24
Iodomethane	9	43
Acrylonitrile	13	78
Dibromomethane	14	97
1,2,3-Trichloropropane**	37	157
total Xylenes**	22	138-144

Footnotes are found on the following page.

TABLE 31 (cont.)

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* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.

** Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.

TABLE 32

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION (METHOD 5041)

Bromochloromethane

Acetone
Acrylonitrile
Bromomethane
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,1-Dichloroethene
Trichloroethene
trans-1,2-Dichloroethene
Iodomethane
Methylene chloride
Trichlorofluoromethane
Vinyl chloride

Chlorobenzene-d_5

4-Bromofluorobenzene (surrogate)
Chlorobenzene
Ethylbenzene
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d_8 (surrogate)
1,2,3-Trichloropropane
Xylenes

1,4-Difluorobenzene

Benzene
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorodibromomethane
Dibromomethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane

TABLE 33

METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Detection Limit[a] (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

[a] Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.

FIGURE 1
GAS CHROMATOGRAM OF VOLATILE ORGANICS

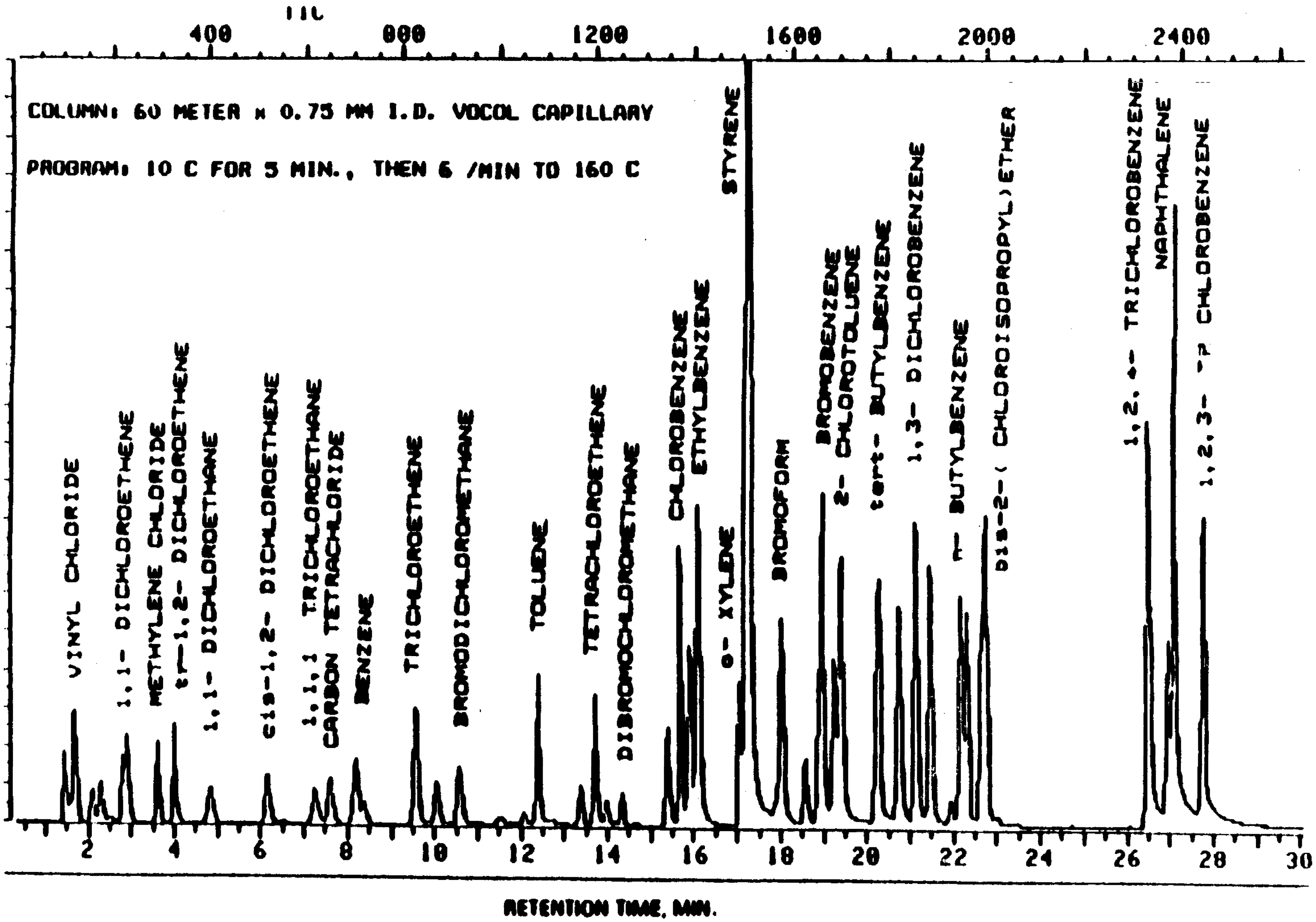


FIGURE 2
GAS CHROMATOGRAM OF VOLATILE ORGANICS

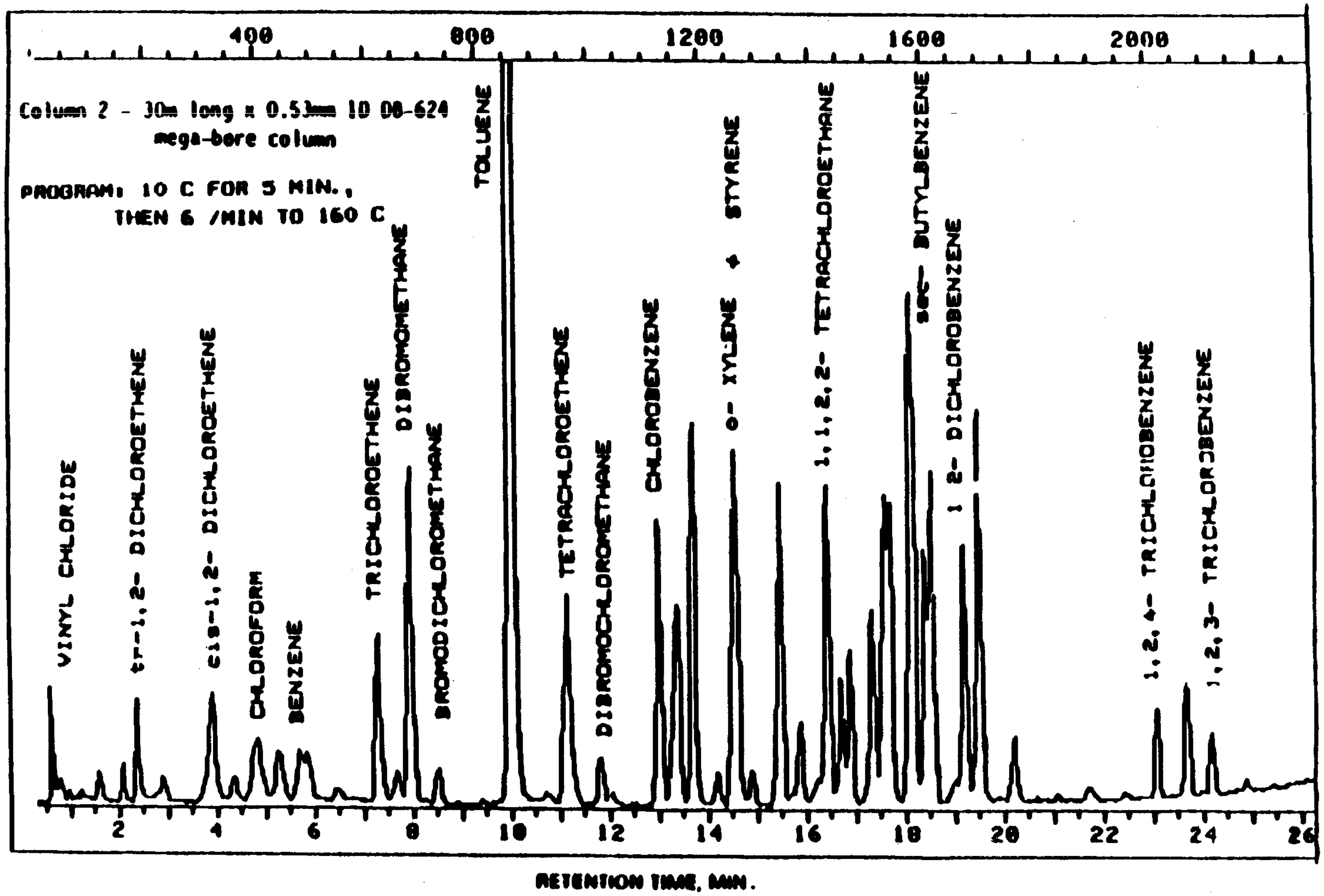


FIGURE 3
GAS CHROMATOGRAM OF VOLATILE ORGANICS

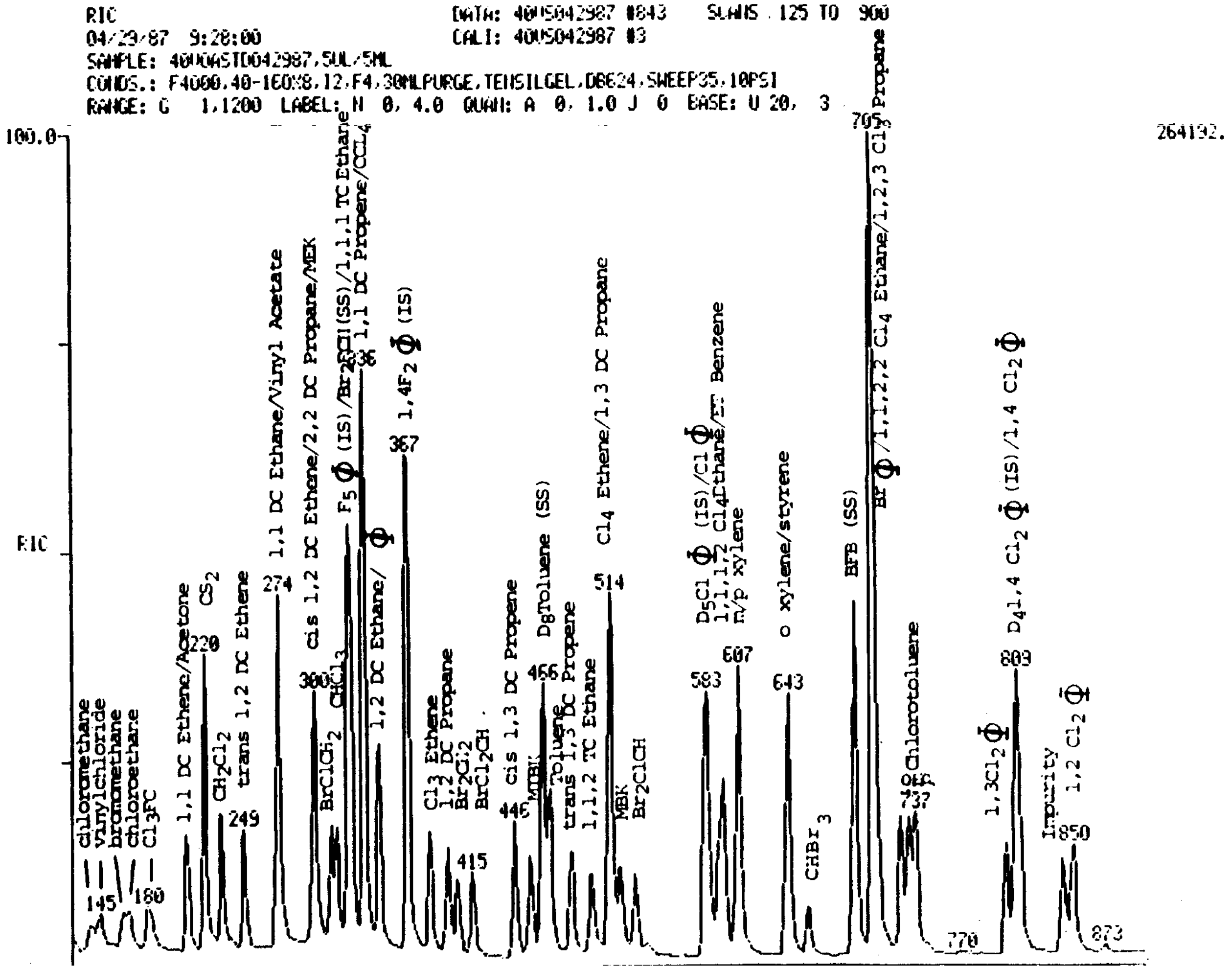
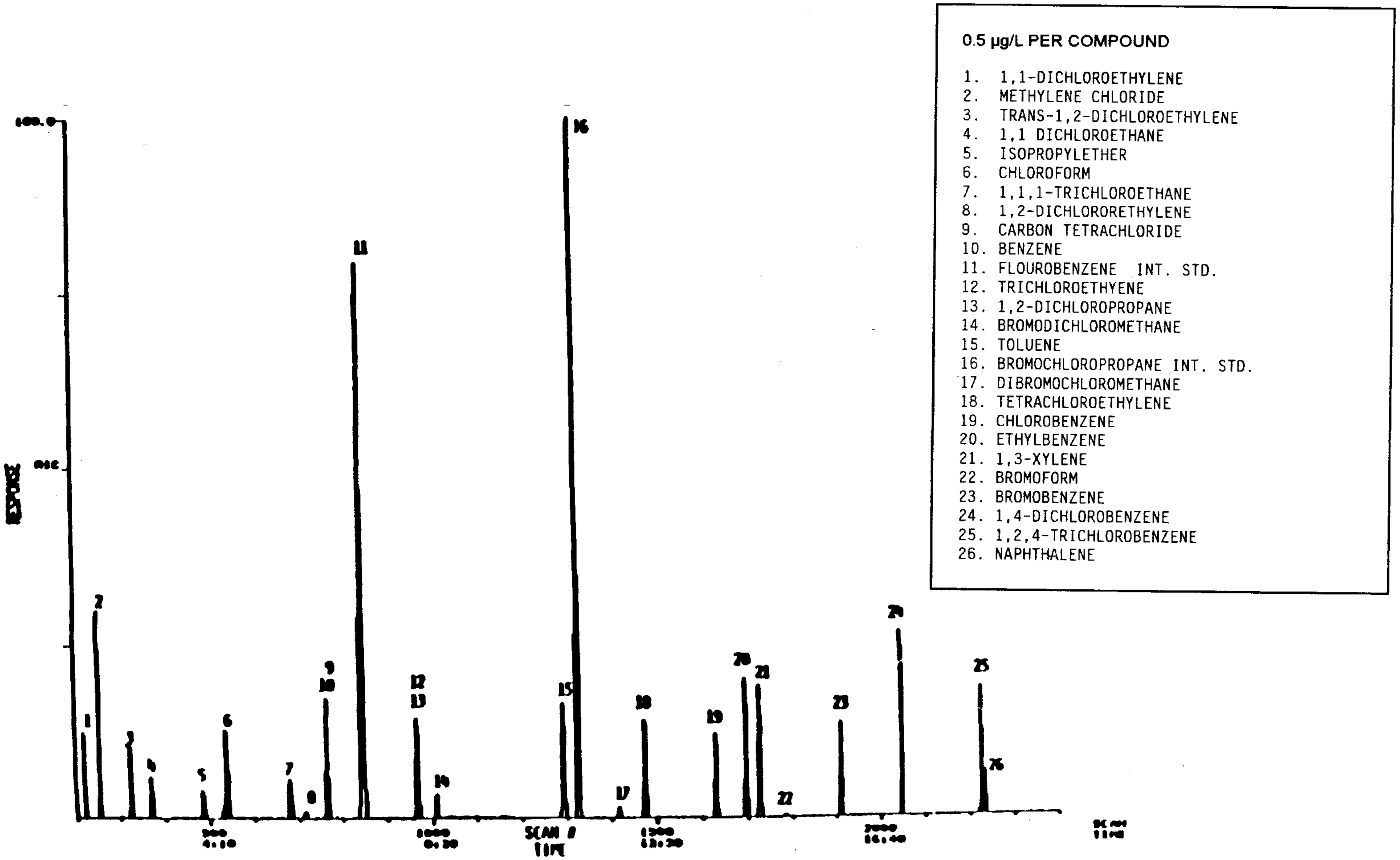
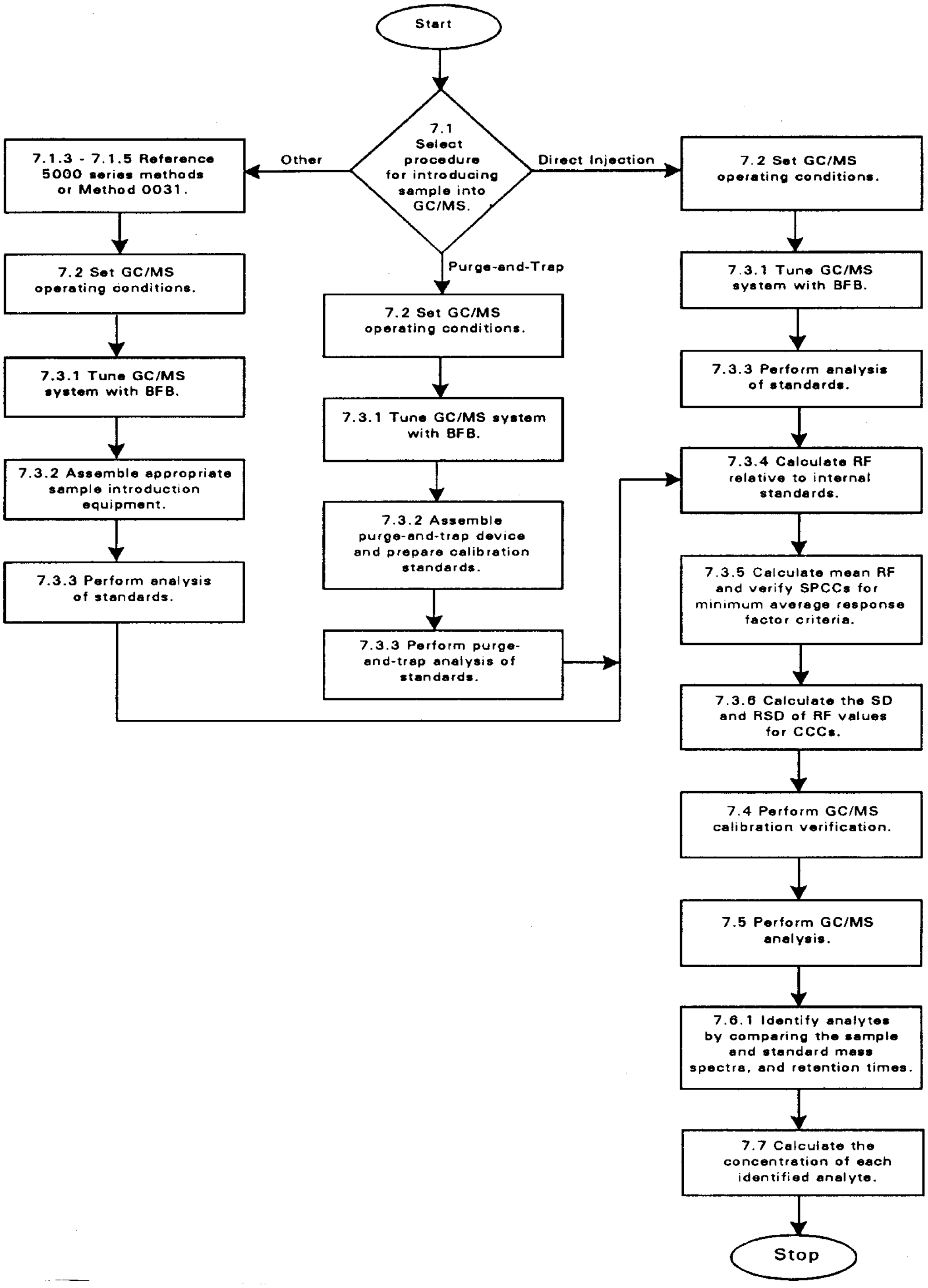


FIGURE 4
GAS CHROMATOGRAM OF TEST MIXTURE



0.5 µg/L PER COMPOUND

1. 1,1-DICHLOROETHYLENE
2. METHYLENE CHLORIDE
3. TRANS-1,2-DICHLOROETHYLENE
4. 1,1 DICHLOROETHANE
5. ISOPROPYLETHER
6. CHLOROFORM
7. 1,1,1-TRICHLOROETHANE
8. 1,2-DICHLORORETHYLENE
9. CARBON TETRACHLORIDE
10. BENZENE
11. FLOUROBENZENE INT. STD.
12. TRICHLOROETHYENE
13. 1,2-DICHLOROPROPANE
14. BROMODICHLOROMETHANE
15. TOLUENE
16. BROMOCHLOROPROPANE INT. STD.
17. DIBROMOCHLOROMETHANE
18. TETRACHLOROETHYLENE
19. CHLOROBENZENE
20. ETHYLBENZENE
21. 1,3-XYLENE
22. BROMOFORM
23. BROMOBENZENE
24. 1,4-DICHLOROBENZENE
25. 1,2,4-TRICHLOROBENZENE
26. NAPHTHALENE


Start
7.1 Select procedure for introducing sample into GC/MS.
Other
Direct Injection
Purge-and-Trap
7.1.3 - 7.1.5 Reference 5000 series methods or Method 0031.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble appropriate sample introduction equipment.
7.3.3 Perform analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble purge-and-trap device and prepare calibration standards.
7.3.3 Perform purge-and-trap analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.3 Perform analysis of standards.
7.3.4 Calculate RF relative to internal standards.
7.3.5 Calculate mean RF and verify SPCCs for minimum average response factor criteria.
7.3.6 Calculate the SD and RSD of RF values for CCCs.
7.4 Perform GC/MS calibration verification.
7.5 Perform GC/MS analysis.
7.6.1 Identify analytes by comparing the sample and standard mass spectra, and retention times.
7.7 Calculate the concentration of each identified analyte.
Stop

METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d$_{10}$ (IS)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
p-Benzoquinone	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC	319-84-6	X	X	X	X	X
β-BHC	319-85-7	X	X	X	X	X
δ-BHC	319-86-8	X	X	X	X	X
γ-BHC (Lindane)	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
Captafol	2425-06-1	HS(55)	ND	ND	ND	X
Captan	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl	63-25-2	X	ND	ND	ND	X
Carbofuran	1563-66-2	X	ND	ND	ND	X
Carbophenothion	786-19-6	X	ND	ND	ND	X
Chlordane (NOS)	57-74-9	X	X	X	X	X
Chlorfenvinphos	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methylaniline	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d_{12} (IS)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxyphos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
2,4-Diaminotoluene	95-80-7	DC,0E(42)	ND	ND	ND	X
Dibenz(a,j)acridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,h)anthracene	53-70-3	X	X	X	X	X
Dibenzofuran	132-64-9	X	X	ND	X	X
Dibenzo(a,e)pyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibromo-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl phthalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlorobenzene	95-50-1	X	X	X	X	X
1,3-Dichlorobenzene	541-73-1	X	X	X	X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X	X
1,4-Dichlorobenzene-d_4 (IS)		X	X	X	X	X
3,3'-Dichlorobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
Dichlorovos	62-73-7	X	ND	ND	ND	X
Dicrotophos	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phthalate	84-66-2	X	X	X	X	X
Diethylstilbestrol	56-53-1	AW,0S(67)	ND	ND	ND	X
Diethyl sulfate	64-67-5	LR	ND	ND	ND	LR
Dihydrosaffrole	56312-13-1	ND	ND	ND	ND	ND
Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethoxybenzidine	119-90-4	X	ND	ND	ND	LR
Dimethylaminoazobenzene	60-11-7	X	ND	ND	ND	X
7,12-Dimethylbenz(a)-anthracene	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethylbenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethylphenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
Dimethyl phthalate	131-11-3	X	X	X	X	X
1,2-Dinitrobenzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobenzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobenzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2-methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylamine	122-39-4	X	X	X	X	X
5,5-Diphenylhydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenylhydrazine	122-66-7	X	X	X	X	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
Di-n-octyl phthalate	117-84-0	X	X	X	X	X
Disulfoton	298-04-4	X	ND	ND	ND	X
Endosulfan I	959-98-8	X	X	X	X	X
Endosulfan II	33213-65-9	X	X	X	X	X
Endosulfan sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin aldehyde	7421-93-4	X	X	X	X	X
Endrin ketone	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl carbamate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl methanesulfonate	62-50-0	X	ND	ND	ND	X
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfothion	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloralin	33245-39-5	X	ND	ND	ND	X
Fluoranthene	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorobiphenyl (surr)	321-60-8	X	X	X	X	X
2-Fluorophenol (surr)	367-12-4	X	X	X	X	X
Heptachlor	76-44-8	X	X	X	X	X
Heptachlor epoxide	1024-57-3	X	X	X	X	X
Hexachlorobenzene	118-74-1	X	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X	X
Hexachlorocyclopentadiene	77-47-4	X	X	X	X	X
Hexachloroethane	67-72-1	X	X	X	X	X
Hexachlorophene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachloropropene	1888-71-7	X	ND	ND	ND	X
Hexamethylphosphoramide	680-31-9	X	ND	ND	ND	X
Hydroquinone	123-31-9	ND	ND	ND	ND	X
Indeno(1,2,3-cd)pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
Isosafrole	120-58-1	DC(46)	ND	ND	ND	X
Kepone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anhydride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrilene	91-80-5	X	ND	ND	ND	X
Methoxychlor	72-43-5	X	ND	ND	ND	X
3-Methylcholanthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methylenebis (2-chloroaniline)	101-14-4	OE,OS(0)	ND	ND	ND	LR

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
4,4'-Methylenebis (N,N-dimethylaniline)	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
Methyl parathion	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
Mevinphos	7786-34-7	X	ND	ND	ND	X
Mexacarbate	315-18-4	HE,HS(68)	ND	ND	ND	X
Mirex	2385-85-5	X	ND	ND	ND	X
Monocrotophos	6923-22-4	HE	ND	ND	ND	X
Naled	300-76-5	X	ND	ND	ND	X
Naphthalene	91-20-3	X	X	X	X	X
Naphthalene-d_8 (IS)		X	X	X	X	X
1,4-Naphthoquinone	130-15-4	X	ND	ND	ND	X
1-Naphthylamine	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d_5 (surr)		X	X	X	X	X
4-Nitrobiphenyl	92-93-3	X	ND	ND	ND	X
Nitrofen	1836-75-5	X	ND	ND	ND	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine	99-55-8	X	X	ND	ND	X
Nitroquinoline-1-oxide	56-57-5	X	ND	ND	ND	X
N-Nitrosodi-n-butylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethylamine	62-75-9	X	X	X	X	X
N-Nitrosomethylethylamine	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenylamine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramide	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline	101-80-4	X	ND	ND	ND	X

		Appropriate Preparation Techniques^b				
Compounds	CAS No^a	3510	3520	3540/ 3541	3550	3580
Parathion	56-38-2	X	X	ND	ND	X
Pentachlorobenzene	608-93-5	X	ND	ND	ND	X
Pentachloronitrobenzene	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d$_{12}$ (IS)		X	X	X	X	X
Phenacetin	62-44-2	X	ND	ND	ND	X
Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d$_{10}$ (IS)		X	X	X	X	X
Phenobarbital	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d$_6$ (surr)		DC(28)	X	X	X	X
1,4-Phenylenediamine	106-50-3	X	ND	ND	ND	X
Phorate	298-02-2	X	ND	ND	ND	X
Phosalone	2310-17-0	HS(65)	ND	ND	ND	X
Phosmet	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline (2-Methylpyridine)	109-06-8	X	X	ND	ND	ND
Piperonyl sulfoxide	120-62-7	X	ND	ND	ND	X
Pronamide	23950-58-5	X	ND	ND	ND	X
Propylthiouracil	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole	94-59-7	X	ND	ND	ND	X
Strychnine	57-24-9	AW,0S(55)	ND	ND	ND	X
Sulfallate	95-06-7	X	ND	ND	ND	X
Terbufos	13071-79-9	X	ND	ND	ND	X
Terphenyl-d$_{14}$(surr)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachlorophenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos	961-11-5	X	ND	ND	ND	X
Tetraethyl dithiopyrophosphate	3689-24-5	X	X	ND	ND	ND
Tetraethyl pyrophosphate	107-49-3	X	ND	ND	ND	X
Thionazine	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)	108-98-5	X	ND	ND	ND	X
Toluene diisocyanate	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine	95-53-4	X	ND	ND	ND	X
Toxaphene	8001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr)	118-79-6	X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X
Trifluralin	1582-09-8	X	ND	ND	ND	X

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
2,4,5-Trimethylaniline	137-17-7	X	ND	ND	ND	X
Trimethyl phosphate	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrobenzene	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibromopropyl) phosphate	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl phosphate	78-32-0	X	ND	ND	ND	X
O,O,O-Triethyl phosphorothioate	126-68-1	X	ND	ND	ND	X

[a] Chemical Abstract Service Registry Number
[b] See Sec. 1.2 for other acceptable preparation methods.

KEY TO ANALYTE LIST

IS = This compound may be used as an internal standard.
surr = This compound may be used as a surrogate.
AW = Adsorption to walls of glassware during extraction and storage.
CP = Nonreproducible chromatographic performance.
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery).
HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
HS = Hydrolysis during storage (number in parenthesis is percent stability).
LR = Low response.
ND = Not determined.
OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
OS = Oxidation during storage (number in parenthesis is percent stability).
X = Greater than 70 percent recovery by this technique.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid extraction of solids for PAHs (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 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.

1.4.7 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. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less then 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 2). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

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 (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

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 a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm ID) 1 µm film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 3 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have 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 should also be available 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 should also be available.

4.1.6 Guard column (optional) - (J&W Deactivated Fused Silica, 0.25 mm ID x 6 m, or equivalent) between the injection port and the analytical column joined with column joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10-µL.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be 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.

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

5.3 Stock standard solutions (1000 mg/L) - Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 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 should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.3.4 It is recommended that nitrosamine compounds be placed together in a separate calibration mix and not combined with other calibration mixes. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.3.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analytical difficulties. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.4 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (see Table 5). Other compounds may be used as internal standards as long as the requirements given in Sec. 7.3.2 are met.

5.4.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfer to a 50 mL volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene-d_{12}. The resulting solution will contain each standard at a concentration of 4,000 ng/µL. Each 1 mL sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not in use. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute internal standard solution may be required. Area counts of the internal standard peaks should be between 50-200% of the area of the target analytes in the mid-point calibration analysis.

5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at -10°C or less when not in use. If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute tuning solution may be necessary. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method.

5.6.1 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.6.2 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the internal standard solution prior to analysis. All standards should be stored at -10°C or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at 4°C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 Surrogate Standard Check: Determine what the appropriate concentration should be for the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

<u>NOTE</u>: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

5.7.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute surrogate solution may be necessary.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS).

5.8.1 Matrix Spike Check: Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

5.8.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute matrix and LCS spiking solution may be necessary.

5.8.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis.

Matrix	Methods
Air	3542
Water	3510, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3550, 3560, 3561
Waste	3540, 3541, 3545, 3550, 3560, 3561, 3580

7.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10-µL syringe may be appropriate. The detection limit is very high (approximately 10,000 µg/L). Therefore, it is only permitted where concentrations in excess of 10,000 µg/L are expected.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

Analytes of interest	Methods
Aniline & aniline derivatives	3620
Phenols	3630, 3640, 8041[a]
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3610, 3620, 3630, 3660, 3665
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620

Analytes of interest	Methods
Petroleum waste	3611, 3650
All base, neutral, and acid priority pollutants	3640

[a] Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	270°C, hold until benzo[g,h,i]perylene elutes
Injector temperature:	250-300°C
Transfer line temperature:	250-300°C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	1-2 µL
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. (See Sec. 8.0 of Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection port and the analytical column may help prolong analytical column performance.

7.3.2 The internal standards selected in Sec. 5.4 should 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 Table 1). 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).

7.3.3 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and Method 8000). The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

Calculate response factors (RFs) for each target analyte relative to one of the internal standards as follows:

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

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) 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.

7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. These SPCCs 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.

7.3.4.3 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. This check must be met before sample analysis begins.

7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are 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 using one of the approaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC (see Table 4) must be less than or equal to 30%.

$$\text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

7.3.5.3 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 beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000.

7.3.6 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

7.3.7 Linearity of target analytes - If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Sec. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 in Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the 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. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000B for method blank performance criteria.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 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. This check must be met before sample analysis begins.

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the 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. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC 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 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 included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification 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, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification 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, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard solution to the 1-mL concentrated sample extract obtained from sample preparation.

7.5.3 Inject a 1-2 µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 7.3). The volume to be injected should contain 100 ng of base/neutral and 200 ng of acid surrogates (assuming 100% recovery), unless a more sensitive GC/MS system is being used and the surrogate solution is less concentrated then that listed in Sec. 5.7. The injection volume must be the same volume used for the calibration standards.

7.5.4 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. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 ng/µL, unless a more sensitive GC/MS system is being used).

<u>NOTE:</u> It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 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 generated by the laboratory 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. Compounds are identified when the following criteria are met.

7.6.1.1 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 will be accepted as meeting this criterion.

7.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 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%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be 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. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

7.6.1.5 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.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 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 will be determined by the purpose of the analyses being conducted. Data system library search routines should 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) should be present in the sample spectrum.

(2) The relative intensities of the major ions should 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 should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be 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.

7.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/µL in the concentrated sample extract.

7.7.6 Structural isomers that produce very similar mass spectra should be quantitated 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 quantitated as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be summed and reported as the sum of both compounds.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Sec. 7.0 of Method 8000 and include calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8270:

8.2.1 The GC/MS system must be tuned to meet the DFTPP criteria listed in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 7.4, each 12 hours.

8.2.4 The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 7.6.1.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be 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. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

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

9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 8.

9.3 Method performance data (using Method 3541 Automated Soxhlet extraction) are presented in Table 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately 1 hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three determinations were performed and each extract was analyzed by gas chromatography/ mass spectrometry following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by Method 8270.

9.5 Single laboratory precision and bias data (using Method 3545 Accelerated Solvent Extraction) for semivolatile organic compounds are presented in Table 12. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by Perstorp Environmental Soxtec™ (automated Soxhlet). The data presented in Table 12 represents seven replicate extractions and analyses for each individual sample and were taken from reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low-101.2%, mid-97.2% and high-99.2%.

9.6 Single laboratory precision and bias data (using Method 3561 SFE Extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (one, EC-1, a lake sediment from Environment Canada and the other, HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally-contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions range from 85 to 148% (overall average of 100%) based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions range from 73 to 133% (overall average of 92%) based on the certified value for the marine sediment. The data are found in Tables 13 and 14 and were taken from Reference 10.

9.7 Single laboratory precision and accuracy data (using Method 3561 SFE Extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions range from 60 to 122% (overall average of 89%) based on the certified value. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO_2; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

10.0 REFERENCES

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TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75[a]	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d_4 (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d_8 (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65[a]	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d_{10} (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00[a]	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramide	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54[a]	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d_{10} (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramide	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α-Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d_{12} (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d_5 (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d_6 (surr)	--	99	42,71
Terphenyl-d_{14} (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard
surr = surrogate
[a] Estimated retention times
[b] Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

	Estimated Quantitation Limits[a]	
Compound	Ground water µg/L	Low Soil/Sediment[b] µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	 Low Soil/Sediment[b] µg/kg
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	 Low Soil/Sediment[b] µg/kg
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	 Low Soil/Sediment[b] µg/kg
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodi-n-butylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramide	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	 Low Soil/Sediment[b] µg/kg
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
O,O,O-Triethyl phosphorothioate	NT	ND

[a] Sample EQLs are highly matrix-dependent. The EQLs listed here are provided for guidance and may not always be achievable.

[b] EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined
NA = Not Applicable
NT = Not Tested

<u>Other Matrices</u>	<u>Factor</u>[c]
High-concentration soil and sludges by ultrasonic extractor	7.5
Non-water miscible waste	75

[c] EQL = (EQL for Low Soil/Sediment given above in Table 2) x (Factor)

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a] Data taken from Reference 3.

[b] Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4

CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d_8 (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

TABLE 5
(continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethylaminoazobenzene	
Hexachlorobenzene	Pyrene	
N-Nitrosodiphenylamine	Terphenyl-d_{14} (surr)	
Pentachlorophenol	7,12-Dimethylbenz(a)anthracene	
Pentachloronitrobenzene	Di-n-octyl phthalate	
Phenacetin	Indeno(1,2,3-cd) pyrene	
Phenanthrene	3-Methylcholanthrene	
Pronamide		

(surr) = surrogate

TABLE 6

MULTILABORATORY PERFORMANCE DATA[a]

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192

TABLE 6

(continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L

p, p_s = Measured percent recovery

D = Detected; result must be greater than zero

[a] Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. <u>These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.</u>

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Acenaphthene	0.96C+0.19	$0.15\bar{x}-0.12$	$0.21\bar{x}-0.67$
Acenaphthylene	0.89C+0.74	$0.24\bar{x}-1.06$	$0.26\bar{x}-0.54$
Aldrin	0.78C+1.66	$0.27\bar{x}-1.28$	$0.43\bar{x}+1.13$
Anthracene	0.80C+0.68	$0.21\bar{x}-0.32$	$0.27\bar{x}-0.64$
Benz(a)anthracene	0.88C-0.60	$0.15\bar{x}+0.93$	$0.26\bar{x}-0.21$
Benzo(b)fluoranthene	0.93C-1.80	$0.22\bar{x}+0.43$	$0.29\bar{x}+0.96$
Benzo(k)fluoranthene	0.87C-1.56	$0.19\bar{x}+1.03$	$0.35\bar{x}+0.40$
Benzo(a)pyrene	0.90C-0.13	$0.22\bar{x}+0.48$	$0.32\bar{x}+1.35$
Benzo(g,h,i)perylene	0.98C-0.86	$0.29\bar{x}+2.40$	$0.51\bar{x}-0.44$
Benzyl butyl phthalate	0.66C-1.68	$0.18\bar{x}+0.94$	$0.53\bar{x}+0.92$
β-BHC	0.87C-0.94	$0.20\bar{x}-0.58$	$0.30\bar{x}+1.94$
δ-BHC	0.29C-1.09	$0.34\bar{x}+0.86$	$0.93\bar{x}-0.17$
Bis(2-chloroethyl) ether	0.86C-1.54	$0.35\bar{x}-0.99$	$0.35\bar{x}+0.10$
Bis(2-chloroethoxy)methane	1.12C-5.04	$0.16\bar{x}+1.34$	$0.26\bar{x}+2.01$
Bis(2-chloroisopropyl) ether	1.03C-2.31	$0.24\bar{x}+0.28$	$0.25\bar{x}+1.04$
Bis(2-ethylhexyl) phthalate	0.84C-1.18	$0.26\bar{x}+0.73$	$0.36\bar{x}+0.67$
4-Bromophenyl phenyl ether	0.91C-1.34	$0.13\bar{x}+0.66$	$0.16\bar{x}+0.66$
2-Chloronaphthalene	0.89C+0.01	$0.07\bar{x}+0.52$	$0.13\bar{x}+0.34$
4-Chlorophenyl phenyl ether	0.91C+0.53	$0.20\bar{x}-0.94$	$0.30\bar{x}-0.46$
Chrysene	0.93C-1.00	$0.28\bar{x}+0.13$	$0.33\bar{x}-0.09$
4,4'-DDD	0.56C-0.40	$0.29\bar{x}-0.32$	$0.66\bar{x}-0.96$
4,4'-DDE	0.70C-0.54	$0.26\bar{x}-1.17$	$0.39\bar{x}-1.04$
4,4'-DDT	0.79C-3.28	$0.42\bar{x}+0.19$	$0.65\bar{x}-0.58$
Dibenzo(a,h)anthracene	0.88C+4.72	$0.30\bar{x}+8.51$	$0.59\bar{x}+0.25$
Di-n-butyl phthalate	0.59C+0.71	$0.13\bar{x}+1.16$	$0.39\bar{x}+0.60$
1,2-Dichlorobenzene	0.80C+0.28	$0.20\bar{x}+0.47$	$0.24\bar{x}+0.39$
1,3-Dichlorobenzene	0.86C-0.70	$0.25\bar{x}+0.68$	$0.41\bar{x}+0.11$
1,4-Dichlorobenzene	0.73C-1.47	$0.24\bar{x}+0.23$	$0.29\bar{x}+0.36$
3,3'-Dichlorobenzidine	1.23C-12.65	$0.28\bar{x}+7.33$	$0.47\bar{x}+3.45$
Dieldrin	0.82C-0.16	$0.20\bar{x}-0.16$	$0.26\bar{x}-0.07$
Diethyl phthalate	0.43C+1.00	$0.28\bar{x}+1.44$	$0.52\bar{x}+0.22$
Dimethyl phthalate	0.20C+1.03	$0.54\bar{x}+0.19$	$1.05\bar{x}-0.92$
2,4-Dinitrotoluene	0.92C-4.81	$0.12\bar{x}+1.06$	$0.21\bar{x}+1.50$
2,6-Dinitrotoluene	1.06C-3.60	$0.14\bar{x}+1.26$	$0.19\bar{x}+0.35$
Di-n-octyl phthalate	0.76C-0.79	$0.21\bar{x}+1.19$	$0.37\bar{x}+1.19$
Endosulfan sulfate	0.39C+0.41	$0.12\bar{x}+2.47$	$0.63\bar{x}-1.03$
Endrin aldehyde	0.76C-3.86	$0.18\bar{x}+3.91$	$0.73\bar{x}-0.62$
Fluoranthene	0.81C+1.10	$0.22\bar{x}-0.73$	$0.28\bar{x}-0.60$
Fluorene	0.90C-0.00	$0.12\bar{x}+0.26$	$0.13\bar{x}+0.61$
Heptachlor	0.87C-2.97	$0.24\bar{x}-0.56$	$0.50\bar{x}-0.23$
Heptachlor epoxide	0.92C-1.87	$0.33\bar{x}-0.46$	$0.28\bar{x}+0.64$

TABLE 7
(continued)

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Hexachlorobenzene	0.74C+0.66	0.18$\bar{x}$-0.10	0.43$\bar{x}$-0.52
Hexachlorobutadiene	0.71C-1.01	0.19$\bar{x}$+0.92	0.26$\bar{x}$+0.49
Hexachloroethane	0.73C-0.83	0.17$\bar{x}$+0.67	0.17$\bar{x}$+0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29$\bar{x}$+1.46	0.50$\bar{x}$-0.44
Isophorone	1.12C+1.41	0.27$\bar{x}$+0.77	0.33$\bar{x}$+0.26
Naphthalene	0.76C+1.58	0.21$\bar{x}$-0.41	0.30$\bar{x}$-0.68
Nitrobenzene	1.09C-3.05	0.19$\bar{x}$+0.92	0.27$\bar{x}$+0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27$\bar{x}$+0.68	0.44$\bar{x}$+0.47
Aroclor 1260	0.81C-10.86	0.35$\bar{x}$+3.61	0.43$\bar{x}$+1.82
Phenanthrene	0.87C+0.06	0.12$\bar{x}$+0.57	0.15$\bar{x}$+0.25
Pyrene	0.84C-0.16	0.16$\bar{x}$+0.06	0.15$\bar{x}$+0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15$\bar{x}$+0.85	0.21$\bar{x}$+0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23$\bar{x}$+0.75	0.29$\bar{x}$+1.31
2-Chlorophenol	0.78C+0.29	0.18$\bar{x}$+1.46	0.28$\bar{x}$+0.97
2,4-Dichlorophenol	0.87C-0.13	0.15$\bar{x}$+1.25	0.21$\bar{x}$+1.28
2,4-Dimethylphenol	0.71C+4.41	0.16$\bar{x}$+1.21	0.22$\bar{x}$+1.31
2,4-Dinitrophenol	0.81C-18.04	0.38$\bar{x}$+2.36	0.42$\bar{x}$+26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10$\bar{x}$+42.29	0.26$\bar{x}$+23.10
2-Nitrophenol	0.07C-1.15	0.16$\bar{x}$+1.94	0.27$\bar{x}$+2.60
4-Nitrophenol	0.61C-1.22	0.38$\bar{x}$+2.57	0.44$\bar{x}$+3.24
Pentachlorophenol	0.93C+1.99	0.24$\bar{x}$+3.03	0.30$\bar{x}$+4.33
Phenol	0.43C+1.26	0.26$\bar{x}$+0.73	0.35$\bar{x}$+0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16$\bar{x}$+2.22	0.22$\bar{x}$+1.81

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{x}$, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{x}$, in µg/L.

C = True value for the concentration, in µg/L.

$\bar{x}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

[a] Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 8

EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

Compound	Percent Recovery on Day 0		Percent Recovery on Day 7	
	Mean	RSD	Mean	RSD
3-Amino-9-ethylcarbazole	80	8	73	3
4-Chloro-1,2-phenylenediamine	91	1	108	4
4-Chloro-1,3-phenylenediamine	84	3	70	3
1,2-Dibromo-3-chloropropane	97	2	98	5
Dinoseb	99	3	97	6
Parathion	100	2	103	4
4,4'-Methylenebis(N,N-dimethylaniline)	108	4	90	4
5-Nitro-o-toluidine	99	10	93	4
2-Picoline	80	4	83	4
Tetraethyl dithiopyrophosphate	92	7	70	1

Data taken from Reference 6.

TABLE 9

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANICS FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)[a]

	Clay Soil		Topsoil	
Compound	Mean Recovery	RSD	Mean Recovery	RSD
1,3-Dichlorobenzene	0	--	0	--
1,2-Dichlorobenzene	0	--	0	--
Nitrobenzene	0	--	0	--
Benzal chloride	0	--	0	--
Benzotrichloride	0	--	0	--
4-Chloro-2-nitrotoluene	0	--	0	--
Hexachlorocyclopentadiene	4.1	15	7.8	23
2,4-Dichloronitrobenzene	35.2	7.6	21.2	15
3,4-Dichloronitrobenzene	34.9	15	20.4	11
Pentachlorobenzene	13.7	7.3	14.8	13
2,3,4,5-Tetrachloronitrobenzene	55.9	6.7	50.4	6.0
Benefin	62.6	4.8	62.7	2.9
alpha-BHC	58.2	7.3	54.8	4.8
Hexachlorobenzene	26.9	13	25.1	5.7
delta-BHC	95.8	4.6	99.2	1.3
Heptachlor	46.9	9.2	49.1	6.3
Aldrin	97.7	12	102	7.4
Isopropalin	102	4.3	105	2.3
Heptachlor epoxide	90.4	4.4	93.6	2.4
trans-Chlordane	90.1	4.5	95.0	2.3
Endosulfan I	96.3	4.4	101	2.2
Dieldrin	129	4.7	104	1.9
2,5-Dichlorophenyl-4-nitrophenyl ether	110	4.1	112	2.1
Endrin	102	4.5	106	3.7
Endosulfan II	104	4.1	105	0.4
p,p'-DDT	134	2.1	111	2.0
2,3,6-Trichlorophenyl-4'-nitrophenyl ether	110	4.8	110	2.8
2,3,4-Trichlorophenyl-4'-nitrophenyl ether	112	4.4	112	3.3
Mirex	104	5.3	108	2.2

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[a] The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 10

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY AUTOMATED SOXHLET (METHOD 3541)[a]

Compound	Mean Recovery	RSD
Phenol	47.8	5.6
Bis(2-chloroethyl)ether	25.4	13
2-Chlorophenol	42.7	4.3
Benzyl alcohol	55.9	7.2
2-Methylphenol	17.6	6.6
Bis(2-chloroisopropyl)ether	15.0	15
4-Methylphenol	23.4	6.7
N-Nitroso-di-n-propylamine	41.4	6.2
Nitrobenzene	28.2	7.7
Isophorone	56.1	4.2
2-Nitrophenol	36.0	6.5
2,4-Dimethylphenol	50.1	5.7
Benzoic acid	40.6	7.7
Bis(2-chloroethoxy)methane	44.1	3.0
2,4-Dichlorophenol	55.6	4.6
1,2,4-Trichlorobenzene	18.1	31
Naphthalene	26.2	15
4-Chloroaniline	55.7	12
4-Chloro-3-methylphenol	65.1	5.1
2-Methylnaphthalene	47.0	8.6
Hexachlorocyclopentadiene	19.3	19
2,4,6-Trichlorophenol	70.2	6.3
2,4,5-Trichlorophenol	26.8	2.9
2-Chloronaphthalene	61.2	6.0
2-Nitroaniline	73.8	6.0
Dimethyl phthalate	74.6	5.2
Acenaphthylene	71.6	5.7
3-Nitroaniline	77.6	5.3
Acenaphthene	79.2	4.0
2,4-Dinitrophenol	91.9	8.9
4-Nitrophenol	62.9	16
Dibenzofuran	82.1	5.9
2,4-Dinitrotoluene	84.2	5.4
2,6-Dinitrotoluene	68.3	5.8
Diethyl phthalate	74.9	5.4
4-Chlorophenyl-phenyl ether	67.2	3.2
Fluorene	82.1	3.4
4-Nitroaniline	79.0	7.9

TABLE 10
(continued)

Compound	Mean Recovery	RSD
4,6-Dinitro-2-methylphenol	63.4	6.8
N-Nitrosodiphenylamine	77.0	3.4
4-Bromophenyl-phenyl ether	62.4	3.0
Hexachlorobenzene	72.6	3.7
Pentachlorophenol	62.7	6.1
Phenanthrene	83.9	5.4
Anthracene	96.3	3.9
Di-n-butyl phthalate	78.3	40
Fluoranthene	87.7	6.9
Pyrene	102	0.8
Butyl benzyl phthalate	66.3	5.2
3,3'-Dichlorobenzidine	25.2	11
Benzo(a)anthracene	73.4	3.8
Bis(2-ethylhexyl) phthalate	77.2	4.8
Chrysene	76.2	4.4
Di-n-octyl phthalate	83.1	4.8
Benzo(b)fluoranthene	82.7	5.0
Benzo(k)fluoranthene	71.7	4.1
Benzo(a)pyrene	71.7	4.1
Indeno(1,2,3-cd)pyrene	72.2	4.3
Dibenzo(a,h)anthracene	66.7	6.3
Benzo(g,h,i)perylene	63.9	8.0
1,2-Dichlorobenzene	0	--
1,3-Dichlorobenzene	0	--
1,4-Dichlorobenzene	0	--
Hexachloroethane	0	--
Hexachlorobutadiene	0	--

[a] Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

TABLE 11

PRECISION AND BIAS VALUES FOR METHOD 3542[1]

Compound	Mean Recovery	Standard Deviation	Relative Standard Deviation Percent
2-Fluorophenol	74.6	28.6	38.3
Phenol-d_5	77.8	27.7	35.6
Nitrobenzene-d_5	65.6	32.5	49.6
2-Fluorobiphenyl	75.9	30.3	39.9
2,4,6-Tribromophenol	67.0	34.0	50.7
Terphenyl-d_{14}	78.6	32.4	41.3

[1] The surrogate values shown in Table 11 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Phenol	93.3	78.7	135.9	73.9	82.8	124.6	108.8	130.6	89.7	**102.0**
Bis(2-chloroethyl) ether	102.1	85.1	109.1	96.0	88.0	103.6	122.3	119.9	90.8	**101.9**
2-Chlorophenol	100.8	82.6	115.0	93.8	88.9	111.1	115.0	115.3	91.9	**101.6**
1,3-Dichlorobenzene	127.7	129.7	110.0	*364.2	129.9	119.0	*241.3	*163.7	107.1	**120.6**
1,4-Dichlorobenzene	127.9	127.0	110.5	*365.9	127.8	116.4	*309.6	*164.1	105.8	**119.2**
1,2-Dichlorobenzene	116.8	115.8	101.3	*159.2	113.4	105.5	*189.3	134.0	100.4	**112.5**
2-Methylphenol	98.9	82.1	119.7	87.6	89.4	111.0	133.2	128.0	92.1	**104.7**
Bis(2-chloroisopropyl)ether	109.4	71.5	108.0	81.8	81.0	88.6	118.1	148.3	94.8	**100.2**
o-Toluidine	100.0	89.7	117.2	100.0	*152.5	120.3	100.0	*199.5	102.7	**110.3**
N-Nitroso-di-n-propylamine	103.0	79.1	107.7	83.9	88.1	96.2	109.9	123.3	91.4	**98.1**
Hexachloroethane	97.1	125.1	111.0	*245.4	117.1	128.1	*566.7	147.9	103.7	**118.6**
Nitrobenzene	104.8	82.4	106.6	86.8	84.6	101.7	119.7	122.1	93.3	**100.2**
Isophorone	100.0	86.4	98.2	87.1	87.5	109.7	135.5	118.4	92.7	**101.7**
2,4-Dimethylphenol	100.0	104.5	140.0	100.0	114.4	123.1	100.0	*180.6	96.3	**109.8**
2-Nitrophenol	80.7	80.5	107.9	91.4	86.7	103.2	122.1	107.1	87.0	**96.3**
Bis(chloroethoxy)methane	94.4	80.6	94.7	86.5	84.4	99.6	130.6	110.7	93.2	**97.2**
2,4-Dichlorophenol	88.9	87.8	111.4	85.9	87.6	103.5	123.3	107.0	92.1	**98.6**
1,2,4-Trichlorobenzene	98.0	97.8	98.8	123.0	93.7	94.5	137.0	99.4	95.3	**104.2**
Naphthalene	101.7	97.2	123.6	113.2	102.9	129.5	*174.5	114.0	89.8	**106.1**
4-Chloroaniline	100.0	*150.2	*162.4	100.0	125.5	*263.6	100.0	*250.8	114.9	**108.1**
Hexachlorobutadiene	101.1	98.7	102.2	124.1	90.3	98.0	134.9	96.1	96.8	**104.7**
4-Chloro-3-methylphenol	90.4	80.2	114.7	79.0	85.2	109.8	131.6	116.2	90.1	**99.7**
2-Methylnaphthalene	93.2	89.9	94.6	104.1	92.2	105.9	146.2	99.1	93.3	**102.1**
Hexachlorocyclopentadiene	100.0	100.0	0.0	100.0	100.0	6.8	100.0	100.0	*238.3	**75.8**
2,4,6-Trichlorophenol	94.6	90.0	112.0	84.2	91.2	103.6	101.6	95.9	89.8	**95.9**
2,4,5-Trichlorophenol	84.4	91.9	109.6	96.1	80.7	103.6	108.9	83.9	87.9	**94.1**
2-Chloronaphthalene	100.0	91.3	93.6	97.6	93.4	98.3	106.8	93.0	92.0	**96.2**
2-Nitroaniline	90.0	83.4	97.4	71.3	88.4	89.9	112.1	113.3	87.7	**92.6**
2,6-Dinitrotoluene	83.1	90.6	91.6	86.4	90.6	90.3	104.3	84.7	90.9	**90.3**
Acenaphthylene	104.9	95.9	100.5	99.0	97.9	108.8	118.5	97.8	92.0	**101.7**
3-Nitroaniline	*224.0	115.6	97.6	100.0	111.8	107.8	0.0	111.7	99.0	**92.9**
Acenaphthene	102.1	92.6	97.6	97.2	96.9	104.4	114.2	92.0	89.0	**98.4**
4-Nitrophenol	0.0	93.2	121.5	18.1	87.1	116.6	69.1	90.5	84.5	**75.6**
2,4-Dinitrotoluene	73.9	91.9	100.2	84.7	93.8	98.9	100.9	84.3	87.3	**90.7**

TABLE 12 (cont.)

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Dibenzofuran	89.5	91.7	109.3	98.5	92.2	111.4	113.8	92.7	90.4	**98.8**
4-Chlorophenyl phenyl ether	83.0	94.5	98.7	95.7	94.3	94.2	111.4	87.7	90.3	**94.4**
Fluorene	85.2	94.9	89.2	102.0	95.5	93.8	121.3	85.7	90.9	**95.4**
4-Nitroaniline	77.8	114.8	94.5	129.6	103.6	95.4	*154.1	89.3	87.5	**99.1**
N-Nitrosodiphenylamine	82.6	96.7	93.8	92.9	93.4	116.4	97.5	110.9	86.7	**96.8**
4-Bromophenyl phenyl ether	85.6	92.9	92.8	91.1	107.6	89.4	118.0	97.5	87.1	**95.8**
Hexachlorobenzene	95.4	91.7	92.3	95.4	93.6	83.7	106.8	94.3	90.0	**93.7**
Pentachlorophenol	68.2	85.9	107.7	53.2	89.8	88.1	96.6	59.8	81.3	**81.2**
Phenanthrene	92.1	93.7	93.3	100.0	97.8	113.3	124.4	101.0	89.9	**100.6**
Anthracene	101.6	95.0	93.5	92.5	101.8	118.4	123.0	94.5	90.6	**101.2**
Carbazole	94.4	99.3	96.6	105.5	96.7	111.4	115.7	83.2	88.9	**99.1**
Fluoranthene	109.9	101.4	94.3	111.6	96.6	109.6	123.2	85.4	92.7	**102.7**
Pyrene	106.5	105.8	107.6	116.7	90.7	127.5	103.4	95.5	93.2	**105.2**
3,3'-Dichlorobenzidine	100.0	*492.3	131.4	100.0	*217.6	*167.6	100.0	*748.8	100.0	**116.5**
Benzo(a)anthracene	98.1	107.0	98.4	119.3	98.6	104.0	105.0	93.4	89.3	**101.5**
Chrysene	100.0	108.5	100.2	116.8	93.0	117.0	106.7	93.6	90.2	**102.9**
Benzo(b)fluoranthene	106.6	109.9	75.6	121.7	100.7	93.9	106.9	81.9	93.6	**99.0**
Benzo(k)fluoranthene	102.4	105.2	88.4	125.5	99.4	95.1	144.7	89.2	78.1	**103.1**
Benzo(a)pyrene	107.9	105.5	80.8	122.3	97.7	104.6	101.7	86.2	92.0	**99.9**
Indeno(1,2,3-cd)pyrene	95.1	105.7	93.8	126.0	105.2	90.4	133.6	82.6	91.9	**102.7**
Dibenz(a,h)anthracene	85.0	102.6	82.0	118.8	100.7	91.9	142.3	71.0	93.1	**98.6**
Benzo(g,h,i)perylene	98.0	0.0	81.2	0.0	33.6	78.6	128.7	83.0	94.2	**66.4**
Average	**95.1**	**94.3**	**101.0**	**95.5**	**96.5**	**104.1**	**113.0**	**100.9**	**92.5**	

* Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	(27.9)[b]	41.3 ± 3.6	(148)	8.7
Acenaphthylene	(0.8)	0.9 ± 0.1	(112)	11.1
Acenaphthene	(0.2)	0.2 ± 0.01	(100)	0.05
Fluorene	(15.3)	15.6 ± 1.8	(102)	11.5
Phenanthrene	15.8 ± 1.2	16.1 ± 1.8	102	11.2
Anthracene	(1.3)	1.1 ± 0.2	(88)	18.2
Fluoranthene	23.2 ± 2.0	24.1 ± 2.1	104	8.7
Pyrene	16.7 ± 2.0	17.2 ± 1.9	103	11.0
Benz(a)anthracene	8.7 ± 0.8	8.8 ± 1.0	101	11.4
Chrysene	(9.2)	7.9 ± 0.9	(86)	11.4
Benzo(b)fluoranthene	7.9 ± 0.9	8.5 ± 1.1	108	12.9
Benzo(k)fluoranthene	4.4 ± 0.5	4.1 ± 0.5	91	12.2
Benzo(a)pyrene	5.3 ± 0.7	5.1 ± 0.6	96	11.8
Indeno(1,2,3-cd)pyrene	5.7 ± 0.6	5.2 ± 0.6	91	11.5
Benzo(g,h,i)perylene	4.9 ± 0.7	4.3 ± 0.5	88	11.6
Dibenz(a,h)anthracene	(1.3)	1.1 ± 0.2	(85)	18.2

[a] Relative standard deviations for the SFE values are based on six replicate extractions.

[b] Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	9.0 ± 0.7	7.4 ± 0.6	82	8.1
Acenaphthylene	0.3 ± 0.1	0.4 ± 0.1	133	25.0
Acenaphthene	4.5 ± 1.5	3.3 ± 0.3	73	9.0
Fluorene	13.6 ± 3.1	10.4 ± 1.3	77	12.5
Phenanthrene	85.0 ± 20.0	86.2 ± 9.5	101	11.0
Anthracene	13.4 ± 0.5	12.1 ± 1.5	90	12.4
Fluoranthene	60.0 ± 9.0	54.0 ± 6.1	90	11.3
Pyrene	39.0 ± 9.0	32.7 ± 3.7	84	11.3
Benz(a)anthracene	14.6 ± 2.0	12.1 ± 1.3	83	10.7
Chrysene	14.1 ± 2.0	12.0 ± 1.3	85	10.8
Benzo(b)fluoranthene	7.7 ± 1.2	8.4 ± 0.9	109	10.7
Benzo(k)fluoranthene	2.8 ± 2.0	3.2 ± 0.5	114	15.6
Benzo(a)pyrene	7.4 ± 3.6	6.6 ± 0.8	89	12.1
Indeno(1,2,3-cd)pyrene	5.0 ± 2.0	4.5 ± 0.6	90	13.3
Benzo(g,h,i)perylene	5.4 ± 1.3	4.4 ± 0.6	82	13.6
Dibenz(a,h)anthracene	1.3 ± 0.5	1.1 ± 0.3	85	27.3

[a] Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 15

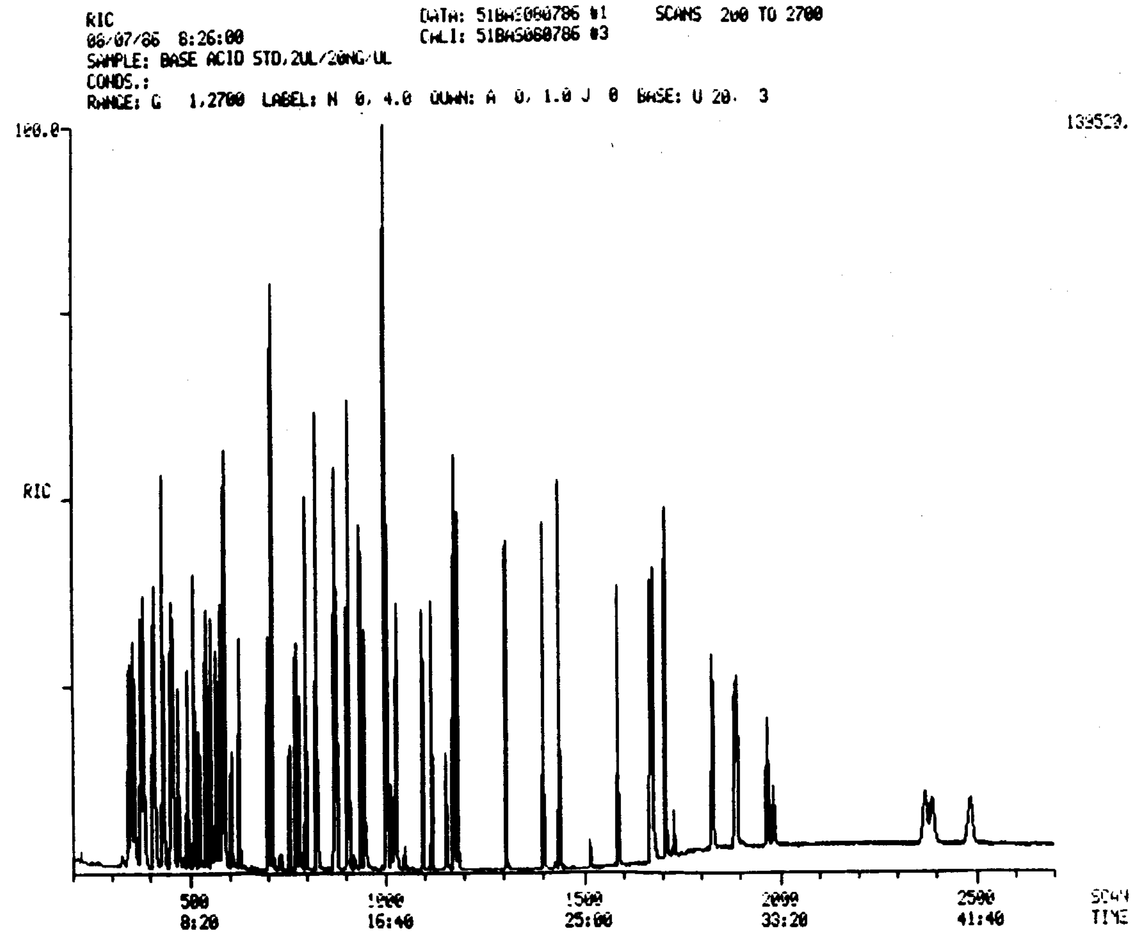
SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561 (SFE - LIQUID TRAP)

Compound	Certified Value (mg/kg)			SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	32.4	±	8.2	29.55	91	10.5
2-Methylnaphthalene	62.1	±	11.5	76.13	122	2.0
Acenaphthene	632	±	105	577.28	91	2.9
Dibenzofuran	307	±	49	302.25	98	4.1
Fluorene	492	±	78	427.15	87	3.0
Phenanthrene	1618	±	340	1278.03	79	3.4
Anthracene	422	±	49	400.80	95	2.6
Fluoranthene	1280	±	220	1019.13	80	4.5
Pyrene	1033	±	285	911.82	88	3.1
Benz(a)anthracene	252	±	38	225.50	89	4.8
Chrysene	297	±	26	283.00	95	3.8
Benzo(b)fluoranthene + Benzo(k)fluoranthene	153	±	22	130.88	86	10.7
Benzo(a)pyrene	97.2	±	17.1	58.28	60	6.5

[a] Relative standard deviations for the SFE values are based on four replicate extractions.

Data are taken from Reference 11.

FIGURE 1
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



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RIC                                    DATA: 51BAS060786 #1     SCANS  200 TO 2700
06/07/86  8:26:00                      CALI: 51BAS060786 #3
SAMPLE: BASE ACID STD,2UL/20NG/UL
CONDS.:
RANGE: G   1,2700  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
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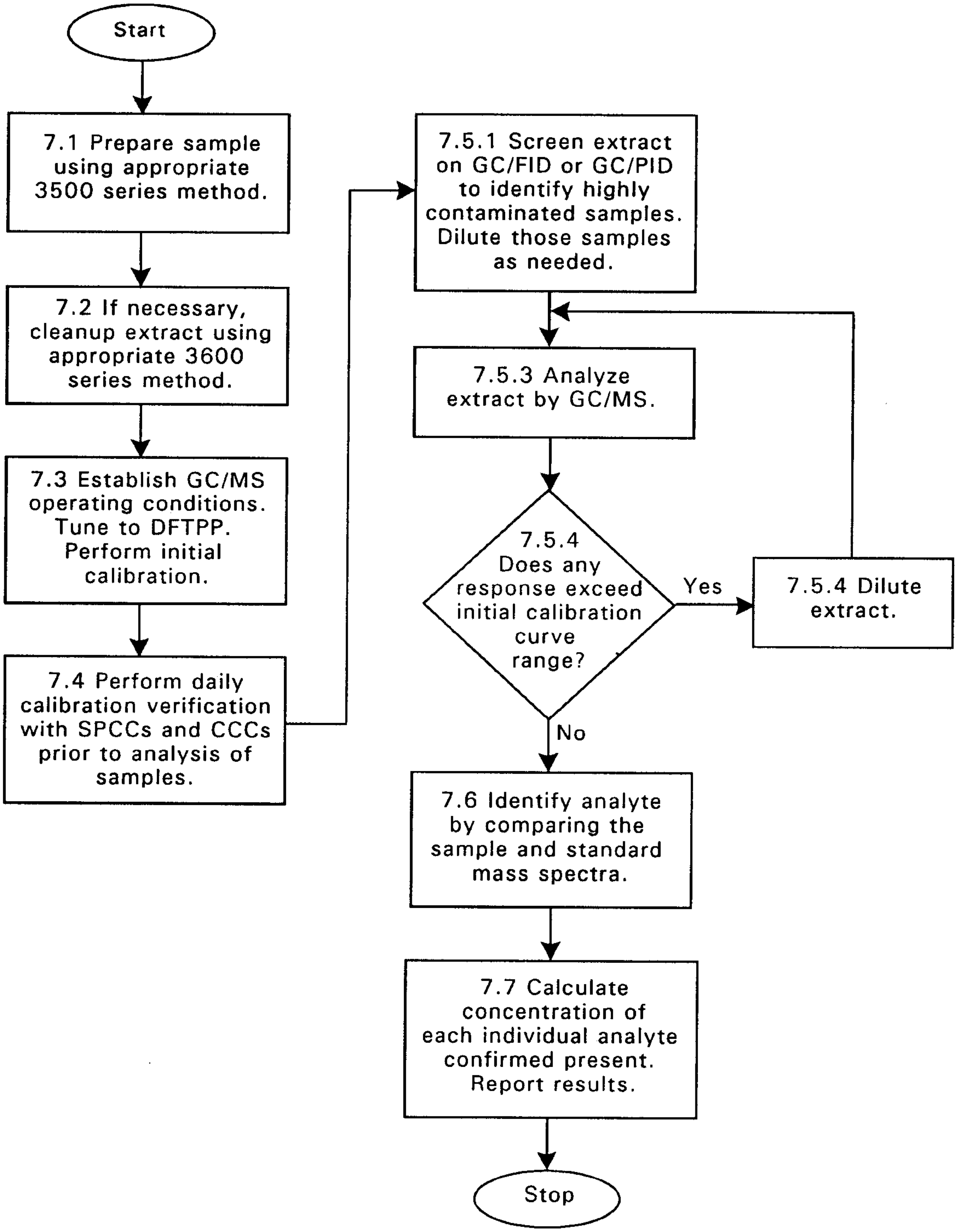
METHOD 8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)



Designation: D2216 – 10

Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass[1]

This standard is issued under the fixed designation D2216; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods cover the laboratory determination of the water (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due to loss of water except as noted in 1.4, 1.5, and 1.7. For simplicity, the word "material" shall refer to soil, rock or aggregate whichever is most applicable.

1.2 Some disciplines, such as soil science, need to determine water content on the basis of volume. Such determinations are beyond the scope of this test method.

1.3 The water content of a material is defined in 3.2.1.

1.4 The term "solid material" 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 etc.) 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 humidity, forming a compound (such as calcium sulfate hemihydrate) that 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/fibrous organic soils, it may be desirable to dry the 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 the standard water content determined at the standard drying temperature of 110°C.

NOTE 1—Test Method D2974 provides an alternate procedure for determining water content of peat materials.

1.5 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 that includes the previously soluble dissolved 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. For example, see Test Method D4542 regarding information on marine sediments.

1.6 This test standard requires several hours for proper drying of the water content specimen. Test Methods D4643, D4944 and D4959 provide less time-consuming processes for determining water content. See Gilbert[2] for details on the background of Test Method D4643.

1.7 Two test methods are provided in this standard. The methods differ in the significant digits reported and the size of the specimen (mass) required. The method to be used may be specified by the requesting authority; otherwise Method A shall be performed.

1.7.1 *Method A*—The water content by mass is recorded to the nearest 1 %. For cases of dispute, Method A is the referee method.

1.7.2 *Method B*—The water content by mass is recorded to the nearest 0.1 %.

1.8 This standard requires the drying of material in an oven. 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.

1.9 *Units*—The values stated in SI units shall be regarded as standard excluding the Alternative Sieve Sizes listed in Table 1. No other units of measurement are included in this test method.

1.10 Refer to Practice D6026 for guidance concerning the use of significant figures that shall determine whether Method, A or B is required. This is especially important if the water content will be used to calculate other relationships such as

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved July 1, 2010. Published August 2010. Originally approved in 1963. Last previous edition approved in 2005 as D2216–05. DOI: 10.1520/D2216-10.

[2] Gilbert, P.A., "Computer Controlled Microwave Oven System for Rapid Water Content Determination," Tech. Report GL-88–21, Department of the Army, Waterways Experiment Station, Corps of Engineers, Vicksburg, MS, November 1988 .

***A Summary of Changes section appears at the end of this standard.**

TABLE 1 Minimum Requirements for Mass of Test Specimen, and Balance Readability[A]

Maximum Particle Size (100 % Passing)		Method A Water Content Recorded to ±1 %		Method B Water Content Recorded to ±0.1 %	
SI Unit Sieve Size	Alternative Sieve Size	Specimen Mass	Balance Readability (g)	Specimen Mass (g)	Balance Readability (g)
75.0 mm	3 in	5 kg	10	50 kg	10
37.5 mm	1-½ in.	1 kg	10	10 kg	10
19.0 mm	¾ in.	250 g	1	2.5 kg	1
9.5 mm	⅜ in.	50 g	0.1	500 g	0.1
4.75 mm	No. 4	20 g	0.1	100 g	0.1
2.00 mm	No. 10	20 g	0.1	20 g	0.01

[A] If water content data is to be used to calculate other relationships, such as moist or dry mass, wet or dry unit weight or total or dry density, then specimen mass up to 200 g must be determined using a balance accurate to 0.01 g.

moist mass to dry mass or vice versa, wet unit weight to dry unit weight or vice versa, and total density to dry density or vice versa. For example, if four significant digits are required in any of the above calculations, then the water content must be recorded to the nearest 0.1 %. This occurs since 1 plus the water content (not in percent) will have four significant digits regardless of what the value of the water content is; that is, 1 plus 0.1/100 = 1.001, a value with four significant digits. While, if three significant digits are acceptable, then the water content can be recorded to the nearest 1 %.

1.11 *This standard does not purport to address all of the safety concerns, 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:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4220 Practices for Preserving and Transporting Soil Samples

D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

D4542 Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer

D4643 Test Method for Determination of Water (Moisture) Content of Soil by Microwave Oven Heating

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D4944 Test Method for Field Determination of Water (Moisture) Content of Soil by the Calcium Carbide Gas Pressure Tester

D4959 Test Method for Determination of Water (Moisture) Content of Soil By Direct Heating

D5079 Practices for Preserving and Transporting Rock Core Samples

D6026 Practice for Using Significant Digits in Geotechnical Data

D7263 Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 Refer to Terminology D653 for standard definitions of terms.

3.2 *Definitions:*

3.2.1 *water content by mass (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. A standard temperature of 110 ± 5°C is used to determine these masses.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *constant dry mass (of a material)*—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 for Method A or B respectively. The time required to obtain constant dry mass will vary depending on numerous factors. The influence of these factors generally can be established by good judgement, and experience with the materials being tested and the apparatus being used.

4. Summary of Test Method

4.1 A test specimen is dried in an oven at a temperature of 110 ± 5°C 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 index 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

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[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

of a soil, along with its liquid and plastic limits as determined by Test Method D4318, is used to express its relative consistency or liquidity index.

NOTE 2—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Drying Oven*—Vented, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E145 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 D4753 and this section. A Class GP1 balance of 0.01 g readability is required for specimens having a mass of up to 200 g (excluding mass of specimen container) and a Class GP2 balance of 0.1 g readability is required for specimens having a mass over 200 g. However, the balance used may be controlled by the number of significant digits needed (see 1.10).

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. Unless a dessicator is used, 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 (see Note 3). One uniquely numbered (identified) container or number-matched container and lid combination as required is needed for each water content determination.

NOTE 3—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 (Optional)*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium sulfate. It is preferable to use a desiccant that changes color when it needs to be reconstituted.

NOTE 4—Anhydrous calcium sulfate is sold under the trade name Drierite.

6.5 *Container Handling Apparatus*, heat resistant gloves, tongs, or suitable holder for moving and handling hot containers after drying.

6.6 *Miscellaneous*, knives, spatulas, scoops, quartering cloth, wire saws, etc., as required.

7. Samples

7.1 Soil samples shall be preserved and transported in accordance with Practice D4220 Section 8 Groups B, C, or D soils. Rock samples shall be preserved and transported in accordance with Practice D5079 section 7.5.2, Special Care Rock. Keep the samples that are stored prior to testing in non-corrodible 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 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 below shall apply. See Howard[4] for background data for the values listed.

8.2 The minimum specimen mass of moist material selected to be representative of the total sample is based on visual maximum particle size in the sample and the Method (Method A or B) used to record the data. Minimum specimen mass and balance readability shall be in accordance with Table 1.

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 on the test data forms or test data sheets.

8.4 When working with a small (less than 200 g) 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 on the test data form/sheet.

8.5 For those samples consisting entirely of intact rock or gravel-size aggregate, the minimum specimen mass shall be 500 g. Representative portions of the sample may be broken into smaller particles. The particle size is dictated by the specimen mass, the container volume and the balance being used to determine constant mass, see 10.4. Specimen masses as small as 200 g may be tested if water contents of only two significant digits are acceptable.

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, etc.).

9.2 For disturbed samples such as trimmings, bag samples, etc; 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 and segregation, the material should be mixed thoroughly. Select a representative

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[4] Howard, A. K., "Minimum Test Specimen Mass for Moisture Content Determination," *Geotechnical Testing Journal*, ASTM., Vol. 12, No. 1, March 1989, pp. 39-44.

portion using a scoop of a size that no more than a few scoopfuls are required to obtain the proper size of specimen defined in 8.2. Combine all the portions for the test specimen.

9.2.2 If the material is such that it cannot be thoroughly mixed or mixed and sampled by a scoop, 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 practical, using 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, etc, obtain the test specimen by one of the following methods depending on the purpose and potential use of the sample:

9.3.1 Using a knife, wire saw, or other sharp cutting device, trim the outside portion of the sample a sufficient distance to see if the material is layered, and to remove material that appears more dry or more wet than the main portion of the sample. If the existence of layering is questionable, slice the sample in half. If the material is layered, see 9.3.3.

9.3.2 If the material is not layered, obtain the specimen meeting the mass requirements in 8.2 by: *(1)* taking all or one-half of the interval being tested; *(2)* trimming a representative slice from the interval being tested; or *(3)* trimming the exposed surface of one-half or from the interval being tested.

NOTE 5—Migration of moisture in some cohesionless soils may require that the entire sample be tested.

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 the test data forms or test data sheets.

10. Procedure

10.1 Determine and record the mass of the clean and dry specimen container and its lid, if used along with its identification number.

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 specimen using a balance (see 8.2 and Table 1) selected on the basis of the specimen mass or required significant digits. Record this value.

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 the moist specimen in the drying oven. Dry the specimen to a constant mass. Maintain the drying oven at 110 ± 5°C unless otherwise specified (see 1.4). 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.

10.4.1 In most cases, drying a test specimen overnight (about 12 to 16 h) is sufficient, especially when using forced draft ovens. In cases where there is doubt concerning the adequacy of drying to a constant dry mass, see 3.3.1 and check for additional loss in mass with additional oven drying over an adequate time period. A minimum time period of two hours should be used, increasing the drying time with increasing specimen mass. A rapid check to see if a relatively large specimen (> than about 100 g of material) is dry; place a small strip of torn paper on top of the material while it is in the oven or just upon removal from the oven. If the paper strip curls the material is **not** dry and requires additional drying time. Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

10.4.2 Since some dry materials may absorb moisture from drying specimens that still retain moisture, dried specimens shall be removed before placing moist specimens in the same oven; unless they are being dried overnight.

10.5 After the specimen has dried to constant mass, remove the container from the oven (and replace the lid if used). Allow the specimen 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 or heat transmission or both. Determine the mass of the container and oven-dried specimen using the same type/capacity balance 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.

10.5.1 Cooling in a desiccator is acceptable in place of tight fitting lids since it greatly reduces absorption of moisture from the atmosphere during cooling.

10.6 A copy of a sample data sheet is shown in Appendix X1. Any data sheet can be used, provided the form contains all the required data.

11. Calculation

11.1 Calculate the water content of the material as follows:

$$w = [(M_{cms} - M_{cds})/(M_{cds} - M_c)] \times 100 = (M_w/M_s) \times 100 \quad (1)$$

where:

w = water content, %,
M_{cms} = mass of container and moist specimen, g,
M_{cds} = mass of container and oven dry specimen, g,
M_c = mass of container, g,
M_w = mass of water ($M_w = M_{cms} - M_{cds}$), g, and
M_s = mass of oven dry specimen ($M_s = M_{cds} - M_c$), g.

12. Report: Test Data Form/Sheet

12.1 The method used to specify how data are recorded on the test data sheets or forms, as given below, is the industry standard, and are representative of the significant digits that should be retained. These requirements do not consider in situ material variation, use of the data, special purpose studies, or any considerations for the user's objectives. It is common practice to increase or reduce significant digits of reported data commensurate with these considerations. It is beyond the scope of the standard to consider significant digits used in analysis method for engineering design.

12.1.1 Test data forms or test data sheets shall include the following:

12.1.2 Identification of the sample (material) being tested, such as boring number, sample number, test number, container number etc.

12.1.3 Water content of the specimen to the nearest 1 % for Method A or 0.1 % for Method B, as appropriate based on the minimum mass of the specimen. 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. Refer to Practice D6026 for guidance concerning significant digits, especially if the value obtained from this test method is to be used to calculate other relationships such as unit weight or density. For instance, if it is desired to express dry unit weight, as determined by D7263 to the nearest 0.1 lbf/f^3 (0.02 kN/m^3), it may be necessary to use a balance with a greater readability or use a larger specimen mass to obtain the required significant digits the mass of water so that the water content can be determined to the required significant digits. Also, the significant digits in Practice D6026 may need to be increased when calculating phase relationships requiring four significant digits.

12.1.4 Indicate if test specimen had a mass less than the minimum indicated in 8.2.

12.1.5 Indicate if test specimen contained more than one material type (layered, etc.).

12.1.6 Indicate the drying temperature if different from 110 ± 5°C.

12.1.7 Indicate if any material (size and amount) was excluded from the test specimen.

12.2 When reporting water content in tables, figures, etc., any data not meeting the requirements of this test method shall be noted, such as not meeting the mass, balance, or temperature requirements or a portion of the material is excluded from the test specimen.

13. Precision and Bias

13.1 *Statements on Precision*[5]:

13.1.1 *Precision*—Test data on precision is not presented due to the nature of the soil or rock materials tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

13.1.2 Subcommittee D18.03 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

13.1.3 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

14. Keywords

14.1 aggregate; consistency; index property; laboratory; moisture analysis; moisture content; soil; water content

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D13-1108.

APPENDIX

(Nonmandatory Information)

X1. WATER CONTENT OF SOIL AND ROCK SAMPLE DATA SHEET

Project Name: __________________ Project Number: __________________

Test Method: __X__ Method A
_____ Method B

Laboratory Number	04-725-S	04-726-S	04-727-S		
Boring Number	B-1	B-2	B-2		
Field Number	SPT-1	SPT-2	SPT-2a		
Container / Lid Number	725	726	727		
Container Mass, g M_c	770.1	731.7	770.6		
Container+Moist Specimen Mass, g M_{cms}	1895.3	2008.4	1827.9		
Date / Time In Oven	8/20/2004 0700	8/20/2004 0700	8/20/2004 0700		
Initial Container+Oven Dry Specimen Mass, g	1721.4	1872.1	1707.6		
Date / Time Out of Oven	8/20/2004 1200	8/20/2004 1200	8/20/2004 1200		
Secondary Container+Oven Dry Specimen Mass, g	1721.4	1801.2	1660.8		
Date / Time Out of Oven	--	8/20/2004 1600	8/20/2004 1600		
Final Container+Oven Dry Specimen Mass, g, M_{cds}	1721.4	1801.2	1660.8		
Date / Time Out of Oven	--	8/21/2004 0700	8/21/2004 0700		
Mass of Water, g, $M_w = M_{cms} - M_{cds}$	173.9	207.2	167.1		
Mass of Solids, g, $M_s = M_{cds} - M_c$	951.3	1069.5	890.2		
Water Content, %, $w = (M_w/M_s) \times 100$	18	19	19		
Unified Soil Classification Group Symbol (Visual)	GC	GC	GC		
Bold Approximate Maximimum Grain Size (Visual)	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., ¾ in., ⅜ in., #4, #10, < #10	3 in., 1½ in., ¾ in., ⅜ in., #4, #10, < #10
Oven Temperature if Other Than 110°C	—	—	—		

Remarks: ____

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Tested By: ______________________ Date: ______________ Checked By: ______________________

Dry Mass By: ____________________ Date: ______________ Spot Checked: ____________________

Calculated By: ___________________ Date: ______________ Reviewed By: ______________________

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to these test methods since the last issue, D2216–05, that may impact the use of these test methods. (Approved July 1, 2010)

(*1*) Replaced "has to" with "must" in 1.10.
(*2*) Added the "heat resistant" to "gloves" in 6.5.
(*3*) Replaced "reduce" with "minimize" in 7.1.
(*4*) Added "or required significant digits" in 10.3.
(*5*) Revised 10.4.1 to clarify the process of obtaining and checking to determine if a specimen had reached constant mass.
(*6*) Added "that still retain moisture" in 10.4.2.
(*7*) Replaced "its being heated" with "heat transmission" in 10.5.
(*8*) Added "as determined by D7263" in 12.1.3.
(*9*) Added Footnote A to Table 1 reflecting balance requirements outlined in 6.2.

Designation: D 7263 – 09

Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens[1]

1. Scope

1.1 These test methods describe two ways of determining the total/moist and dry densities (unit weights) of intact, disturbed, remolded, and reconstituted (compacted) soil specimens. Density (unit weight) as used in this standard means the same as "bulk density" of soil as defined by the Soil Science Society of America. Intact specimens may be obtained from thin-walled sampling tubes, block samples, or clods. Specimens that are remolded by dynamic or static compaction procedures may also be measured by these methods. These methods apply to soils that will retain their shape during the measurement process and may also apply to other materials such as soil-cement, soil-lime, soil-bentonite or solidified soil-bentonite-cement slurries. It is common for the density (unit weight) of specimens after removal from sampling tubes and compaction molds to be less than the value based on tube or mold volumes, or of in-situ conditions. This is due to the specimen swelling after removal of lateral pressures.

1.1.1 Method A covers the procedure for measuring the volume of wax coated specimens by determining the quantity of water displaced.

1.1.1.1 This method only applies to specimens in which the wax will not penetrate the outer surface of the specimen.

1.1.2 Method B covers the procedure by means of the direct measurement of the dimensions and mass of a specimen, usually one of cylindrical shape. Intact and reconstituted/remolded specimens may be tested by this method in conjunction with strength, permeability (air/water) and compressibility determinations.

1.2 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are approximate.

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026.

1.3.1 The method used to specify how data are collected, calculated, or recorded in this standard is not directly related to the accuracy with which the data can be applied in design or other uses, or both. How one applies the results obtained using this standard is beyond its scope.

1.4 *This standard does not purport to address all of the safety concerns, 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:*[2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 698 Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 ft-lbf/ft^3(600 kN-m/m^3))

D 854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D 1557 Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft^3(2,700 kN-m/m^3))

D 1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D 2166 Test Method for Unconfined Compressive Strength of Cohesive Soil

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)

D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

D 3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D 4220 Practices for Preserving and Transporting Soil Samples

D 4318 Test Methods for Liquid Limit, Plastic Limit, and

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[1] These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved March 15, 2009. Published April 2009.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Plasticity Index of Soils

D 4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D 6026 Practice for Using Significant Digits in Geotechnical Data

E 2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *Other Reference:*

Soil Science Society of America Glossary of Soil Science Terms[3]

3. Terminology

3.1 Refer to Terminology D 653 for standard definitions of terms.

4. Significance and Use

4.1 Dry density, as defined as "density of soil or rock" in Terminology D 653 and "bulk density" by soil scientists, can be used to convert the water fraction of soil from a mass basis to a volume basis and vise-versa. When particle density, that is, specific gravity (Test Methods D 854) is also known, dry density can be used to calculate porosity and void ratio (see Appendix X1). Dry density measurements are also useful for determining degree of soil compaction. Since moisture content is variable, moist soil density provides little useful information except to estimate the weight of soil per unit volume, for example, pounds per cubic yard, at the time of sampling. Since soil volume shrinks with drying of swelling soils, bulk density will vary with moisture content. Hence, the water content of the soil should be determined at the time of sampling.

4.2 Densities (unit weights) of remolded/reconstituted specimens are commonly used to evaluate the degree of compaction of earthen fills, embankments, etc. Dry density values are usually used in conjunction with compaction curve values (Test Methods D 698 and D 1557).

4.3 Density (unit weight) is one of the key components in determining the mass composition/phase relations of soil, see Appendix X1.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D 3740 does not in itself assure reliable results. Reliable results depend on several factors; Practice D 3740 provides a means of evaluating some of these factors.

5. Apparatus

5.1 For Method A the following apparatus are required:

5.1.1 *Balance*—All balances must meet the requirements of Specification D 4753 and this section. A Class GP1 balance of 0.01 g readability is required for specimens having a mass up to 200 grams and a Class GP2 balance of 0.1 g readability is required for specimens having a mass over 200 grams. For method A, the balance must be capable of measuring the mass of the specimen suspended in water. This is usually accomplished by a weighing hook built into the balance for that purpose, or a yoke assemblage is placed upon the pan which suspends a thin, non-absorbent string or wire, that is, a nylon line, etc., below the balance into the water reservoir.

5.1.2 *Drying Oven*—A thermostatically controlled, preferably of the forced-draft type, capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

5.1.3 *Wax*—Non-shrinking, paraffin and/or microcrystalline wax that has a known and constant density, ρ_ρ, to four significant figures and that does not change after repeated melting and cooling cycles.

NOTE 2—The waxes generally used are commercially available and have density values in the range of 0.87 to 0.91 g/cm^3 or Mg/m^3.

5.1.4 *Wax-Melting Container*—Used to melt the wax, but should not allow the wax to overheat. A container heated by hot water, preferably thermostatically controlled, is satisfactory. The wax should be heated to only slightly above the melting point to avoid flashing of the wax vapors and to permit quickly forming a uniform surface coating of wax. **Warning—**Vapors given off by molten wax ignite spontaneously above 205°C (400°F) and should not be allowed to come in contact with the heating element or open flame.

5.1.5 *Wire Basket*—A wire basket of 3.35 mm or finer mesh of approximately equal width and height of sufficient size to contain the specimen. The basket shall be constructed to prevent trapping air when it is submerged. The basket is suspended from the balance by a fine thread or string. A hairnet may also be used in lieu of the basket for smaller soil specimens.

5.1.6 *Container*—A container or tank of sufficient size to contain the submerged basket and specimen.

5.1.7 *Specimen Container*—A corrosion-resistant container of sufficient size to contain the specimen for water content determination.

5.1.8 *Thermometer*—Capable of measuring the temperature range within which the test is being performed graduated in a 0.1 degree C division scale and meeting the requirements of Specification E 2251.

5.1.9 *Container Handling Apparatus*—Gloves or suitable holder for moving and handling hot containers.

5.1.10 *Miscellaneous*—Paintbrush, trimming tools, specimen containers, and data sheets provided as required.

5.2 For Method B the following apparatus are needed:

5.2.1 *Balance*—See 5.1.1.

5.2.2 *Drying Oven*—See 5.1.2.

5.2.3 *Specimen-Size Measurement Devices*—Devices used to determine the height and width or diameter of the specimen shall measure the respective dimensions to four significant digits and shall be constructed so that their use will not indent or penetrate into the specimen.

NOTE 3—Circumferential measuring tapes are recommended over calipers for measuring the diameter of cylindrical specimens.

5.2.4 *Apparatus for Preparing Reconstituted or Remolded Specimens (Optional)*—Such apparatus is only required if these types of specimens are being tested.

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[3] Available online: www.soils.org/sssagloss/index.php.

5.2.5 *Miscellaneous Apparatus*—Specimen trimming and carving tools including a wire saw, steel straightedge, miter box and vertical trimming lathe, specimen containers, and data sheets shall be provided as required.

6. Samples and Test Specimens

6.1 *Samples*—Intact samples shall be preserved and transported in accordance with Practice D 4220 Groups C and D soil. Compacted or remolded specimens shall be preserved in accordance with Practice D 4220 Group B soil. Maintain the samples that are stored prior to testing in non-corrodible airtight containers at a temperature between approximately 3° and 30°C and in an area that prevents direct contact with sunlight.

6.2 *Specimens*—Specimens for testing shall be sufficiently cohesive and firm to maintain shape during the measuring procedure if Method A is used, see 1.1.1.1. Specimens shall have a minimum dimension of 30 mm (1.3 in.) and the largest particle contained within the test specimen shall be smaller than one-tenth of the specimen's smallest dimension. For specimens having a dimension of 72 mm (2.8 in.) or larger, the largest particle size shall be smaller than one-sixth of the specimen's smallest dimension. If, after completion of a test on an intact specimen, visual observations indicate that larger particles than permitted are present, indicate this information in the remarks section of the report of test data.

7. Procedure

7.1 Record all identifying information for the specimen, such as project, boring number, depth, sample type (that is, tube, trimmed, etc.), visual soil classification (Practice D 2488), or other pertinent data.

7.2 *Method A—Water Displacement*:

7.2.1 Determine, if not previously established, the density of the wax to be used to four significant digits (see 5.1.3).

7.2.2 Prepare specimens in an environment that minimizes any changes in water content. For some soils, changes in water content are minimized by trimming specimens in a controlled environment, such as a controlled high-humidity room/enclosure.

7.2.3 If required, cut a specimen meeting the size requirements given in 6.2 from the sample to be tested. If required, trim the specimen to a fairly regular shape. Re-entrant angles should be avoided, and any cavities formed by large particles being pulled out should be patched carefully with material from the trimmings. Handle specimens carefully to minimize disturbance, change in shape, or change in water content. Typically, for most samples, changes in water content are minimized by trimming specimens, in a controlled environment, such as a controlled high-humidity room/enclosure.

7.2.4 Determine and record the moist mass of the soil specimen (M_t) to four significant figures in g or kg.

7.2.5 Cover the specimen with a thin coat of melted wax, either with a paintbrush or by dipping the specimen in a container of melted wax. Apply a second coat of wax after the first coat has hardened. The wax should be sufficiently warm to flow when brushed on the specimen, yet it should not be so hot that it dries the soil.

NOTE 4—If overheated wax comes in contact with the soil specimen, it may cause the moisture to vaporize and form air bubbles under the wax. Bubbles may be trimmed out and filled with wax.

7.2.6 Determine and record the mass of the wax-coated specimen in air (M_C) to four significant figures in g or kg.

7.2.7 Determine and record the submerged mass of the wax-coated specimen (M_{sub}) to four significant digits in g or kg. This is done by placing the specimen in a wire basket hooked onto a balance and immersing the basket and specimen in a container of water. In order to directly measure the submerged mass of the wet soil and wax, the balance must have been previously balanced (tared to zero) with the wire basket completely submerged in the container of water. Make sure that the specimen and basket is fully submerged, and that the basket is not touching the sides or bottom of the container.

7.2.8 Record the temperature of the water to 0.1 degrees C.

NOTE 5—Maintain water bath temperature and submerged basket depth the same as when calibrated or zeroed.

7.2.9 Remove the wax from the specimen. It can be peeled off after a break is made in the wax surface.

7.2.10 Determine the water content to the nearest 0.1 percent in accordance with Method D 2216.

NOTE 6—The water content may be determined from an adjacent piece of soil or from trimmings if appropriate, for example, if the wax becomes difficult to remove from the specimen. Note in the report if water content is not from the specimen itself.

7.3 *Method B—Direct Measurement*:

7.3.1 *Intact Specimens*—Prepare intact specimens from large block samples or from samples secured in accordance with Practice D 1587 or other acceptable tube sampling procedures, such as Practice D 3550. Specimens can be obtained from intact block samples using a sharp cutting ring. Handle samples/specimens carefully to minimize disturbance, changes in cross section, or change in water content, see 6.1. Specimens are usually cubical or cylindrical in shape.

NOTE 7—Core sampling might be difficult or impossible in gravelly or hard dry soils. Wet soils tend to be more plastic and subject to compression.

7.3.1.1 Specimens obtained by tube sampling may be tested without extrusion except for cutting the end surfaces plane and perpendicular to the longitudinal axis of the sampling tube. The height and inner dimensions of the tube may be taken to represent specimen dimensions.

NOTE 8—Some soils may expand into the sampling tube with a resultant change in volume from the original in-situ condition.

7.3.1.2 Trim specimens in an environment that minimizes any change in water content, see 7.2.2. Where removal of gravel or crumbling resulting from trimming causes voids on the surface of the specimen, carefully fill the voids with remolded soil obtained from the trimmings. When the sample condition permits, a vertical trimming lathe may be used to reduce cylindrical specimens to a uniform diameter.

7.3.1.3 After obtaining uniform dimensions, place the specimen in a miter box or trimming collar (especially for friable soils) and cut the specimen to a uniform height with a wire saw or other suitable device, such as a sharpened steel straightedge.

Perform one or more water content determinations on material obtained during the trimming of the specimen in accordance with Test Method D 2216 for the estimated water content(s). Final water content shall be performed on the whole specimen or representative slice (if other testing such as plasticity limits, Test Methods D 4318, are required) at the end of the test. Determine and record the mass (g) and dimensions (mm) of the specimen to four significant digits using the applicable apparatus described in 5.2. A minimum of three height measurements (approximately 120° apart if three, 90° apart if four, etc.) and at least three diameter measurements at the quarter points of the height shall be made to determine each the average height and diameter of cylindrical specimens. A minimum of three measurements each of length, width and height shall be made to determine the volume of cubical specimens.

NOTE 9—Test Method D 2166, section 6.2, describes a procedure for preparing intact test specimens for strength testing.

7.3.2 *Remolded/Reconstituted (Compacted) Specimens*—Specimens shall be prepared as prescribed by the individual assigning the test or as prescribed by the applicable related test procedure. After a specimen is formed, trim (if necessary) the ends perpendicular to the longitudinal axis, remove the mold, and determine the mass and dimensions of the test specimens in accordance with 7.3.1.3. The height and inner dimensions of the mold may be taken to represent specimen dimensions.

NOTE 10—It is common for the density (unit weight) of the specimen after removal from the mold to be less than the value based on the volume of the mold. This occurs as a result of the specimen swelling after removal of the lateral confinement due to the mold.

8. Calculations

8.1 *Water Content, w*—Calculate in accordance with Test Method D 2216 to four significant digits.

8.2 Calculate the moist density to four significant figures as follows:

8.2.1 *Method A—Water Displacement*:

$$\rho_m = M_t / [((M_c - M_{sub})/\rho_w) - ((M_c - M_t)/\rho_\rho)] \tag{1}$$

where:

M_t = mass of moist/total soil specimen, g,
M_c = mass of wax-coated specimen, g,
M_{sub} = mass of submerged paraffin-coated specimen, g,
ρ_ρ = density of paraffin, g/cm^3 or Mg/m^3,
ρ_w = density of water at test temperature, g/cm^3 or Mg/m^3, (see Test Methods D 854, Table 1), and
ρ_m = density of total (moist) soil specimen, g/cm^3 or Mg/m^3.

8.2.2 *Method B—Direct Measurement*:

$$\rho_m = (M_t / V) \tag{2}$$

where:

V = volume of moist soil specimen, cm^3.

8.2.2.1 *Cylindrical Shape*:

$$V = (\pi\, d^2\, h) / 4000 \tag{3}$$

where:

d = average specimen diameter, mm, and
h = average specimen height, mm.

8.2.2.2 *Cubical Shape*:

$$V = (l\, w\, h) / 4000 \tag{4}$$

where:

l = average length, mm,
w = average width, mm, and
h = average height, mm.

8.3 Calculate the dry density for either method A or B as follows:

$$\rho_d = \rho_m / (1 + w / 100) \tag{5}$$

where:

ρ_d = dry density of soil, g/cm^3 or Mg/m^3, and
w = water content of soil specimen (in percent), to nearest four significant digits.

8.4 Calculate the moist/total and dry unit weights to four significant figures for either method A or B as follows:

$$\gamma_m = 62.428\, \rho_m \text{ in lbf/ft}^3 \quad \text{and} \quad \gamma_d = 62.428\, \rho_d \text{ in lbf/ft}^3 \tag{6}$$
$$\gamma_m = 9.80665\, \rho_m \text{ in kN/m}^3 \quad \text{and} \quad \gamma_d = 9.80665\, \rho_d \text{ in kN/m}^3$$

where:

γ_m = moist/total unit weight of specimen, and
γ_d = dry unit weight of soil specimen.

9. Report: Test Data Sheet/Form

9.1 The report (data sheet) shall contain the following (see Appendix X2 and Appendix X3):

9.1.1 Identification of the sample (material) being tested, such as project, boring number, sample number, test number, container number, etc.,

9.1.2 Sample depth in meters (feet) below ground surface or elevation in meters (feet) (if applicable),

9.1.3 Classification of soil by Practice D 2487, if determined, or visual classification of soil (group name and symbol) as determined by Practice D 2488,

9.1.4 Moist/total and dry density (unit weight), to four significant digits,

9.1.5 Water content (in percent), to four significant digits,

9.1.6 Method used (A or B), and

9.1.7 Whether the specimen was intact, disturbed, remolded, or reconstituted (compacted).

10. Precision and Bias

10.1 *Precision*—The precision of the procedure in this test method for measuring the density (unit weight) of cohesive soil specimens is being determined. In addition, Subcommittee D18.03 is seeking pertinent data from users of the test method.

10.2 *Bias*—Since there is not an accepted reference material suitable for measuring the bias for this procedure, a statement on bias cannot be made.

11. Keywords

11.1 density; porosity; saturation; specimen; unit weight; void ratio

APPENDIXES

(Nonmandatory Information)

X1. RELATIONSHIPS AMONG SOIL PHASES; POROSITY, VOID RATIO, & SATURATION USING METRIC UNITS

X1.1 Let:

n = porosity, %,
e = void ratio,
S = saturation, %,
V = volume of soil specimen, cm^3,
V_v = volume of voids in soil specimen, cm^3,
V_s = volume of solids in soil specimen, cm^3,
w = water content of soil specimen, %,
G_s = specific gravity of soil solids in soil specimen as determined by Test Methods D 854,
ρ_d = dry density of soil specimen, Mg/m^3, and
M_d = dry mass of soil in soil specimen, g.

X1.2 Then:

$$n = \frac{V_v}{V} \times 100 = \frac{V - V_s}{V} \times 100 = \frac{V - \frac{M_d}{G_s}}{V} \times 100 = \frac{e}{1 + e} \times 100 \tag{X1.1}$$

$$e = \frac{V_v}{V_s} = \frac{V - V_s}{V_s} = \frac{V - \frac{M_d}{G_s}}{\frac{M_d}{G_s}} = \frac{n}{100 - n} = \frac{G_s w}{S} \tag{X1.2}$$

$$S = \frac{wG_s\rho_d}{G_s - \rho_d} = \frac{G_s w}{e} \tag{X1.3}$$

X2. EXAMPLE DATA SHEET
DISPLACEMENT METHOD—A

UNIT WEIGHTS, VOID RATIO, POROSITY, AND DEGREE OF SATURATION
(DISPLACEMENT METHOD—A)

NAME ______________________________ DATE ______________ JOB NO. ________________

LOCATION ____

BORING NO. ________________ SAMPLE NO. ________________ DEPTH/ELEV. ________________

DESCRIPTION OF SAMPLE ____

WATER CONTENT						
SAMPLE OR SPECIMEN NO.						
TARE NO.						
MASS IN GRAMS	TARE PLUS WET SOIL					
	TARE PLUS DRY SOIL					
	WATER	M_w				
	TARE					
	DRY SOIL	M_d				
WATER CONTENT		w	%	%	%	%
WEIGHT-VOLUME RELATIONS						
SAMPLE OR SPECIMEN NO.						
TEST TEMPERATURE OF WATER, T, °C						
MASS IN GRAMS	SOIL AND WAX IN AIR					
	WET SOIL	M_t				
	WAX					
	WET SOIL AND WAX IN WATER					
	DRY SOIL[A]	M_d				
SPECIFIC GRAVITY OF SOIL		G_s				
VOLUME IN CC	WET SOIL AND WAX[B]					
	WAX					
	WET SOIL	V				
	DRY SOIL = M_d / G_s	V_s				
LBS PER CU FT	WET UNIT WEIGHT = $(M_t / V) \times 62.4$	γ_m				
	DRY UNIT WEIGHT = $(M_d / V) \times 62.4$	γ_d				
VOID RATIO = $(V - V_s) / V_s$		e				
POROSITY,% = $[(V - V_s) / V] \times 100$		n	%	%	%	%
DEGREE OF SATURATION = $[V_w / (V - V_s)] \times 100$		S	%	%	%	%

VOLUME OF WAX = WEIGHT OF WAX / SPECIFIC GRAVITY OF WAX = ________________
VOLUME OF WATER = $V_w = M_w$ / SPECIFIC GRAVITY OF WATER[C]

[A] IF NOT MEASURED DIRECTLY, MAY BE COMPUTED AS FOLLOWS: $M_d = M_t / (1 + 0.01w)$

[B] VOLUME OF WET SOIL AND WAX = $\frac{\text{(WT OF WET SOIL \& WAX IN AIR)} - \text{(WT OF WET SOIL \& WAX IN WATER)}}{\text{DENSITY OF WATER AT TEST TEMPERATURE}}$

[C] SPECIFIC GRAVITY OF WATER IN METRIC SYSTEM = 1 (APPROX)

REMARKS ______________________ COMPUTED BY ______________ CHECKED BY ______________

X3. EXAMPLE DATA SHEET
VOLUMETRIC METHOD—B

UNIT WEIGHTS, VOID RATIO, POROSITY, AND DEGREE OF SATURATION
(VOLUMETRIC METHOD—B)

NAME ______________________ DATE ______________ JOB NO. ________________

LOCATION ____

BORING NO. ________________ SAMPLE NO. ________________ DEPTH/ELEV. ________________

DESCRIPTION OF SAMPLE ____________________________________

WATER CONTENT						
SAMPLE OR SPECIMEN NO.						
TARE NO.						
MASS IN GRAMS	TARE PLUS WET SOIL					
	TARE PLUS DRY SOIL					
	WATER	M_w				
	TARE					
	DRY SOIL	M_d				
WATER CONTENT		w	%	%	%	%
WEIGHT-VOLUME RELATIONS						
SAMPLE OR SPECIMEN NO.						
CYLINDER NO.						
CENTIMETERS	HEIGHT OF CYLINDER	H				
	INSIDE DIAMETER OF CYLINDER	D				
MASS IN GRAMS	WET SOIL AND TARE					
	TARE					
	WET SOIL	M_t				
	DRY SOIL[A]	M_d				
SPECIFIC GRAVITY OF SOIL		G_s				
VOLUME IN CC	WET SOIL (VOLUME OF CYLINDER)					
	DRY SOIL = M_d / G_s	V_s				
LBS PER CU FT	WET UNIT WEIGHT = $(M_t / V) \times 62.4$	γ_m				
	DRY UNIT WEIGHT = $(M_d / V) \times 62.4$	γ_d				
VOID RATIO = $(V - V_s) / V_s$		e				
POROSITY,% = $[(V - V_s) / V] \times 100$		n	%	%	%	%
DEGREE OF SATURATION = $[V_w / (V - V_s)] \times 100$		S	%	%	%	%

VOLUME OF CYLINDER, $V = (\pi D^2 H) / 4$
VOLUME OF WATER = $V_w = M_w$ / SPECIFIC GRAVITY OF WATER[B]

[A] IF NOT MEASURED ON ENTIRE SPECIMEN, MAY BE COMPUTED AS FOLLOWS: $M_d = M_t / (1 + 0.01w)$

[B] SPECIFIC GRAVITY OF WATER IN METRIC SYSTEM = 1 (APPROX)

REMARKS ____________________ COMPUTED BY ______________ CHECKED BY ______________

METHOD 9045D

SOIL AND WASTE pH

1.0 SCOPE AND APPLICATION

1.1 This method is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

2.0 SUMMARY OF METHOD

2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.0 APPARATUS AND MATERIALS

4.1 pH meter with means for temperature compensation.

4.2 Glass electrode.

4.3 Reference electrode -- A silver-silver chloride or other reference electrode of constant potential may be used.

NOTE: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

4.4 Beaker -- 50-mL.

4.5 Thermometer and/or temperature sensor for automatic compensation.

4.6 Analytical balance -- capable of weighing 0.1 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be 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.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Primary standard buffer salts are available from the National Institute of Standards and Technology (NIST) and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

5.4 Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NIST standards, are recommended for routine use.

6.0 SAMPLE PRESERVATION AND HANDLING

Samples should be analyzed as soon as possible.

7.0 PROCEDURE

7.1 Calibration

7.1.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value. If an accurate pH reading based on the conventional pH scale [0 to 14 at 25 EC] is required, the analyst should control sample temperature at 25 ± 1 EC when sample pH approaches the alkaline end of the scale (e.g., a pH of 11 or above).

7.2 Sample preparation and pH measurement of soils:

7.2.1 To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

7.2.2 Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

7.2.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.2.4 If the sample temperature differs by more than 2 EC from the buffer solution, the measured pH values must be corrected.

7.2.5 Report the results as "soil pH measured in water at _EC" where "_EC" is the temperature at which the test was conducted.

7.3 Sample preparation and pH measurement of waste materials

7.3.1 To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.

7.3.2 Let the waste suspension stand for about 15 min to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material.

7.3.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.3.4 If the sample temperature differs by more than 2 EC from the buffer solution, the measured pH values must be corrected.

7.3.5 Report the results as "waste pH measured in water at _EC" where "_EC" is the temperature at which the test was conducted.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for the appropriate QC protocols.

8.2 Electrodes must be thoroughly rinsed between samples.

9.0 METHOD PERFORMANCE

9.1 No data provided.

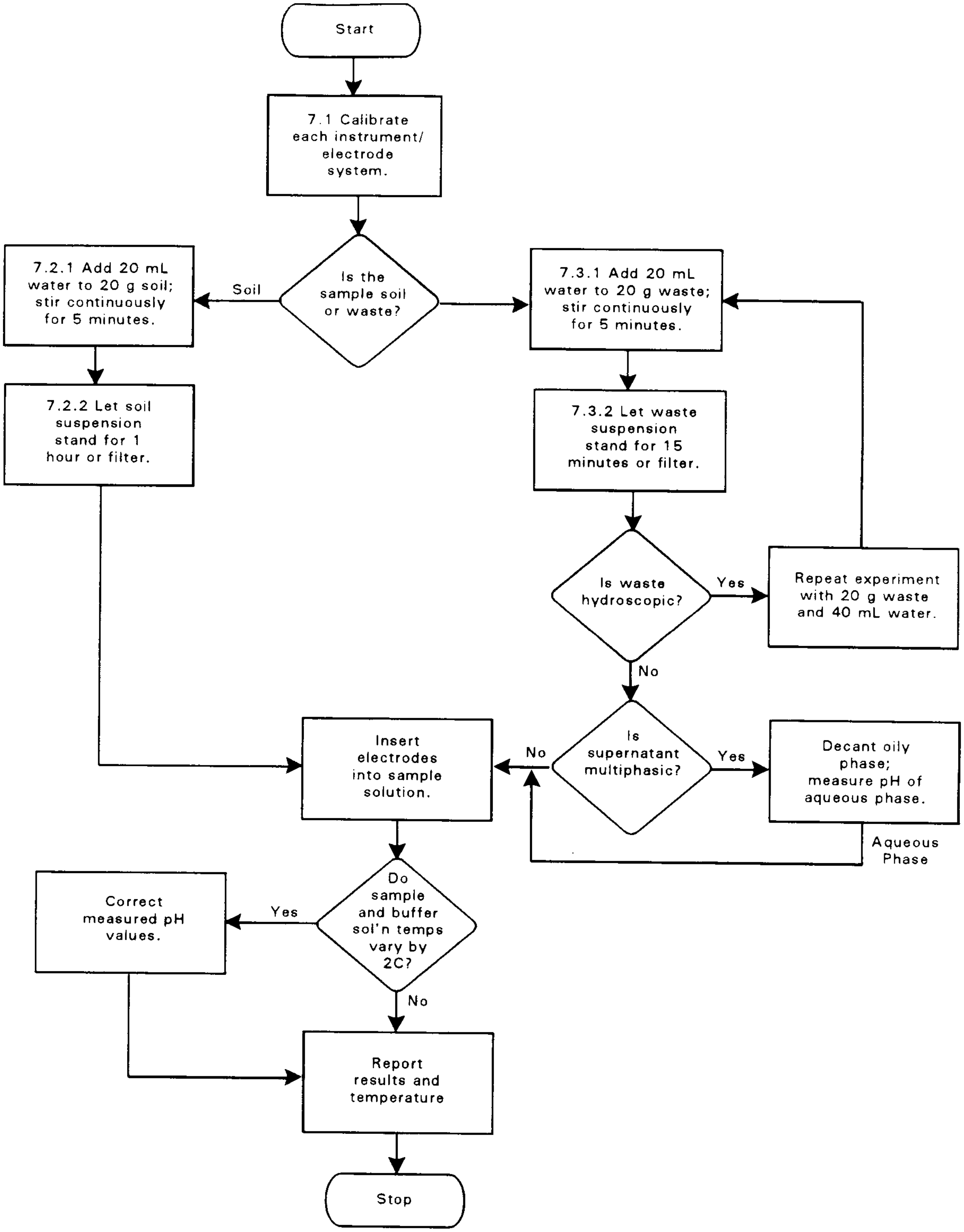
10.0 REFERENCES

1. Black, Charles Allen; Methods of Soil Analysis; American Society of Agronomy: Madison, WI, 1973.

2. National Bureau of Standards, Standard Reference Material Catalog, 1986-87, Special Publication 260.

METHOD 9045D

SOIL AND WASTE pH



Designation: D422 – 63 (Reapproved 2007)

Standard Test Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D422; 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 quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 µm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 µm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Note 1 and Note 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-µm), or No. 200 (75-µm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-µm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 µm.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E100 Specification for ASTM Hydrometers

2.2 *ASTM Adjuncts:*

Air-Jet Dispersion Cup for Grain-Size Analysis of Soil[3]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup (See drawing2.2[3]) (Note 3) conforming to the general details shown in Fig. 3 (Note 4 and Note 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

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[1] This test method is under the jurisdiction of ASTM Committee D18 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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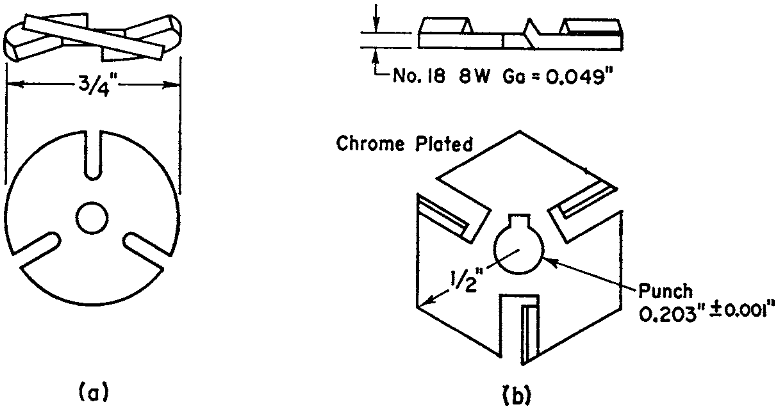
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

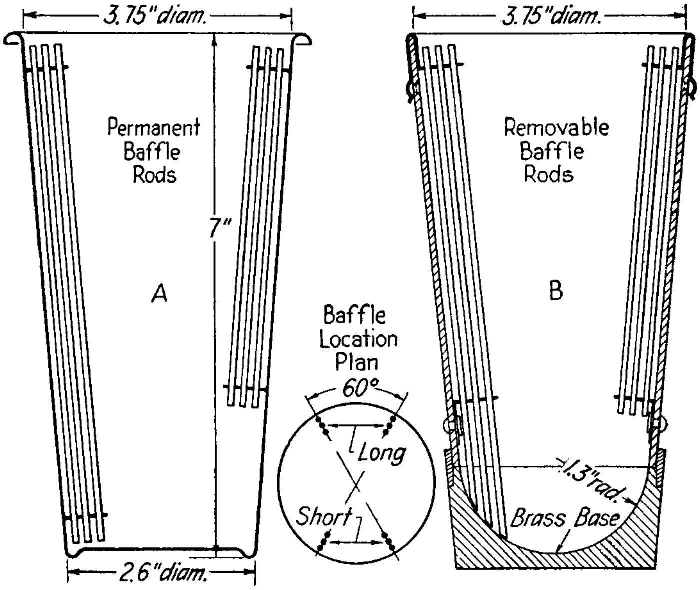
[3] Available from ASTM International Headquarters. Order Adjunct No. ADJD0422.



Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E11. A full set of sieves includes the following (Note 6):

3-in. (75-mm)
2-in. (50-mm)
1½-in. (37.5-mm)
1-in. (25.0-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 20 (850-µm)
No. 40 (425-µm)
No. 60 (250-µm)
No. 140 (106-µm)
No. 200 (75-µm)

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)
1½-in. (37.5-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 8 (2.36-mm)
No. 16 (1.18-mm)
No. 30 (600-µm)
No. 50 (300-µm)
No. 100 (150-µm)
No. 200 (75-µm)

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

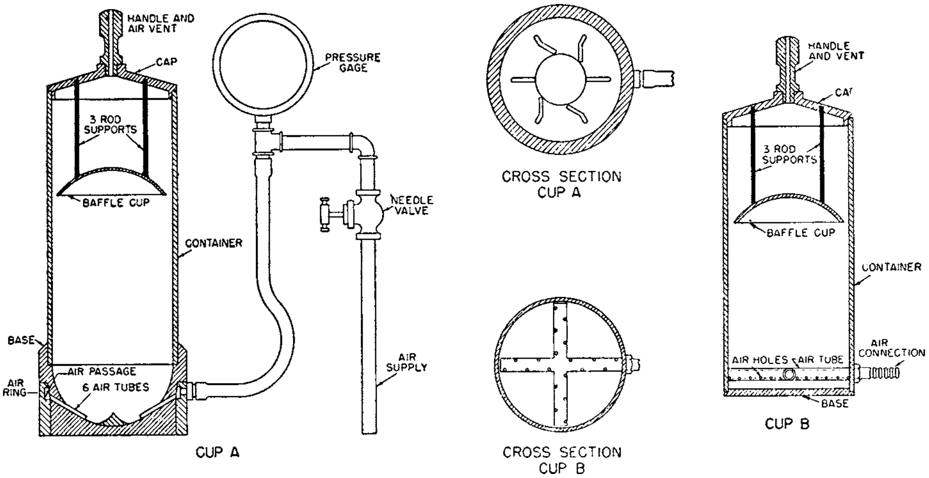
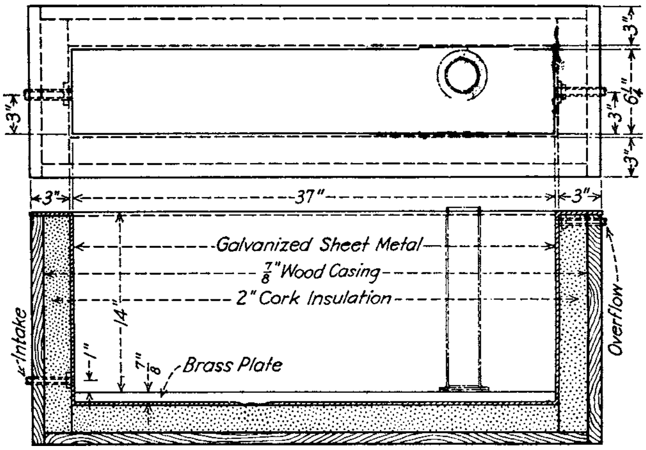


FIG. 3 Air-Jet Dispersion Cups of Apparatus B



Metric Equivalents

in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Practice D421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil-water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil-water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil-water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil-water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil-water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-µm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1) \quad (1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100 \tag{2}$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

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

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

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

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), 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. According to Stokes' law: see Table 2

$$D = \sqrt{[30n/980(G - G_1)] \times L/T} \tag{3}$$

where:

D = diameter of particle, mm,

n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (see Table 2)),

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of soil particles, and

G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

TABLE 1 Values of Correction Factor, α, for Different Specific Gravities of Soil Particles^A

Specific Gravity	Correction Factor^A
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

^{A} For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

15.2 For convenience in calculations the above equation may be written as follows: see Table 3

$$D = K\sqrt{L/T} \tag{4}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the *A*- and *B*-scales, the square root being indicated on the *D*-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the *C*- or *CI*-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

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

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + 1/2\,[L_2 - (V_B/A)] \qquad (5)$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm^3, and

A = cross-sectional area of sedimentation cylinder, cm^2

Values used in calculating the values in Table 2 are as follows:
For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm^3

A = 27.8 cm^2

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(*1*) Gravel, passing 3-in. and retained on No. 4 sieve . . . . . %
(*2*) Sand, passing No. 4 sieve and retained on No. 200 sieve . . . . . %
  (*a*) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve . . . . . %
  (*b*) Medium sand, passing No. 10 sieve and retained on No. 40 sieve . . . . . %
  (*c*) Fine sand, passing No. 40 sieve and retained on No. 200 sieve . . . . . %
(*3*) Silt size, 0.074 to 0.005 mm . . . . . %
(*4*) Clay size, smaller than 0.005 mm . . . . . %
  Colloids, smaller than 0.001 mm . . . . . %

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01530	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	. . . .
2-in.	. . . .
1½-in.	. . . .
1-in.	. . . .
¾-in.	. . . .
⅜-in.	. . . .
No. 4 (4.75-mm)	. . . .
No. 10 (2.00-mm)	. . . .
No. 40 (425-µm)	. . . .
No. 200 (75-µm)	. . . .

HYDROMETER ANALYSIS

0.074 mm	. . . .
0.005 mm	. . . .
0.001 mm	. . . .

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-µm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

Designation: D2166 – 06

Standard Test Method for Unconfined Compressive Strength of Cohesive Soil[1]

This standard is issued under the fixed designation D2166; 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 unconfined compressive strength of cohesive soil in the intact, remolded, or reconstituted condition, using strain-controlled application of the axial load.

1.2 This test method provides an approximate value of the strength of cohesive soils in terms of total stresses.

1.3 This test method is applicable only to cohesive materials which will not expel or bleed water (water expelled from the soil due to deformation or compaction) during the loading portion of the test and which will retain intrinsic strength after removal of confining pressures, such as clays or cemented soils. Dry and crumbly soils, fissured or varved materials, silts, peats, and sands cannot be tested with this method to obtain valid unconfined compression strength values.

NOTE 1—The determination of the unconsolidated, undrained strength of cohesive soils with lateral confinement is covered by Test Method D2850.

1.4 This test method is not a substitute for Test Method D2850.

1.5 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.5.1 The procedures used to specify how data are collected/recorded and calculated in this test method are regarded as the industry standard. In addition, they are representative of the significant digits that should generally be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to commensurate with these considerations. It is beyond the scope of this test method to consider significant digits used in analysis methods for engineering design.

1.6 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are approximate.

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:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer
D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes
D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
D2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)
D2850 Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D4220 Practices for Preserving and Transporting Soil Samples
D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils
D6026 Practice for Using Significant Digits in Geotechnical Data
D6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:* Refer to Terminology D653 for standard definitions of terms.

3.2 *Definitions of Terms Specific to This Standard:*

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Strength and Compressibility of Soils.

Current edition approved July 15, 2006. Published January 2007. Originally approved in 1963. Last previous edition approved in 2000 as D2166 – $00^{\varepsilon 1}$. DOI: 10.1520/D2166-06.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

3.2.1 *unconfined compressive strength* (q_u)—the compressive stress at which an unconfined cylindrical specimen of soil will fail in a simple compression test. In this test method, unconfined compressive strength is taken as the maximum load attained per unit area or the load per unit area at 15 % axial strain, whichever is secured first during the performance of a test.

3.2.2 *shear strength* (s_u)—for unconfined compressive strength test specimens, the shear strength is calculated to be ½ of the compressive stress at failure, as defined in 3.2.1.

4. Significance and Use

4.1 The primary purpose of the unconfined compression test is to quickly obtain a measure of compressive strength for those soils that possess sufficient cohesion to permit testing in the unconfined state.

4.2 Samples of soils having slickensided or fissured structure, samples of some types of loess, very soft clays, dry and crumbly soils and varved materials, or samples containing significant portions of silt or sand, or both (all of which usually exhibit cohesive properties), frequently display higher shear strengths when tested in accordance with Test Method D2850. Also, unsaturated soils will usually exhibit different shear strengths when tested in accordance with Test Method D2850.

4.3 If tests on the same sample in both its intact and remolded states are performed, the sensitivity of the material can be determined. This method of determining sensitivity is suitable only for soils that can retain a stable specimen shape in the remolded state.

NOTE 2—For soils that will not retain a stable shape, a vane shear test or Test Method D2850 can be used to determine sensitivity.

NOTE 3—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

5. Apparatus

5.1 *Compression Device*, The compression device may be a platform weighing scale equipped with a screw-jack-activated load yoke, a hydraulic loading device, or any other compression device with sufficient capacity and control to provide the rate of loading prescribed in 7.1. For soil with an unconfined compressive strength of less than 100 kPa (1.0 ton/ft ^{2}) the compression device shall be capable of measuring the compressive stress to within 1 kPa (0.01 ton/ft ^{2}). For soil with an unconfined compressive strength of 100 kPa (1.0 ton/ft ^{2}) or greater, the compression device shall be capable of measuring the compressive stress to the nearest 5 kPa (0.05 ton/ft ^{2}).

5.2 *Sample Extruder*, capable of extruding the soil core from the sampling tube at a uniform rate in the same direction of travel in which the sample entered the tube, and with negligible disturbance of the sample. Conditions at the time of sample removal may dictate the direction of removal, but the principal concern is to reduce the potential for additional disturbance beyond that incurred during initial sampling.

5.3 *Deformation Indicator*, The deformation indicator shall be a dial indicator graduated to 0.03 mm (0.001 in.) or better and having a travel range of at least 20 % of the length of the test specimen, or some other measuring device, such as an electronic deformation measuring device, meeting these requirements.

5.4 *Dial Comparator*, or other suitable device, for measuring the physical dimensions of the specimen to within 0.1 % of the measured dimension.

NOTE 4—Vernier calipers are not recommended for soft specimens, which will deform as the calipers are applied on the specimen.

5.5 *Timer*, A timing device indicating the elapsed testing time to the nearest second shall be used for establishing the rate of strain application prescribed in 7.1.

5.6 *Balance*, The balance used to weigh specimens shall determine the mass of the specimen to within 0.1 % of its total mass.

5.7 *Equipment*, as specified in Test Method D2216.

5.8 *Miscellaneous Apparatus*, including specimen trimming and carving tools, remolding apparatus, water content cans, and data sheets, as required.

6. Preparation of Test Specimens

6.1 *Specimen Size*—Specimens shall have a minimum diameter of 30 mm (1.3 in.) and the largest particle contained within the test specimen shall be smaller than one tenth of the specimen diameter. For specimens having a diameter of 72 mm (2.8 in.) or larger, the largest particle size shall be smaller than one sixth of the specimen diameter. If, after completion of a test on an intact specimen, it is found, based on visual observation, that larger particles than permitted are present, indicate this information in the remarks section of the report of test data (Note 5). The height-to-diameter ratio shall be between 2 and 2.5. Determine the average height and diameter of the test specimen using the apparatus specified in 5.4. Take a minimum of three height measurements (120° apart), and at least three diameter measurements at the quarter points of the height.

NOTE 5—If large soil particles are found in the specimen after testing, a particle-size analysis performed in accordance with Test Method D6913 may be performed to confirm the visual observation and the results provided with the test report.

6.2 *Intact Specimens*—Prepare intact specimens from large samples or from samples secured in accordance with Practice D1587 and preserved and transported in accordance with the practices for Group C samples in Practices D4220. Tube specimens may be tested without trimming except for the squaring of ends, if conditions of the sample justify this procedure. Handle specimens carefully to reduce the potential for additional disturbance, changes in cross section, or loss of water content. If compression or any type of noticeable disturbance would be caused by the extrusion device, split the sample tube lengthwise or cut it off in small sections to facilitate removal of the specimen with minimal disturbance. Prepare carved specimens with minimal disturbance, and whenever possible, in a humidity-controlled room. Make every effort to prevent a change in water content of the soil. Specimens shall be of uniform circular cross section with ends perpendicular to the longitudinal axis of the specimen. When carving or trimming, remove any small pebbles or shells encountered. Carefully fill voids on the surface of the specimen with remolded soil obtained from the trimmings. When pebbles or crumbling result in excessive irregularity at the ends, cap the specimen with a minimum thickness of plaster of paris, hydrostone, or similar material. When sample condition permits, a vertical lathe that will accommodate the total sample may be used as an aid in carving the specimen to the required diameter. Where prevention of the development of appreciable capillary forces is deemed important, seal the specimen with a rubber membrane, thin plastic coatings, or with a coating of grease or sprayed plastic immediately after preparation and during the entire testing cycle. Determine the mass and dimensions of the test specimen. If the specimen is to be capped, its mass and dimensions should be determined before capping. If the entire test specimen is not to be used for determination of water content, secure a representative sample of trimmings for this purpose, placing them immediately in a covered container. The water content determination shall be performed in accordance with Test Method D2216.

6.3 *Remolded Specimens*—Specimens may be prepared either from a failed intact specimen or from a disturbed sample, providing it is representative of the failed intact specimen. In the case of failed intact specimens, wrap the material in a thin rubber membrane and work the material thoroughly with the fingers to assure complete remolding. Avoid entrapping air in the specimen. Exercise care to obtain a uniform density, to remold to the same void ratio as the intact specimen, and to preserve the natural water content of the soil. Form the disturbed material into a mold of circular cross section having dimensions meeting the requirements of 6.1. After removal from the mold, determine the mass and dimensions of the test specimens.

6.4 *Reconstituted Specimens*—Specimens shall be prepared to the predetermined water content and density prescribed by the individual assigning the test (Note 6). After a specimen is formed, trim the ends perpendicular to the longitudinal axis, remove from the mold, and determine the mass and dimensions of the test specimen.

NOTE 6—Experience indicates that it is difficult to compact, handle, and obtain valid results with specimens that have a degree of saturation that is greater than 90 %.

7. Procedure

7.1 Place the specimen in the loading device so that it is centered on the bottom platen. Adjust the loading device carefully so that the upper platen just makes contact with the specimen. Zero the deformation indicator or record the initial reading of the electronic deformation device. Apply the load so as to produce an axial strain at a rate of ½ to 2 %/min. Record load, deformation, and time values at sufficient intervals to define the shape of the stress-strain curve (usually 10 to 15 points are sufficient). The rate of strain should be chosen so that the time to failure does not exceed about 15 min (Note 7). Continue loading until the load values decrease with increasing strain, or until 15 % strain is reached. Indicate the rate of strain in the report of the test data, as required in 9.1.7. Determine the water content of the test specimen using the entire specimen, unless representative trimmings are obtained for this purpose, as in the case of intact specimens. Indicate on the test report whether the water content sample was obtained before or after the shear test, as required in 9.1.2.

NOTE 7—Softer materials that will exhibit larger deformation at failure should be tested at a higher rate of strain. Conversely, stiff or brittle materials that will exhibit small deformations at failure should be tested at a lower rate of strain.

7.2 Make a sketch, or take a photo, of the test specimen at failure showing the slope angle of the failure surface if the angle is measurable.

7.3 A copy of a example data sheet is included in Appendix X1. Any data sheet can be used, provided the form contains all the required data.

8. Calculation

8.1 Calculate the axial strain, ε_1, to the nearest 0.1 %, for a given applied load, as follows:

$$\varepsilon_1 = \frac{\Delta L}{L_0} \times 100$$

where:

ΔL = length change of specimen as read from deformation indicator or computed from the electronic device, mm (in.), and

L_0 = initial length of test specimen, mm (in).

8.2 Calculate the average cross-sectional area, A, for a given applied load, as follows:

$$A = \frac{A_0}{\left(1 - \frac{\varepsilon_1}{100}\right)}$$

where:

A_0 = initial average cross-sectional area of the specimen, mm ^{2}(in. ^{2}), and

ε_1 = axial strain for the given load, expressed as a decimal.

8.3 Calculate the compressive stress, σ_c, to three significant figures or nearest 1 kPa (0.01 ton/ft ^{2}), for a given applied load, as follows:

$$\sigma_c = (P/A)$$

where:

P = given applied load, kN (lbf),

A = corresponding average cross-sectional area mm ^{2}(in. ^{2}).

8.4 *Graph*—If desired, a graph showing the relationship between compressive stress (ordinate) and axial strain (abscissa) may be plotted. Select the maximum value of compressive stress, or the compressive stress at 15 % axial strain, whichever is secured first, and report as the unconfined compressive strength, q_u. Whenever it is considered necessary for proper interpretation, include the graph of the stress-strain data as part of the data reported.

8.5 If both the intact and remolded compressive strengths are measured, determine the sensitivity, S_T, as follows:

$$S_T = \frac{q_u\ \text{(intact specimen)}}{q_u\ \text{(remolded specimen)}}$$

9. Report

9.1 The report should include the following:

9.1.1 Identification and visual description of the specimen, including soil classification, symbol, and whether the specimen is intact, remolded, reconstituted, etc. Also include specimen identifying information, such as project, location, boring number, sample number, depth, etc. Visual descriptions shall be made in accordance with Practice D2488,

9.1.2 Initial dry density and water content (specify if the water content specimen was obtained before or after shear, and whether from trimmings or the entire specimen),

9.1.3 Degree of saturation (Note 8), if computed,

NOTE 8—The specific gravity determined in accordance with Test Method D854 is required for calculation of the degree of saturation.

9.1.4 Unconfined compressive strength and shear strength,

9.1.5 Average height and diameter of specimen,

9.1.6 Height-to-diameter ratio,

9.1.7 Average rate of strain to failure, %,

9.1.8 Strain at failure, %,

9.1.9 Liquid and plastic limits, if determined, in accordance with Test Method D4318,

9.1.10 Failure sketch or photo,

9.1.11 Stress-strain graph, if prepared,

9.1.12 Sensitivity, if determined,

9.1.13 Particle size analysis, if determined, in accordance with Test Method D6913, and

9.1.14 *Remarks*—Note any unusual conditions or other data that would be considered necessary to properly interpret the results obtained, for example, slickensides, stratification, shells, pebbles, roots, or brittleness, the type of failure (that is, bulge, diagonal shear, etc.).

10. Precision and Bias

10.1 *Precision*—Criteria for judging the acceptability of test results obtained by this test method on rigid polyurethane foam (density about 0.09 g/cm^3) is given in Table 1. These estimates of precision are based on the results of the interlaboratory program conducted by the ASTM Reference Soils and Testing Program.[3] The precision estimates will vary with the material/soil type being tested, and judgement is required when applying these estimates to soil.

TABLE 1 Summary of Test Results from Each Laboratory (Compressive Strength Data on Rigid Polyurethane Foam (density about 0.09 g/cm^3))

(1) Number of Triplicate Test Laboratories	(2) Test Parameter^A	(3) Average Value^B	(4) Standard Deviation^C	(5) Acceptable Range of Two Results^D
Single-Operator Results (Wiithin-Laboratory Repeatability):				
22	Strength, kPa	989	42	120
22	Strain, %	4.16	0.32	0.9
Multilaboratory Results (Between- Laboratory Reproducibility):				
22	Strength, kPa	989	53	150
22	Strain, %	4.16	0.35	1.0

^{A}Strength = peak compressive stress and strain = axial strain at peak compressive stress.

^{B}The number of significant digits and decimal places presented are representative of the input data. In accordance with Practice D6026, the standard deviation and acceptable range of results can not have more decimal places than the input data.

^{C}Standard deviation is calculated in accordance with Practice E691 and is referred to as the 1*s* limit.

^{D}Acceptable range of two results is referred to as the *d*2*s* limit. It is calculated as $1.960\sqrt{2}\cdot 1s$, as defined by Practice E177. The difference between two properly conducted tests should not exceed this limit. The number of significant digits/decimal places presented is equal to that prescribed by this test method or Practice D6026. In addition, the value presented can have the same number of decimal places as the standard deviation, even if that result has more significant digits than the standard deviation.

10.1.1 The data in Table 1 are based on three replicate tests performed by each test laboratory. The single-operator and multilaboratory standard deviation shown in Table 1, Column 4, were obtained in accordance with Practice E691. Results of two properly conducted tests performed by the same operator on the same material, using the same equipment, and in the shortest practical period of time should not differ by more than the single-operator *d*2*s* limits shown in Table 1, Column 5. For definition of *d*2*s* see Footnote D in Table 1. Results of two properly conducted tests performed by different operators and on different days should not differ by more than the multilaboratory *d*2*s* limits shown in Table 1, Column 5.

10.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

11. Keywords

11.1 cohesive soil; sensitivity; strain-controlled loading; strength; stress-strain relationships; unconfined compression

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1014.

APPENDIX

(Nonmandatory Information)

X1. Example Data Sheet

UNCONFINED COMPRESSION TEST—UI

Name ____________ Date ____________ Job No. ____________

Location ____________

Boring No. ____________ Sample No. ____________ Depth/Elev. ____________

Description of Sample ____________

Proving Ring No. ____________ Apparatus No. ____________

Water Content Determination

Tare No. ____________

Wt. Specimen Wet + Tare

Wt. Specimen Dry + Tare

Wt. Water ____________

Wt. Tare ____________

Wt. Specimen Wet ____________

Wt. Specimen Dry ____________

Water Content in % Dry Wt.

at 105°C ____________ %

Wet Density ____________

Dry Density ____________

Unconfined Compressive Strength

Initial Diameter D_o ____________

Initial Area A_o ____________

Initial Height L_o ____________

Initial Volume V_o ____________

Specific Gravity ____________

Stress = $\frac{\text{Load}}{\text{Corr. Area}}$

Test Data

Unit Strain $\frac{\Delta L}{L_o}$

$\frac{L}{D}$ = ____________

Corr. Area = $\frac{A_o}{1 - \text{Unit Strain}}$

Elapsed Time-min	Load Dial	Axial Load	Strain Dial	Total Strain	Unit Strain	Corrected Area	Stress	

Type of Sample ____________

Attach a photo or sketch of the specimen after failure to this form

Strain Rate ____________ %/Min

Remarks ____________

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (00^{e1}) that may impact the use of this standard.

(*1*) Replaced instances of the term "undisturbed" with the term "intact" and instances of the term "compacted" with "reconstituted." Replaced instances of "remove-the-water-content can" with "water content can."
(*2*) In Referenced Documents, added Test Method D6913.
(*3*) In the Significance and Use section, changed 4.1 to read "to quickly obtain *a measure of* compressive strength *for those* soils…," and changed 4.3 to read "If *tests on the same sample in* both *its intact* and remolded *states* are performed…" [italics indicate new wording]
(*4*) In the Apparatus section, changed 5.2 to read "…sampling tube *at a uniform rate* in the same…" and …"principal concern is to *reduce the potential for additional* disturbance *beyond that incurred during initial sampling*." Note 4 was changed to read "…calipers are *applied* on the specimen." [italics indicate new wording].
(*5*) In the Procedure section, changed 7.1 to read "…deformation indicator *or record the initial reading of the electronic deformation device*," and the sentence "The rate of strain used for testing sealed specimens…for better test results" was deleted. [italics indicate new wording]
(*6*) In the Calculation section, corrected the equations to calculate strain and average cross-sectional area, and changed the definition of ΔL to include the following "…indicator o*r computed from the electronic device*,…" [italics indicate new wording]

Designation: D5084 – 10

Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter[1]

1. Scope*

1.1 These test methods cover laboratory measurement of the hydraulic conductivity (also referred to as *coefficient of permeability*) of water-saturated porous materials with a flexible wall permeameter at temperatures between about 15 and 30°C (59 and 86°F). Temperatures outside this range may be used; however, the user would have to determine the specific gravity of mercury and R_T (see 10.3) at those temperatures using data from *Handbook of Chemistry and Physics*. There are six alternate methods or hydraulic systems that may be used to measure the hydraulic conductivity. These hydraulic systems are as follows:

1.1.1 *Method A*—Constant Head

1.1.2 *Method B*—Falling Head, constant tailwater elevation

1.1.3 *Method C*—Falling Head, rising tailwater elevation

1.1.4 *Method D*—Constant Rate of Flow

1.1.5 *Method E*—Constant Volume–Constant Head (by mercury)

1.1.6 *Method F*—Constant Volume–Falling Head (by mercury), rising tailwater elevation

1.2 These test methods use water as the permeant liquid; see 4.3 and Section 6 on Reagents for water requirements.

1.3 These test methods may be utilized on all specimen types (undisturbed, reconstituted, remolded, compacted, etc.) that have a hydraulic conductivity less than about 1×10^{-6} m/s (1×10^{-4} cm/s), providing the head loss requirements of 5.2.3 are met. For the constant-volume methods, the hydraulic conductivity typically has to be less than about 1×10^{-7} m/s.

1.3.1 If the hydraulic conductivity is greater than about 1×10^{-6} m/s, but not more than about 1×10^{-5} m/s; then the size of the hydraulic tubing needs to be increased along with the porosity of the porous end pieces. Other strategies, such as using higher viscosity fluid or properly decreasing the cross-sectional area of the test specimen, or both, may also be possible. The key criterion is that the requirements covered in Section 5 have to be met.

1.3.2 If the hydraulic conductivity is less than about 1×10^{-11} m/s, then standard hydraulic systems and temperature environments will typically not suffice. Strategies that may be possible when dealing with such impervious materials may include the following: *(a)* controlling the temperature more precisely, *(b)* adoption of unsteady state measurements by using high-accuracy equipment along with the rigorous analyses for determining the hydraulic parameters (this approach reduces testing duration according to Zhang et al. (**1**)[2]), and *(c)* shortening the length or enlarging the cross-sectional area, or both, of the test specimen. Other items, such as use of higher hydraulic gradients, lower viscosity fluid, elimination of any possible chemical gradients and bacterial growth, and strict verification of leakage, may also be considered.

1.4 The hydraulic conductivity of materials with hydraulic conductivities greater than 1×10^{-5} m/s may be determined by Test Method D2434.

1.5 All observed and calculated values shall conform to the guide for significant digits and rounding established in Practice D6026.

1.5.1 The procedures used to specify how data are collected, recorded, and calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that should generally be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.6 This standard also contains a Hazards section about using mercury, see Section 7.

1.7 The time to perform this test depends on such items as the Method (A, B, C, D, E, or F) used, the initial degree of saturation of the test specimen and the hydraulic conductivity of the test specimen. The constant volume Methods (E and F) and Method D require the shortest period-of-time. Typically a test can be performed using Methods D, E, or F within two to three days. Methods A, B, and C take a longer period-of-time,

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[1] This standard is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.04 on Hydrologic Properties and Hydraulic Barriers.

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[2] The boldface numbers in parentheses refer to the list of references appended to this standard.

***A Summary of Changes section appears at the end of this standard.**

from a few days to a few weeks depending on the hydraulic conductivity. Typically, about one week is required for hydraulic conductivities on the order of 1×10^{-9} m/s. The testing time is ultimately controlled by meeting the equilibrium criteria for each Method (see 9.5).

1.8 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are mathematical conversions, which are provided for information purposes only and are not considered standard, unless specifically stated as standard, such as 0.5 mm or 0.01 in.

1.9 *This standard does not purport to address all of the safety concerns, 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:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D698 Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 ft-lbf/ft^3(600 kN-m/m^3))

D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D1140 Test Methods for Amount of Material in Soils Finer than No. 200 (75-µm) Sieve

D1557 Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft^3(2,700 kN-m/m^3))

D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D2113 Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2434 Test Method for Permeability of Granular Soils (Constant Head)

D2435 Test Methods for One-Dimensional Consolidation Properties of Soils Using Incremental Loading

D3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4220 Practices for Preserving and Transporting Soil Samples

D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D4767 Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils

D5079 Practices for Preserving and Transporting Rock Core Samples

D6026 Practice for Using Significant Digits in Geotechnical Data

D6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling

D6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of technical terms in this standard, refer to Terminology D653.

3.1.2 *head loss, Δh*—the change in total head of water across a given distance.

3.1.2.1 *Discussion*—In hydraulic conductivity testing, typically the change in total head is across the influent and effluent lines connected to the permeameter, while the given distance is typically the length of the test specimen.

3.1.3 *permeameter*—the apparatus (cell) containing the test specimen in a hydraulic conductivity test.

3.1.3.1 *Discussion*—The apparatus in this case is typically a triaxial-type cell with all of its components (top and bottom specimen caps, stones, and filter paper; membrane; chamber; top and bottom plates; valves; etc.).

3.1.4 *hydraulic conductivity, k*—the rate of discharge of water under laminar flow conditions through a unit cross-sectional area of porous medium under a unit hydraulic gradient and standard temperature conditions (20°C).

3.1.4.1 *Discussion*—In hydraulic conductivity testing, the term *coefficient of permeability* is often used instead of *hydraulic conductivity*, but *hydraulic conductivity* is used exclusively in this standard. A more complete discussion of the terminology associated with Darcy's law is given in the literature. (**2, 3**)

3.1.5 *pore volume of flow—in hydraulic conductivity testing*, the cumulative quantity of flow into a test specimen divided by the volume of voids in the specimen.

4. Significance and Use

4.1 These test methods apply to one-dimensional, laminar flow of water within porous materials such as soil and rock.

4.2 The hydraulic conductivity of porous materials generally decreases with an increasing amount of air in the pores of the material. These test methods apply to water-saturated porous materials containing virtually no air.

4.3 These test methods apply to permeation of porous materials with water. Permeation with other liquids, such as chemical wastes, can be accomplished using procedures similar to those described in these test methods. However, these test methods are only intended to be used when water is the permeant liquid. See Section 6.

4.4 Darcy's law is assumed to be valid and the hydraulic conductivity is essentially unaffected by hydraulic gradient.

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[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

4.5 These test methods provide a means for determining hydraulic conductivity at a controlled level of effective stress. Hydraulic conductivity varies with varying void ratio, which changes when the effective stress changes. If the void ratio is changed, the hydraulic conductivity of the test specimen will likely change, see Appendix X2. To determine the relationship between hydraulic conductivity and void ratio, the hydraulic conductivity test would have to be repeated at different effective stresses.

4.6 The correlation between results obtained using these test methods and the hydraulic conductivities of in-place field materials has not been fully investigated. Experience has sometimes shown that hydraulic conductivities measured on small test specimens are not necessarily the same as larger-scale values. Therefore, the results should be applied to field situations with caution and by qualified personnel.

4.7 In most cases, when testing high swell potential materials and using a constant-volume hydraulic system, the effective confining stress should be about 1.5 times the swell pressure of the test specimen or a stress which prevents swelling. If the confining stress is less than the swell pressure, anomalous flow conditions my occur; e.g., mercury column(s) move in the wrong direction.

NOTE 1—The quality of the result produced by this standard is dependent of the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing, sampling, inspection, etc.. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

5. Apparatus

5.1 *Hydraulic System*—Constant head (Method A), falling head (Methods B and C), constant rate of flow (Method D), constant volume-constant head (Method E), or constant volume-falling head (Method F) systems may be utilized provided they meet the following criteria:

5.1.1 *Constant Head*—The system must be capable of maintaining constant hydraulic pressures to ±5 % or better and shall include means to measure the hydraulic pressures to within the prescribed tolerance. In addition, the head loss across the permeameter must be held constant to ±5 % or better and shall be measured with the same accuracy or better. A pressure gage, electronic pressure transducer, or any other device of suitable accuracy shall measure pressures to a minimum of three significant digits. The last digit may be due to estimation, see 5.1.1.1.

5.1.1.1 Practice D6026 discusses the use or application of estimated digits. When the last digit is estimated and that reading is a function of the eye's elevation/location, then a mirror or another device is required to reduce the reading error caused by parallax.

5.1.2 *Falling Head*—The system shall allow for measurement of the applied head loss, thus hydraulic gradient, to ±5 % or better at any time. In addition, the ratio of initial head loss divided by final head loss over an interval of time shall be measured such that this computed ratio is accurate to ±5 % or better. The head loss shall be measured with a pressure gage, electronic pressure transducer, engineer's scale, graduated pipette, or any other device of suitable accuracy to a minimum of three significant digits. The last digit may be due to estimation, see 5.1.1.1. Falling head tests may be performed with either a constant tailwater elevation (Method B) or a rising tailwater elevation (Method C), see Fig. 1. This schematic of a hydraulic system presents the basic components needed to meet the objectives of Method C. Other hydraulic systems or schematics that meet these objectives are acceptable.

5.1.3 *Constant Rate of Flow*—The system must be capable of maintaining a constant rate of flow through the specimen to ±5 % or better. Flow measurement shall be by calibrated syringe, graduated pipette, or other device of suitable accuracy. The head loss across the permeameter shall be measured to a minimum of three significant digits and to an accuracy of ±5 % or better using an electronic pressure transducer(s) or other device(s) of suitable accuracy. The last digit may be due to estimation, see 5.1.1.1. More information on testing with a constant rate of flow is given in the literature (**4**).

5.1.4 *Constant Volume-Constant Head (CVCH)*—The system, with mercury to create the head loss, must be capable of maintaining a constant head loss cross the permeameter to ±5 % or better and shall allow for measurement of the applied head loss to ±5 % or better at any time. The head loss shall be measured to a minimum of three significant digits with an electronic pressure transducer(s) or equivalent device, (**5**) or based upon the pressure head caused by the mercury column, see 10.1.2. The last digit may be due to estimation, see 5.1.1.1.

5.1.4.1 Schematics of two CVCH systems are shown in Fig. 2 and Fig. 3. In each of these systems, the mercury-filled portion of the tubing may be continuous for constant head loss to be maintained. For the system showed in Fig. 2, the head loss remains constant provided the mercury column is vertical and is retained in only one half of the burette system (left burette in Fig. 2). In the system shown in Fig. 3, the head loss remains constant provided the water-mercury interface on the effluent end remains in the upper horizontal tube, and the water-mercury interface on the influent end remains in the lower horizontal tube. These schematics present the basic components needed to meet the objectives of Method E. Other hydraulic systems or schematics that meet these objectives are acceptable.

5.1.4.2 These types of hydraulic systems are typically not used to study the temporal or pore-fluid effect on hydraulic conductivity. The total volume of the specimen is maintained constant using this procedure, thereby significantly reducing effects caused by seepage stresses, pore fluid interactions, etc. Rather, these systems are intended for determining the hydraulic conductivity of a material as rapidly as possible.

5.1.4.3 *Hazards*—Since this hydraulic system contains mercury, special health and safety precautions have to be considered. See Section 7.

5.1.4.4 *Caution*—For these types of hydraulic systems to function properly, the separation of the mercury column has to be prevented. To prevent separation, the mercury and "constant head" tube have to remain relatively clean, and the inside diameter of this tube cannot be too large; typically a capillary tube is used. The larger diameter flushing tube (Fig. 2) is added

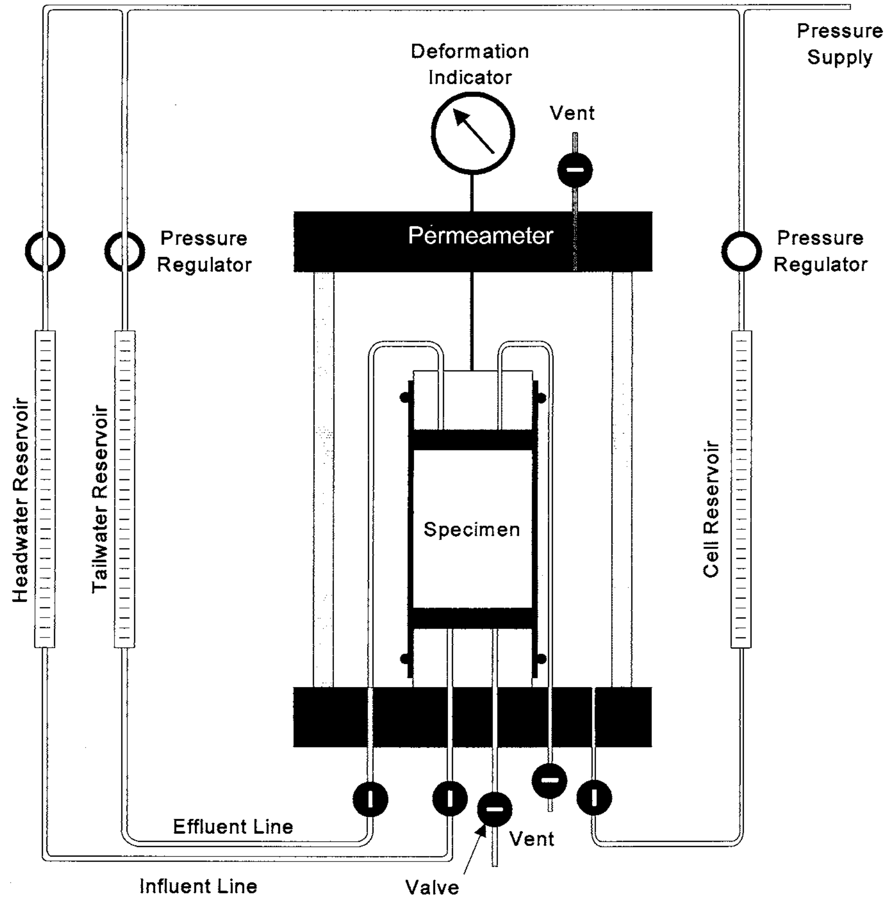

FIG. 1 Falling Head – Rising Tail System, Method C

to enable flushing clean water through the system without excessive mercury displacement. Traps to prevent the accidental flow of mercury out of the "Constant Head" tube or flushing tube are not shown in Fig. 2 and Fig. 3.

5.1.5 *Constant Volume-Falling Head (CVFH)*—The system, with mercury to create the head loss, shall meet the criteria given in 5.1.2. The head loss shall be measured to a minimum of three significant digits with an electronic pressure transducer(s) or equivalent device(s), (**5**) or based upon the differential elevation between the top surfaces of the mercury level in the headwater and tailwater tubes. The last digit may be due to estimation, see 5.1.1.1.

5.1.5.1 A schematic drawing of a typical CVFH hydraulic system is shown in Fig. 4 (**5**). Typically, the tailwater tube has a smaller area than the headwater tube to increase the sensitivity of flow measurements, and to enable flushing clean water through the system without excessive mercury displacement in the headwater tube. The schematic of the hydraulic system in Fig. 4 presents the basic components needed to meet the objectives of Method F. Other hydraulic systems or schematics that meet these objectives are acceptable. The development of the hydraulic conductivity equation for this type of system is given in Appendix X1.

5.1.5.2 See 5.1.4.2.

5.1.5.3 *Hazards*—Since this hydraulic system contains mercury, special health and safety precautions have to be considered. See Section 7.

5.1.5.4 *Caution*—For these types of hydraulic systems to function properly, the separation of the mercury column and entrapment of water within the mercury column have to be prevented. To prevent such problems, the mercury and tubes have to remain relatively clean. In addition, if different size headwater and tailwater tubes are used, capillary head might have to be accounted for, see Appendix X1, X1.2.3.2, and X1.4. Traps to prevent the accidental flow of mercury out of the tubes are not shown in Fig. 4.

5.1.6 *System De-airing*—The hydraulic system shall be designed to facilitate rapid and complete removal of free air bubbles from flow lines; e.g., using properly sized tubing and ball valves and fittings without pipe threads. Properly sized tubing, etc., means they are small enough to prevent entrapment of air bubbles, but not so small that the requirements of 5.2.3 cannot be met.

5.1.7 *Back Pressure System*—The hydraulic system shall have the capability to apply back pressure to the specimen to facilitate saturation. The system shall be capable of maintaining the applied back pressure throughout the duration of

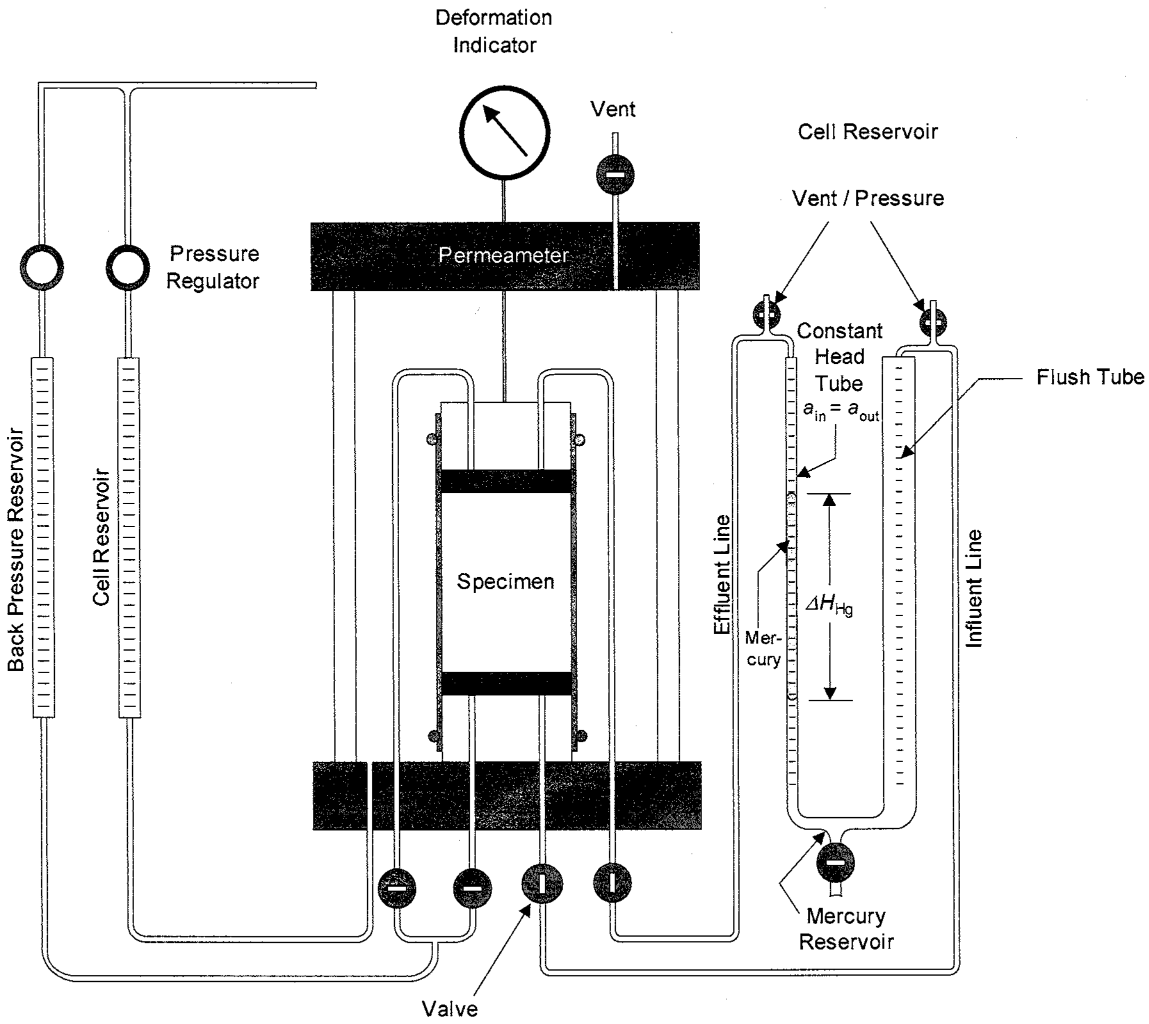

FIG. 2 Constant Volume – Constant Head System, Method E (5)

hydraulic conductivity measurements. The back pressure system shall be capable of applying, controlling, and measuring the back pressure to ±5 % or better of the applied pressure. The back pressure may be provided by a compressed gas supply, a deadweight acting on a piston, or any other method capable of applying and controlling the back pressure to the tolerance prescribed in this paragraph.

NOTE 2—Application of gas pressure directly to a fluid will dissolve gas in the fluid. A variety of techniques are available to minimize dissolution of gas in the back pressure fluid, including separation of gas and liquid phases with a bladder and frequent replacement of the liquid with de-aired water.

5.2 *Flow Measurement System*—Both inflow and outflow volumes shall be measured unless the lack of leakage, continuity of flow, and cessation of consolidation or swelling can be verified by other means. Flow volumes shall be measured by a graduated accumulator, graduated pipette, vertical standpipe in conjunction with an electronic pressure transducer, or other volume-measuring device of suitable accuracy.

5.2.1 *Flow Accuracy*—Required accuracy for the quantity of flow measured over an interval of time is ±5 % or better.

5.2.2 *De-airing and Compliance of the System*—The flow-measurement system shall contain a minimum of dead space and be capable of complete and rapid de-airing. Compliance of the system in response to changes in pressure shall be minimized by using a stiff flow measurement system. Rigid tubing, such as metallic or rigid thermoplastic tubing, or glass shall be used.

5.2.3 *Head Losses*—Head losses in the tubes, valves, porous end pieces, and filter paper may lead to error. To guard against such errors, the permeameter shall be assembled with no specimen inside and then the hydraulic system filled.

5.2.3.1 *Constant or Falling Head*—If a constant or falling head test is to be used, the hydraulic pressures or heads that will be used in testing a specimen shall be applied, and the rate of flow measured with an accuracy of ±5 % or better. This rate of flow shall be at least ten times greater than the rate of flow that is measured when a specimen is placed inside the permeameter and the same hydraulic pressures or heads are applied.

5.2.3.2 *Constant Rate of Flow*—If a constant rate of flow test is to be used, the rate of flow to be used in testing a

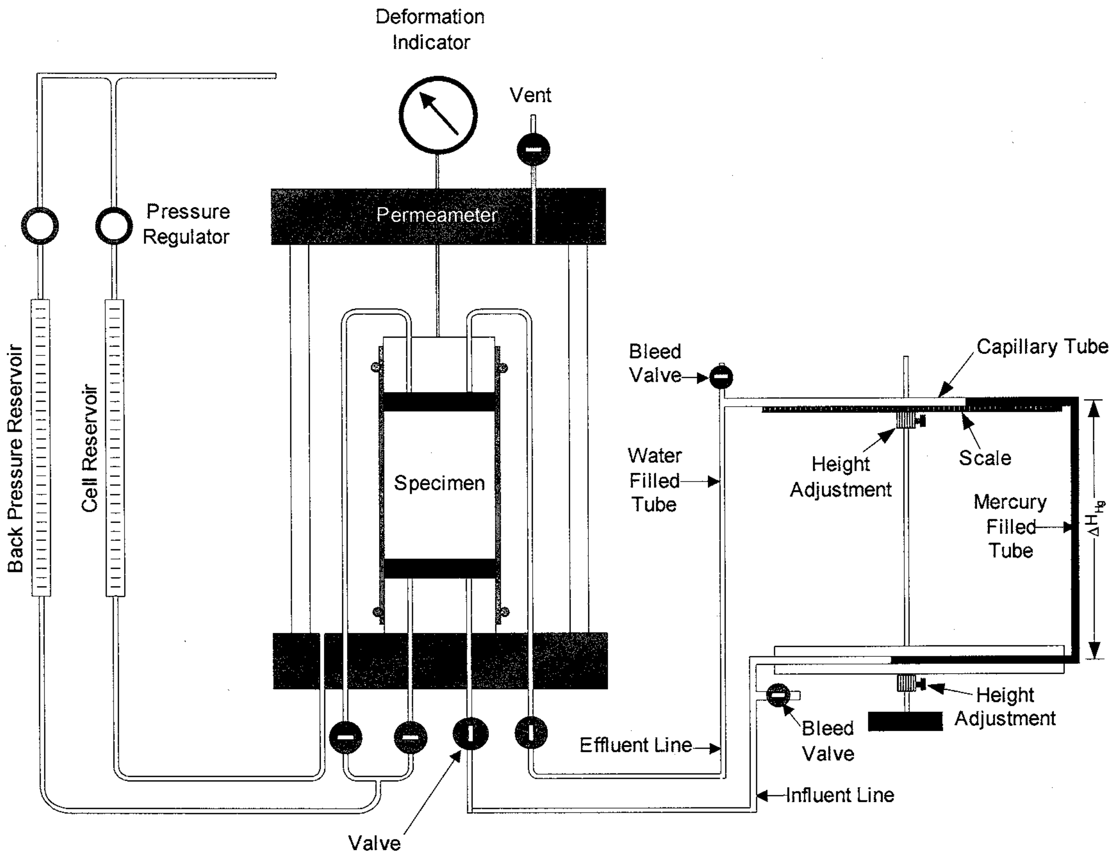

FIG. 3 Constant Volume—Constant Head System, Method E

specimen shall be supplied to the permeameter and the head loss measured. The head loss without a specimen shall be less than 0.1 times the head loss when a specimen is present.

5.3 *Permeameter Cell Pressure System*—The system for pressurizing the permeameter cell shall be capable of applying and controlling the cell pressure to ±5 % or better of the applied pressure. However, the effective stress on the test specimen (which is the difference between the cell pressure and the pore water pressure) shall be maintained to the desired value with an accuracy of ±10 % or better. The device for pressurizing the cell may consist of a reservoir connected to the permeameter cell and partially filled with de-aired water, with the upper part of the reservoir connected to a compressed gas supply or other source of pressure (see Note 3). The gas pressure shall be controlled by a pressure regulator and measured by a pressure gage, electronic pressure transducer, or any other device capable of measuring to the prescribed tolerance. A hydraulic system pressurized by deadweight acting on a piston or any other pressure device capable of applying and controlling the permeameter cell pressure within the tolerance prescribed in this paragraph may be used.

NOTE 3—De-aired water is commonly used for the cell fluid to minimize potential for diffusion of air through the membrane into the specimen. Other fluids that have low gas solubilities such as oils, are also acceptable, provided they do not react with components of the permeameter. Also, use of a long (approximately 5 to 7 m) tube connecting the pressurized cell liquid to the cell helps to delay the appearance of air in the cell fluid and to reduce the flow of dissolved air into the cell.

5.4 *Permeameter Cell*—An apparatus shall be provided in which the specimen and porous end pieces, enclosed by a membrane sealed to the cap and base, are subjected to controlled fluid pressures. A schematic diagram of a typical permeameter cell and falling head (raising tailwater) hydraulic system is shown in Fig. 1.

5.4.1 The permeameter cell may allow for observation of changes in height of the specimen, either by observation through the cell wall using a cathetometer or other instrument, or by monitoring of either a loading piston or an extensometer extending through the top plate of the cell bearing on the top cap and attached to a dial indicator or other measuring device. The piston or extensometer should pass through a bushing and seal incorporated into the top plate and shall be loaded with sufficient force to compensate for the cell pressure acting over the cross-sectional area of the piston where it passes through the seal. If deformations are measured, the deformation indicator shall be a dial indicator or cathetometer graduated to 0.5 mm or 0.01 in. or better and having an adequate travel range. Any other measuring device meeting these requirements is acceptable.

5.4.2 In order to facilitate gas removal, and thus saturation of the hydraulic system, four drainage lines leading to the specimen, two each to the base and top cap, are recommended. The drainage lines shall be controlled by no-volume-change valves, such as ball valves, and shall be designed to minimize dead space in the lines.

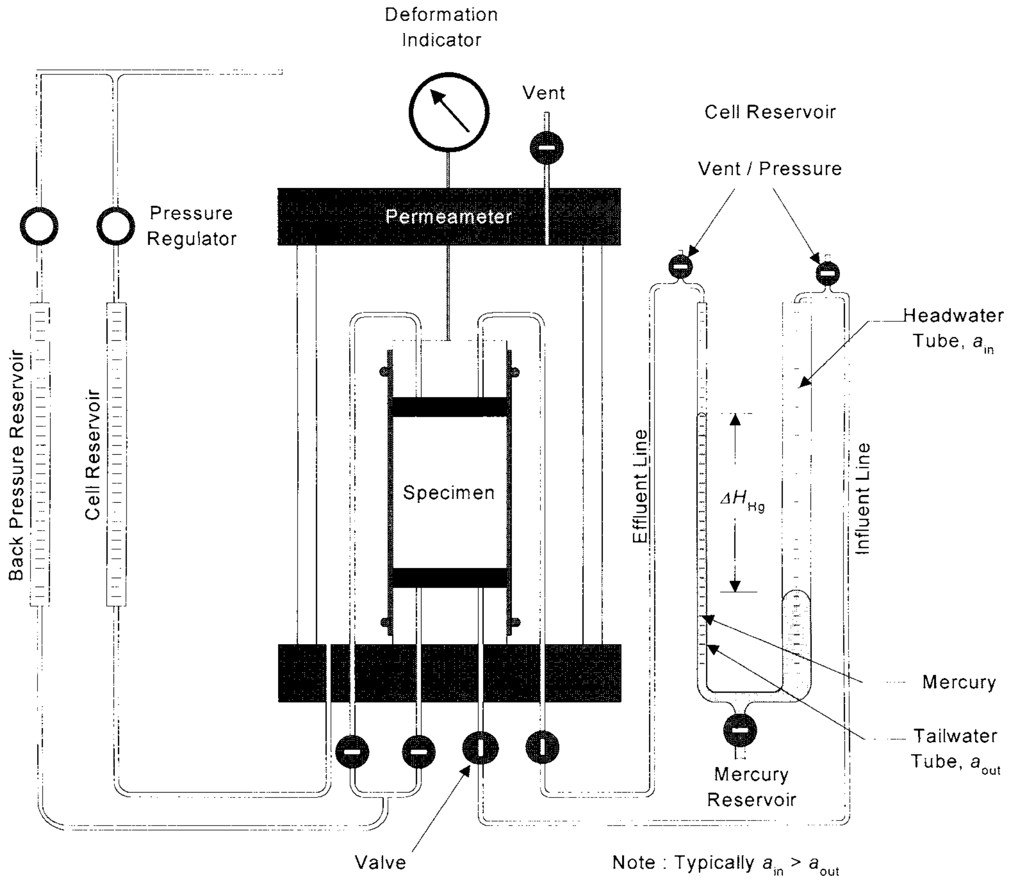

FIG. 4 Constant Volume – Falling Head System, Method F (5)

5.4.3 *Top Cap and Base*—An impermeable, rigid top cap and base shall be used to support the specimen and provide for transmission of permeant liquid to and from the specimen. The diameter or width of the top cap and base shall be equal to the diameter or width of the specimen to ±5 % or better. The base shall prevent leakage, lateral motion, or tilting, and the top cap shall be designed to receive the piston or extensometer, if used, such that the piston-to-top cap contact area is concentric with the cap. The surface of the base and top cap that contacts the membrane to form a seal shall be smooth and free of scratches.

5.4.4 *Flexible Membranes*—The flexible membrane used to encase the specimen shall provide reliable protection against leakage. The membrane shall be carefully inspected prior to use. If any flaws or pinholes are evident, the membrane shall be discarded. To minimize restraint to the specimen, the diameter or width of the non-stretched membrane shall be between 90 and 95 % of that of the specimen. The membrane shall be sealed to the specimen base and cap with rubber O-rings for which the unstressed, inside diameter or width is less than 90 % of the diameter or width of the base and cap, or by any other method that will produce an adequate seal.

NOTE 4—Membranes may be tested for flaws by placing them around a form sealed at both ends with rubber O-rings, subjecting them to a small air pressure on the inside, and then dipping them into water. If air bubbles come up from any point on the membrane, or if any visible flaws are observed, the membrane shall be discarded.

5.4.5 *Porous End Pieces*—The porous end pieces shall be of silicon carbide, aluminum oxide, or other material that is not attacked by the specimen or permeant liquid. The end pieces shall have plane and smooth surfaces and be free of cracks, chips, and discontinuities. They shall be checked regularly to ensure that they are not clogged.

5.4.5.1 The porous end pieces shall be the same diameter or width (±5 % or better) as the specimen, and the thickness shall be sufficient to prevent breaking.

5.4.5.2 The hydraulic conductivity of the porous end pieces shall be significantly greater than that of the specimen to be tested. The requirements outlined in 5.2.3 ensure this criterion is met.

5.4.6 *Filter Paper*—If necessary to prevent intrusion of material into the pores of the porous end pieces, one or more sheets of filter paper shall be placed between the top and bottom porous end pieces and the specimen. The paper shall have a negligibly small hydraulic impedance. The requirements outlined in 5.2.3 ensure that the impedance is small.

5.5 *Equipment for Compacting a Specimen*—Equipment (including compactor and mold) suitable for the method of compaction specified by the requester shall be used.

5.6 *Sample Extruder*—When the material being tested is a soil core, the soil core shall usually be removed from the sampler with an extruder. The sample extruder shall be capable of extruding the soil core from the sampling tube in the same direction of travel in which the sample entered the tube and with minimum disturbance of the sample. If the soil core is not extruded vertically, care should be taken to avoid bending stresses on the core due to gravity. Conditions at the time of sample extrusion may dictate the direction of removal, but the principal concern is to keep the degree of disturbance minimal.

5.7 *Trimming Equipment*—Specific equipment for trimming the specimen to the desired dimensions will vary depending on quality and characteristics of the sample (material). However, the following items listed may be used: lathe, wire saw with a wire about 0.3 mm (0.01 in.) in diameter, spatulas, knives, steel rasp for very hard clay specimens, cradle or split mold for trimming specimen ends, and steel straight edge for final trimming of specimen ends.

5.8 *Devices for Measuring the Dimensions of the Specimen*—Devices used to measure the dimensions of the specimen shall be capable of measuring to the nearest 0.5 mm or 0.01 in. or better (see 8.1.1) and shall be constructed such that their use will not disturb the specimen.

5.9 *Balances*—The balance shall be suitable for determining the mass of the specimen and shall be selected as discussed in Specification D4753. The mass of specimens less than 100 g shall be determined to the nearest 0.01 g. The mass of specimens between 100 g and 999 g shall be determined to the nearest 0.1 g. The mass of specimens equal to or greater than 1000 g shall be determined to the nearest gram.

5.10 *Equipment for Mounting the Specimen*—Equipment for mounting the specimen in the permeameter cell shall include a membrane stretcher or cylinder, and ring for expanding and placing O-rings on the base and top cap to seal the membrane.

5.11 *Vacuum Pump*—To assist with de-airing of permeant liquid (water) and saturation of specimens.

NOTE 5—For guidance or avoiding excessive consolidation in the use of vacuum for specimen saturation, consult 8.2 of Test Method D4767.

5.12 *Temperature Maintaining Device*— The temperature of the permeameter, test specimen, and reservoir of permeant liquid shall not vary more than ±3°C or ±6°F or better. Normally, this is accomplished by performing the test in a room with a relatively constant temperature. If such a room is not available, the apparatus shall be placed in a water bath, insulated chamber, or other device that maintains a temperature within the tolerance specified above. The temperature shall be periodically measured and recorded.

5.13 *Water Content Containers*—The containers shall be in accordance with Method D2216.

5.14 *Drying Oven*—The oven shall be in accordance with Test Method D2216.

5.15 *Time Measuring Device(s)*—Devices to measure the duration of each permeation trial, such as either a clock with a second hand or a stopwatch (or equivalent), or both.

6. Reagents

6.1 *Permeant Water*:

6.1.1 The permeant water is the liquid used to permeate the test specimen and is also the liquid used in backpressuring the specimen.

6.1.2 The type of permeant water should be specified by the requestor. If no specification is made, potable tap water shall be used for the permeant liquid. The type of water utilized shall be indicated in the test data sheet/form.

6.1.2.1 Chemical interactions between a permeant liquid and the porous material may lead to variations in hydraulic conductivity. Distilled water can significantly lower the hydraulic conductivity of clayey soils (2). For this reason, distilled water is not usually recommended as a permeant liquid. A permeant liquid used by some is a 0.01 molar $CaCl_2$ solution, which can be obtained for example, by dissolving 11.1 g of reagent-grade $CaCl_2$ in 10 L of de-aired, distilled water (commercial grade) or deionized water. This $CaCl_2$ solution is thought to neither increase nor decrease significantly the hydraulic conductivity of clayey soils. In areas with extremely hard or soft water, the $CaCl_2$ solution is recommended. Its use is also recommended when the flow of permeant water is significant (greater than about ⅓ to ½ times the volume of voids). Additional de-airing may modify the concentration of this solution slightly, but this should not affect the hydraulic conductivity.

6.1.3 *Deaired Water*—To aid in removing as much air from the test specimen as possible, deaired water shall be used. The water is usually deaired by boiling, by spraying a fine mist of water into an evacuated vessel attached to a vacuum source, or by forceful agitation of water in a container attached to a vacuum source. If boiling is used, care shall be taken not to evaporate an excessive amount of water, which can lead to a larger salt concentration in the permeant water than desired. To prevent dissolution of air back into the water, deaired water shall not be exposed to air for prolonged periods.

7. Hazards

7.1 *Warning*—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury or mercury-containing products or both into your state may be prohibited by state law.

7.1.1 Tubing composed of glass or other brittle materials may explode/shatter when under pressure, especially air. Therefore, such tubing should be enclosed. Establish allowable working pressures and make sure they are not exceeded.

7.2 *Precaution*—In addition to other precautions, store mercury in sealed shatterproof containers to control evaporation. When adding/subtracting mercury to/from the hydraulic system used in Method E or F, work in a well-ventilated area (preferably under a fume hood), and avoid contact with skin. Rubber gloves should be worn at all times when contact with mercury is possible.

7.2.1 Minimize uncontrolled flow of mercury out of the specialized hydraulic system by installing mercury traps or an inline check-valve mechanism. Minimize uncontrolled spills by using shatterproof materials or protective shields, or both.

7.2.2 If mercury comes into contact with brass/copper fittings, valves, etc., such items may rapidly become leaky. Therefore, where-ever practical use stainless steel fittings, etc.

7.2.3 Clean up spills immediately using a recommended procedure explicitly for mercury.

7.2.4 Dispose of contaminated waste materials containing mercury in a safe and environmentally acceptable manner.

8. Test Specimens

8.1 *Size*—Specimens shall have a minimum diameter of 25 mm (1.0 in.) and a minimum height of 25 mm. The height and diameter of the specimen shall be measured to three significant digits or better (see 8.1.1). The length shall vary by no more than ±5 %. The diameter shall vary by no more than ±5 %. The surface of the test specimen may be uneven, but indentations must not be so deep that the length or diameter vary by more than ±5 %. The diameter and height of the specimen shall each be at least 6 times greater than the largest particle size within the specimen. If, after completion of a test, it is found based on visual observation that oversized particles are present, that information shall be indicated on the data sheet(s)/form(s).

8.1.1 If the density or unit weight needs to be determined/recorded to four significant digits, or the void ratio to three significant digits; then the test specimens dimensions need to have four significant digits; i.e., typically measured to the nearest 0.01 mm or 0.001 in.

8.1.2 Specimens of soil-cement and mixtures of cement, bentonite, and soils often have more irregular surfaces than specimens of soil. Thus, for these specimens the length and the diameter may vary by no more than ±10 %.

NOTE 6—Most hydraulic conductivity tests are performed on cylindrical test specimens. It is possible to utilize special equipment for testing prismatic test specimens, in which case reference to “diameter” in 8.1 applies to the least width of the prismatic test specimen.

8.2 *Undisturbed Specimens*—Undisturbed test specimens shall be prepared from a representative portion of undisturbed samples secured in accordance with Practice D1587, Practice D3550, Practice D6151, or Practice D2113. In addition, undisturbed samples may be obtained by “block sampling” (**6**). Additional guidance on other drilling and sampling methods is given in Guide D6169. Samples shall be preserved and transported in accordance with these requirements; for soils follow Group C in Practice D4220, while for rock follow either “special care” or “soil-like care,” as appropriate in Practice D5079. Specimens obtained by tube sampling or coring may be tested without trimming except for cutting the end surfaces plane and perpendicular to the longitudinal axis of the specimen, provided soil characteristics are such that no significant disturbance results from sampling. Where the sampling operation has caused disturbance of the soil, the disturbed material shall be trimmed. Where removal of pebbles or crumbling resulting from trimming causes voids on the surface of the specimen that cause the length or diameter to vary by more than ±5 %, the voids shall be filled with remolded material obtained from the trimmings. The ends of the test specimen shall be cut and not troweled (troweling can seal off cracks, slickensides, or other secondary features that might conduct water flow). Specimens shall be trimmed, whenever possible, in an environment where changes in water content are minimized. A controlled high-humidity room is usually used for this purpose. The mass and dimensions of the test specimen shall be determined to the tolerances given in 5.8 and 5.9. The test specimen shall be mounted immediately in the permeameter. The water content of the trimmings shall be determined in accordance with Method D2216, to the nearest 0.1 % or better.

8.3 *Laboratory-Compacted Specimens*—The material to be tested shall be prepared and compacted inside a mold in a manner specified by the requester. If the specimen is placed and compacted in layers, the surface of each previously-compacted layer shall be lightly scarified (roughened) with a fork, ice pick, or other suitable object, unless the requester specifically states that scarification is not to be performed. Test Methods D698 and D1557 describe two methods of compaction, but any other method specified by the requester may be used as long as the method is described in the report. Large clods of material should not be broken down prior to compaction unless it is known that they will be broken in field construction, as well, or the requester specifically requests that the clod size be reduced. Neither hard clods nor individual particles of the material shall exceed ⅙ of either the height or diameter of the specimen. After compaction, the test specimen shall be removed from the mold, the ends scarified, and the dimensions and weight determined within the tolerances given in 5.8 and 5.9. After the dimensions and mass are determined, the test specimen shall be immediately mounted in the permeameter. The water content of the trimmings shall be determined in accordance with Method D2216 to the nearest 0.1 % or better.

8.4 *Other Preparation Methods*—Other methods of preparation of a test specimen are permitted if specifically requested. The method of specimen preparation shall be identified in the data sheet(s)/form(s).

8.5 After the height, diameter, mass, and water content of the test specimen have been determined, the dry unit weight shall be calculated. Also, the initial degree of saturation shall be estimated (this information may be used later in the back-pressure stage).

8.6 In some cases, the horizontal hydraulic conductivity of a sample needs to be determined. In that case, the specimen may be trimmed such that its longitudinal axis is perpendicular to the longitudinal axis of the sample. Obtaining a specimen having a diameter of 36 mm (1.4 in.) typically requires a cylindrical sample with a diameter equal to or greater than about 70 mm (2.8 in.) or a rectangular sample with a minimum dimension of about 40 mm (1.6 in.).

9. Procedure

9.1 *Specimen Setup*:

9.1.1 Cut two filter paper sheets to approximately the same shape as the cross section of the test specimen. Soak the two porous end pieces and filter paper sheets, if used, in a container of permeant water.

9.1.2 Place the membrane on the membrane expander. Apply a thin coat of silicon high-vacuum grease to the sides of the end caps. Place one porous end piece on the base and place one filter paper sheet, if used, on the porous end piece, followed by the test specimen. Place the second filter paper sheet, if used, on top of the specimen followed by the second porous end piece and the top cap. Place the membrane around the specimen, and using the membrane expander or other suitable O-ring expander, place one or more O-rings to seal the membrane to the base and one or more additional O-rings to seal the membrane to the top cap.

9.1.3 Attach flow tubing to the top cap, if not already attached, assemble the permeameter cell, and fill it with de-aired water or other cell fluid. Attach the cell pressure reservoir to the permeameter cell line and the hydraulic system to the influent and effluent lines. Fill the cell pressure reservoir with deaired water, or other suitable liquid, and the hydraulic system with deaired permeant water. Apply a small confining pressure of 7 to 35 kPa (1 to 5 psi) to the cell and apply a pressure less than the confining pressure to both the influent and effluent systems, and flush permeant water through the flow system. After all visible air has been removed from the flow lines, close the control valves. At no time during saturation of the system and specimen or hydraulic conductivity measurements shall the maximum applied effective stress be allowed to exceed that to which the specimen is to be consolidated.

9.2 *Specimen Soaking (Optional)*—To aid in saturation, specimens may be soaked under partial vacuum applied to the top of the specimen. Water under atmospheric pressure shall be applied to the specimen base through the influent lines, and the magnitude of the vacuum set to generate a hydraulic gradient across the specimen less than that which will be used during hydraulic conductivity measurements.

NOTE 7—Soaking under vacuum is applicable when there are continuous air voids in the specimen e.g., specimens having a degree of saturation of less than about 85%. The specimen may swell when exposed to water; the effective stress will tend to counteract the swelling. However, for materials that tend to swell, unless the applied effective stress is greater than or equal to the swell pressure, the specimen will swell. In addition, see Note 5.

9.3 *Back-Pressure Saturation*—To saturate the specimen, back pressuring is usually necessary. Fig. 5 (**7**) provides guidance on back pressure required to attain saturation. Additional guidance on the back-pressure process is given by Black and Lee (**8**) and Head (**9**).

NOTE 8—The relationships presented in Fig. 5 are based on the assumption that the water used for back pressuring is deaired and that the only source for air to dissolve into the water is air from the test specimen. If air pressure is used to control the back pressure, pressurized air will dissolve into the water, thus reducing the capacity of the water used for back pressure to dissolve air located in the pores of the test specimen. The problem is minimized by using a long (>5 m) tube that is impermeable to air between the air-water interface and test specimen, by separating the back-pressure water from the air by a material or fluid that is relatively impermeable to air, by periodically replacing the back-pressure water with deaired water, or by other means.

9.3.1 During the saturation process, any change in the volume (swelling or compression of the void ratio, density, etc.) of the test specimen should be minimized. The easiest way to verify that volume changes are minor is to measure the height of the specimen during the back-pressuring process. Volume changes are considered minor if the resulting change in hydraulic conductivity is less than about one-half the acceptable error of 25 % given in 9.5.4, unless more stringent control on density or hydraulic conductivity, or both, is required. For this to occur the axial strain should be less than about 0.4 % for normally consolidated soils, or about 0.1 % for overconsolidated soils. See Appendix X2.

9.3.2 Take and record an initial reading of specimen height, if being monitored. Open the flow line valves and flush out of the system any free air bubbles using the procedure outlined in 9.1.3. If an electronic pressure transducer or other measuring device is to be used during the test to measure pore pressures or applied hydraulic gradient, bleed any trapped air from the device.

9.3.3 Adjust the applied confining pressure to the value to be used during saturation of the specimen. Apply back pressure by simultaneously increasing the cell pressure and the influent

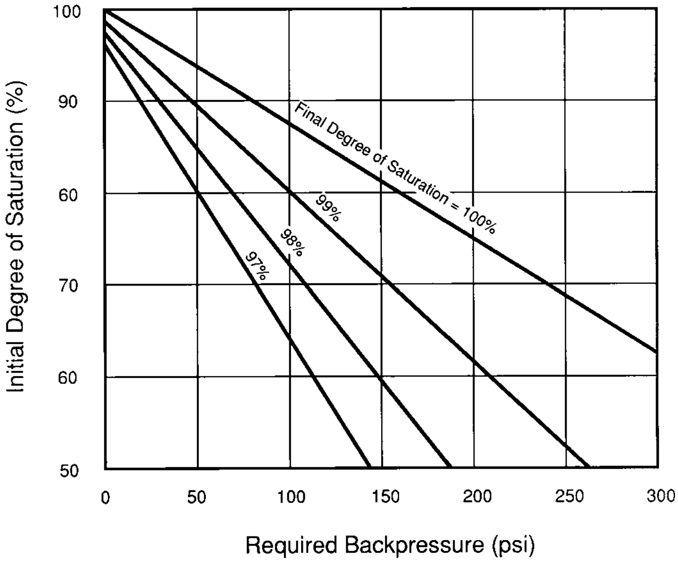

FIG. 5 Back Pressure to Attain Various Degrees of Saturation (7)

and effluent pressures in increments. The maximum value of an increment in back pressure shall be sufficiently low such that no point in the specimen is exposed to an effective stress in excess of that to which the specimen will be subsequently consolidated. At no time shall a head be applied such that the effective confining stress is <7 kPa (1 psi) because of the danger of separation of the membrane from the test specimen. Maintain each increment of pressure for a period of a few minutes to a few hours, depending upon the characteristics of the specimen. To assist in removal of trapped air, a small hydraulic gradient may be applied across the specimen to induce flow.

9.3.4 Saturation shall be verified with one of the three following techniques:

9.3.4.1 Saturation may be verified by measuring the *B* coefficient as described in Test Method D4767 (see Note 9). The test specimen shall be considered to be adequately saturated if the *B* value is ≥0.95, or for relatively incompressible materials, for example, rock, if the *B* value remains unchanged with application of larger values of back pressure. The *B* value may be measured prior to or after completion of the consolidation phase (see 9.4). An accurate *B*-value determination can only be made if no gradient is acting on the specimen and all pore-water pressure induced by consolidation has dissipated. That is, conform completion of primary consolidation before this determination; see Test Method D2435 or D4767 on how to confirm completion of primary consolidation.

NOTE 9—The *B* coefficient is defined for this type of test as the change in pore-water pressure in the porous material divided by the change in confining pressure. Compressible materials that are fully saturated with water will have a *B* value of 1.0. Relatively incompressible, saturated materials have *B* values that are somewhat less than 1.0 (**10**).

9.3.4.2 Saturation of the test specimen may be confirmed at the completion of the test by calculation of the final degree of saturation. The final degree of saturation shall be 100 ± 5 %. However, measurement of the *B* coefficient as described in 9.3.4.1 or use of some other technique (9.3.4.3) is strongly recommended because it is much better to confirm saturation prior to permeation than to wait until after the test to determine if the test was valid.

9.3.4.3 Other means for verifying saturation, such as observing the flow of water into the specimen when the back pressure is increased, can be used for verifying saturation provided data are available for similar materials to establish that the procedure used confirms saturation as required in 9.3.4.1 or 9.3.4.2.

9.4 *Consolidation*—The specimen shall be consolidated to the effective stress specified by the requester. Consolidation shall be accomplished in stages, with the increase in cell pressure minus back pressure (effective stress) in each new stage equal to or less than the effective stress in the previous stage i.e., consolidation increment ratio of one or less.

NOTE 10—The test specimen may be consolidated prior to application of back pressure. Also, the back pressure and consolidation phases may be completed concurrently if back pressures are applied sufficiently slowly to minimize potential for overconsolidation of the specimen.

9.4.1 Record the specimen height, if being monitored, prior to application of consolidation pressure and periodically during consolidation.

9.4.2 Increase the cell pressure to the level necessary to develop the desired effective stress, and begin consolidation. Drainage may be allowed from the base or top of the specimen, or simultaneously from both ends.

9.4.3 (Optional) Record outflow volumes to confirm that primary consolidation has been completed prior to initiation of the hydraulic conductivity test. Alternatively, measurements of the change in height of the test specimen can be used to confirm completion of consolidation.

NOTE 11—The procedure in 9.4.3 is optional because the requirements of 9.5 ensure that the test specimen is adequately consolidated during permeation because if it is not, inflow and outflow volumes will differ significantly. However, for accurate *B*-value determination, saturation should be confirmed at the completion of consolidation (see 9.3.4.1). Recording outflow volumes or height changes is recommended as a means for verifying the completion of consolidation prior to initialization of permeation. Also, measurements in the change in height of the test specimen, coupled with knowledge of the initial height, provide a means for checking the final height of the specimen.

9.5 *Permeation*:

9.5.1 *Hydraulic Gradient*—When possible, the hydraulic gradient ($i = \Delta h/L$, for definitions of notation see 10.1) used for hydraulic conductivity measurements should be similar to that expected to occur in the field. In general, hydraulic gradients from <1 to 5 cover most field conditions. However, the use of small hydraulic gradients can lead to very long testing times for materials having low hydraulic conductivity (less than about 1×10^{-8} m/s). Somewhat larger hydraulic gradients are usually used in the laboratory to accelerate testing, but excessive gradients must be avoided because high seepage pressures may consolidate the material, material may be washed from the specimen, or fine particles may be washed downstream and plug the effluent end of the test specimen. These effects could increase or decrease hydraulic conductivity. If no gradient is specified by the requestor, the following guidelines may be followed:

Hydraulic Conductivity, m/s	Recommended Maximum Hydraulic Gradient
1×10^{-5} to 1×10^{-6}	2
1×10^{-6} to 1×10^{-7}	5
1×10^{-7} to 1×10^{-8}	10
1×10^{-8} to 1×10^{-9}	20
less than 1×10^{-9}	30

9.5.1.1 A higher gradient than given above may be used if the higher gradient can be shown not to change the hydraulic conductivity. For example, on a representative specimen, perform a hydraulic conductivity determination at $i = 30$ than at $i = 50$ or 100, or more. Determine which, if any, of the hydraulic conductivities (k) determined at these gradients are similar (i.e., within the acceptable steady-state range given for the Method (A, B, C, D, E, or F). Any gradient equal to or less than the highest gradient yielding a similar hydraulic conductivity may be used for testing.

NOTE 12—Seepage pressures associated with large hydraulic gradients can consolidate soft, compressible specimens and reduce their hydraulic conductivity. Smaller hydraulic gradients (<10) may be necessary for such specimens.

9.5.2 *Initialization*—Initiate permeation of the specimen by increasing the influent (headwater) pressure (see 9.3.3). The effluent (tailwater) pressure shall not be decreased because air bubbles that were dissolved by the specimen water during backpressuring may come out of solution if the pressure is decreased. The back pressure shall be maintained throughout the permeation phase.

9.5.2.1 The maximum increase in headwater pressure cannot exceed 95 % of the effective consolidation stress. Alternatively, the difference between the cell pressure and the total headwater pressure cannot be less than 5 % of the effective consolidation stress.

9.5.2.2 At the start and end of each permeation trial, at t_1 and t_2, read and record the test temperature to the nearest 0.1°C. See Section 10. If the number of significant digits in the calculation of hydraulic conductivity at 20°C can be one, then the test temperature can be measured to the nearest degree Celsius.

9.5.3 *Time Measurements*—Measure and record the time at the start and end of each permeation trial (or its interval) to two or more significant digits. That is the time interval has to be greater than 9 s unless the time is recorded to the nearest 0.1 s.

9.5.4 *Constant Head Tests*:

9.5.4.1 *(Method A)*—Measure and record the required head loss across the tolerances and significant digits stated in 5.1.1 and 5.2.3 at the start and end of each permeation trial (as a minimum). The head loss across the permeameter shall be kept constant to ±5 % or better. Measure and record periodically the quantity of inflow as well as the quantity of outflow to a minimum of three significant digits. Also measure and record any changes in height of the test specimen, if being monitored (see Note 12). Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which: (*1*) the ratio of outflow to inflow rate is between 0.75 and 1.25, and (*2*) the hydraulic conductivity is steady. The hydraulic conductivity shall be considered steady if four or more consecutive hydraulic conductivity determinations fall within ±25 % or better of the mean value for $k \geq 1 \times 10^{-10}$ m/s or within ±50 % or better for $k < 1 \times 10^{-10}$ m/s, and a plot or tabulation of the hydraulic conductivity versus time shows no significant upward or downward trend.

9.5.4.2 *Method E (Constant Volume)*—Measure and record the required head loss across the permeameter to the tolerances and significant digits stated in 5.1.4. The head loss across the permeameter shall be kept constant to ±5 % or better. Measure and record, to a minimum of three significant digits, the quantity of either inflow (influent) or outflow (effluent). In this measurement the last digit may be due to estimation, see 5.1.1.1. In addition, measure and record any changes in the height of the test specimen, if being monitored (see Note 12). Continue permeation until at least two or more values of hydraulic conductivity (k) are steady. The hydraulic conductivity shall be considered steady if two or more consecutive k determinations fall within ±15 % or better of the mean value (two or more determinations) for $k \geq 1 \times 10^{-10}$ m/s or within ±50 % or better for $k < 1 \times 10^{-10}$ m/s.

9.5.5 *Falling-Head Tests (Methods B, C, and F)*—Measure and record the required head loss across the permeameter to the tolerances and significant digits stated in 5.1.2. Measure and record these head losses at the start and end of each permeation trial (as a minimum). At no time shall the applied head loss across the specimen be less than 75 % of the initial (maximum) head loss during the hydraulic conductivity determination (see Note 13). At the start and end of each trial, as a minimum, measure and record any changes in the height of the test specimen, if being monitored. To meet these requirements, especially for Method F, the initial head loss in each trial will most likely have to be reset to the same value (±5 %) used in the first trial. In addition, the "75 % criterion" mentioned above has to be adhered to closely.

9.5.5.1 *Methods B and C*—The volumes of outflow and inflow shall be measured and recorded to three significant digits (the last digit may be due to estimation, see 5.1.1.1). Measure and record these volumes at the start and end of each permeation trial (as a minimum). Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which: the ratio of outflow to inflow rate is between 0.75 and 1.25, and the hydraulic conductivity is steady (see 9.5.4.1).

NOTE 13—When the water pressure in a test specimen changes and the applied total stress is constant, the effective stress in the test specimen changes, which can cause volume changes that can invalidate the test results. The requirement that the head loss not decrease very much is intended to keep the effective stress from changing too much. For extremely soft, compressible test specimens, even more restrictive criteria may be needed. Also, when the initial and final head losses across the test specimen do not differ by much, great accuracy is needed to comply with the requirement of 5.1.2 that the ratio of initial to final head loss be determined with an accuracy of ±5 % or better. When the initial and final head loss over an interval of time do not differ very much, it may be possible to comply with the requirements for a constant head test (9.5.4) in which the head loss must not differ by more than ±5 % and to treat the test as a constant head test.

9.5.5.2 *Method F (Constant Volume)*—Continue permeation until at least two or more values of hydraulic conductivity (k) meet the requirements stated in 9.5.4.2.

9.5.6 *Constant Rate of Flow Tests (Method D)*—Initiate permeation of the specimen by imposing a constant flow rate. Choose the flow rate so the hydraulic gradient does not exceed the value specified, or if none is specified, the value recommended in 9.5.1. Periodically measure the rate of inflow, the rate of outflow, and head loss across the test specimen to the tolerances and significant digits given in 5.1.3. Also, measure and record any changes in specimen height, if being monitored. Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which the ratio of inflow to outflow rates is between 0.75 and 1.25, and hydraulic conductivity is steady (see 9.5.4.1).

9.6 *Final Dimensions of the Specimen*—After completion of permeation, reduce the applied confining, influent, and effluent pressures in a manner that does not generate significant volume change of the test specimen. Then carefully disassemble the permeameter cell and remove the specimen. Measure and record the final height, diameter, and total mass of the specimen. Then determine the final water content of the specimen by the procedure of Method D2216. Dimensions and mass of the test specimen shall be measured to the tolerances specified in 5.8 and 5.9.

NOTE 14—The specimen may swell after removal of back pressure as a result of air coming out of solution. A correction may be made for this effect, provided that changes in the length of the specimen are monitored during the test. The strain caused by dismantling the cell is computed from the length of the specimen before and after dismantling the cell. The same strain is assumed to have occurred in the diameter. The corrected diameter and actual length before the back pressure was removed are used to compute the volume of the test specimen prior to dismantling the cell. The volume prior to dismantling the cell is used to determine the final dry density and degree of saturation.

10. Calculation

10.1 *Constant Head and Constant Rate of Flow Tests*:

10.1.1 *Methods A and D*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{\Delta Q \cdot L}{A \cdot \Delta h \cdot \Delta t} \tag{1}$$

where:

k = hydraulic conductivity, m/s,
ΔQ = quantity of flow for given time interval Δt, taken as the average of inflow and outflow, m^3,
L = length of specimen, m,
A = cross-sectional area of specimen, m^2,
Δt = interval of time, *s*, over which the flow ΔQ occurs $(t_2 - t_1)$,
t_1 = time at start of permeation trial, date: hr:min:sec,
t_2 = time at end of permeation trial, date: hr:min:sec,
Δh = average head loss across the permeameter/specimen $((\Delta h_1 + \Delta h_2)/2)$, m of water,
Δh_1 = head loss across the permeameter/specimen at t_1, m of water, and
Δh_2 = head loss across the permeameter/specimen at t_2, m of water.

NOTE 15—The interval of time, Δt, can be measured directly using a stop watch or equivalent device, see 11.5.1. Units other than second(s), meters (m), etc., may be used providing an appropriate unit conversion factor (UCF) is used so *k* is in m/s or other units, if requested or customary (see Section 11).

10.1.2 *Method E*—Use the above Eq 1. If the height of the mercury column in the "Constant Head" tube is used to determine the head loss, Δh, use the following equation.

$$\Delta h = \Delta H_{Hg} \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1 \right) = \Delta H_{Hg} \cdot (G_{Hg} - 1) \tag{2}$$

where:

ΔH_{Hg} = the peak to peak height of mercury column (see Fig. 2), m, and
ρ_{Hg} = the density of mercury, g/cm^3,
ρ_{wg} = the density of water, g/cm^3,
G_{Hg} = the ratio of the density of mercury to the density of water (specific gravity of mercury) at the test/trial temperature. See Table 1.

NOTE 16—For the constant-volume hydraulic systems, there is no head loss across the permeameter/specimen due to elevation head. Units other than seconds (s), meters (m), etc., may be used providing an appropriate UCF is used so *k* is in m/s or other units, if requested or customary (see Section 11).

10.2 *Falling-Head Tests*:

TABLE 1 Specific Gravity of Mercury (G_{Hg})

Temperature (°C)	$G_{Hg} = (\rho_{Hg}/\rho_w)$
15	13.570
16	13.570
17	13.570
18	13.570
19	13.570
20	13.570
21	13.571
22	13.571
23	13.572
24	13.573
25	13.574
26	13.575
27	13.576
28	13.577
29	13.579
30	13.580

10.2.1 *Constant Tailwater Pressure (Method B)*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{a \cdot L}{A \cdot \Delta t} \ln \left(\frac{\Delta h_1}{\Delta h_2} \right) \tag{3}$$

where:

a = cross-sectional area of the reservoir containing the influent liquid, m^2, and
ln = natural logarithm (base e = 2.71828).

See Note 15.

10.2.2 *Increasing Tailwater Pressure (Method C)*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{a_{in} \cdot a_{out} \cdot L}{(a_{in} + a_{out}) \cdot A \cdot \Delta t} \ln \left(\frac{\Delta h_1}{\Delta h_2} \right) \tag{4}$$

where:

a_{in} = cross-sectional area of the reservoir containing the influent/inflow liquid, m^2, and
a_{out} = cross-sectional area of the reservoir containing the effluent/outflow liquid, m^2.

See Note 15.

NOTE 17—For the case in which $a_{out} = a_{in} = a$, the equation for calculating *k* for a falling head test with a rising tailwater level is:

$$k = \frac{a \cdot L}{2 \cdot A \cdot \Delta t} \ln \left(\frac{\Delta h_1}{\Delta h_2} \right) \tag{5}$$

where:

a = area of the reservoirs containing either the influent/inflow or effluent/outflow liquid, m^2

10.2.3 *Constant-Volume System (Method F)*— Calculate the hydraulic conductivity, *k*, as follows:

$$k = \left(\frac{a_{in} \cdot a_{out}}{(a_{out} + a_{in})} \cdot \frac{1}{(G_{Hg} - 1)} \right) \cdot \frac{L}{A} \cdot \frac{1}{\Delta t} \cdot \ln \left(\frac{\Delta h_1}{\Delta h_2} \right) \tag{6}$$

10.2.3.1 If the differential elevation between the top surfaces of the mercury level in the headwater and tailwater tubes is used to determine the head loss, Δh, use the following equations.

a) For the head loss at the start of the permeation trial, h_1:

$$h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1 \right) = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1) \tag{7}$$

where:

$\Delta H_{Hg,1}$ = difference in elevation between the top surfaces of the mercury level in the tailwater and headwater tubes at the start of the permeation trial, t_1(see Fig. 3), m, and

$\Delta H_{Hg,c}$ = difference in elevation of mercury in the headwater and tailwater tubes of the manometer with equal pressures applied to both tubes, m. This value is positive if the inside diameter (ID) of the headwater tube is larger than the ID of the tailwater tube, and negative if the opposite is true. A discussion on capillary head is given in Appendix X1, X1.2.3.2 and X1.4. See Note 16.

b) For the head loss at the end of the permeation trial, Δh_2,:

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1 \right) \cdot (G_{\text{Hg}} - 1) \right) \tag{8}$$

where:

$-\Delta Hg_{\text{tail}}$ = the negative change in elevation of the mercury levels in the tailwater tube during the permeation trial, m.

The reason why ΔHg_{tail} is used instead of $\Delta Hg_{\text{Hg},2}$ (difference in mercury levels at end of trial) is explained in Appendix X1, X1.3.2.1.

10.3 *Hydraulic Conductivity at Standard Temperature*—Correct the hydraulic conductivity to that for 20°C (68°F), k_{20}, by multiplying k by the ratio of the viscosity of water at test temperature to the viscosity of water at 20°C (68°F), R_T:

$$k_{20} = R_T \cdot K \tag{9}$$

with

$$R_T = 2.2902\,(0.9842^{T})/T^{0.1702} \tag{10}$$

where:

k_{20} = hydraulic conductivity corrected to 20°C, m/s

R_T = ratio of the viscosity of water at test temperature to the viscosity of water at 20°C

T = average test temperature during the permeation trial $((T_1 + T_2)/2)$, to the nearest 0.1°C.

T_1 = test temperature at start of permeation trial, to nearest 0.1 °C, and

T_2 = test temperature at end of permeation trial, to nearest 0.1 °C

10.3.1 The equation for R_T is only accurate to three significant digits between 5 and 50°C (41 and 122°F), see 1.1.

10.3.2 If the number of significant digits in the calculation of hydraulic conductivity at 20°C can be one, then the test temperature can be measured to the nearest °C.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.5.

11.2 Record as a minimum the following general information (data):

11.2.1 Sample/specimen identifying information, such as Project No., Boring No., Sample No., Depth, etc.

11.2.2 Any special selection and preparation process, such as removal of gravel or other materials, or identification of their presence, if "undisturbed" specimen.

11.2.3 If the specimen is reconstituted, remolded or trimmed in a specialized manner (determine horizontal hydraulic conductivity, see 8.6), provide information on method of reconstitution, remolding, etc.

11.3 Record as a minimum the following test specimen data:

11.3.1 The measured specific gravity test (Test Method D854) or assumed value.

11.3.2 The initial mass, dimensions (length and diameter), area, and volume of the specimen, to either three or four significant digits (see 8.1 and 8.1.1).

11.3.3 The initial water content (nearest 0.1 percent), dry unit weight (three or four significant digits, see 8.1.1) and saturation (nearest percent) of the test specimen.

11.3.4 The final mass, dimensions (length and diameter), area, and volume of the specimen, to either three or four significant digits (see 8.1 and 8.1.1).

11.3.5 The final water content (nearest 0.1 percent), dry unit weight (three or four significant digits, see 8.1.1) and saturation (nearest percent) of the test specimen.

11.4 Record as a minimum the following test boundary conditions:

11.4.1 The type of permeant liquid used.

11.4.2 The magnitude of total back pressure (two significant digits or three if used in the head loss determination).

11.4.3 The effective consolidation stress (two or more significant digits).

11.4.4 The area of the headwater and tailwater tubes (such as burettes, reservoirs, U-tube manometers, etc.), as applicable (three or more significant digits).

11.4.5 The length (L) and area (A) of the test specimen during permeation (minimum of three significant digits).

11.4.5.1 These values can be determined based on either *a)* the initial dimensions of specimen plus any length/height and volume changes occurring during saturation and consolidation; or *b)* final dimensions of the test specimen, see 11.3.4.

11.5 Record as a minimum the following permeation data:

11.5.1 The date, time (or start and elapsed time), temperature (nearest 0.1°C see 10.3.2), head loss reading(s), flow reading(s) (if applicable), and deformation gage (if applicable) at the start and end of each trial/determination. Applicable measurements/readings and any averages/differences calculated using measurements/readings obtained shall have two or more significant digits, unless specified differently in Section 9.

11.5.2 The calculated initial hydraulic gradient and ending value if falling head Method B, C, or F is being used, and the hydraulic conductivity to two or more significant digits.

11.5.3 The average corrected hydraulic conductivity (k_{20}, see 10.3) for the values meeting the applicable requirements in 9.5.4 to 9.5.6. Record this value to two or three significant digits in units of m/s or other units, if requested or customary, for example, 7.1×10^{-10} or 7.13×10^{-10}m/s.

11.5.4 A graph or table of hydraulic conductivity versus time or pore volumes of flow is recommended, unless a constant-volume hydraulic system is used.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is based on an interlaboratory study of D5084, Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter, conducted in 2008. Each of twelve laboratories tested three different soil types:

ML-1: Vicksburg silt (ASTM Reference Soil ML-1)
CH-1: Vicksburg clay (ASTM Reference Soil CH-1)
CL-1: Annapolis clay (ASTM Reference Soil CL-1)

All three soils are from the D18 ISR Reference Soils and Testing Program. Index properties for the soils are shown in Table 2. These properties are from the ASTM Reference Soils and Testing Program.

Every "test result" represents an individual determination. Each laboratory reported three replicate test results for the analyses. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D18-D1018.[4]

12.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 3.

12.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits are listed in Table 3.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 would have an approximately 95 % probability of being correct.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method. Therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 104 results, from twelve laboratories, on the three soils described in 12.1.

13. Keywords

13.1 coefficient of permeability; constant head; constant rate of flow; constant volume; falling head; hydraulic barriers; hydraulic conductivity; liner; permeability; permeameter

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1018.

TABLE 2 Index Properties for ASTM Reference Soils Used in Interlaboratory Study on Saturated Hydraulic Conductivity

ASTM Reference Soil ID	Liquid Limit per D4318 (%)	Plasticity Index per D4318 (%)	Specific Gravity of Soils per D854 (-)	Percent Finer than No. 200 Sieve per D1140 (%)
ML-1	27.3 ± 2.5	3.9 ± 4.5	2.725 ± 0.043	99.0 ± 0.3
CL-1	33.2 ± 1.4	13.4 ± 3.7	2.675 ± 0.030	88.5 ± 0.8
CH-1	59.7 ± 2.8	39.3 ± 7.0	2.726 ± 0.032	98.8 ± 0.4

TABLE 3 Hydraulic Conductivity Statistics from ILS Report RR:D18-D1018 (All Units in cm/s)

ILS Soil	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
ML-1	1.2×10^{-6}	3.3×10^{-7}	4.4×10^{-7}	9.3×10^{-7}	1.2×10^{-6}
CL-1	3.8×10^{-8}	4.4×10^{-9}	6.2×10^{-9}	1.2×10^{-8}	1.8×10^{-8}
CH-1	3.6×10^{-9}	2.9×10^{-9}	4.7×10^{-9}	8.2×10^{-9}	1.3×10^{-8}

APPENDIXES

(Nonmandatory Information)

X1. DEVELOPMENT OF HYDRAULIC CONDUCTIVITY EQUATION FOR THE MERCURY CONSTANT VOLUME-FALLING HEAD HYDRAULIC SYSTEM

X1.1 *Introduction*—A schematic of a mercury constant volume-falling head hydraulic system is given in Fig. 4. In this figure, the falling head is applied by the difference in elevation between the mercury levels in the tailwater and headwater tubes of the mercury U-tube manometer. In designing this type of hydraulic system, the area of the tailwater tube (a_{out}) is made significantly smaller than that of the headwater tube (a_{in}). This is done for three reasons:

First, to increase the sensitivity of the flow/volume measurement;

Second, to decrease the time required to measure the hydraulic conductivity; and,

Third, so one can clean the hydraulic system by flushing water through the tailwater tube and out the headwater tube of the mercury U-tube manometer without loosing mercury.

X1.1.1 The tubing lines leading from the test specimen to mercury U-tube manometer are filled with water, as well as the spaces above the mercury in the manometer. Therefore, the volume of the saturated test specimen remains constant during permeation. This occurs because the components (water, tubing, and manometer) of the hydraulic system are relatively incompressible compared to soil. In addition, there is continuity of inflow and outflow of permeant water during permeation.

X1.1.2 The presentation of determining heads and head losses in a mercury constant volume-falling head hydraulic system is presented before the development of the hydraulic conductivity equation. This allows one to become familiar with the notation and required parameters before addressing that relatively complicated equation development process. The heads involved are the total head (H), pressure head (H_p), elevation head (H_e), capillary head (H_c), velocity head (H_v), and total head loss (Δh).

X1.2 *Determination of Total Head and Total Head Loss*

X1.2.1 *Total Head*—The total head is equal to the sum of the pressure head (H_p), elevation head (H_e), velocity head (H_v), and capillary head (H_c). Usually it is expressed in height of water, such as m or cm of water. In addition, the velocity head is assumed to be zero/insignificant providing the conditions specified in 4.4 through 5.2.3.2 are met. The pressure head is the height of a vertical column of static water that can be supported by the static pressure (p) at a given point. It may be expressed as

$$H_p = \mathrm{UCF} \cdot \frac{p}{\rho_w \cdot g} \quad \text{(X1.1)}$$

where:

H_p = the pressure head at given point (units of vertical height of water column, m),
UCF = a unit conversion factor,
p = the static pressure at a given point (units of force per unit area, kPa),
ρ_w = the density of the water (units of mass per unit volume, Mg/m^3), and
g = the acceleration of gravity, convert mass to force (9.80665 m/s^2 or 980.665 cm/s^2).

X1.2.2 The static pressure at any point within a confined fluid may be calculated as shown in Fig. X1.1, assuming there is no drop in pressure due to velocity head loss. This figure shows that fluid pressure for Point O at elevation El. O on the "out" or tailwater side may be expressed as:

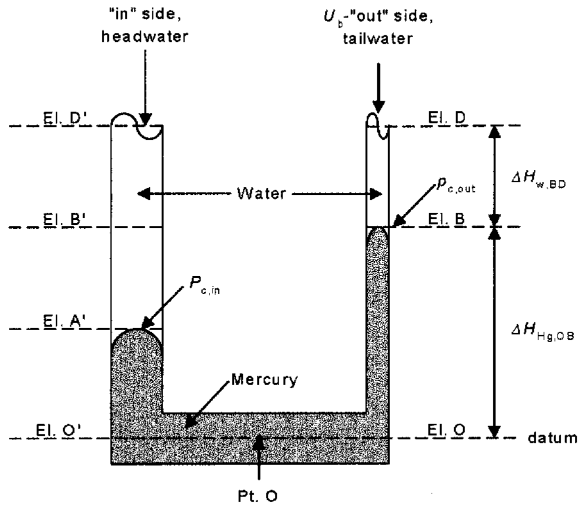
$$p_{o,\,out} \approx \mathrm{UCF} \cdot (\Delta H_{Hg,OB} \cdot \rho_{Hg} \cdot g + \Delta p_{c,out} + \Delta H_{w,BD} \cdot \rho_w \cdot g + U_b) \quad \text{(X1.2)}$$

or

$$p \approx \mathrm{UCF} \cdot (\Delta H_{Hg} \cdot \rho_{Hg} \cdot g + \Delta p_c + \Delta H_w \cdot \rho_w \cdot g + U_b)$$

where:

ΔH_{Hg} = the differential height of the mercury column, m,
ρ_{Hg} = the density of the mercury, Mg/m^3,
ΔH_w = the differential height of the water column, m,
Δp_c = the change in pressure due to capillarity at the tube-water-mercury interface, kPa, and
U_b = the applied back pressure, kPa.



Mercury U-tube Manometer

Determination of pressure for Pt. O at EL. O on the "out" or tailwater side, $p_{O,out}$.

$$p_{O,\,out} = \mathrm{UCF} \cdot (OB \cdot \rho_{Hg} \cdot g + \Delta p_{c,\,out} + BD \cdot \rho_W \cdot g + U_b)$$

or

$$= \mathrm{UCF} \cdot (\Delta H_{Hg,\,OB} \cdot \rho_{Hg} \cdot g + \Delta p_{c,\,out} + \Delta H_{W,BD} \cdot \rho_W \cdot g + U_b)$$

where:

UCF = a unit conversion factor,
ρ_{Hg} = the density of mercury, g/cm^3 or Mg/m^3,
ρ_W = the density of water, g/cm^3 or Mg/m^3,
g = the acceleration of gravity, m/s^2 or cm/s^2,
$\Delta p_{c,out}$ = the change in pressure due to capillarity at the tube-water-mercury interface in the outflow or tailwater tube, and
U_b = the applied back pressure, kPa or kN/m^2

FIG. X1.1 Static Pressure Calculations

X1.2.3 For the above case, the pressure head (in height of water) is

$$H_p \approx \mathrm{UCF} \cdot \left(H_{Hg} \times \frac{\rho_{Hg} \cdot g}{\rho_w \cdot g} + H_w \times \frac{\rho_w \cdot g}{\rho_w \cdot g} + \frac{\Delta p_c}{\rho_w \cdot g} + \frac{U_b}{\rho_w \cdot g} \right) \quad \text{(X1.3)}$$

or

$$H_p \approx \mathrm{UCF} \cdot \left(H_{Hg} \times G_{Hg} + \Delta H_w + \Delta H_c + \frac{U_b}{\rho_w \cdot g} \right)$$

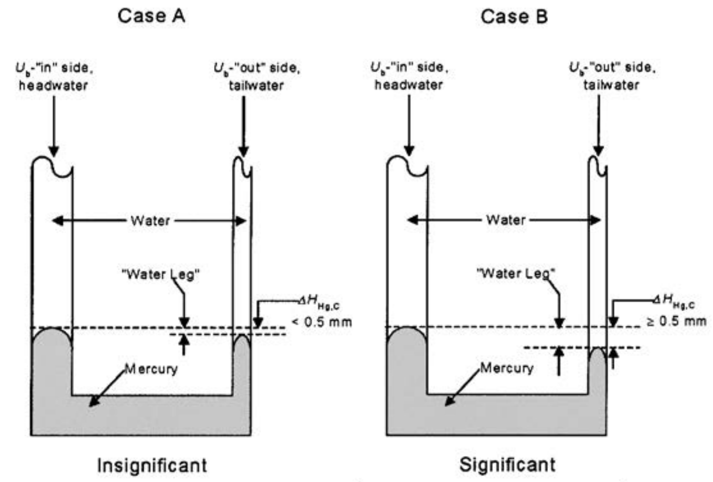
where:

G_{Hg} = the specific gravity of mercury at a given temperature, and
ΔH_c = the change in head due to capillarity, m of water, see X1.2.3.2

X1.2.3.1 *Velocity Head*—In most cases, the velocity head or velocity head loss is assumed equal to zero or insignificant, providing the requirements specified in 5.2.3 are met.

X1.2.3.2 *Capillary Head*—In most cases, the capillary head or capillary head loss is assumed equal to zero. However, in some mercury U-tube manometers, the difference in capillary head between the headwater and tailwater tubes; i.e., the capillary head loss, ΔH_c is significant. Therefore, it has to be accounted for as shown in Fig. X1.2. To help one understand the derivation of ΔH_c in this figure, one has to remember to account for the "water leg" in the mercury U-tube manometer containing water instead of air. Subtracting away the "water leg" pressure does this. Also, the pressure difference measured by the manometer has to be converted to a pressure head by dividing it by $\rho_w \cdot$ g. As shown in Fig. X1.2, the capillary head loss is

Capillary Head for
Mercury U-tube Manometer at No Flow or Equilibrium



$$\text{Capillary Head} = \Delta H_c = \text{UCF} \cdot \left(\frac{\Delta H_{Hg,c} \cdot \rho_{Hg} \cdot g - \Delta H_{Hg,c} \cdot \rho_w \cdot g}{\rho_w \cdot g} \right)$$

or

$$\Delta H_c = \text{UCF} \cdot \Delta H_{Hg,c} \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1 \right) = \text{UCF} \cdot \Delta H_{Hg,c} \cdot (G_{Hg} - 1)$$

Notes:
1) For definitions of notation, see X1.2.1 through X1.2.3.2.
2) For this case, the capillary head loss is a positive value since the total head on the headwater side would have to be increased to make the mercury levels equal.

NOTE—For this case capillary head loss is a positive value since the total head on the head water side would have to be increased to make the mercury levels equal.

FIG. X1.2 Difference in Capillary Head in Mercury U-tube Manometer

$$\Delta H_c = \Delta H_{Hg,c} \cdot (G_{Hg} - 1) \quad \text{(X1.4)}$$

where:

ΔH_c = capillary head loss in hydraulic system, m of water,
$\Delta H_{Hg,c}$ = differential height of mercury in the tailwater/outflow and headwater/inflow tubes of the manometer with equal pressure applied to each tube, m of mercury, and
G_{Hg} = specific gravity of mercury at test/trial temperature, see Table 1.

X1.2.3.2.1 As shown in Fig. X1.2, the application of either ΔH_c or $\Delta H_{Hg,c}$ is only necessary when $\Delta H_{Hg,c}$ is equal to or greater than 0.0005 m or 0.5 mm (0.02 in.). An explanation of how to measure ΔH_c is given in X1.4.

X1.2.4 *Total Head*—As stated above, the total head equals the sum of the pressure, elevation, velocity, and capillary heads. Since the change in velocity head is assumed to be zero and the change in capillary head is included in the pressure head calculation given above (Eq X1.3), the total head relationships at various points/elevations, as shown in Fig. X1.3, may be expressed as follows.

X1.2.4.1 Assuming there are no head losses in the tubing, the pressure head at Point Z ($H_{p,Z}$) equals the pressure head just above Point B ($H_{p,B}$), before the effect of capillary head; therefore

$$H_{p,Z} = H_{p,B} \text{ and } H_{p,X} = H_{p,B'}$$

X1.2.4.2 By definition, the total head just above Point B equals the pressure plus elevation heads at that point, therefore

$$H_B = H_{p,B} + H_{e,OB} = \frac{U_b}{\rho_w \cdot g} + H_{e,OB}$$

and

$$H_{B'} = H_{p,B'} + H_{e,O'B'}$$

Assuming continuity in hydraulics, the pressure head at Point B′ equals

$$H_{p,B'} = \frac{U_b}{\rho_w \cdot g} + \Delta H_{p,c,B} + H_{p,BA} + H_{p,AO} - H_{p,O'A'} - \Delta H_{p,c,A'} - H_{p,A'B'}$$

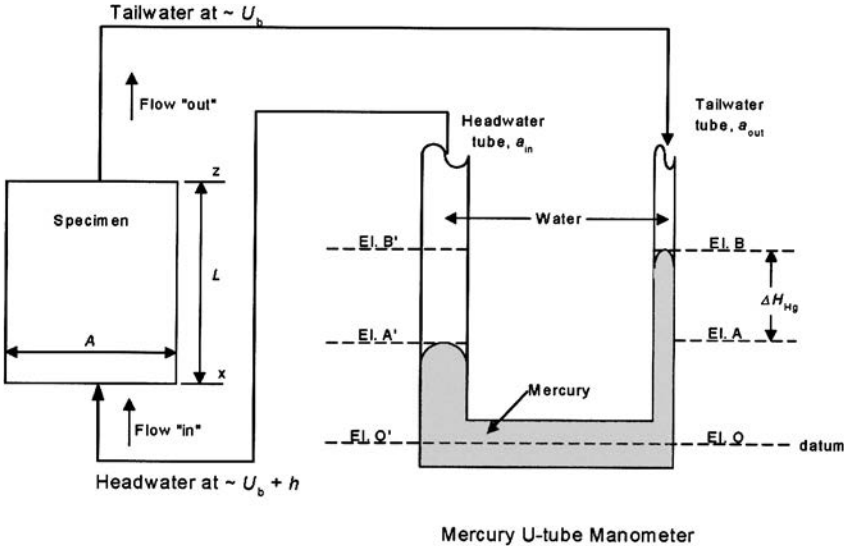


FIG. X1.3 Schematic of Mercury Constant Volume – Falling Head Hydraulic System for Head and Head Loss Equations

where:

$\Delta H_{p,c,B}$ = the change in capillary pressure head going from just above Point B to just below it, and

$\Delta H_{p,c,A'}$ = the change in capillary pressure head going from just below Point A′ to just above it.

X1.3 *Total Head Loss*—Based on a detailed review of the hydraulic systems shown in Fig. X1.1, Fig. X1.2, and Fig. X1.3, one can come to the conclusion that the flow of fluid (permeant) will only occur when the difference in the mercury heights in the U-tube manometer is greater then equilibrium value, as shown in Fig. X1.2.

X1.3.1 *Initial Head Loss*—Using the total head discussion given above and the notation given in Fig. X1.3; the initial total-head loss (Δh_1) across the specimen is

$$\Delta h_1 = H_X - H_Z = H_{B'} - H_B \tag{X1.5}$$

or

$$\Delta h_1 = H_{p,B'} - H_{p,B} =$$

$$= \frac{U_b}{\rho_w \cdot g} + \Delta H_{p,c,B} + H_{p,BA} + H_{p,AO} - H_{p,O'A'} - \Delta H_{p,c,A'} - H_{p,A'B'} - H_{p,B}$$

Since

$$H_{p,b} = \frac{U_b}{\rho_w \cdot g}, \tag{X1.6}$$

$$H_{p,AO} = H_{p,O'A'}, \text{ and}$$

$$\Delta H_c = \Delta H_{p,c,B} - \Delta H,$$

therefore

$$\Delta h_1 = H_{p,BA} - H_{p,A'B'} + \Delta H_c \tag{X1.7}$$

Using generic notation instead of specific notation as given in Fig. X1.1 to Fig. X1.4, therefore

$$\Delta h_1 = \frac{\Delta H_{Hg,1} \cdot g}{\rho_W} - \Delta H_{Hg,1} + \Delta H_{Hg,c} \times \left(\frac{\rho_{Hg}}{\rho_W} - 1\right) \tag{X1.8}$$

and rearranging, therefore

$$\Delta h_1 = (\Delta H_{Hg} + \Delta H_{Hg,c}) \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1\right) \tag{X1.9}$$

$$\Delta h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1)$$

where:

Δh_{Hg} = the initial total-head loss at the start (t_1) of a given permeation trial, in m of water,

ΔH_{Hg} = the initial differential height of mercury in the tailwater and headwater tubes of the manometer at the start (t_1) of a given permeation trial, in m,

$\Delta H_{Hg,c}$ = the positive differential height of mercury in the tailwater and headwater tubes of the manometer with equal pressures applied to both tubes, in m. This height differential is caused by the difference in capillary pressure heads within the two tubes making up the mercury U-tube manometer, see X1.2.3.2 and X1.4

ΔH

X1.3.2 *Final Head Loss*—The final total-head loss (Δh_2) across the specimen is

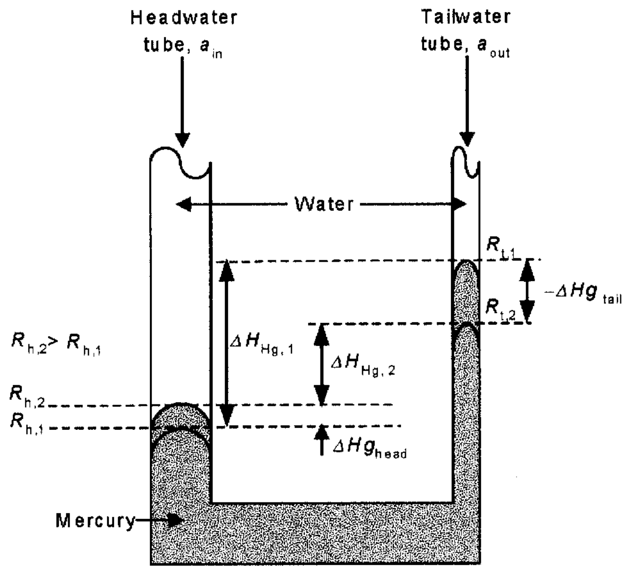
$$\Delta h_2 = (\Delta H_{Hg,2} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1) \tag{X1.10}$$

where:

Δh_2 = the final total-head loss at the end (t_2) Of a given permeation trial, in m of water,

$\Delta H_{Hg,2}$ = the final differential height of mercury in the tailwater and headwater tubes of the manometer at the end (t_2) of a given permeation trial, in m.

X1.3.2.1 The determination of $\Delta H_{Hg,2}$ requires two readings; i.e., the elevation of the top surfaces of the mercury (meniscus) in the tailwater and headwater tubes. Each of these readings will have some error, especially the headwater reading. In addition, the change in the headwater readings between t_1 and t_2 is typically very small and at about the sensitivity to which readings can be made/estimated. Because of these factors, it is assumed that the accuracy of Δh_2 can increased by just measuring the change in elevation of the top surface



"Flow" Functions:

Flow "in" = $\Delta Q_{in} = \Delta Hg_{head} \cdot a_{in}$

Flow "out" = $-\Delta Q_{out} = -\Delta Hg_{tail} \cdot a_{out}$

Based on continuity, $\Delta Q_{in} = -\Delta Q_{out}$

Therefore, $\Delta Hg_{head} \cdot a_{in} = -\Delta Hg_{tail} \cdot a_{out}$

or

$$\Delta Hg_{head} = -\Delta Hg_{tail} \cdot \frac{a_{out}}{a_{in}}$$

"Head" Functions:

$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) - \Delta Hg_{head}$

Substituting for ΔHg_{head},

$$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) + \left(-\Delta Hg_{tail} \cdot \frac{a_{out}}{a_{in}}\right)$$

$$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right)$$

Substituting for $\Delta H_{Hg,2}$ in the final head loss equation (X1.10) and rearranging,

$$\Delta h_2 = \left(\Delta H_{Hg,1} + \Delta H_{Hg,c} + (-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right)\right) \cdot (G_{Hg} - 1)$$

or

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right) \cdot (G_{Hg} - 1)\right)$$

FIG. X1.4 Relationship Between Change in Flow and Total Head Loss

mercury level in the tailwater tube and calculating what $\Delta H_{Hg,2}$ should be based on the area relationships between the tailwater and headwater tubes. As shown in Fig. X1.4, the following flow relationships can be established.

$$\Delta Q_{in} = \Delta Hg_{\ head} \cdot a_{in}$$

,

$$-\Delta Q_{out} = -\Delta Hg_{tail} \cdot a_{out}$$

NOTE X1.1—the symbol for height (H) has been omitted to keep the notation simpler, and

ΔQ_{in} = inflow of permeant water for given time interval (positive units of volume),

$-\Delta Q_{out}$ = outflow of permeant water for given time interval (negative units of volume),

ΔHg_{head} = positive change in elevation of the mercury level (top of meniscus) in the headwater tube (units of distance)

$-\Delta Hg_{tail}$ = negative change in elevation of the mercury level (top of meniscus) in the tailwater tube (units of distance),

a_{in} = area of the headwater/inflow tube containing mercury (units of area), and

a_{out} = area of the tailwater/outflow tube containing mercury (units of area)

Based on continuity of flow in a saturated specimen at constant volume,

$$\Delta Q\text{in} = -\Delta Q\text{out}$$

therefore,

$$\Delta Hg_{\text{head}} \cdot a_{\text{in}} = -\Delta Hg_{\text{tail}} \cdot a_{\text{out}} \quad \text{(X1.11)}$$

or

$$\Delta Hg_{\text{head}} = -\Delta Hg_{\text{tail}} \cdot \frac{a_{\text{out}}}{a_{\text{in}}}$$

therefore,

$$\Delta H_{\text{Hg,2}} = \Delta H_{Hg,1} + (-\Delta Hg_{\text{tail}}) + \left(\Delta Hg_{\text{tail}} \cdot \frac{a_{\text{out}}}{a_{\text{in}}}\right) \quad \text{(X1.12)}$$

or

$$\Delta H_{\text{Hg,2}} = \Delta H_{\text{Hg,1}} + (-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1\right)$$

Substituting for $\Delta H_{\text{Hg,2}}$ and rearranging

$$\Delta h_2 = \left(\Delta H_{\text{Hg,1}} + \Delta H_{\text{Hg,c}} + (-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1\right)\right) \cdot (G_{\text{Hg}} - 1) \quad \text{(X1.13)}$$

or

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1\right) \cdot (G_{\text{Hg}} - 1)\right)$$

X1.4 *Capillary Head Measurements*—The key to measuring the difference in capillary head (ΔH_c) between the headwater and tailwater tubes of the mercury U-tube manometer is to ensure that an equal water pressure is applied to both tubes. In addition, flow of water can occur under that equal water pressure. This can be accomplished by individually connecting the tailwater and headwater tubing lines to clean burettes containing water at equal elevation. These lines can not have any air bubbles in them. Then, apply the same air pressure to these two burettes. This air pressure should be similar to the back pressure applied during testing. Finally, adjust the height of one burette (typically the one connected to the headwater line) so the water level within each burette is equal. In making this height adjustment, make sure the water level in the headwater burette starts out below that of the tailwater burette. This simulates the direction of fluid flow during the test.

X1.4.1 Once the water level in the two burettes are level, determine the difference in elevation of the two mercury columns at the tops of their meniscuses. The mercury level in the tailwater tube (one with a smaller ID) should be below that in the headwater tube. If it is not, there is an error in applying equal pressures to the two tubes of the U-tube manometer, check for air in the lines, external pressure source, etc..

X1.4.2 If the mercury U-tube manometer being used is the version in which the tailwater tube is contained within the headwater tube, a different approach has to be used. A different approach is required since the mercury level in the tailwater tube is not visible at equilibrium. One approach would be to raise the headwater burette until the mercury levels (top of menisci) in the U-tube manometer (headwater and tailwater columns) are equal. Then determine the difference in elevation of the water levels in the headwater and tailwater burettes in m of water,

Next, convert ΔH_c to $\Delta H_{\text{Hg,c}}$ with

$\Delta H_{\text{Hg,c}} = \Delta H_c/(G_{\text{Hg}} - 1) = \Delta H_c/12.74$, in m of mercury. The value of 12.57 is good for temperatures ranging between 15°C and 25°C.

X1.5 *Falling-Head Hydraulic Conductivity Equation*—Darcy's law for hydraulic conductivity in a saturated medium requires that:

$$q = k \cdot i \cdot A = k \cdot \frac{\Delta h}{L} \cdot A \quad \text{(X1.14)}$$

or

$$\Delta Q = k \cdot \frac{\Delta h}{L} \cdot A \cdot \Delta t$$

where:

q = rate of flow of the fluid (units of volume over time, m^3/s),

k = hydraulic conductivity or coefficient of permeability (units of length over time, m/s),

i = hydraulic gradient (no unit),

Δh = total head loss across a given length/test specimen (unit of height of water, m),

L = given length (test specimen) over which the total head loss occurs (unit of distance, m),

ΔQ = volume of flow for a given time interval (unit of volume, m^3), and

Δt = time interval (unit of time, s).

X1.5.1 For a differential volume of flow and time period, this equation becomes

$$d\Delta Q = k \cdot \frac{\Delta h}{L} \cdot A \cdot d\Delta t \quad \text{(X1.15)}$$

where:

$d\Delta Q$ = differential volume of flow in a differential time period, and

$d\Delta t$ = differential time period.

X1.5.2 It can be demonstrated that the differential volume of flow is a function of the differential head loss, as shown below.

From Fig. X1.4 or X1.3.2.1;

$$-\Delta Q = -\Delta Hg_{\text{tail}} \cdot a_{\text{out}} \quad \text{(X1.16)}$$

or

$$-d\Delta Q = -d\Delta Hg_{\text{tail}} \cdot a_{\text{out}}$$

and from Fig. X1.4,

$$-d\Delta Hg_{\text{tail}} = \frac{dh}{1} \cdot \frac{a_{\text{in}}}{a_{\text{out}} + a_{\text{in}}} \cdot \frac{1}{(G_{\text{Hg}}-1)} \quad \text{(X1.17)}$$

By substituting for $-d\Delta Q$ and $-d\Delta Hg_{\text{tail}}$ from the above equations in Eq X1.15 we get,

$$-\frac{dh}{1} \cdot \frac{a_{\text{in}} \cdot a_{\text{out}}}{a_{\text{out}} + a_{\text{in}}} \cdot \frac{1}{(G_{\text{Hg}} - 1)} = k \cdot \frac{\Delta h}{L} \cdot A \cdot d\Delta t \quad \text{(X1.18)}$$

or

$$d\Delta t = -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{\Delta h} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a_{\text{out}}}{a_{\text{out}} + a_{\text{in}}} \cdot dh$$

By integrating between times t_1 and t_2 and h_1 and h_2 we get,

$$\int_{t_1}^{t_2} d\Delta t = \int_{\Delta h_1}^{\Delta h_2} -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{\Delta h} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a}{a_{out} + a_{in}} \cdot dh$$

which yields the general constant volume-falling head equation,

$$\Delta t = -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a_{out}}{a_{\text{out}} + a_{\text{in}}} \, ln \left(\frac{\Delta h_2}{\Delta h_1}\right) \quad \text{(X1.19)}$$

or

$$k = -\frac{L}{A} \cdot \frac{1}{\Delta t} \cdot \frac{1}{(G_{Hg} - 1)} \cdot \frac{a_{in} \cdot a_{out}}{a_{out} + a_{in}}\, ln\left(\frac{\Delta h_2}{\Delta h_1}\right)$$

Noting that,

$$-\, ln \frac{\Delta h_2}{\Delta h_1} = ln \frac{\Delta h_1}{\Delta h_2}$$

the equation becomes,

$$k = \left(\frac{a_{in} \cdot a_{out}}{(a_{out} + a_{in})} \cdot \frac{1}{(G_{Hg} - 1)}\right) \cdot \frac{L}{A} \cdot \frac{1}{\Delta t} \cdot ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \qquad (X1.20)$$

where:

k = hydraulic conductivity of the test specimen at the test temperature, m/s,

Δh_1 = total head loss across length L at the start of a permeation trial; i.e., initial total head loss, m of water,

Δh_2 = final total head loss across length L at the end of a permeation trial; i.e., final total head loss, m of water,

Δt = elapsed time during a permeation trial; i.e., $\Delta t = t_2 - t_1$, s,

a_{out} = area of the tailwater tube (tube with smaller ID), m^2,

a_{in} = area of the headwater tube, in m^2,

G_{Hg} = specific gravity of mercury (ρ_{Hg}/ρ_w) at the test temperature,

ρ_{Hg} = density of mercury at the test temperature, in Mg/m^3,

ρ_W = density of water at the test temperature, Mg/m^3,

$(G_{Hg}-1)$ = constant equal to 12.57 between 15 and 25°C,

L = length/height of the test specimen, in m,

A = area of the test specimen, in m^2,

ln = natural logarithm (base e),

and head loss equations from X1.3.1 and X1.3.2 are

$$\Delta h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1) \qquad (X1.21)$$

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right) \cdot (G_{Hg} - 1)\right) \qquad (X1.22)$$

where for Δh_1 and Δh_2:

$\Delta H_{Hg,1}$ = the initial difference in height of mercury in the tailwater and headwater tubes of the manometer at the start (t_1) of a given permeation trial, m;

$\Delta H_{Hg,c}$ = the positive difference in height of mercury in the tailwater and headwater tubes of the manometer with equal pressure applied to both tubes, m, (This height differential is caused by the difference in capillary pressure of the two tubes making up the mercury U-tube manometer); and,

$-\Delta Hg_{tail}$ = the negative change in height of the mercury level in the tailwater tube of the manometer during a given permeation trial, m.

X2. RELATIONSHIP BETWEEN CHANGE IN AXIAL STRAIN AND HYDRAULIC CONDUCTIVITY OF TEST SPECIMEN

X2.1 *Introduction*—It is important to understand how hydraulic conductivity (k) changes with changes in void ratio, dry unit weight, or volume change of a given test specimen. With this understanding, one can thereby know the accuracy to which volume changes need to be controlled while testing. For instance, if k is very sensitive to volume changes then the effective consolidation stress needs to be accurately controlled; also, any volume changes during the back-pressuring process would have to be minimized.

X2.1.1 The relationships presented in this appendix are for relatively plastic clays, CL or CH, with a plasticity index greater than about 10, but less than about 50.

X2.2 *Change in Void Ratio versus Hydraulic Conductivity*—A graphical representation of how hydraulic conductivity (k) varies with void ratio (e) is presented in Fig. X2.1. This figure graphically shows that the k decreases as e decreases. In addition, for a given change in void ratio (Δe) the rate of change in k is much more dramatic in the overconsolidated range than in the normally consolidated range. The definition for the terms/notation presented in this figure are:

k= given hydraulic conductivity, m/s,

k_L= lower bound k for given percent change in k, m/s,

k_U= upper bound k for given percent change in k, m/s,

Δk_L= lower bound change in k for given percent change (decimal form) in k, m/s/

% Change = the percent change (decimal form) in k,

Δe_L= the compressive change in void ration (decreasing change)

e_U= the swelling change in void ratio (increasing change), and

m = the ratio of Δe to $\Delta log\ k = \Delta e / log\ (k/k_L)$, 1/(m/s).

X2.3 Mathematical Relationship Between e and k—It can be shown that:

$$k_L = k + (-\Delta k_L) = k - \%Change \times k = (1 - \%Change) \times k \qquad (X2.1)$$

or

$$k_U = (1 + \%Change) \times k$$

$$-\Delta k_L = log(1 - \%Change) \qquad (X2.2)$$

or

$$\Delta k_U = log(1 + \%Change)$$

$$e_L = e + (-\Delta e_L) \qquad (X2.3)$$

or

$$e_U = e + \Delta e_U$$

$$-\Delta e_L = m \times -\Delta k_L = m \times log(1 - \%Change), \qquad (X2.4)$$

or

$$\Delta e_U = m \times \Delta k_U = m \times log(1 + \%Change)$$

X2.4 Based upon the theory of elasticity, the following relationships between axial strain (ε_a) and volumetric strain (ε_v), and volumetric strain and change in void ratio ($\Delta\varepsilon$) are:

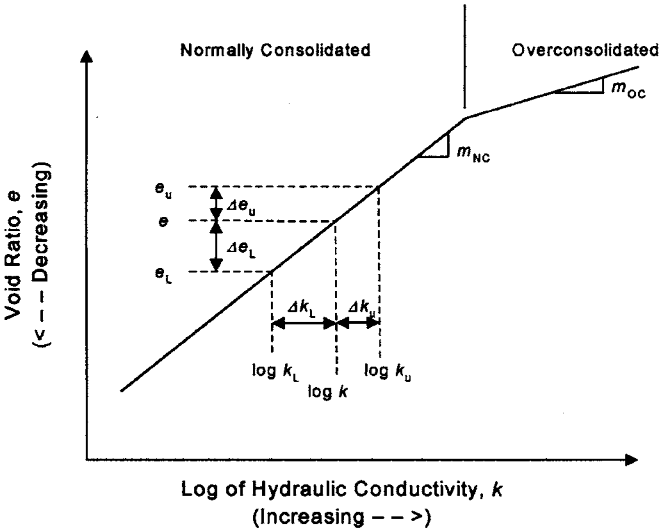


FIG. X2.1 Typical Relationship Between Void Ratio and Logarithm of Hydraulic Conductivity

$$\Delta\varepsilon_a = \frac{1}{3}\cdot E_c\cdot \Delta\varepsilon_v = \left(\frac{1}{3}\cdot E_c\cdot - \Delta\varepsilon_L\right)/(1-e_o) \qquad \text{(X2.5)}$$

$\Delta\varepsilon_a$ = the change in compressive axial strain ($\Delta L/L_o$), m/m.
E_c = a constant to correct for the non-elastic response of hydraulic-conductivity test specimens. For test specimens having a height to diameter ratio of about one, this value is about 0.8 for normally consolidated (NC) specimens and 0.6 for overconsolidated (OC) specimens,
$\Delta\varepsilon_v$ = the change in compressive volumetric strain ($\Delta V/V_o$), m^3/m^3.
$-\Delta e$ = the compressive change in void ratio; i.e., decreasing change,
e_o = the initial void ratio.

X2.5 Combining the above equations and rearranging:

$$\Delta\varepsilon_{a,NC} = \frac{-1}{3\cdot E_c}\cdot\frac{m_{NC}}{1+e_o}\cdot log(1-\%Change) \qquad \text{(X2.6)}$$

or

$$\Delta\varepsilon_{a,OC} = \frac{-1}{3\cdot E_c}\cdot\frac{m_{OC}}{1+e_o}\cdot log(1-\%Change)$$

also

$$-\%Change = 10^{-\left(3\cdot E_c\cdot\left(\frac{1+e_o}{m}\right)\cdot\Delta\varepsilon_{a,L}\right)} \qquad \text{(X2.7)}$$

$$+\%Change = 10^{-\left(3\cdot E_c\cdot\left(\frac{1+e_o}{m}\right)\cdot-\Delta\varepsilon_{a,U}\right)}$$

where:

m_{NC} = m value in normally consolidated region, 1/[m/s], and
m_{OC} = m value in the overconsolidated region, 1/[m/s].

X2.5.1 Tavenas, et al. (**10**) indicates that m is about (⅓ to ½) × e_o for normally consolidated clays. For overconsolidated clay, it is assumed that m in the overconsolidated range is reduced by the same ratio that the compression index (C) is when going from the normally consolidated region (C_{NC}) to the overconsolidated region (C_{OC}). Therefore,

$$m_{OC}/m_{NC} = C_{OC}/C_{NC} \sim 0.185 + 0.002 \times PI$$

where PI = plasticity index. This assumption is based on limited data, and in some cases, there was not any significant difference in m between the normally consolidated and overconsolidated regions. Based on the above, an initial void ratio of 0.8 and a plasticity index (PI) of 30, m_{NC} is about 0.33; while m_{OC} is about 0.082.

X2.5.2 Using the above m values and equations, and a %Change equal to 12.5 %, one could assume excess axial strains caused by a poor testing protocol should be less than the following values:

For normally consolidated soils:

$$\Delta\varepsilon_{a,NC} = \frac{-1}{3\cdot 0.8}\cdot\frac{0.33}{1.8}\cdot log\ 0.875 = 0.0044 = 0.4\ \%$$

For overconsolidated consolidated soils:

$$\Delta\varepsilon_{a,OC} = \frac{-1}{3\cdot 0.6}\cdot\frac{0.082}{1.8}\cdot log\ 0.875 = 0.00147 = 0.1\ \%$$

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SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to these test methods since the last issue, D5084–03, that may impact the use of these test methods. (Approved July 1, 2010)

(*1*) Replaced "h" with "Δh" throughout. Deleted part of X1.1.2 and Note X1.2.

(*2*) Revised 1.8 to comply with the D18 Standards Preparation Manual.

(*3*) Revised 3.1.1 to comply with the D18 Standards Preparation Manual.

(*4*) Revised the "Mercury Warning" in 7.1 with the wording in the new ASTM "Mercury Caveat."

(*5*) Revised 8.1 to clarify the statement regarding dimensions of specimens.

(*6*) Revised 11.5.1 to ensure that any differences/averages calculated using measured values comply with the significant digit requirements in the standard.

(*7*) Rewrote Section 12 Precision and Bias, including new Tables 2 and 3.

Designation: D2974 – 07a

Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils[1]

This standard is issued under the fixed designation D2974; 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 These test methods cover the measurement of moisture content, ash content, and organic matter in peats and other organic soils, such as organic clays, silts, and mucks.

1.1.1 Method A – moisture is determined by drying peat or organic sample at 105 °C.

1.1.2 Method B – Alternative moisture method which removes the total moisture in two steps: (1) evaporation of moisture at room temperature, (2) subsequent oven drying of air dried sample at 105 °C.

1.1.3 Method C – Ash content of a peat or organic soil sample is determined by igniting oven dried sample from moisture content determination in a muffle furnace at 440 °C.

1.1.4 Method D – Ash content of a peat or organic soil sample is determined by igniting oven dried sample from moisture content determination in a muffle furnace at 750 °C.

1.2 This test method should be used for geotechnical and general classification. In addition, the test method should be used when peats are being evaluated for use as a fuel.

1.3 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard. Use Practice D6026 for determining significant digits to report.

1.4 *This standard does not purport to address all of the safety concerns, 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:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions*:

3.1.1 For common definitions of terms in this standard, refer to Terminology D653.

4. Summary of Test Methods

4.1 *Test Method A*—Moisture is determined by drying a peat or organic soil sample at 105°C. The moisture content is expressed either as a percent of the oven dry mass or of the as-received mass.

4.2 *Test Method B*—This is an alternative moisture method which removes the total moisture in two steps: (*1*) evaporation of moisture in air at room temperature (air-drying), and (*2*) the subsequent oven drying of the air-dried sample at 105°C. This method provides a more stable sample, the air-dried sample, when tests for nitrogen, pH, cation exchange, and the like are to be made.

4.3 *Test Methods C and D*—Ash content of a peat or organic soil sample is determined by igniting the oven-dried sample from the moisture content determination in a muffle furnace at 440°C (Method C) or 750°C (Method D). The substance remaining after ignition is the ash. The ash content is expressed as a percentage of the mass of the oven-dried sample.

4.4 Organic matter is determined by subtracting percent ash content from one hundred.

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[1] These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.22 on Soil as a Medium for Plant Growth.

Current edition approved March 15, 2007. Published May 2007. Originally approved in 1971. Last previous edition approved in 2007 as D2974 – 07. DOI: 10.1520/D2974-07A.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

5. Significance and Use

5.1 This test method can be used to determine the moisture content, ash content, and percent organic matter in soil.

5.2 The percent organic matter is important in the following: (1) classifying peat or other organic soil, (2) geotechnical and general classification purposes, and (3) when peats are being evaluated as a fuel

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Oven*, meeting the requirements of E145 and capable of being regulated to a constant temperature of 105 ± 5°C.

6.2 The temperature of 105 °C is quite critical for organic soils. The oven should be checked for “hot spots” to avoid possible ignition of the specimen.

6.3 *Muffle Furnace*, capable of producing constant temperatures of 440 °C ± 22 °C and 750 °C ± 38°C

6.4 *Balance or Scale*, a balance or scale for determining the mass of the soil having a minimum capacity of 500 g and meeting the requirements of Standard D4753 for a balance or scale of 0.01 g readability.

6.5 *Rubber Sheet, Oil Cloth*, or other non-absorbent material.

6.6 *Evaporating Dishes*, of high silica or porcelain of not less than 100-mL capacity.

6.7 *Blender*, high-speed.

6.8 *Aluminum Foil*, heavy-duty.

6.9 *Porcelain Pan, Spoons*, and equipment of the like.

6.10 *Desiccator*.

7. Sampling and Test Specimens

7.1 Place a representative field sample on a rubber sheet, oil cloth, or equivalent material and mix thoroughly.

7.2 Reduce the sample to the quantity required for a test specimen by quartering.

7.3 Place the test specimen and the remaining sample in separate waterproof containers.

7.4 Work rapidly to prevent moisture loss or perform the operation in a room with a high humidity.

8. Procedure

8.1 *Moisture Content Determination*

8.1.1 *Test Method A*

8.1.1.1 Record to the nearest 0.01 g the mass of a high silica or porcelain evaporating dish fitted with a heavy-duty aluminum foil cover. The dish shall have a capacity of not less than 100 mL.

8.1.1.2 Following the instruction in section 7.1 above, place a test specimen of at least 50 g in the container described in 8.1.1.1. Crush soft lumps with a spoon or spatula. The thickness of peat in the container should not exceed 3 cm.

8.1.1.3 Cover immediately with the aluminum foil cover and record the mass to the nearest 0.01 g.

8.1.1.4 Dry uncovered for at least 16 h at 105°C or until there is no change in mass of the sample after further drying periods in excess of 1 h. Remove from the oven, cover tightly, cool in a desiccator, and record the mass to the nearest 0.01 g.

8.1.2 *Calculations for Test Method A*

8.1.2.1 Calculate the moisture content as follows:

$$\text{Moisture Content, \%} = [(A - B) \times 100]/A \qquad (1)$$

where:

A = mass of the as-received test specimen, g, and

B = mass of the oven-dried specimen, g.

8.1.2.1.1 This calculation is used primarily for agriculture, forestry, energy, and horticultural purposes and the result should be referred to as the moisture content as a percentage of as-received or total mass.

8.1.2.2 An alternative calculation is as follows:

$$\text{Moisture Content, \%} = [(A - B) \times 100]/B \qquad (2)$$

where:

A = mass of the as-received test specimen, g, and

B = mass of the oven-dried specimen, g.

8.1.2.2.1 This calculation is used primarily for geotechnical purposes and the result should be referred to as the moisture content as a percentage of oven-dried mass.

8.1.2.3 Take care to indicate the calculation method used.

8.1.3 *Test Method B*

8.1.3.1 This test method should be used if pH, nitrogen content, cation exchange capacity, and the like are to be tested.

8.1.3.2 Following the instructions in section 7.1 above, select a 100 to 300 g representative test specimen. Determine the mass of this test specimen to the nearest 0.01 g and spread it evenly on a large flat pan. Crush soft lumps with a spoon or spatula and let the sample come to moisture equilibrium with room air. This will require at least 24 h. Stir occasionally to maintain maximum air exposure of the entire sample. When there is no change in mass of the sample after further drying periods in excess of 1 h, calculate the moisture removed during air drying as a percentage of the as-received mass.

8.1.3.3 Grind a representative portion of the air-dried sample for 1 to 2 min in a high-speed blender. Use the ground portion for moisture, ash, nitrogen, cation exchange capacity tests, and the like.

8.1.3.4 Thoroughly mix the air-dried, ground sample. Weigh to the nearest 0.01 g the equivalent of 50 g of test specimen on an as-received basis. Determine the amount, in grams, of air-dried sample equivalent to 50 g of as-received sample, as follows:

$$\text{Equivalent Sample Mass, g} = 50.0 - [(50 \times M)/100] \qquad (3)$$

where:

M = moisture removed in air drying, % (on as received basis).

8.1.3.5 Place the sample in a container as described in 8.1.1 and proceed as in Method A.

8.1.4 *Calculations for Test Method B*

8.1.4.1 Calculate the moisture content as follows:

$$\text{Moisture Content, \%} = (50 - B) \times 2 \qquad (4)$$

where:

B = mass of the oven-dried sample, g.

8.1.4.1.1 This calculation gives moisture content as a percentage of as-received mass.

8.1.4.2 An alternative calculation is as follows:

$$\text{Moisture Content, \%} = [(50 - B) \times 100]/B \quad (5)$$

8.1.4.2.1 This calculation gives moisture content as a percentage of oven-dried mass.

8.2 *Ash Content Determination*

8.2.1 *Test Method C*

8.2.1.1 Determine the mass of a covered high-silica or porcelain dish to the nearest 0.01 g.

8.2.1.2 Place a part of or all of the oven-dried test specimen from a moisture determination in the dish and determine the mass of the dish and specimen to the nearest 0.01 g.

8.2.1.3 Remove the cover and place the dish in a muffle furnace. Gradually bring the temperature in the muffle furnace to 440 °C ± 22 °C and hold until the specimen is completely ashed (no change of mass occurs after at least 1 hr. period of heating).

8.2.1.4 Cover with the retained aluminum foil cover, cool in a desiccator, and determine the mass to the nearest 0.01 g.

8.2.1.5 This test method should be used for geotechnical and general classification purposes.

8.2.2 *Test Method D*

8.2.2.1 Determine the mass of a covered high-silica or porcelain dish to the nearest 0.01 g.

8.2.2.2 Place a part of or all of the oven-dried test specimen from a moisture determination in the dish and determine the mass of the dish and specimen to the nearest 0.01 g.

8.2.2.3 Remove the cover and place the dish in a muffle furnace. Gradually bring the temperature in the muffle furnace to 750 °C ± 38 °C and hold until the specimen is completely ashed (no change in mass of the sample after further drying periods in excess of 1 h).

8.2.2.4 Cover with the retained aluminum foil cover, cool in a desiccator, and determine the mass to the nearest 0.01 g.

8.2.2.5 This test method should be used when peats are being evaluated for use as a fuel.

8.2.3 *Calculation for Test Methods C and D*

8.2.3.1 Calculate the ash content as follows:

$$\text{Ash Content, \%} = (C \times 100)/B \quad (6)$$

where:

C = mass of ash, g, and

B = oven-dried test specimen, g.

8.3 *Organic Matter Determination*

8.3.1 *Calculation*

8.3.1.1 Determine the amount of organic matter to the nearest 0.1 % by difference, as follows:

$$\text{Organic matter, \%} = 100.0 - D \quad (7)$$

where:

D = ash content, % (nearest 0.1%).

9. Report

9.1 Report the following information:

9.1.1 Identify sample: project, boring or location, depth, and method used in sampling.

9.1.2 Results for organic matter and ash content, to the nearest 0.1 %. Use Practice D6026 to determine significant digits

9.1.3 Furnace temperature used for ash content determinations.

9.1.4 Whether moisture contents are by proportion of as-received mass or oven-dried mass.

9.1.4.1 Express results for moisture content as a percentage of as-received mass to the nearest 0.1 %.

9.1.4.2 Express results for moisture content as a percentage of oven-dried mass as follows:

(a) Below 100 % to the nearest 1 %.

(b) Between 100 % and 500 % to the nearest 5 %.

(c) Between 500 % and 1000 % to the nearest 10 %.

(d) Above 1000 % to the nearest 20 %.

10. Precision and Bias

10.1 *Precision*—Test data on precision is not presented due to the nature of the soil materials tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program.

10.1.1 The Subcommittee D 18.22 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

10.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

11. Keywords

11.1 ash content; moisture content; organic soil; peat; percent organic matter

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (07) that may impact the use of the standard.

(1) In 8.1.1.2 changed 4.1 to 7.1.
(2) In 8.1.3.2 changed 4.1 to 7.1.
(3) In 8.1.3.4 added (on as received basis) to end of sentence.
(4) In 8.1.4.1 add "mass of the" to B.
(5) In 8.2.1.2 added "to the nearest 0.01 g " to the end of the sentence.
(6) In 8.1.2.2 added "mass of the" to A.
(7) In 8.2.1.3 In sentence 2 add "at least 1 hr" to sentence.
(8) In 8.2.1.5 removed "all" in sentence.
(9) Changed D18.06 to D18.22 in Section 10.1.1
(10) In 11 changed "%" to "percent".
(11) In 7.1 deleted "rubber" from sentence.

Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[1]

This standard is issued under the fixed designation D4318; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods cover the determination of the liquid limit, plastic limit, and the plasticity index of soils as defined in Section 3 on Terminology.

1.2 Two methods for preparing test specimens are provided as follows: *Wet preparation method*, as described in 10.1. *Dry preparation method*, as described in 10.2. The method to be used shall be specified by the requesting authority. If no method is specified, use the wet preparation method.

1.2.1 The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on non-dried samples. If the liquid and plastic limits of soils are used to correlate or estimate the engineering behavior of soils in their natural moist state, samples should not be permitted to dry before testing unless data on dried samples are specifically desired.

1.3 Two methods for determining the liquid limit are provided as follows: *Method A*, Multipoint test as described in Sections 11 and 12. *Method B*, One-point test as described in Sections 13 and 14. The method to be used shall be specified by the requesting authority. If no method is specified, use Method A.

1.3.1 The multipoint liquid limit method is generally more precise than the one-point method. It is recommended that the multipoint method be used in cases where test results may be subject to dispute, or where greater precision is required.

1.3.2 Because the one-point method requires the operator to judge when the test specimen is approximately at its liquid limit, it is particularly not recommended for use by inexperienced operators.

1.3.3 The correlation on which the calculations of the one-point method are based may not be valid for certain soils, such as organic soils or soils from a marine environment. It is strongly recommended that the liquid limit of these soils be determined by the multipoint method.

1.4 The plastic limit test is performed on material prepared for the liquid limit test.

1.5 The liquid limit and plastic limit of soils (along with the shrinkage limit) are often collectively referred to as the Atterberg limits. These limits distinguished the boundaries of the several consistency states of plastic soils.

1.6 The composition and concentration of soluble salts in a soil affect the values of the liquid and plastic limits as well as the water content values of soils (see Method D4542). Special consideration should therefore be given to soils from a marine environment or other sources where high soluble salt concentrations may be present. The degree to which the salts present in these soils are diluted or concentrated must be given careful consideration.

1.7 The methods described herein are performed only on that portion of a soil that passes the 425-µm (No. 40) sieve. Therefore, the relative contribution of this portion of the soil to the properties of the sample as a whole must be considered when using these tests to evaluate properties of a soil.

1.8 The values stated in SI units are to be regarded as the standard, except as noted below. The values given in parentheses are for information only.

1.8.1 The standard units for the resilience tester covered in Annex A1 are inch-pound, not SI. The SI values given are for information only.

1.9 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.9.1 For purposes of comparing a measured or calculated value(s) with specified limits, the measured or calculated value(s) shall be rounded to the nearest decimal or significant digits in the specified limits

1.9.2 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the

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[1] This standard is under the jurisdiction of ASTM Committee D18 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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***A Summary of Changes section appears at the end of this standard.**

user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.10 *This standard does not purport to address all of the safety concerns, 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:*[2]

C702 Practice for Reducing Samples of Aggregate to Testing Size

D75 Practice for Sampling Aggregates

D420 Guide to Site Characterization for Engineering Design and Construction Purposes

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1241 Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D3282 Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4542 Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of terms in this standard, refer to Terminology D653.

3.1.2 *Atterberg Limits*—Originally, six "limits of consistency" of fine-grained soils were defined by Albert Atterberg: the upper limit of viscous flow, the liquid limit, the sticky limit, the cohesion limit, the plastic limit, and the shrinkage limit. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limit.

3.1.3 *consistency*—the relative ease with which a soil can be deformed.

3.1.4 *liquid limit (LL, w_L)*—the water content, in percent, of a soil at the arbitrarily defined boundary between the semi-liquid and plastic states.

3.1.4.1 *Discussion*—The undrained shear strength of soil at the liquid limit is considered to be approximately 2 kPa (0.28 psi).

3.1.5 *plastic limit (PL, w_p)*—the water content, in percent, of a soil at the boundary between the plastic and semi-solid states.

3.1.6 *plastic soil*—a soil which has a range of water content over which it exhibits plasticity and which will retain its shape on drying.

3.1.7 *plasticity index (PI)*—the range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

3.1.8 *liquidity index*—the ratio, expressed as a percentage of (1) the water content of a soil minus its plastic limit, to (2) its plasticity index.

3.1.9 *activity number (A)*—the ratio of (1) the plasticity index of a soil to (2) the percent by mass of particles having an equivalent diameter smaller than 2 µm.

4. Summary of Test Method

4.1 The specimen is processed to remove any material retained on a 425-µm (No. 40) sieve. The liquid limit is determined by performing trials in which a portion of the specimen is spread in a brass cup, divided in two by a grooving tool, and then allowed to flow together from the shocks caused by repeatedly dropping the cup in a standard mechanical device. The multipoint liquid limit, Method A, requires three or more trials over a range of water contents to be performed and the data from the trials plotted or calculated to make a relationship from which the liquid limit is determined. The one-point liquid limit, Method B, uses the data from two trials at one water content multiplied by a correction factor to determine the liquid limit.

4.2 The plastic limit is determined by alternately pressing together and rolling into a 3.2-mm (⅛-in.) diameter thread a small portion of plastic soil until its water content is reduced to a point at which the thread crumbles and can no longer be pressed together and re-rolled. The water content of the soil at this point is reported as the plastic limit.

4.3 The plasticity index is calculated as the difference between the liquid limit and the plastic limit.

5. Significance and Use

5.1 These test methods are used as an integral part of several engineering classification systems to characterize the fine-grained fractions of soils (see Practices D2487 and D3282) and to specify the fine-grained fraction of construction materials (see Specification D1241). The liquid limit, plastic limit, and plasticity index of soils are also used extensively, either individually or together, with other soil properties to correlate

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

with engineering behavior such as compressibility, hydraulic conductivity (permeability), compactibility, shrink-swell, and shear strength.

5.2 The liquid and plastic limits of a soil and its water content can be used to express its relative consistency or liquidity index. In addition, the plasticity index and the percentage finer than 2-µm particle size can be used to determine its activity number.

5.3 These methods are sometimes used to evaluate the weathering characteristics of clay-shale materials. When subjected to repeated wetting and drying cycles, the liquid limits of these materials tend to increase. The amount of increase is considered to be a measure of a shale's susceptibility to weathering.

5.4 The liquid limit of a soil containing substantial amounts of organic matter decreases dramatically when the soil is oven-dried before testing. Comparison of the liquid limit of a sample before and after oven-drying can therefore be used as a qualitative measure of organic matter content of a soil (see Practice D2487.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740, generally, are considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Liquid Limit Device*—A mechanical device consisting of a brass cup suspended from a carriage designed to control its drop onto the surface of a block of resilient material that serves as the base of the device. Fig. 1 shows the essential features and critical dimensions of the device. The device may be operated by either a hand crank or electric motor.

6.1.1 *Base*—A block of material having a resilience rebound of at least 77 % but no more than 90 %. Conduct resilience tests on the finished base with the feet attached. Details for measuring the resilience of the base are given in Annex A1.

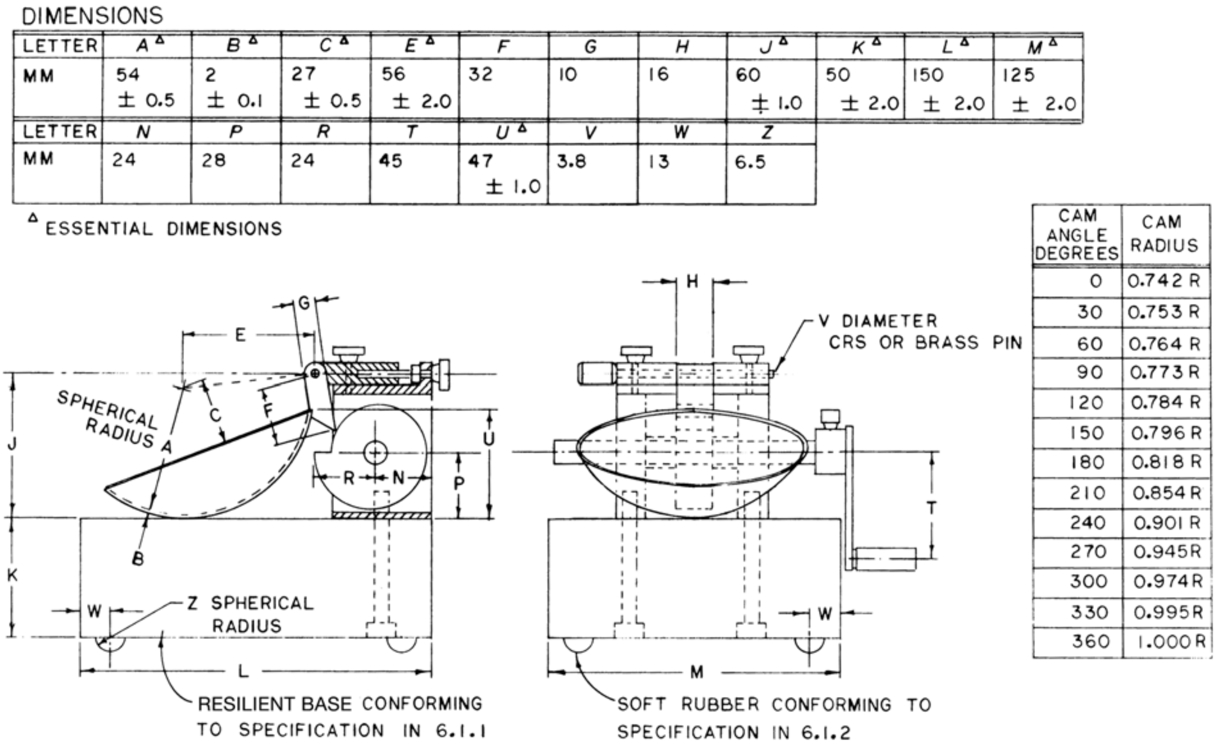
6.1.2 *Rubber Feet*, supporting the base, designed to provide dynamic isolation of the base from the work surface.

6.1.3 *Cup*, brass, with a mass, including cup hanger, of 185 to 215 g.

6.1.4 *Cam*—Designed to raise the cup smoothly and continuously to its maximum height, over a distance of at least 180° of cam rotation, without developing an upward or downward velocity of the cup when the cam follower leaves the cam. (The preferred cam motion is a uniformly accelerated lift curve.)

NOTE 2—The cam and follower design in Fig. 1 is for uniformly accelerated (parabolic) motion after contact and assures that the cup has no velocity at drop off. Other cam designs also provide this feature and may be used. However, if the cam-follower lift pattern is not known, zero velocity at drop off can be assured by carefully filing or machining the cam and follower so that the cup height remains constant over the last 20 to 45° of cam rotation.

6.1.5 *Carriage*, constructed in a way that allows convenient but secure adjustment of the height-of-drop of the cup to 10 mm (0.394 in.), and designed such that the cup and cup hanger assembly is only attached to the carriage by means of a



DIMENSIONS

LETTER	A$^{\Delta}$	B$^{\Delta}$	C$^{\Delta}$	E$^{\Delta}$	F	G	H	J$^{\Delta}$	K$^{\Delta}$	L$^{\Delta}$	M$^{\Delta}$
MM	54 ± 0.5	2 ± 0.1	27 ± 0.5	56 ± 2.0	32	10	16	60 ± 1.0	50 ± 2.0	150 ± 2.0	125 ± 2.0

LETTER	N	P	R	T	U$^{\Delta}$	V	W	Z
MM	24	28	24	45	47 ± 1.0	3.8	13	6.5

$^{\Delta}$ ESSENTIAL DIMENSIONS

CAM ANGLE DEGREES	CAM RADIUS
0	0.742 R
30	0.753 R
60	0.764 R
90	0.773 R
120	0.784 R
150	0.796 R
180	0.818 R
210	0.854 R
240	0.901 R
270	0.945R
300	0.974R
330	0.995R
360	1.000R

FIG. 1 Hand-Operated Liquid Limit Device

removable pin. See Fig. 2 for definition and determination of the height-of-drop of the cup.

6.1.6 *Motor Drive (Optional)*—As an alternative to the hand crank shown in Fig. 1, the device may be equipped with a motor to turn the cam. Such a motor must turn the cam at 2 ± 0.1 revolutions per second and must be isolated from the rest of the device by rubber mounts or in some other way that prevents vibration from the motor being transmitted to the rest of the apparatus. It must be equipped with an ON-OFF switch and a means of conveniently positioning the cam for height-of-drop adjustments. The results obtained using a motor-driven device must not differ from those obtained using a manually operated device.

6.2 *Flat Grooving Tool*—A tool made of plastic or noncorroding-metal having the dimensions shown in Fig. 3. The design of the tool may vary as long as the essential dimensions are maintained. The tool may, but need not, incorporate the gauge for adjusting the height-of-drop of the liquid limit device.

NOTE 3—Prior to the adoption of this test method, a curved grooving tool was specified as part of the apparatus for performing the liquid limit test. The curved tool is not considered to be as accurate as the flat tool described in 6.2 since it does not control the depth of the soil in the liquid limit cup. However, there are some data which indicate that typically the liquid limit is slightly increased when the flat tool is used instead of the curved tool.

6.3 *Gauge*—A metal gauge block for adjusting the height-of-drop of the cup, having the dimensions shown in Fig. 4. The design of the tool may vary provided the gauge will rest securely on the base without being susceptible to rocking, and the edge which contacts the cup during adjustment is straight, at least 10 mm (⅜ in.) wide, and without bevel or radius.

6.4 *Water Content Containers*—Small corrosion-resistant containers with snug-fitting lids for water content specimens. Aluminum or stainless steel cans 2.5 cm (1 in.) high by 5 cm (2 in.) in diameter are appropriate.

6.5 *Balance*, conforming to Specification D4753, Class GP1 (readability of 0.01 g).

6.6 *Mixing and Storage Container*—A container to mix the soil specimen (material) and store the prepared material. During mixing and storage, the container shall not contaminate the material in any way, and prevent moisture loss during storage. A porcelain, glass, or plastic dish about 11.4 cm (4½ in.) in diameter and a plastic bag large enough to enclose the dish and be folded over is adequate.

6.7 *Plastic Limit:*

6.7.1 *Ground Glass Plate*—A ground glass plate of sufficient size for rolling plastic limit threads.

6.7.2 *Plastic Limit-Rolling Device (optional)*—A device made of acrylic conforming to the dimensions shown in Fig. 5.[3,4] The type of unglazed paper attached to the top and bottom plate (see 16.2.2) shall be such that it does not add foreign matter (fibers, paper fragments, etc.) to the soil during the rolling process.

6.8 *Spatula*—A spatula or pill knife having a blade about 2 cm (¾ in.) wide, and about 10 to 13 cm (3 to 4 in.) long.

6.9 *Sieve(s)*—A 200-mm (8-in.) diameter, 425-μm (No. 40) sieve conforming to the requirements of Specification E11 and having a rim at least 5 cm (2 in.) above the mesh. A 2.00-mm (No. 10) sieve meeting the same requirements may also be needed.

6.10 *Wash Bottle*, or similar container for adding controlled amounts of water to soil and washing fines from coarse particles.

6.11 *Drying Oven*, thermostatically controlled, preferably of the forced-draft type, capable of continuously maintaining a temperature of 110 ± 5°C (230 ± 9°F) throughout the drying chamber.

6.12 *Washing Pan*, round, flat-bottomed, at least 7.6 cm (3 in.) deep, and slightly larger at the bottom than a 20.3-cm (8-in.) diameter sieve.

7. Reagents and Materials

7.1 *Purity of Water*—Where distilled water is referred to in this test method, either distilled or demineralized water may be used. See Note 7 covering the use of tap water.

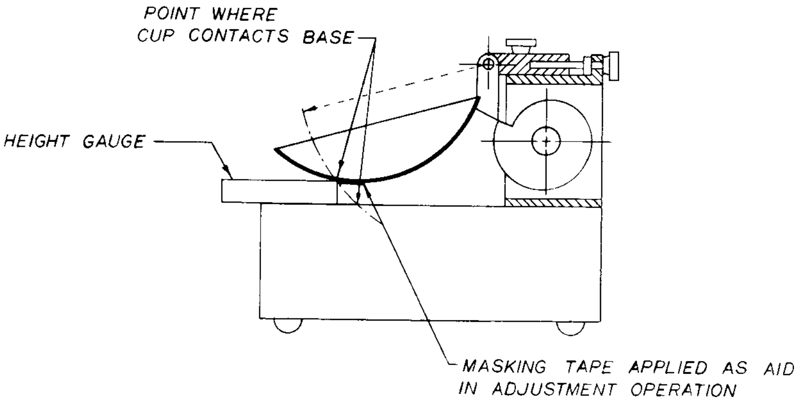


FIG. 2 Calibration for Height-of-Drop

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[3] The plastic limit-rolling device is covered by a patent (U.S. Patent No. 5,027,660).[7] Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible subcommittee, which you may attend.

[4] Bobrowski, L. J., Jr. and Griekspoor, D. M., "Determination of the Plastic Limit of a Soil by Means of a Rolling Device," *Geotechnical Testing Journal*, GTJODJ, Vol 15, No. 3, September 1992, pp. 284–287.

DIMENSIONS

LETTER	A△	B△	C△	D△	E△	F△
MM	2 ± 0.1	11 ± 0.2	40 ± 0.5	8 ± 0.1	50 ± 0.5	2 ± 0.1
LETTER	G	H	J	K△	L△	N
MM	10 MINIMUM	13	60	10 ±0.05	60 DEG ± 1 DEG	20

△ *ESSENTIAL DIMENSIONS*

□ *BACK AT LEAST 15 MM FROM TIP*

NOTE: DIMENSION A SHOULD BE 1.9–2.0 AND DIMENSION D SHOULD BE 8.0–8.1 WHEN NEW TO ALLOW FOR ADEQUATE SERVICE LIFE

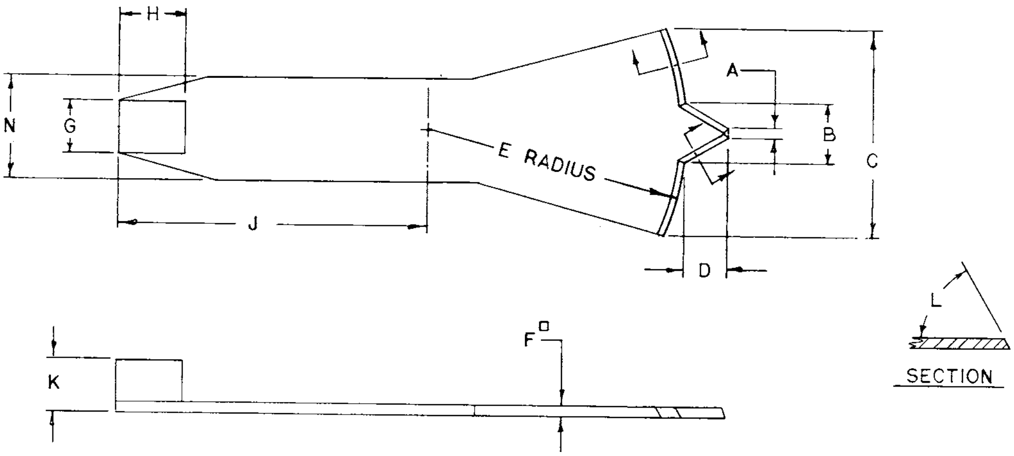
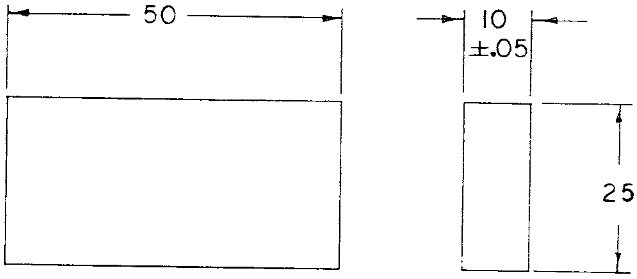


FIG. 3 Grooving Tool (Optional Height-of-Drop Gauge Attached)



DIMENSIONS IN MILLIMETRES

FIG. 4 Height-of-Drop Gauge

8. Sampling and Specimen

8.1 Samples may be taken from any location that satisfies testing needs. However, Practices C702, D75, and D420 should be used as guides for selecting and preserving samples from various types of sampling operations. Samples in which specimens will be prepared using the wet-preparation method (10.1) must be kept at their as–sampled water content prior to preparation.

8.1.1 Where sampling operations have preserved the natural stratification of a sample, the various strata must be kept separated and tests performed on the particular stratum of interest with as little contamination as possible from other strata. Where a mixture of materials will be used in construction, combine the various components in such proportions that the resultant sample represents the actual construction case.

8.1.2 Where data from these test methods are to be used for correlation with other laboratory or field test data, use the same material as used for those tests where possible.

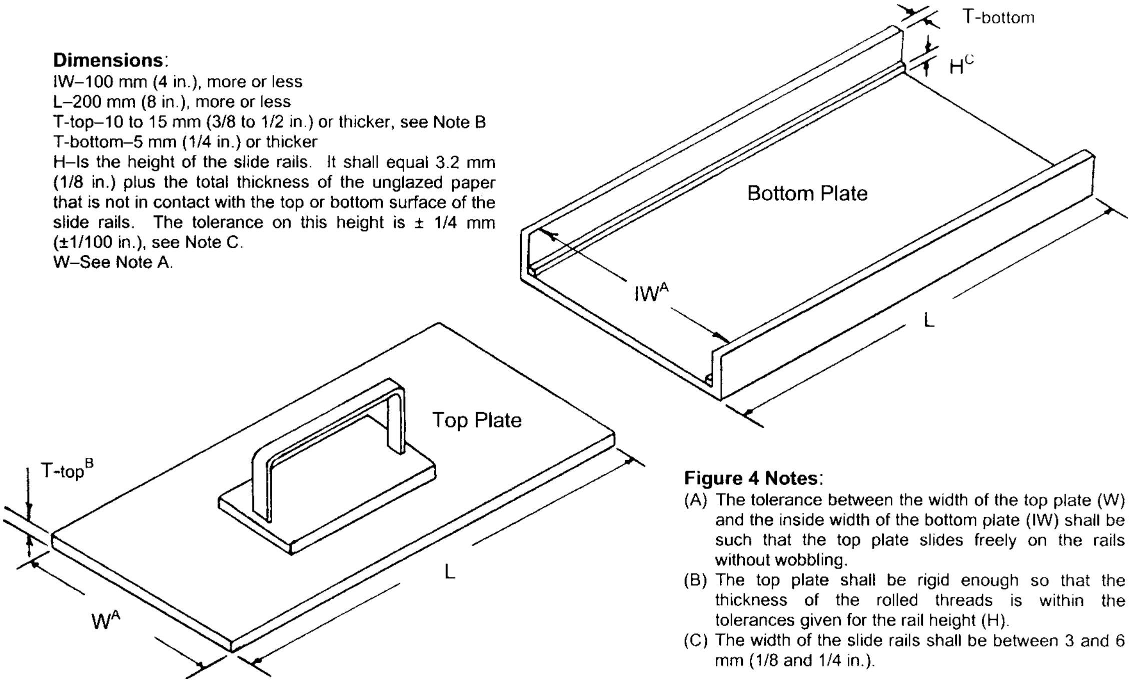
8.2 *Specimen*—Obtain a representative portion from the total sample sufficient to provide 150 to 200 g of material passing the 425-µm (No. 40) sieve. Free flowing samples (materials) may be reduced by the methods of quartering or splitting. Non-free flowing or cohesive materials shall be mixed thoroughly in a pan with a spatula or scoop and a representative portion scooped from the total mass by making one or more sweeps with a scoop through the mixed mass.

9. Calibration of Apparatus

9.1 *Inspection of Wear:*

9.1.1 *Liquid Limit Device*—Determine that the liquid limit device is clean and in good working order. Check the following specific points.

9.1.1.1 *Wear of Base*—The spot on the base where the cup makes contact should be worn no greater than 10 mm (3⁄8 in.) in diameter. If the wear spot is greater than this, the base can be machined to remove the worn spot provided the resurfacing does not make the base thinner than specified in 6.1 and the other dimensional relationships are maintained.



Dimensions:
IW–100 mm (4 in.), more or less
L–200 mm (8 in.), more or less
T-top–10 to 15 mm (3/8 to 1/2 in.) or thicker, see Note B
T-bottom–5 mm (1/4 in.) or thicker
H–Is the height of the slide rails. It shall equal 3.2 mm (1/8 in.) plus the total thickness of the unglazed paper that is not in contact with the top or bottom surface of the slide rails. The tolerance on this height is ± 1/4 mm (±1/100 in.), see Note C.
W–See Note A.

Figure 4 Notes:
(A) The tolerance between the width of the top plate (W) and the inside width of the bottom plate (IW) shall be such that the top plate slides freely on the rails without wobbling.
(B) The top plate shall be rigid enough so that the thickness of the rolled threads is within the tolerances given for the rail height (H).
(C) The width of the slide rails shall be between 3 and 6 mm (1/8 and 1/4 in.).

FIG. 5 Plastic Limit-Rolling Device

9.1.1.2 *Wear of Cup*—Replace the cup when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep or when the rim of the cup has been reduced to half its original thickness. Verify that the cup is firmly attached to the cup hanger.

9.1.1.3 *Wear of Cup Hanger*—Verify that the cup hanger pivot does not bind and is not worn to an extent that allows more than 3 mm (1⁄8 in.) side-to-side movement of the lowest point on the rim.

9.1.1.4 *Wear of Cam*—The cam shall not be worn to an extent that the cup drops before the cup hanger (cam follower) loses contact with the cam.

9.1.1.5 *Rubber Feet*—The feet should prevent the base from bouncing or sliding on the work surface. Replace rubber feet that become hard, cracked, or brittle from age.

9.1.2 *Grooving Tools*—Inspect grooving tools for wear on a frequent and regular basis. The rapidity of wear depends on the material from which the tool is made, and the types of soils being tested. Soils containing a large proportion of fine sand particles may cause rapid wear of grooving tools; therefore, when testing these materials, tools should be inspected more frequently than for other soils.

NOTE 4—The width of the tip of grooving tools is conveniently checked using a pocket-sized measuring magnifier equipped with a millimeter scale. Magnifiers of this type are available from most laboratory supply companies. The depth of the tip of grooving tools can be checked using the depth-measuring feature of vernier calipers.

9.2 *Adjustment of Height-of-Drop*—Adjust the height-of-drop of the cup so that the point on the cup that comes in contact with the base rises to a height of 10 ± 0.2 mm. See Fig. 2 for proper location of the gauge relative to the cup during adjustment.

NOTE 5—A convenient procedure for adjusting the height-of-drop is as follows: place a piece of masking tape across the outside bottom of the cup parallel with the axis of the cup hanger pivot. The edge of the tape away from the cup hanger should bisect the spot on the cup that contacts the base. For new cups, placing a piece of carbon paper on the base and allowing the cup to drop several times will mark the contact spot. Attach the cup to the device and turn the crank until the cup is raised to its maximum height. Slide the height gauge under the cup from the front, and observe whether the gauge contacts the cup or the tape. (See Fig. 2.) If the tape and cup are both simultaneously contacted, the height-of-drop is ready to be checked. If not, adjust the cup until simultaneous contact is made. Check adjustment by turning the crank at 2 revolutions per second while holding the gauge in position against the tape and cup. If a faint ringing or clicking sound is heard without the cup rising from the gauge, the adjustment is correct. If no ringing is heard or if the cup rises from the gauge, readjust the height-of-drop. If the cup rocks on the gauge during this checking operation, the cam follower pivot is excessively worn and the worn parts should be replaced. Always remove tape after completion of adjustment operation.

10. Preparation of Test Specimen

10.1 *Wet Preparation Method*—Except where the dry method of specimen preparation is specified (10.2), prepare the specimen for testing as described in the following sections.

10.1.1 *Material Passes the 425-µm (No. 40) Sieve:*

10.1.1.1 Determine by visual and manual methods that the specimen from 8.2 has little or no material retained on a 425-µm (No. 40) sieve. If this is the case, prepare 150 to 200 g of material by mixing thoroughly with distilled or demineralized water on the glass plate or mixing dish using the spatula. If desired, soak the material in a mixing/storage dish with a small amount of water to soften the material before the start of mixing. If using Method A, adjust the water content of the material to bring it to a consistency that would require about 25 to 35 blows of the liquid limit device to close the groove (Note 6). For Method B, the number of blows should be between about 20 and 30 blows.

10.1.1.2 If, during mixing, a small percentage of material is encountered that would be retained on a 425-µm (No. 40) sieve, remove these particles by hand (if possible). If it is impractical to remove the coarser material by hand, remove small percentages (less than about 15 %) of coarser material by working the material (having the above consistency) through a 425-µm sieve. During this procedure, use a piece of rubber sheeting, rubber stopper, or other convenient device provided the procedure does not distort the sieve or degrade material that would be retained if the washing method described in 10.1.2 were used. If larger percentages of coarse material are encountered during mixing, or it is considered impractical to remove the coarser material by the procedures just described, wash the sample as described in 10.1.2. When the coarse particles found during mixing are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-µm sieve, but remove by hand or by washing.

10.1.1.3 Place the prepared material in the mixing/storage dish, check its consistency (adjust if required), cover to prevent loss of moisture, and allow to stand (cure) for at least 16 h (overnight). After the standing period and immediately before starting the test, thoroughly remix the soil.

NOTE 6—The time taken to adequately mix a soil will vary greatly, depending on the plasticity and initial water content. Initial mixing times of more than 30 min may be needed for stiff, fat clays.

10.1.2 *Material Containing Particles Retained on a 425-µm (No. 40) Sieve:*

10.1.2.1 Place the specimen (see 8.2) in a pan or dish and add sufficient water to cover the material. Allow the material to soak until all lumps have softened and the fines no longer adhere to the surfaces of the coarse particles (Note 7).

NOTE 7—In some cases, the cations of salts present in tap water will exchange with the natural cations in the soil and significantly alter the test results if tap water is used in the soaking and washing operations. Unless it is known that such cations are not present in the tap water, distilled or demineralized water should be used. As a general rule, water containing more than 100 mg/L of dissolved solids should not be used for either the soaking or washing operations.

10.1.2.2 When the material contains a large percentage of particles retained on the 425-µm (No. 40) sieve, perform the following washing operation in increments, washing no more than 0.5 kg (1 lb) of material at one time. Place the 425-µm sieve in the bottom of the clean pan. Transfer, without any loss of material, the soil-water mixture onto the sieve. If gravel or coarse sand particles are present, rinse as many of these as possible with small quantities of water from a wash bottle, and discard. Alternatively, transfer the soil-water mixture over a 2.00-mm (No. 10) sieve nested atop the 425-µm sieve, rinse the fine material through and remove the 2.00-mm sieve. After washing and removing as much of the coarser material as possible, add sufficient water to the pan to bring the level to about 13 mm (½ in.) above the surface of the 425-µm sieve. Agitate the slurry by stirring with the fingers while raising and lowering the sieve in the pan and swirling the suspension so that fine material is washed from the coarser particles. Disaggregate fine soil lumps that have not slaked by gently rubbing them over the sieve with the fingertips. Complete the washing operation by raising the sieve above the water surface and rinsing the material retained with a small amount of clean water. Discard material retained on the 425-µm sieve.

10.1.2.3 Reduce the water content of the material passing the 425–µm (No. 40) sieve until it approaches the liquid limit. Reduction of water content may be accomplished by one or a combination of the following methods: (*a*) exposing to air currents at room temperature, (*b*) exposing to warm air currents from a source such as an electric hair dryer, (*c*) decanting clear water from surface of the suspension, (*d*) filtering in a Büchner funnel or using filter candles, or (*e*) draining in a colander or plaster of Paris dish lined with high retentivity,[5] high wet-strength filter paper. If a plaster of Paris dish is used, take care that the dish never becomes sufficiently saturated that it fails to absorb water into its surface. Thoroughly dry dish between uses. During evaporation and cooling, stir the material often enough to prevent over-drying of the fringes and soil pinnacles on the surface of the mixture. For materials containing soluble salts, use a method of water reduction (*a* or *b*) that will not eliminate the soluble salts from the test specimen.

10.1.2.4 If applicable, remove the material retained on the filter paper. Thoroughly mix this material or the above material on the glass plate or in the mixing dish using the spatula. Adjust the water content of the mixture, if necessary, by adding small increments of distilled or demineralized water or by allowing the mixture to dry at room temperature while mixing on the glass plate. If using Method A, the material should be at a water content that would require about 25 to 35 blows of the liquid limit device to close the groove. For Method B, the number of blows should be between about 20 and 30. Put, if necessary, the mixed material in the storage dish, cover to prevent loss of moisture, and allow to stand (cure) for at least 16 h. After the standing period and immediately before starting the test, thoroughly remix the specimen.

10.2 *Dry Preparation Method:*

10.2.1 Dry the specimen from 8.2 at room temperature or in an oven at a temperature not exceeding 60°C until the soil clods will pulverize readily. Disaggregation is expedited if the material is not allowed to completely dry. However, the material should have a dry appearance when pulverized.

10.2.2 Pulverize the material in a mortar with a rubber-tipped pestle or in some other way that does not cause breakdown of individual particles. When the coarse particles found during pulverization are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-µm (No. 40) sieve, but remove by hand or other suitable means, such as washing. If a washing procedure is used, follow 10.1.2.1-10.1.2.4.

10.2.3 Separate the material on a 425-µm (No. 40) sieve, shaking the sieve by hand to assure thorough separation of the finer fraction. Return the material retained on the 425-µm sieve to the pulverizing apparatus and repeat the pulverizing and sieving operations. Stop this procedure when most of the fine material has been disaggregated and material retained on the 425-µm sieve consists of individual particles.

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[5] S and S 595 filter paper available in 320-mm circles has proven satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

10.2.4 Place material retained on the 425-µm (No. 40) sieve after the final pulverizing operations in a dish and soak in a small amount of water. Stir this mixture and transfer it to a 425-µm sieve, catching the water and any suspended fines in the washing pan. Pour this suspension into a dish containing the dry soil previously sieved through the 425-µm sieve. Discard material retained on the 425-µm sieve.

10.2.5 Proceed as described in 10.1.2.3 and 10.1.2.4.

MULTIPOINT LIQUID LIMIT—METHOD A

11. Procedure

11.1 Thoroughly remix the specimen (soil) in its mixing dish, and, if necessary, adjust its water content until the consistency requires about 25 to 35 blows of the liquid limit device to close the groove. Using a spatula, place a portion(s) of the prepared soil in the cup of the liquid limit device at the point where the cup rests on the base, squeeze it down, and spread it into the cup to a depth of about 10 mm at its deepest point, tapering to form an approximately horizontal surface. Take care to eliminate air bubbles from the soil pat, but form the pat with as few strokes as possible. Keep the unused soil in the mixing/storage dish. Cover the dish with a wet towel (or use other means) to retain the moisture in the soil.

11.2 Form a groove in the soil pat by drawing the tool, beveled edge forward, through the soil on a line joining the highest point to the lowest point on the rim of the cup. When cutting the groove, hold the grooving tool against the surface of the cup and draw in an arc, maintaining the tool perpendicular to the surface of the cup throughout its movement. See Fig. 6. In soils where a groove cannot be made in one stroke without tearing the soil, cut the groove with several strokes of the grooving tool. Alternatively, cut the groove to slightly less than required dimensions with a spatula and use the grooving tool to bring the groove to final dimensions. Exercise extreme care to prevent sliding the soil pat relative to the surface of the cup.

11.3 Verify that no crumbs of soil are present on the base or the underside of the cup. Lift and drop the cup by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 13 mm (½ in.). See Fig. 7 and Fig. 8. The base of the machine shall not be held with the hand, or hands, while the crank is turned.

NOTE 8—Use of a scale is recommended to verify that the groove has closed 13 mm (½ in.).

11.4 Verify that an air bubble has not caused premature closing of the groove by observing that both sides of the groove have flowed together with approximately the same shape. If a bubble has caused premature closing of the groove, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving operation and repeat 11.1-11.3. If the soil slides on the surface of the cup, repeat 11.1-11.3 at a higher water content. If, after several trials at successively higher water contents, the soil pat continues to slide in the cup or if the number of blows required to close the groove is always less than 25, record that the liquid limit could not be determined, and report the soil as nonplastic without performing the plastic limit test.

11.5 Record the number of drops, *N*, required to close the groove. Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at right

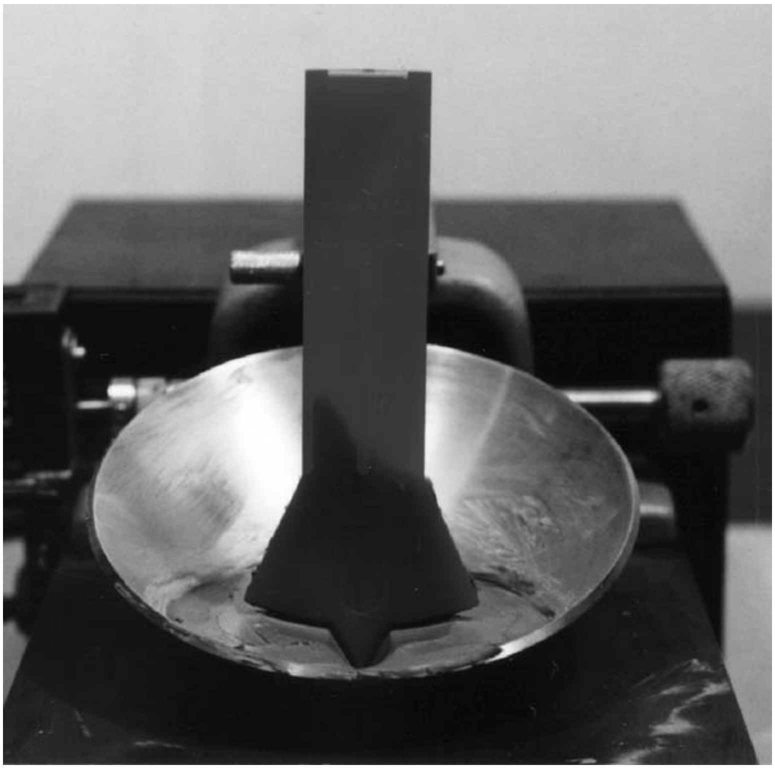

FIG. 6 Example of Grooving Tool Placed in a Properly Grooved Soil Pat

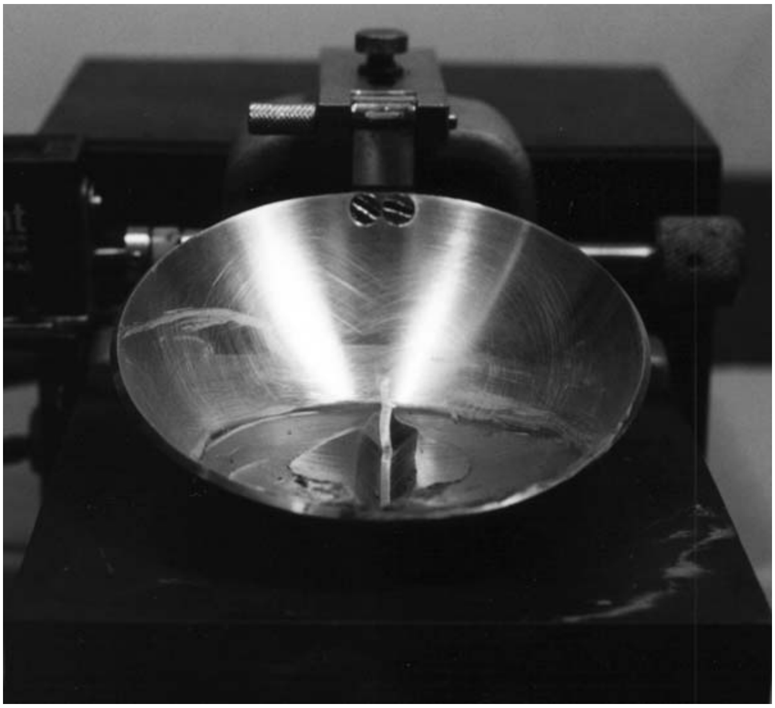

FIG. 7 Grooved Soil Pat in Liquid Limit Device

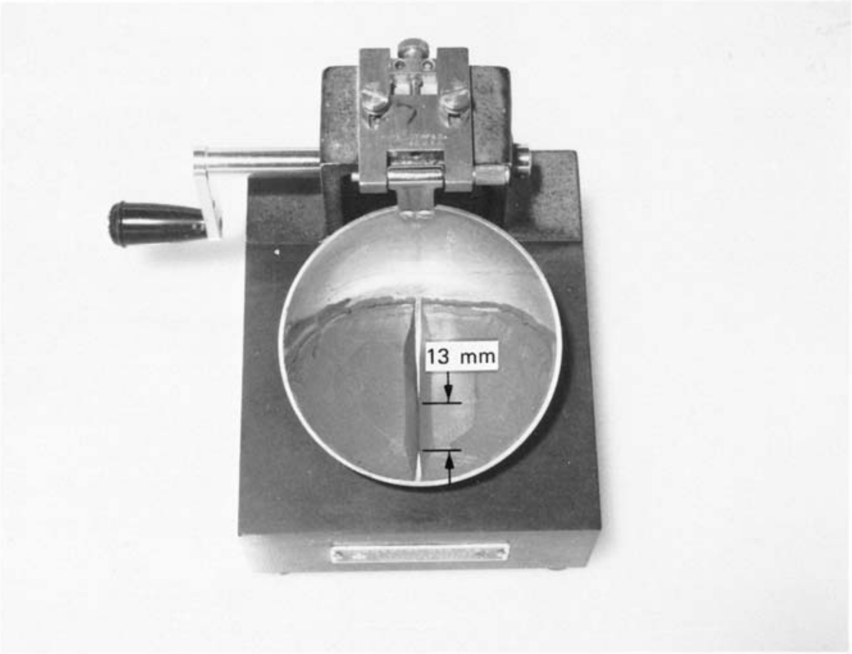

FIG. 8 Soil Pat After Groove Has Closed

angles to the groove and including that portion of the groove in which the soil flowed together, place in a container of known mass, and cover.

11.6 Return the soil remaining in the cup to the dish. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.

11.7 Remix the entire soil specimen in the dish adding distilled water to increase the water content of the soil and decrease the number of blows required to close the groove. Repeat 11.1-11.6 for at least two additional trials producing successively lower numbers of blows to close the groove. One of the trials shall be for a closure requiring 25 to 35 blows, one for closure between 20 and 30 blows, and one trial for a closure requiring 15 to 25 blows.

11.8 Determine the water content, W^n, of the soil specimen from each trial in accordance with Test Method D2216.

11.8.1 Determination of initial masses (container plus moist soil) should be performed immediately after completion of the test. If the test is to be interrupted for more than about 15 minutes, determine the mass of the water content specimens already obtained at the time of the interruption.

12. Calculation

12.1 Plot the relationship between the water content, W^n, and the corresponding number of drops, N, of the cup on a semilogarithmic graph with the water content as ordinates on the arithmetical scale, and the number of drops as abscissas on a logarithmic scale. Draw the best straight line through the three or more plotted points.

12.2 Take the water content corresponding to the intersection of the line with the 25-drop abscissa as the liquid limit of the soil and round to the nearest whole number. Computational methods may be substituted for the graphical method for fitting a straight line to the data and determining the liquid limit.

ONE-POINT LIQUID LIMIT—METHOD B

13. Procedure

13.1 Proceed as described in 11.1-11.5 except that the number of blows required to close the groove shall be 20 to 30. If less than 20 or more than 30 blows are required, adjust the water content of the soil and repeat the procedure.

13.2 Immediately after removing a water content specimen as described in 11.5, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving and water content sampling processes.

13.2.1 As an alternative to reforming the soil in the brass cup after removing the water content specimen, the soil remaining in the cup can be removed from the cup, remixed with the soil in the mixing container and a new specimen placed in the cup as described in 11.1.

13.3 Repeat 11.2-11.5

13.4 If the second closing of the groove requires the same number of drops or no more than two drops difference, secure another water content specimen. If the difference of the number of drops between the first and second closings of the groove is greater than two, remix the entire specimen and repeat the procedure, beginning at 13.1, until two successive closures having the same number of drops or no more than two drops difference are obtained.

NOTE 9—Excessive drying or inadequate mixing will cause the number of blows to vary.

13.5 Determine water contents of the two specimens in accordance with 11.8.

14. Calculation

14.1 Determine the liquid limit for each water content specimen using one of the following equations:

$$LL^n = W^n \cdot \left(\frac{N}{25}\right)^{0.121}$$

or

$$LL^n = k \cdot W^n$$

where:

LL^n = one point liquid limit for given trial, %,
N = number of blows causing closure of the groove for given trial,
W^n = water content for given trial, %, and
k = factor given in Table 1.

14.1.1 The liquid limit, LL, is the average of the two trial liquid-limit values, to the nearest whole number (without the percent designation).

14.2 If the difference between the two trial liquid-limit values is greater than one percentage point, repeat the test as described in 13.1 through 14.1.1.

PLASTIC LIMIT

15. Preparation of Test Specimen

15.1 Select a 20-g or more portion of soil from the material prepared for the liquid limit test; either, after the second mixing before the test, or from the soil remaining after completion of the liquid limit test. Reduce the water content of the soil to a consistency at which it can be rolled without sticking to the hands by spreading or mixing continuously on the glass plate or in the mixing/storage dish. The drying process may be accelerated by exposing the soil to the air current from an electric fan, or by blotting with paper, that does not add any fiber to the soil. Paper such as hard surface paper toweling or high wet-strength filter paper is adequate.

16. Procedure

16.1 From this plastic-limit specimen, select a 1.5 to 2.0 g portion. Form the selected portion into an ellipsoidal mass.

16.2 Roll the soil mass by one of the following methods (hand or rolling device):

16.2.1 *Hand Method*—Roll the mass between the palm or fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length (see Note 10). The thread shall be further deformed on each stroke so that its diameter reaches 3.2 mm (⅛ in.), taking no more than 2 min (see Note 11). The amount of hand or finger pressure required will vary greatly according to the soil being tested, that is, the required pressure typically increases with increasing plasticity. Fragile soils of low plasticity are best rolled under the outer edge of the palm or at the base of the thumb.

TABLE 1 Factors for Obtaining Liquid Limit from Water Content and Number of Drops Causing Closure of Groove

N (Number of Drops)	k (Factor for Liquid Limit)
20	0.973
21	0.979
22	0.985
23	0.990
24	0.995
25	1.000
26	1.005
27	1.009
28	1.014
29	1.018
30	1.022

NOTE 10—A normal rate of rolling for most soils should be 80 to 90 strokes per minute, counting a stroke as one complete motion of the hand forward and back to the starting position. This rate of rolling may have to be decreased for very fragile soils.

NOTE 11—A 3.2-mm (⅛-in.) diameter rod or tube is useful for frequent comparison with the soil thread to ascertain when the thread has reached the proper diameter.

16.2.2 *Rolling Device Method*—Attach smooth unglazed paper to both the top and bottom plates of the plastic limit-rolling device. Place the soil mass on the bottom plate at the midpoint between the slide rails. Place the top plate in contact with the soil mass(es). Simultaneously apply a slight downward force and back and forth motion to the top plate so that the top plate comes into contact with the side rails within 2 min (see Notes 10 and 12). During this rolling process, the end(s) the soil thread(s) shall not contact the side rail(s). If this occurs, roll a smaller mass of soil (even if it is less than that mentioned in Section 16.1).

NOTE 12—In most cases, two soil masses (threads) can be rolled simultaneously in the plastic limit-rolling device.

16.3 When the diameter of the thread becomes 3.2 mm, break the thread into several pieces. Squeeze the pieces together, knead between the thumb and first finger of each hand, reform into an ellipsoidal mass, and re-roll. Continue this alternate rolling to a thread 3.2 mm in diameter, gathering together, kneading and re-rolling, until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a 3.2-mm diameter thread (see Fig. 9). It has no significance if the thread breaks into threads of shorter length. Roll each of these shorter threads to 3.2 mm in diameter. The only requirement for continuing the test is that these threads can be reformed into an ellipsoidal mass and rolled out again. The operator shall at no time attempt to produce failure at exactly 3.2-mm diameter by allowing the thread to reach 3.2 mm, then reducing the rate of rolling or the hand pressure, or both, while continuing the rolling without further deformation until the thread falls apart. It is permissible, however, to reduce the total amount of deformation for feebly plastic soils by making the initial diameter of the ellipsoidal mass nearer to the required 3.2-mm final diameter. If crumbling occurs when the thread has a diameter greater than 3.2 mm, this shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread 3.2 mm in diameter. Crumbling of the thread will manifest itself differently with the various types of soil. Some soils fall apart in numerous small aggregations of particles, others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally, the thread falls apart in many small platy particles. Fat clay soils require much pressure to deform the thread, particularly as they approach the plastic limit. With these soils, the thread breaks into a series of barrel-shaped segments about 3.2 to 9.5 mm (⅛ to ⅜ in.) in length.

16.4 Gather the portions of the crumbled thread together and place in a container of known mass. Immediately cover the container.

16.5 Select another 1.5 to 2.0-g portion of soil from the plastic–limit specimen and repeat the operations described in 16.1 and 16.2 until the container has at least 6 g of soil.

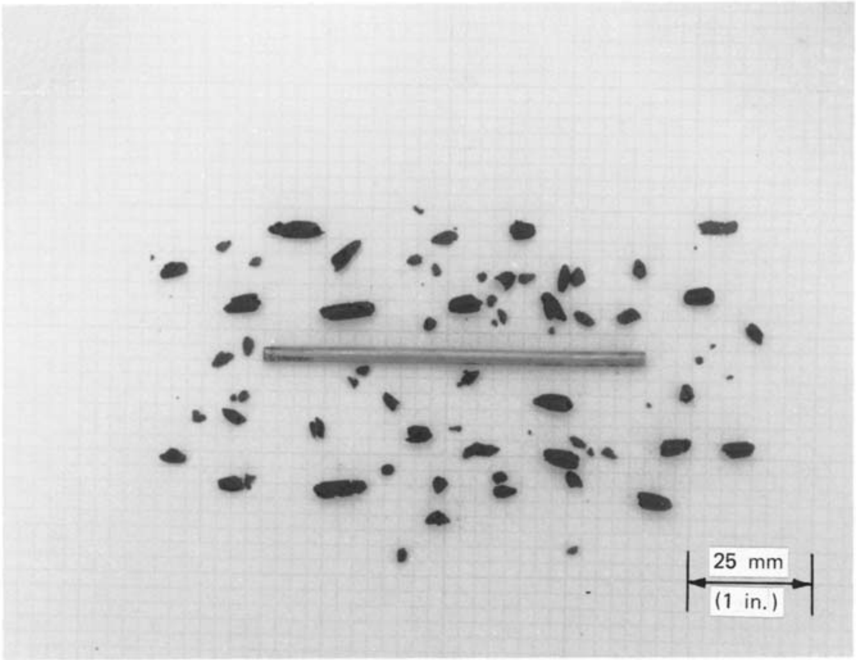

FIG. 9 Lean Clay Soil at the Plastic Limit

16.6 Repeat 16.1-16.5 to make another container holding at least 6 g of soil. Determine the water content of the soil contained in the containers in accordance with Test Method D2216. See 11.8.1.

17. Calculation

17.1 Compute the average of the two water contents (trial plastic limits) and round to the nearest whole number. This value is the plastic limit, *PL*. Repeat the test if the difference between the two trial plastic limits is greater than the acceptable range for two results listed in Table 2 for single-operator precision, that is, 1.4 percentage points; i.e., (2.8 × 0.5).

PLASTICITY INDEX

18. Calculation

18.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:

LL = liquid limit (whole number), and
PL = plastic limit (whole number).

18.1.1 Both LL and PL are whole numbers. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

19. Report: Test Data Sheet(s)/Form(s)

19.1 The terminology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.9.

19.2 Record as a minimum the following information:

19.2.1 Sample/specimen identifying information, such as project name , project number, boring number, depth (m or ft).

19.2.2 Description of sample, such as approximate maximum grain size, estimate of the percentage of sample retained on the 425-µm (No. 40) sieve, as-received water content.

19.2.3 Details of specimen preparation, such as wet or dry (air-dried or oven-dried), method of removing particles larger than the 425-µm (No. 40) sieve.

19.2.4 Any special specimen selection process used, such as removal of sand lenses from an intact (undisturbed) sample.

19.2.5 Equipment used, such as hand rolled or mechanical rolling device for plastic limit, manual or mechanical liquid limit device, metal or plastic grooving tool.

19.2.6 Liquid limit, plastic limit, and plasticity index to the nearest whole number, omitting the percent designation. If the liquid limit or plastic limit tests could not be performed, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

19.2.7 Procedure by which liquid limit was performed, if it differs from the multipoint method.

20. Precision and Bias

20.1 *Precision*—Criteria for judging the acceptability of test results obtained by these test methods on a range of soil types are given in Tables 2 and 3. In performing these test methods, Method A and the Wet Preparation Method (except soil was air-dried) were used.

20.1.1 These estimates of precision are based on the results of the interlaboratory program conducted by the ASTM Reference Soils and Testing Program.[6] In this program, some laboratories performed three replicate tests per soil type (triplicate test laboratory), while other laboratories performed a single test per soil type (single-test laboratory). A description of the soils tested is given in 20.1.5. The precision estimates vary with soil type and method(s) used. Judgment is required when applying these estimates to another soil and method used (Method A or B, or Wet or Dry Preparation Method).

20.1.2 The data in Table 2 are based on three replicate tests performed by each triplicate test laboratory on each soil type. The single operator and multilaboratory standard deviation shown in Table 2, Column 4, were obtained in accordance with Practice E691, which recommends each testing laboratory perform a minimum of three replicate tests. Results of two properly conducted tests performed by the same operator on

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1013.

TABLE 2 Summary of Test Results from Triplicate Test Laboratories (Atterberg Limits)

(1) Soil Type	(2) Number of Triplicate Test Laboratories			(3) Average Value^A (Percentage Points)			(4) Standard Deviation^B (Percentage Points)			(5) Acceptable Range of Two Results^C (Percentage Points)		
	Type Test											
	LL	PL	PI	LL	PL	PI	LL	PL	PI	LL	PL	PI
	Single-Operator Results (Within-Laboratory Repeatability)											
CH	13	13	13	59.8	20.6	39.2	0.7	0.5	0.8	2	1	2
CL	14	13	13	33.4	19.9	13.6	0.3	0.4	0.5	1	1	1
ML	12	11	11	27.4	23.4^D	4.1^D	0.5	0.3	0.6	2	1	2
	Multilaboratory Results (Between-Laboratory Reproducibility)											
CH	13	13	13	59.8	20.6	39.2	1.3	2.0	2.5	4	6	7
CL	14	13	13	33.4	19.9	13.6	1.0	1.2	1.7	3	3	5
ML	12	11	11	27.4	23.4^D	4.1^D	1.3	0.9	1.9	4	3	5

[A] The number of significant digits and decimal places presented are representative of the input data. In accordance with Practice D6026, the standard deviation and acceptable range of results can not have more decimal places than the input data.

[B] Standard deviation is calculated in accordance with Practice E691 and is referred to as the 1*s* limit.

[C] Acceptable range of two results is referred to as the d2*s* limit. It is calculated as $-1.960 \cdot \sqrt{2} \cdot 1s$, as defined by Practice E177. The difference between two properly conducted tests should not exceed this limit. The number of significant digits/decimal places presented is equal to that prescribed by this test method or Practice D6026. In addition, the value presented can have the same number of decimal places as the standard deviation, even if that result has more significant digits than the standard deviation.

[D] For the ML soil, 2 out of 14 triplicate test laboratories reported the soil as nonplastic.

TABLE 3 Summary of Single-Test Result from Each Laboratory (Atterberg Limits)[A]

(1)	(2)	(3)			(4)			(5)		
Soil Type	Number of Test Laboratories	Average Value (Percentage Points)			Standard Deviation (Percentage Points)			Acceptable Range of Two Results (Percentage Points)		
		Type Test								
		LL	PL	PI	LL	PL	PI	LL	PL	PI
CH	24	59.9	20.4	39.5	2.1	2.7	3.1	6	7	9
CL	24	33.3	19.9	13.4	0.8	1.3	1.6	2	4	4
ML	18	27.1	23.2[B]	3.9[B]	1.3	1.2	1.8	4	3	5

[A] For column footnotes, see Table 3.

[B] For the ML soil, 6 out of 24 laboratories reported the soil as nonplastic.

the same material, using the same equipment, and in the shortest practical period of time should not differ by more than the single-operator d2*s* limits shown in Table 2, Column 5. For definition of d2*s* see Footnote C in Table 2. Results of two properly conducted tests performed by different operators and on different days should not differ by more than the multilaboratory d2*s* limits shown in Table 2, Column 5.

20.1.3 In the ASTM Reference Soils and Testing Program, many of the laboratories performed only a single test on each soil type. This is common practice in the design and construction industry. The data for each soil type in Table 3 are based upon the first test results from the triplicate test laboratories and the single test results from the other laboratories. Results of two properly conducted tests performed by two different laboratories with different operators using different equipment and on different days should not vary by more than the d2*s* limits shown in Table 3, Column 5. The results in Table 2 and Table 3 are dissimilar because the data sets are different.

20.1.4 Table 2 presents a rigorous interpretation of triplicate test data in accordance with Practice E691 from pre-qualified laboratories. Table 3 is derived from test data that represents common practice.

20.1.5 *Soil Types*—Based on the multilaboratory test results, the soils used in the program are described below in accordance with Practice D2487. In addition, the local names of the soils are given.

CH—Fat clay, CH, 99 % fines, LL=60, PI=39, grayish brown, soil had been air dried and pulverized. Local name—Vicksburg Buckshot Clay

CL—Lean clay, CL, 89 % fines, LL=33, PI=13, gray, soil had been air dried and pulverized. Local name—Annapolis Clay

ML—Silt, ML, 99 % fines, LL=27, PI=4, light brown, soil had been air dried and pulverized. Local name—Vicksburg Silt

20.2 *Bias*—There is no acceptable reference value for these test methods; therefore, bias cannot be determined.

21. Keywords

21.1 activity; Atterberg limits; liquid limit; plasticity index; plastic limit

ANNEX

(Mandatory Information)

A1. Resilience Tester

A1.1 A device for measuring the resilience of liquid limit device bases is shown in Fig. A1.1. The device consists of a clear acrylic plastic tube and cap, a 5⁄16-in. diameter steel ball, and a small bar magnet. The cylinder may be cemented to the cap or threaded as shown. The small bar magnet is held in the recess of the cap and the steel ball is fixed into the recess in the underside of the cap with the bar magnet. The cylinder is then turned upright and placed on the top surface of the base to be tested. Holding the tube lightly against the liquid limit device base with one hand, release the ball by pulling the magnet out of the cap. Use the scale markings on the outside of the cylinder to determine the highest point reached by the bottom of the ball. Repeat the drop at least three times, placing the tester in a different location for each drop. Tests should be conducted at room temperature.

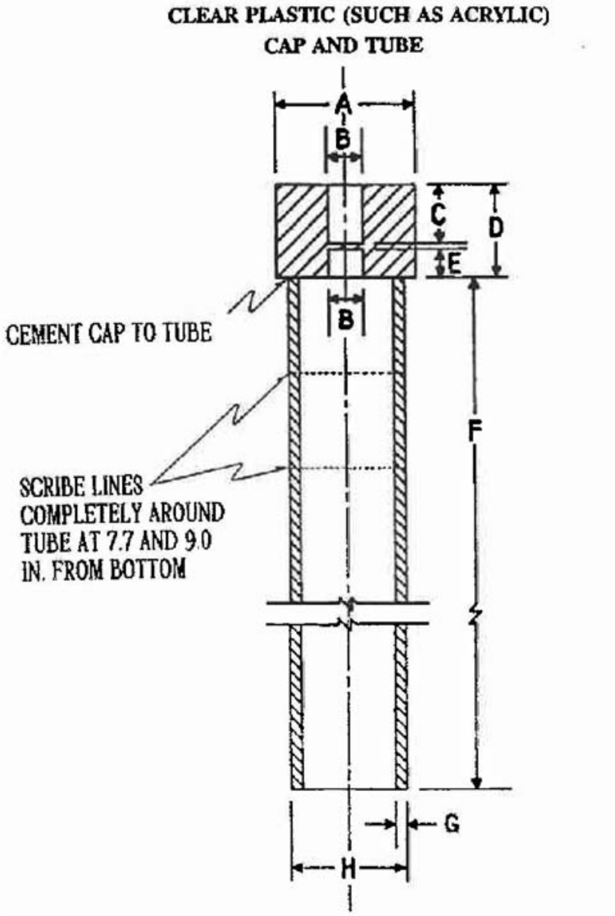


TABLE OF MEASUREMENTS

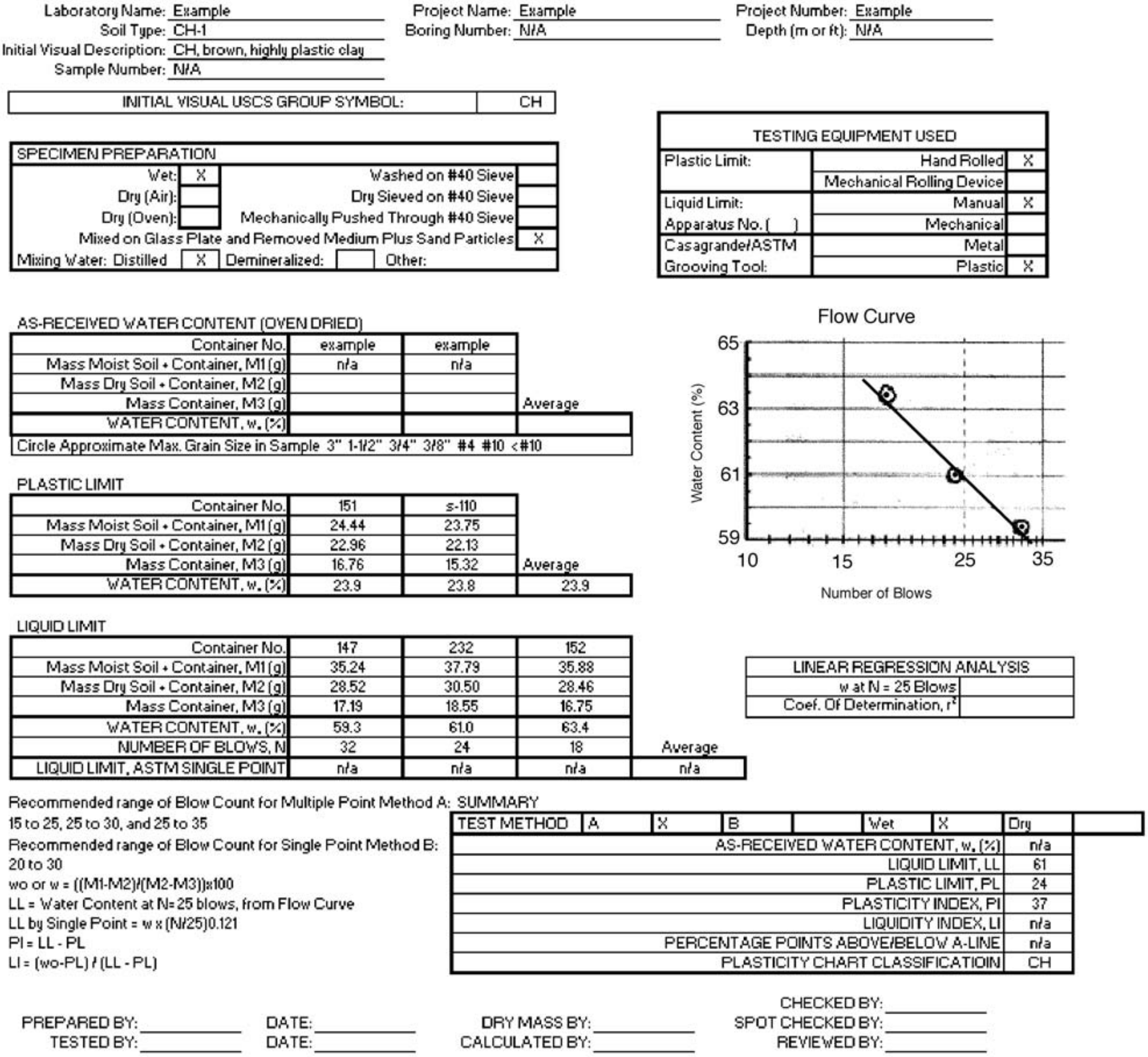
DIMENSION	DESCRIPTION	ENGLISH, in.	SI, mm
A	DIAM. OF CAP	1 1/2	38.10
B	DIAM. OF HOLE	3/8	9.52
C	DEPTH OF HOLE	10/16	15.88
D	HEIGHT OF CAP	1	25.40
E	DEPTH OF HOLE	5/16	7.94
F	LENGTH OF TUBE	10	254.00
G	WALL THICKNESS	1/8	3.18
H	O.D. OF TUBE	1 1/4	31.75

FIG. A1.1 Resilience Tester

APPENDIX

X1. Sample Data Sheet

X1.1 See Fig. X1.1.



Laboratory Name: Example
Soil Type: CH-1
Initial Visual Description: CH, brown, highly plastic clay
Sample Number: N/A

Project Name: Example
Boring Number: N/A

Project Number: Example
Depth (m or ft): N/A

INITIAL VISUAL USCS GROUP SYMBOL:	CH

SPECIMEN PREPARATION			
Wet:	X	Washed on #40 Sieve	
Dry (Air):		Dry Sieved on #40 Sieve	
Dry (Oven):		Mechanically Pushed Through #40 Sieve	
		Mixed on Glass Plate and Removed Medium Plus Sand Particles	X

Mixing Water: Distilled ☒ Demineralized: ☐ Other:

TESTING EQUIPMENT USED		
Plastic Limit:	Hand Rolled	X
	Mechanical Rolling Device	
Liquid Limit:	Manual	X
Apparatus No. ( )	Mechanical	
Casagrande/ASTM	Metal	
Grooving Tool:	Plastic	X

AS-RECEIVED WATER CONTENT (OVEN DRIED)

Container No.	example	example	
Mass Moist Soil + Container, M1 (g)	n/a	n/a	
Mass Dry Soil + Container, M2 (g)			
Mass Container, M3 (g)			Average
WATER CONTENT, w, (%)			

Circle Approximate Max. Grain Size in Sample 3" 1-1/2" 3/4" 3/8" #4 #10 <#10

PLASTIC LIMIT

Container No.	151	s-110	
Mass Moist Soil + Container, M1 (g)	24.44	23.75	
Mass Dry Soil + Container, M2 (g)	22.96	22.13	
Mass Container, M3 (g)	16.76	15.32	Average
WATER CONTENT, w, (%)	23.9	23.8	23.9

Flow Curve

LIQUID LIMIT

Container No.	147	232	152	
Mass Moist Soil + Container, M1 (g)	35.24	37.79	35.88	
Mass Dry Soil + Container, M2 (g)	28.52	30.50	28.46	
Mass Container, M3 (g)	17.19	18.55	16.75	
WATER CONTENT, w, (%)	59.3	61.0	63.4	
NUMBER OF BLOWS, N	32	24	18	Average
LIQUID LIMIT, ASTM SINGLE POINT	n/a	n/a	n/a	n/a

LINEAR REGRESSION ANALYSIS	
w at N = 25 Blows	
Coef. Of Determination, r²	

Recommended range of Blow Count for Multiple Point Method A:
15 to 25, 25 to 30, and 25 to 35
Recommended range of Blow Count for Single Point Method B:
20 to 30
wo or w = ((M1-M2)/(M2-M3))x100
LL = Water Content at N=25 blows, from Flow Curve
LL by Single Point = w x (N/25)0.121
PI = LL - PL
LI = (wo-PL) / (LL - PL)

SUMMARY

TEST METHOD	A	X	B		Wet	X	Dry	
AS-RECEIVED WATER CONTENT, w, (%)							n/a	
LIQUID LIMIT, LL							61	
PLASTIC LIMIT, PL							24	
PLASTICITY INDEX, PI							37	
LIQUIDITY INDEX, LI							n/a	
PERCENTAGE POINTS ABOVE/BELOW A-LINE							n/a	
PLASTICITY CHART CLASSIFICATIOIN							CH	

PREPARED BY: ____________ DATE: ____________
TESTED BY: ____________ DATE: ____________
DRY MASS BY: ____________
CALCULATED BY: ____________
CHECKED BY: ____________
SPOT CHECKED BY: ____________
REVIEWED BY: ____________

FIG. X1.1 Sample Data Sheet

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D4318 – 05) that may impact the use of this standard. (Approved January 15, 2010.)

(*1*) Corrected 1.6 to reference D4542 and added D4542 to Referenced Documents in Section 2.
(*2*) In 1.8 and 1.8.1, clarified use of SI units.
(*3*) Added 1.9 referencing D6026 and the use of significant digits and renumbered 1.9 as 1.10.
(*4*) In 6.1 and 6.1.1 reworded the requirements for the composition of the base and removed the word "rubber." "Rubber" was also removed from the label in Fig. 1.
(*5*) In 6.1.2 removed the Durometer hardness requirement for the rubber feet.
(*6*) In 6.7.1 removed the dimensional requirements for the Ground Glass Plate.
(*7*) In 9.1.1.5 added guidance for replacement of rubber feet.
(*8*) In 11.1 changed "cup" to "dish" for consistency.
(*9*) In 11.3 added instruction that the base shall not be held during testing.
(*10*) In 13.2 to 13.5 clarified the instructions to allow two alternative test procedures.
(*11*) Section 19 was updated to comply with the D18.91 Special Memorandum on Report Section.

Designation: D4767 – 04

Standard Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils[1]

1. Scope*

1.1 This test method covers the determination of strength and stress-strain relationships of a cylindrical specimen of either an undisturbed or remolded saturated cohesive soil. Specimens are isotropically consolidated and sheared in compression without drainage at a constant rate of axial deformation (strain controlled).

1.2 This test method provides for the calculation of total and effective stresses, and axial compression by measurement of axial load, axial deformation, and pore-water pressure.

1.3 This test method provides data useful in determining strength and deformation properties of cohesive soils such as Mohr strength envelopes and Young's modulus. Generally, three specimens are tested at different effective consolidation stresses to define a strength envelope.

1.4 The determination of strength envelopes and the development of relationships to aid in interpreting and evaluating test results are beyond the scope of this test method and must be performed by a qualified, experienced professional.

1.5 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.5.1 The method used to specify how data are collected, calculated, or recorded in this standard is not directly related to the accuracy to which the data can be applied in design or other uses, or both. How one applies the results obtained using this standard is beyond its scope.

1.6 The values stated in SI units shall be regarded as the standard. The values stated in inch-pound units are approximate.

1.7 *This standard does not purport to address all of the safety concerns, 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:*[2]

D422 Test Method for Particle-Size Analysis of Soils
D653 Terminology Relating to Soil, Rock, and Contained Fluids
D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer
D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes
D2166 Test Method for Unconfined Compressive Strength of Cohesive Soil
D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
D2435 Test Methods for One-Dimensional Consolidation Properties of Soils Using Incremental Loading
D2850 Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D4220 Practices for Preserving and Transporting Soil Samples
D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils
D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing
D6026 Practice for Using Significant Digits in Geotechnical Data

3. Terminology

3.1 *Definitions*—The definitions of terms used in this test method shall be in accordance with Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *back pressure*—a pressure applied to the specimen pore-water to cause air in the pore space to compress and to

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Strength and Compressibility of Soils.

Current edition approved Nov. 1, 2004. Published December 2004. Originally approved in 1988. Last previou edition approved in 2002 as D4767 – 02. DOI: 10.1520/D4767-04.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

pass into solution in the pore-water thereby increasing the percent saturation of the specimen.

3.2.2 *effective consolidation stress*—the difference between the cell pressure and the pore-water pressure prior to shearing the specimen.

3.2.3 *failure*—the stress condition at failure for a test specimen. Failure is often taken to correspond to the maximum principal stress difference (maximum deviator stress) attained or the principal stress difference (deviator stress) at 15 % axial strain, whichever is obtained first during the performance of a test. Depending on soil behavior and field application, other suitable failure criteria may be defined, such as maximum effective stress obliquity, $\sigma'1/\sigma'3$, or the principal stress difference (deviator stress) at a selected axial strain other than 15 %.

4. Significance and Use

4.1 The shear strength of a saturated soil in triaxial compression depends on the stresses applied, time of consolidation, strain rate, and the stress history experienced by the soil.

4.2 In this test method, the shear characteristics are measured under undrained conditions and is applicable to field conditions where soils that have been fully consolidated under one set of stresses are subjected to a change in stress without time for further consolidation to take place (undrained condition), and the field stress conditions are similar to those in the test method.

NOTE 1—If the strength is required for the case where the soil is not consolidated during testing prior to shear, refer to Test Method D2850 or Test Method D2166.

4.3 Using the pore-water pressure measured during the test, the shear strength determined from this test method can be expressed in terms of effective stress. This shear strength may be applied to field conditions where full drainage can occur (drained conditions) or where pore pressures induced by loading can be estimated, and the field stress conditions are similar to those in the test method.

4.4 The shear strength determined from the test expressed in terms of total stresses (undrained conditions) or effective stresses (drained conditions) is commonly used in embankment stability analyses, earth pressure calculations, and foundation design.

NOTE 2—Notwithstanding the statements on precision and bias contained in this test method. The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies which meet the criteria of Practice D3740 are generally considered capable of competent testing. Users of this test method are cautioned that compliance with Practice D3740 does not ensure reliable testing. Reliable testing depends on several factors; Practice D3740 provides a means of evaluating some of those factors.

5. Apparatus

5.1 The requirements for equipment needed to perform satisfactory tests are given in the following sections. See Fig. 1 and Fig. 2

5.2 *Axial Loading Device*—The axial loading device shall be a screw jack driven by an electric motor through a geared transmission, a hydraulic loading device, or any other compression device with sufficient capacity and control to provide the rate of axial strain (loading) prescribed in 8.4.2. The rate of advance of the loading device shall not deviate by more than ±1 % from the selected value. Vibration due to the operation of the loading device shall be sufficiently small to not cause dimensional changes in the specimen or to produce changes in pore-water pressure when the drainage valves are closed.

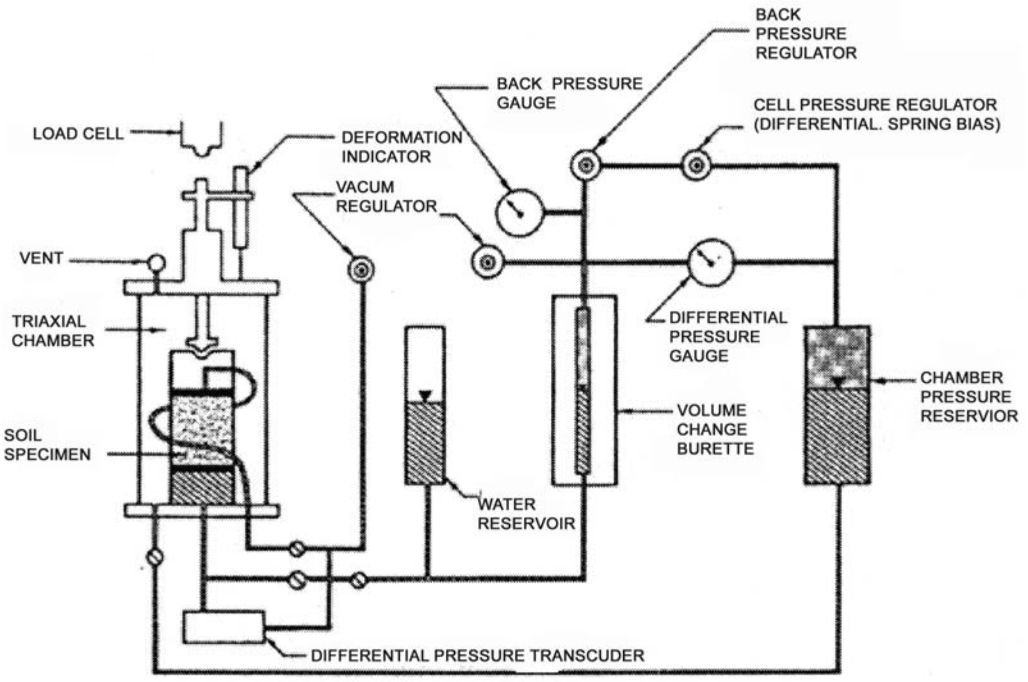

FIG. 1 Schematic Diagram of a Typical Consolidated Undrained Triaxial Apparatus

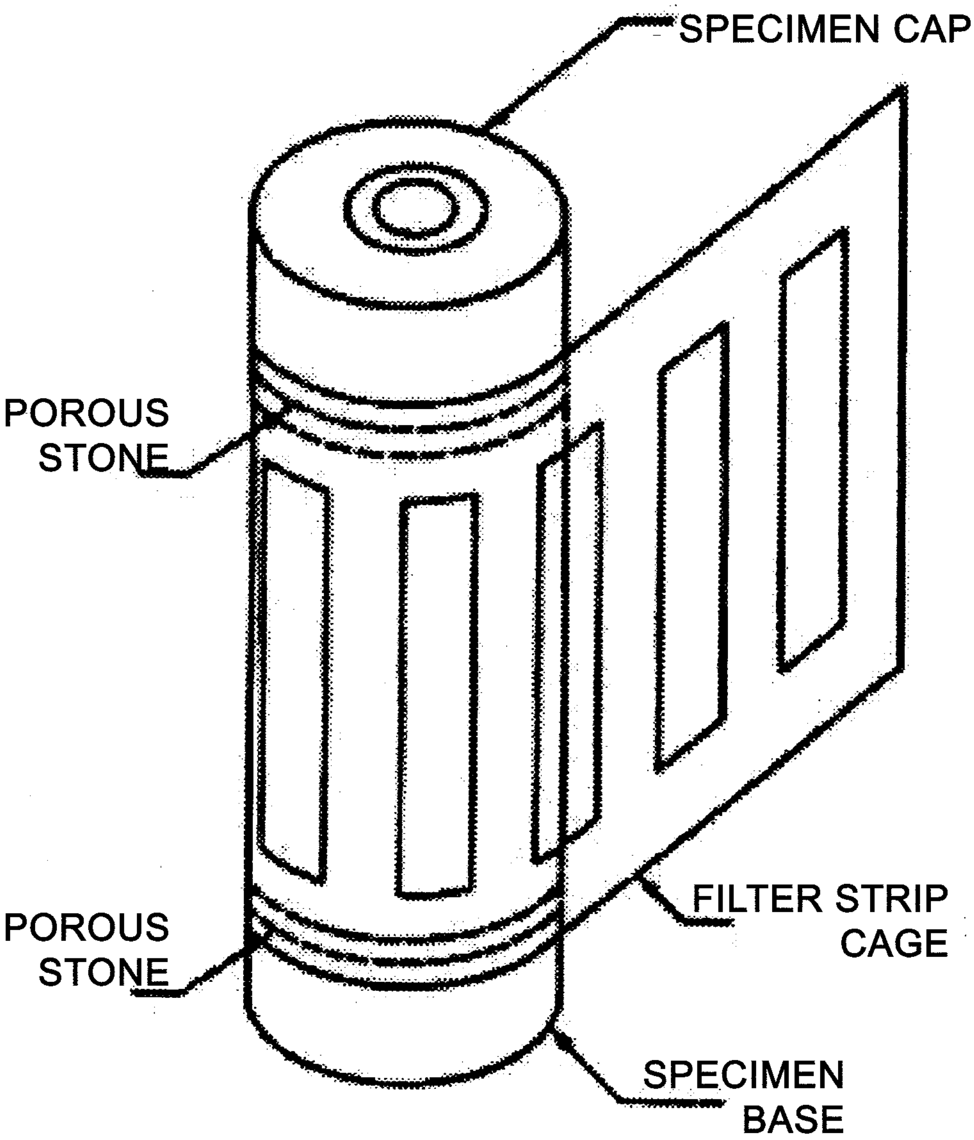

FIG. 2 Filter Strip Cage

NOTE 3—A loading device may be judged to produce sufficiently small vibrations if there are no visible ripples in a glass of water placed on the loading platform when the device is operating at the speed at which the test is performed.

5.3 *Axial Load-Measuring Device*—The axial load-measuring device shall be a load ring, electronic load cell, hydraulic load cell, or any other load-measuring device capable of the accuracy prescribed in this paragraph and may be a part of the axial loading device. The axial load-measuring device shall be capable of measuring the axial load to an accuracy of within 1 % of the axial load at failure. If the load-measuring device is located inside the triaxial compression chamber, it shall be insensitive to horizontal forces and to the magnitude of the chamber pressure.

5.4 *Triaxial Compression Chamber*—The triaxial chamber shall have a working chamber pressure equal to the sum of the effective consolidation stress and the back pressure. It shall consist of a top plate and a base plate separated by a cylinder. The cylinder may be constructed of any material capable of withstanding the applied pressures. It is desirable to use a transparent material or have a cylinder provided with viewing ports so the behavior of the specimen may be observed. The top plate shall have a vent valve such that air can be forced out of the chamber as it is filled. The baseplate shall have an inlet through which the pressure liquid is supplied to the chamber, and inlets leading to the specimen base to the cap to allow saturation and drainage of the specimen when required. The chamber shall provide a connection to the cap.

5.5 *Axial Load Piston*—The piston passing through the top of the chamber and its seal must be designed so the variation in axial load due to friction does not exceed 0.1 % of the axial load at failure and so there is negligible lateral bending of the piston during loading.

NOTE 4—The use of two linear ball bushings to guide the piston is recommended to minimize friction and maintain alignment.

NOTE 5—A minimum piston diameter of ⅙ the specimen diameter has been used successfully in many laboratories to minimize lateral bending.

5.6 *Pressure and Vacuum-Control Devices*—The chamber pressure and back pressure control devices shall be capable of applying and controlling pressures to within ±2 kPa (0.25 lb/in. ^{2}) for effective consolidation pressures less than 200 kPa (28 lb/in. ^{2}) and to within ±1 % for effective consolidation pressures greater than 200 kPa. The vacuum-control device shall be capable of applying and controlling partial vacuums to within ±2 kPa. The devices shall consist of pressure/volume controllers, self-compensating mercury pots, pneumatic pressure regulators, combination pneumatic pressure and vacuum regulators, or any other device capable of applying and controlling pressures or partial vacuums to the required tolerances. These tests can require a test duration of several day. Therefore, an air/water interface is not recommended for either the chamber pressure or back pressure systems, unless isolated from the specimen and chamber (e.g. by long tubing).

5.7 *Pressure- and Vacuum-Measurement Devices*—The chamber pressure-, back pressure-, and vacuum-measuring devices shall be capable of measuring pressures or partial vacuums to the tolerances given in 5.6. They may consist of Bourdon gages, pressure manometers, electronic pressure transducers, or any other device capable of measuring pressures, or partial vacuums to the stated tolerances. If separate devices are used to measure the chamber pressure and back pressure, the devices must be calibrated simultaneously and against the same pressure source. Since the chamber and back pressure are the pressures taken at the mid-height of the specimen, it may be necessary to adjust the calibration of the devices to reflect the hydraulic head of fluids in the chamber and back pressure control systems.

5.8 *Pore-Water Pressure-Measurement Device*—The specimen pore-water pressure shall also be measured to the tolerances given in 5.6. During undrained shear, the pore-water pressure shall be measured in such a manner that as little water as possible is allowed to go into or out of the specimen. To achieve this requirement, a very stiff electronic pressure transducer or null-indicating device must be used. With an electronic pressure transducer the pore-water pressure is read directly. With a null-indicating device a pressure control is continuously adjusted to maintain a constant level of the water/mercury interface in the capillary bore of the device. The pressure required to prevent movement of the water is equal to the pore-water pressure. Both measuring devices shall have a compliance of all the assembled parts of the pore-water pressure-measurement system relative to the total volume of the specimen, satisfying the following requirement:

$$(\Delta V/V)/\Delta u < 3.2 \times 10^{-6}\ \text{m}^2 / \text{kN}\ (2.2 \times 10^{-5}\ \text{in.}^2 / \text{lb}) \tag{1}$$

where:

ΔV = change in volume of the pore-water measurement system due to a pore pressure change, mm^3(in.^{3}),

V = total volume of the specimen, mm^3(in.^{3}), and

Δu = change in pore pressure, kPa (lb/in.^{2}).

NOTE 6—To meet the compliance requirement, tubing between the specimen and the measuring device should be short and thick-walled with small bores. Thermoplastic, copper, and stainless steel tubing have been used successfully.

5.9 *Volume Change Measurement Device*— The volume of water entering or leaving the specimen shall be measured with an accuracy of within ±0.05 % of the total volume of the specimen. The volume measuring device is usually a burette connected to the back pressure but may be any other device meeting the accuracy requirement. The device must be able to withstand the maximum back pressure.

5.10 *Deformation Indicator*—The vertical deformation of the specimen is usually determined from the travel of the piston acting on the top of the specimen. The piston travel shall be measured with an accuracy of at least 0.25 % of the initial specimen height. The deformation indicator shall have a range of at least 15 % of the initial height of the specimen and may be a dial indicator, linear variable differential transformer (LVDT), extensiometer, or other measuring device meeting the requirements for accuracy and range.

5.11 *Specimen Cap and Base*—The specimen cap and base shall be designed to provide drainage from both ends of the specimen. They shall be constructed of a rigid, noncorrosive, impermeable material, and each shall, except for the drainage provision, have a circular plane surface of contact with the porous disks and a circular cross section. It is desirable for the mass of the specimen cap and top porous disk to be as minimal as possible. However, the mass may be as much as 10 % of the axial load at failure. If the mass is greater than 0.5 % of the applied axial load at failure and greater than 50 g (0.1 lb), the axial load must be corrected for the mass of the specimen cap and top porous disk. The diameter of the cap and base shall be equal to the initial diameter of the specimen. The specimen base shall be connected to the triaxial compression chamber to prevent lateral motion or tilting, and the specimen cap shall be designed such that eccentricity of the piston-to-cap contact relative to the vertical axis of the specimen does not exceed 1.3 mm (0.05 in.). The end of the piston and specimen cap contact area shall be designed so that tilting of the specimen cap during the test is minimal. The cylindrical surface of the specimen base and cap that contacts the membrane to form a seal shall be smooth and free of scratches.

5.12 *Porous Discs*—Two rigid porous disks shall be used to provide drainage at the ends of the specimen. The coefficient of permeability of the disks shall be approximately equal to that of fine sand (1×10^{-4} cm/s (4×10^{-5} in./s)). The disks shall be regularly cleaned by ultrasonic or boiling and brushing and checked to determine whether they have become clogged.

5.13 *Filter-Paper Strips and Disks*— Filter-paper strips are used by many laboratories to decrease the time required for testing. Filter-paper disks of a diameter equal to that of the specimen may be placed between the porous disks and specimen to avoid clogging of the porous disks. If filter strips or disks are used, they shall be of a type that does not dissolve in water. The coefficient of permeability of the filter paper shall not be less than 1×10^{-5} cm/s (4×10^{-6} cm/s) for a normal pressure of 550 kPa (80 lb/in.^{2}). To avoid hoop tension, filter strips should cover no more than 50 % of the specimen periphery. Filter-strip cages have been successfully used by many laboratories. An equation for correcting the principal stress difference (deviator stress) for the effect of the strength of vertical filter strips is given in 10.4.3.1.

NOTE 7—Whatman's No. 54 Filter Paper has been found to meet the permeability and durability requirements.

5.14 *Rubber Membrane*—The rubber membrane used to encase the specimen shall provide reliable protection against leakage. Membranes shall be carefully inspected prior to use and if any flaws or pinholes are evident, the membrane shall be discarded. To offer minimum restraint to the specimen, the unstretched membrane diameter shall be between 90 and 95 % of that of the specimen. The membrane thickness shall not exceed 1 % of the diameter of the specimen. The membrane shall be sealed to the specimen cap and base with rubber O-rings for which the unstressed inside diameter is between 75 and 85 % of the diameter of the cap and base, or by other means that will provide a positive seal. An equation for correcting the principal stress difference (deviator stress) for the effect of the stiffness of the membrane is given in 10.4.3.2.

5.15 *Valves*—Changes in volume due to opening and closing valves may result in inaccurate volume change and pore-water pressure measurements. For this reason, valves in the specimen drainage system shall be of the type that produce minimum volume changes due to their operation. A valve may be assumed to produce minimum volume change if opening or closing the valve in a closed, saturated pore-water pressure system does not induce a pressure change of greater than 0.7 kPa (±0.1 lb/in.^{2}). All valves must be capable of withstanding applied pressures without leakage.

NOTE 8—Ball valves have been found to provide minimum volume-change characteristics; however, any other type of valve having suitable volume-change characteristics may be used.

5.16 *Specimen-Size Measurement Devices*— Devices used to determine the height and diameter of the specimen shall measure the respective dimensions to within ±0.1 % of the total dimension and shall be constructed such that their use will not disturb the specimen.

NOTE 9—Circumferential measuring tapes are recommended over calipers for measuring the diameter.

5.17 *Recorders*—Specimen behavior may be recorded manually or by electronic digital or analog recorders. If electronic recorders are used, it shall be necessary to calibrate the measuring devices through the recorder using known input standards.

5.18 *Sample Extruder*—The sample extruder shall be capable of extruding the soil core from the sampling tube at a uniform rate in the same direction of travel as the sample entered the tube and with minimum disturbance of the sample. If the soil core is not extruded vertically, care should be taken to avoid bending stresses on the core due to gravity. Conditions at the time of sample removal may dictate the direction of removal, but the principal concern is to minimize the degree of disturbance.

5.19 *Timer*—A timing device indicating the elapsed testing time to the nearest 1 s shall be used to obtain consolidation data (8.3.3).

5.20 *Balance*—A balance or scale conforming to the requirements of Specification D4753 readable (with no estimate) to 0.1 % of the test mass or better.

5.21 *Water Deaeration Device*—The amount of dissolved gas (air) in the water used to saturate the specimen shall be decreased by boiling, by heating and spraying into a vacuum, or by any other method that will satisfy the requirement for saturating the specimen within the limits imposed by the available maximum back pressure and time to perform the test.

5.22 *Testing Environment*—The consolidation and shear portion of the test shall be performed in an environment where temperature fluctuations are less than ±4°C (±7.2°F) and there is no direct contact with sunlight.

5.23 *Miscellaneous Apparatus*—Specimen trimming and carving tools including a wire saw, steel straightedge, miter box, vertical trimming lathe, apparatus for preparing compacted specimens, membrane and O-ring expander, water content cans, and data sheets shall be provided as required.

6. Test Specimen Preparation

6.1 *Specimen Size*—Specimens shall be cylindrical and have a minimum diameter of 33 mm (1.3 in.). The average height-to-average diameter ratio shall be between 2 and 2.5. An individual measurement of height or diameter shall not vary from average by more than 5 %. The largest particle size shall be smaller than ⅙ the specimen diameter. If, after completion of a test, it is found based on visual observation that oversize particles are present, indicate this information in the report of test data (11.2.23).

NOTE 10—If oversize particles are found in the specimen after testing, a particle-size analysis may be performed on the tested specimen in accordance with Test Method D422 to confirm the visual observation and the results provided with the test report (11.2.4).

6.2 *Undisturbed Specimens*—Prepare undisturbed specimens from large undisturbed samples or from samples secured in accordance with Practice D1587 or other acceptable undisturbed tube sampling procedures. Samples shall be preserved and transported in accordance with the practices for Group C samples in Practices D4220. Specimens obtained by tube sampling may be tested without trimming except for cutting the end surfaces plane and perpendicular to the longitudinal axis of the specimen, provided soil characteristics are such that no significant disturbance results from sampling. Handle specimens carefully to minimize disturbance, changes in cross section, or change in water content. If compression or any type of noticeable disturbance would be caused by the extrusion device, split the sample tube lengthwise or cut the tube in suitable sections to facilitate removal of the specimen with minimum disturbance. Prepare trimmed specimens, in an environment such as a controlled high-humidity room where soil water content change is minimized. Where removal of pebbles or crumbling resulting from trimming causes voids on the surface of the specimen, carefully fill the voids with remolded soil obtained from the trimmings. If the sample can be trimmed with minimal disturbance, a vertical trimming lathe may be used to reduce the specimen to the required diameter. After obtaining the required diameter, place the specimen in a miter box, and cut the specimen to the final height with a wire saw or other suitable device. Trim the surfaces with the steel straightedge. Perform one or more water content determinations on material trimmed from the specimen in accordance with Test Method D2216. Determine the mass and dimensions of the specimen using the devices described in 5.16 and 5.20. A minimum of three height measurements (120° apart) and at least three diameter measurements at the quarter points of the height shall be made to determine the average height and diameter of the specimen.

6.3 *Compacted Specimens*—Soil required for compacted specimens shall be thoroughly mixed with sufficient water to produce the desired water content. If water is added to the soil, store the material in a covered container for at least 16 h prior to compaction. Compacted specimens may be prepared by compacting material in at least six layers using a split mold of circular cross section having dimensions meeting the requirements enumerated in 6.1. Specimens may be compacted to the desired density by either: (*1*) kneading or tamping each layer until the accumulative mass of the soil placed in the mold is compacted to a known volume; or (*2*) by adjusting the number of layers, the number of tamps per layer, and the force per tamp. The top of each layer shall be scarified prior to the addition of material for the next layer. The tamper used to compact the material shall have a diameter equal to or less than ½ the diameter of the mold. After a specimen is formed, with the ends perpendicular to the longitudinal axis, remove the mold and determine the mass and dimensions of the specimen using the devices described in 5.16 and 5.20. Perform one or more water content determinations on excess material used to prepare the specimen in accordance with Test Method D2216.

NOTE 11—It is common for the unit weight of the specimen after removal from the mold to be less than the value based on the volume of the mold. This occurs as a result of the specimen swelling after removal of the lateral confinement due to the mold.

7. Mounting Specimen

7.1 *Preparations*—Before mounting the specimen in the triaxial chamber, make the following preparations:

7.1.1 Inspect the rubber membrane for flaws, pinholes, and leaks.

7.1.2 Place the membrane on the membrane expander or, if it is to be rolled onto the specimen, roll the membrane on the cap or base.

7.1.3 Check that the porous disks and specimen drainage tubes are not obstructed by passing air or water through the appropriate lines.

7.1.4 Attach the pressure-control and volume-measurement system and a pore-pressure measurement device to the chamber base.

7.2 Depending on whether the saturation portion of the test will be initiated with either a wet or dry drainage system, mount the specimen using the appropriate method, as follows in either 7.2.1 or 7.2.2. The dry mounting method is strongly recommended for specimens with initial saturation less than 90 %. The dry mounting method removes air prior to adding backpressure and lowers the backpressure needed to attain an adequate percent saturation.

NOTE 12—It is recommended that the dry mounting method be used for specimens of soils that swell appreciably when in contact with water. If the wet mounting method is used for such soils, it will be necessary to obtain the specimen dimensions after the specimen has been mounted. In such cases, it will be necessary to determine the double thickness of the membrane, the double thickness of the wet filter paper strips (if used), and the combined height of the cap, base, and porous disks (including the thickness of filter disks if they are used) so that the appropriate values may be subtracted from the measurements.

7.2.1 *Wet Mounting Method*:

7.2.1.1 Fill the specimen drainage lines and the pore-water pressure measurement device with deaired water.

7.2.1.2 Saturate the porous disks by boiling them in water for at least 10 min and allow to cool to room temperature.

7.2.1.3 If filter-paper disks are to be placed between the porous disks and specimen, saturate the paper with water prior to placement.

7.2.1.4 Place a saturated porous disk on the specimen base and wipe away all free water on the disk. If filter-paper disks are used, placed on the porous disk. Place the specimen on the disk. Next, place another filter-paper disk (if used), porous disk and the specimen cap on top of the specimen. Check that the specimen cap, specimen, filter-paper disks (if used) and porous disks are centered on the specimen base.

7.2.1.5 If filter-paper strips or a filter-paper cage are to be used, saturate the paper with water prior to placing it on the specimen. To avoid hoop tension, do not cover more than 50 % of the specimen periphery with vertical strips of filter paper.

7.2.1.6 Proceed with 7.3.

7.2.2 *Dry Mounting Method*:

7.2.2.1 Dry the specimen drainage system. This may be accomplished by allowing dry air to flow through the system prior to mounting the specimen.

7.2.2.2 Dry the porous disks in an oven and then place the disks in a desiccator to cool to room temperature prior to mounting the specimen.

7.2.2.3 Place a dry porous disk on the specimen base and place the specimen on the disk. Next, place a dry porous disk and the specimen cap on the specimen. Check that the specimen cap, porous disks, and specimen are centered on the specimen base.

NOTE 13—If desired, dry filter-paper disks may be placed between the porous disks and specimen.

7.2.2.4 If filter-paper strips or a filter-paper cage are to be used, the cage or strips may be held in place by small pieces of tape at the top and bottom.

7.3 Place the rubber membrane around the specimen and seal it at the cap and base with two rubber O-rings or other positive seal at each end. A thin coating of silicon grease on the vertical surfaces of the cap and base will aid in sealing the membrane. If filter-paper strips or a filter-paper cage are used, do not apply grease to surfaces in contact with the filter-paper.

7.4 Attach the top drainage line and check the alignment of the specimen and the specimen cap. If the dry mounting method has been used, apply a partial vacuum of approximately 35 kPa (5 lb/in.^{2}) (not to exceed the consolidation stress) to the specimen through the top drainage line prior to checking the alignment. If there is any eccentricity, release the partial vacuum, realign the specimen and cap, and then reapply the partial vacuum. If the wet mounting method has been used, the alignment of the specimen and the specimen cap may be checked and adjusted without the use of a partial vacuum.

8. Procedure

8.1 *Prior to Saturation*—After assembling the triaxial chamber, perform the following operations:

8.1.1 Bring the axial load piston into contact with the specimen cap several times to permit proper seating and alignment of the piston with the cap. During this procedure, take care not to apply an axial load to the specimen exceeding 0.5 % of the estimated axial load at failure. When the piston is brought into contact, record the reading of the deformation indicator to three significant digits.

8.1.2 Fill the chamber with the chamber liquid, being careful to avoid trapping air or leaving an air space in the chamber.

8.2 *Saturation*—The objective of the saturation phase of the test is to fill all voids in the specimen with water without undesirable prestressing of the specimen or allowing the specimen to swell. Saturation is usually accomplished by applying back pressure to the specimen pore water to drive air into solution after saturating the system by either: (*1*) applying vacuum to the specimen and dry drainage system (lines, porous disks, pore-pressure device, filter-strips or cage, and disks) and then allowing deaired water to flow through the system and specimen while maintaining the vacuum; or (*2*) saturating the drainage system by boiling the porous disks in water and allowing water to flow through the system prior to mounting the specimen. It should be noted that placing the air into solution is a function of both time and pressure. Accordingly, removing as much air as possible prior to applying back pressure will decrease the amount of air that will have to be placed into solution and will also decrease the back pressure required for saturation. In addition, air remaining in the specimen and drainage system just prior to applying back pressure will go into solution much more readily if deaired water is used for saturation. The use of deaired water will also decrease the time and back pressure required for saturation. Many procedures have been developed to accomplish saturation. The following are suggested procedures:

8.2.1 *Starting with Initially Dry Drainage System*—Increase the partial vacuum acting on top of the specimen to the maximum available vacuum. If the effective consolidation stress under which the strength is to be determined is less than the maximum partial vacuum, apply a lower partial vacuum to the chamber. The difference between the partial vacuum applied to the specimen and the chamber should never exceed the effective consolidation stress for the test and should not be less than 35 kPa (5 lb/in.^{2}) to allow for flow through the sample. After approximately 10 min, allow deaired water to percolate from the bottom to the top of the specimen under a differential vacuum of less than 20 kPa (3 lb/in.^{2}) (Note 14).

8.2.1.1 There should always be a positive effective stress of at least 13 kPa (2 lb/in.^{2}) at the bottom of the specimen during this part of the procedure. When water appears in the burette connected to the top of the specimen, close the valve to the bottom of the specimen and fill the burette with deaired water. Next, reduce the vacuum acting on top of the specimen through the burette to atmospheric pressure while simultaneously increasing the chamber pressure by an equal amount. This process should be performed slowly such that the difference between the pore pressure measured at the bottom of the specimen and the pressure at the top of the specimen should be allowed to equalize. When the pore pressure at the bottom of the specimen stabilizes, proceed with back pressuring of the specimen pore-water as described in . To check for equalization, close the drainage valves to the specimen and measure the pore pressure change until stable. If the change is less than 5 % of the chamber pressure, the pore pressure may be assumed to be stabilized.

NOTE 14—For saturated clays, percolation may not be necessary and water can be added simultaneously at both top and bottom.

8.2.2 *Starting with Initially Saturated Drainage System*—After filling the burette connected to the top of the specimen with deaired water, apply a chamber pressure of 35 kPa (5 lb/in.^{2}) or less and open the specimen drainage valves. When the pore pressure at the bottom of the specimen stabilizes, according to the method described in 8.2.1, or when the burette reading stabilizes, back pressuring of the specimen pore-water may be initiated.

8.2.3 *Back-Presuure Saturation*—To saturate the specimen, back pressuring is usually necessary. Fig. 3 [3] provides guidance on back pressure required to attain saturation. Additional guidance on the back-pressure process is given by Black[4] and Lee.[5]

8.2.3.1 *Applying Back Pressure*—Simultaneously increase the chamber and back pressure in steps with specimen drainage valves opened so that deaired water from the burette connected to the top and bottom of the specimen may flow into the specimen. To avoid undesirable prestressing of the specimen while applying back pressure, the pressures must be applied incrementally with adequate time between increments to permit equalization of pore-water pressure throughout the specimen. The size of each increment may range from 35 kPa (5 lb/in.^{2}) up to 140 kPa (20 lb/in. ^{2}), depending on the magnitude of the desired effective consolidation stress, and the percent saturation of the specimen just prior to the addition of the increment. The difference between the chamber pressure and the back pressure during back pressuring should not exceed 35 kPa unless it is deemed necessary to control swelling of the specimen during the procedure. The difference between the chamber and back pressure must also remain within ±5 % when the pressures are raised and within± 2 % when the

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[3] Lowe, J., and Johnson, T. C., "Use of Back Pressure to Increase Degree of Saturation of Triaxial Test Specimens," *Proceedings, ASCE Research Conference on Shear Strength of Cohesive Soils* , Boulder, CO, 1960

[4] Black, A. W. and Lee, K. L. (1973), "Saturating Laboratory Samples by Back Pressure," *Journal of the Soil Mechanics and Foundation Division,* ASCE, Vol. 99, No. SM1, Proc. Paper 9484, Jan., pp. 75–93.

[5] Head, K. H., (1986), *Manual of Soil Laboratory Testing, Volume 3: Effective Stress Tests*, Pentech Press Limited, Graham Lodge, London, United Kingdom, pp. 787–796.

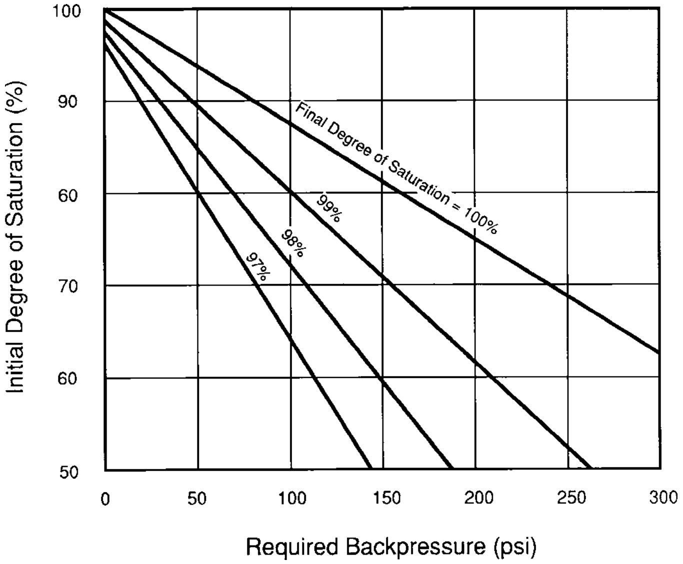

FIG. 3 Pressure to Attain Various Degrees of Saturation

pressures are constant. To check for equalization after application of a back pressure increment or after the full value of back pressure has been applied, close the specimen drainage valves and measure the change in pore-pressure over a 1-min interval. If the change in pore pressure is less than 5 % of the difference between the chamber pressure and the back pressure, another back pressure increment may be added or a measurement may be taken of the pore pressure Parameter B (see 8.2.4) to determine if saturation is completed. Specimens shall be considered to be saturated if the value of *B* is equal to or greater than 0.95, or if *B* remains unchanged with addition of back pressure increments.

NOTE 15—The relationships presented in Fig. 4 are based on the assumption that the water used for back pressuring is deaired and that the only source for air to dissolve into the water is air from the test specimen. If air pressure is used to control the back pressure, pressurized air will dissolve into the water, thus reducing the capacity of the water used for back pressure to dissolve air located in the pores of the test specimen. The problem is minimized by using a long (>5 m) tube that is impermeable to air between the air-water interface and test specimen, by separating the back-pressure water from the air by a material or fluid that is relatively impermeable to air, by periodically replacing the back-pressure water with deaired water, or by other means.

NOTE 16—Although the pore pressure Parameter *B* is used to determine adequate saturation, the *B*-value is also a function of soil stiffness. If the saturation of the sample is 100 %, the *B*-value measurement will increase with decreasing soil stiffness. Therefore, when testing soft soil samples, a *B*-value of 95 % may indicate a saturation less than 100 %.

NOTE 17—The back pressure required to saturate a compacted specimen may be higher for the wet mounting method than for the dry mounting method and may be as high as 1400 kPa (200 lb/in.^{2}).

NOTE 18—Many laboratories use differential pressure regulators and transducers to achieve the requirements for small differences between chamber and back pressure.

8.2.4 *Measurement of the Pore Pressure Parameter B*—Determine the value of the pore pressure Parameter *B* in accordance with 8.2.4.1 through 8.2.4.4. The pore pressure Parameter *B* is defined by the following equation:

$$B = \Delta u / \Delta \sigma_3 \tag{2}$$

where:

Δu = change in the specimen pore pressure that occurs as a result of a change in the chamber pressure when the specimen drainage valves are closed, and

$\Delta \sigma_3$ = change in the chamber pressure.

8.2.4.1 Close the specimen drainage valves, record the pore pressure, to the nearest 0.7 kPa (0.1 psi), and increase the chamber pressure by 70 kPa (10 lb/in.^{2}).

8.2.4.2 After approximately 2 min, determine and record the maximum value of the induced pore pressure to the nearest 0.7 kPa (0.1 psi),. For many specimens, the pore pressure may decrease after the immediate response and then increase slightly with time. If this occurs, values of Δu should be plotted with time and the asymptotic pore pressure used as the change in pore pressure. A large increase in Δu with time or values of Δu greater than $\Delta \sigma_3$ indicate a leak of chamber fluid into the specimen. Decreasing values of Δu with time may indicate a leak in that part of the pore pressure measurement system located outside of the chamber.

8.2.4.3 Calculate the *B*-value using Eq 2.

8.2.4.4 Reapply the same effective consolidation stress as existed prior to the *B*-value by reducing the chamber pressure by 70 kPa (10 lb/in.^{2}) or by alternatively, increasing the back pressure by 70 kPa. If *B* is continuing to increase with increasing back pressure, continue with back pressure saturation. If *B* is equal to or greater than 0.95 or if a plot of *B* versus back pressure indicates no further increase in *B* with increasing back pressure, initiate consolidation.

8.3 *Consolidation*—The objective of the consolidation phase of the test is to allow the specimen to reach equilibrium in a drained state at the effective consolidation stress for which a strength determination is required. During consolidation, data is obtained for use in determining when consolidation is complete and for computing a rate of strain to be used for the shear portion of the test. The consolidation procedure is as follows:

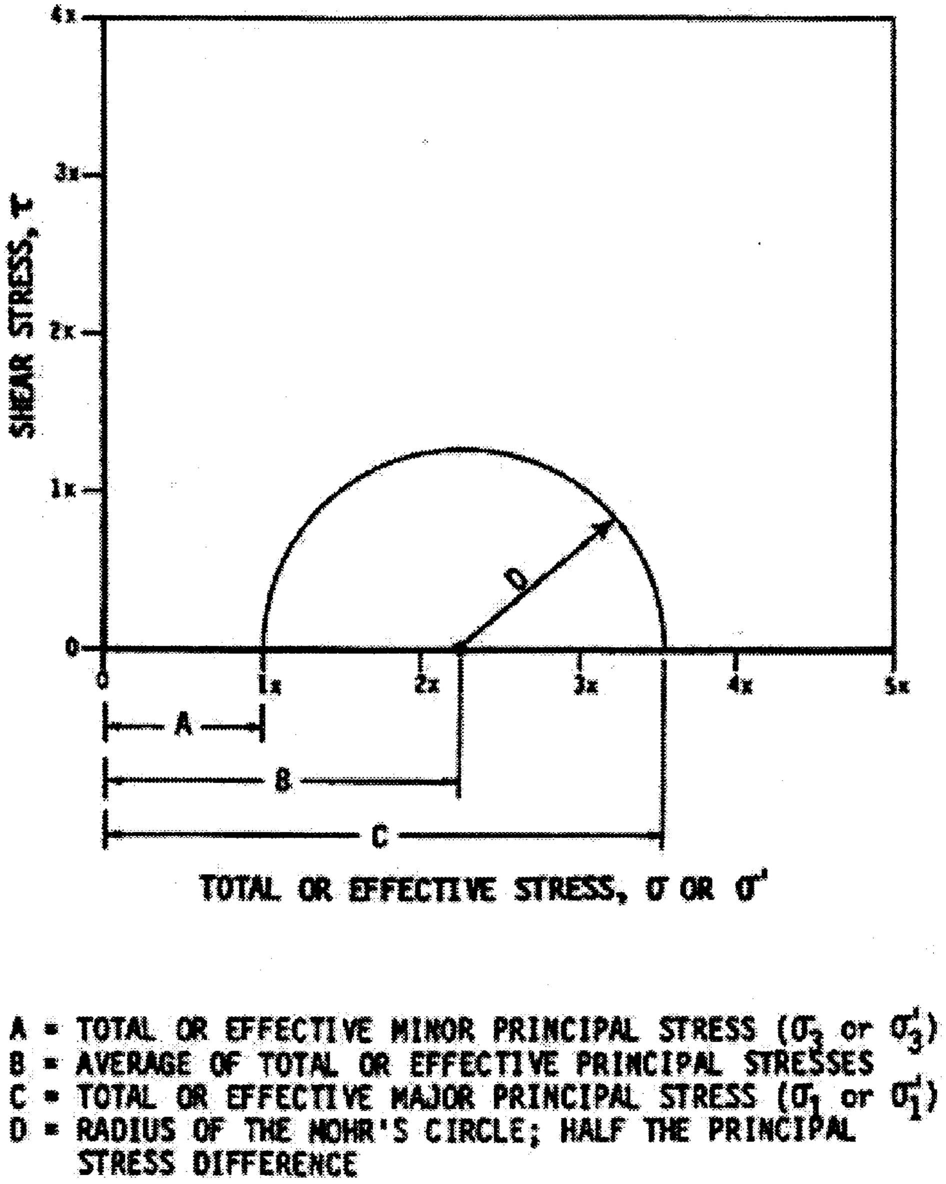

FIG. 4 Construction of Mohr Stress Circle

8.3.1 When the saturation phase of the test is completed, bring the axial load piston into contact with the specimen cap, and record the reading on the deformation indicator to three significant digits. During this procedure, take care not to apply an axial load to the specimen exceeding 0.5 % of the estimated axial load at failure. After recording the reading, raise the piston a small distance above the specimen cap, and lock the piston in place.

8.3.2 With the specimen drainage valves closed, hold the maximum back pressure constant and increase the chamber pressure until the difference between the chamber pressure and the back pressure equals the desired effective consolidation pressure. Consolidation in stages is required when filter strips for radial drainage are used, and the load increment ratio shall not exceed two.

8.3.3 Obtain an initial burette reading, and, then, open appropriate drainage valves so that the specimen may drain from both ends into the burette. At increasing intervals of elapsed time (0.1, 0.2, 0.5, 1, 2, 4, 8, 15, and 30 min and at 1, 2, 4, and 8 h, and so forth) observe and record the burette readings, and, after the 15-min reading, record the accompanying deformation indicator readings obtained by carefully bringing the piston in contact with the specimen cap. If burette and deformation indicator readings are to be plotted against the square root of time, the time intervals at which readings are taken may be adjusted to those that have easily obtained square roots, for example, 0.09, 0.25, 0.49, 1, 4, and 9 min, and so forth. Depending on soil type, time intervals may be changed to convenient time intervals which allow for adequate definition of volume change versus time.

NOTE 19—In cases where significant amounts of fines may be washed from the specimen because of high initial hydraulic gradients, it is permissible to gradually increase the chamber pressure to the total desired pressure over a period with the drainage valves open. If this is done, recording of data should begin immediately after the total pressure is reached.

8.3.4 Plot the burette and deformation indicator readings versus either the logarithm or square root of elapsed time. Allow consolidation to continue for at least one log cycle of time or one overnight period after 100 % primary consolidation has been achieved as determined in accordance with one of the procedures outlined in Test Method D2435. A marked deviation between the slopes of the burette and deformation indicator curves toward the end of consolidation based on deformation indicator readings indicates leakage of fluid from the chamber into the specimen, and the test shall be terminated.

8.3.5 Determine the time for 50 % primary consolidation, t_{50}, in accordance with one of the procedures outlined in Test Method D2435.

8.4 *Shear*—During shear, the chamber pressure shall be kept constant while advancing the axial load piston downward against the specimen cap using controlled axial strain as the loading criterion. Specimen drainage is not permitted during shear.

8.4.1 *Prior to Axial Loading*—Before initiating shear, perform the following:

8.4.1.1 By opening or closing the appropriate valves, isolate the specimen so that during shear the specimen pore-water pressure will be measured by the pore-pressure measurement device and no drainage will occur.

8.4.1.2 Place the chamber in position in the axial loading device. Be careful to align the axial loading device, the axial load-measuring device, and the triaxial chamber to prevent the application of a lateral force to the piston during shear.

8.4.1.3 Bring the axial load piston into contact with the specimen cap to permit proper seating and realignment of the piston with the cap. During this procedure, care should be taken not to apply an axial load to the specimen exceeding 0.5 % of the estimated axial load at failure. If the axial load-measuring device is located outside of the triaxial chamber, the chamber pressure will produce an upward force on the piston that will react against the axial loading device. In this case, start shear with the piston slightly above the specimen cap, and before the piston comes into contact with the specimen cap, either (*1*) measure and record the initial piston friction and upward thrust of the piston produced by the chamber pressure and later correct the measured axial load, or (*2*) adjust the axial load-measuring device to compensate for the friction and thrust. The variation in the axial load-measuring device reading should not exceed 0.1 % of the estimated failure load when the piston is moving downward prior to contacting the specimen cap. If the axial load-measuring device is located inside the chamber, it will not be necessary to correct or compensate for the uplift force acting on the axial loading device or for piston friction. However, if an internal load-measuring device of significant flexibility is used in combination with an external deformation indicator, correction of the deformation readings may be necessary. In both cases, record the initial reading on the pore-water pressure measurement device to the nearest 0.7 kPa (0.1 psi) immediately prior to when the piston contacts the specimen cap and the reading on the deformation indicator to three significant digits when the piston contacts the specimen cap.

8.4.1.4 Check for pore pressure stabilization. Record the pore pressure to the nearest 0.7 kPa (0.1 psi). Close the drainage valves to the specimen, and measure the pore pressure change until stable. If the change is less than 5 % of the chamber pressure, the pore pressure may be assumed to be stabilized.

8.4.2 *Axial Loading*—Apply axial load to the specimen using a rate of axial strain that will produce approximate equalization of pore pressures throughout the specimen at failure. Assuming failure will occur after 4 %, a suitable rate of strain, $'\varepsilon$, may be determined from the following equation:

$$'\varepsilon = 4\ \%/(10\ t_{50}) \tag{3}$$

where:

t_{50} = time value obtained in 8.3.5.

If, however, it is estimated that failure will occur at a strain value lower than 4 %, a suitable strain rate may be determined using Eq 3 by replacing 4 % with the estimated failure strain. This rate of strain will provide for determination of accurate effective stress paths in the range necessary to define effective strength envelopes.

8.4.2.1 At a minimum, record load and deformation to three significant digits, and pore-water pressure values to the nearest 0.7 kPa (0.1 psi), at increments of 0.1 to 1 % strain and, thereafter, at every 1 %. Take sufficient readings to define the stress-strain curve; hence, more frequent readings may be required in the early stages of the test and as failure is approached. Continue the loading to 15 % strain, except loading may be stopped when the principal stress difference (deviator stress) has dropped 20 % or when 5 % additional axial strain occurs after a peak in principal stress difference (deviator stress).

NOTE 20—The use of a manually adjusted null-indicating device will require nearly continuous attention to ensure the criterion for undrained shear.

9. Removing Specimen

9.1 When shear is completed, perform the following:

9.1.1 Remove the axial load and reduce the chamber and back pressures to zero.

9.1.2 With the specimen drainage valves remaining closed, quickly remove the specimen from the apparatus so that the specimen will not have time to absorb water from the porous disks.

9.1.3 Remove the rubber membrane (and the filter-paper strips or cage from the specimen if they were used), and determine the water content of the total specimen in accordance with the procedure in Test Method D2216. (Free water remaining on the specimen after removal of the membrane should be blotted away before obtaining the water content.) In cases where there is insufficient material from trimmings for index property tests, that is, where specimens have the same diameter as the sampling tube, the specimen should be weighed prior to removing material for index property tests and a representative portion of the specimen used to determine its final water content. Prior to placing the specimen (or portion thereof) in the oven to dry, sketch or photograph the specimen showing the mode of failure (shear plane, bulging, and so forth).

10. Calculation

10.1 Measurements and calculations shall contain three significant digits.

10.2 *Initial Specimen Properties*—Using the dry mass of the total specimen, calculate and record the initial water content, volume of solids, initial void ratio, initial percent saturation, and initial dry unit weight. Calculate the specimen volume from values measured in 6.2 or 6.3. Calculate the volume of solids by dividing the dry mass of the specimen by the specific gravity of the solids (Note 20) and dividing by the density of water. Calculate the void ratio by dividing the volume of voids by the volume of solids where the volume of voids is assumed to be the difference between the specimen volume and the volume of the solids. Calculate dry density by dividing the dry mass of the specimen by the specimen volume.

NOTE 21—The specific gravity of solids can be determined in accordance with Test Method D854 or it may be assumed based on previous test results.

10.3 *Specimen Properties After Consolidation*—Calculate the specimen height and area after consolidation as follows:

10.3.1 Height of specimen after consolidation, H_c, is determined from the following equation:

$$H_c = H_o - \Delta H_o \tag{4}$$

where:
H_o = initial height of specimen, and
ΔH_o = change in height of specimen at end of consolidation.

See Fig. 4.

10.3.2 The cross-sectional area of the specimen after consolidation, A_c, shall be computed using one of the following methods. The choice of the method to be used depends on whether shear data are to be computed as the test is performed (in which case Method A would be used) or on which of the two methods, in the opinion of a qualified person, yield specimen conditions considered to be most representative of those after consolidation. Alternatively, the average of the two calculated areas may be appropriate.

10.3.2.1 *Method A*:

$$A_c = (V_o - \Delta V_{sat} - \Delta V_c) / H_c \tag{5}$$

where:
V_o = initial volume of specimen,
ΔV_c = change in volume of specimen during consolidation as indicated by burette readings, and
ΔV_{sat} = change in volume of specimen during saturation as follows:
ΔV_{sat} = $3V_o[\Delta H_s/H_o]$

where:
ΔH_s = change in height of the specimen during saturation.

10.3.2.2 *Method B*:

$$Ac = (V_{wf} + V_s) / H_c \tag{6}$$

where:
V_{wf} = final volume of water (based on final water content), and
V_s = volume of solids as follows:
V_s = $w_s/(G_s p_w)$

where:
w_s = specimen dry mass,
G_s = specific gravity of solids, and
p_w = density of water.

10.3.3 Using the calculated dimensions of the specimen after consolidation, and assuming that the water content after consolidation is the same as the final water content, calculate the consolidated void ratio and percent saturation.

NOTE 22—The specimen will absorb water from the porous disks and drainage lines during the time it is being removed from the apparatus. When this effect is significant, Method A will yield more reasonable values.

NOTE 23— In this test method, the equations are written such that compression and consolidation are considered positive.

10.4 *Shear Data*:

10.4.1 Calculate the axial strain, ε_1, for a given applied axial load as follows:

$$\varepsilon_1 = \Delta H / H_c \tag{7}$$

where:
ΔH = change in height of specimen during loading as determined from deformation indicator readings, and
H_c = height of specimen after consolidation.

10.4.2 Calculate the cross-sectional area, A, for a given applied axial load as follows:

$$A = A_c / (1 - \varepsilon_1) \tag{8}$$

where:
A_c = average cross-sectional area of the specimen after consolidation, and
ε_1 = axial strain for the given axial load.

NOTE 24—The cross-sectional area computed in this manner is based on the assumption that the specimen deforms as a right circular cylinder during shear. In cases where there is localized bulging, it may be possible to determine more accurate values for the area based on specimen dimension measurements obtained after shear.

10.4.3 Calculate the principal stress difference (deviator stress), $\sigma_1 - \sigma_3$, for a given applied axial load as follows:

$$\sigma_1 - \sigma_3 = P / A \tag{9}$$

where:
P = given applied axial load (corrected for uplift and piston friction if required as obtained in 8.4.1.3), and
A = corresponding cross-sectional area.

10.4.3.1 *Correction for Filter-Paper Strips*— For vertical filter-paper strips which extend over the total length of the specimen, apply a filter-paper strip correction to the computed values of the principal stress difference (deviator stress), if the error in principal stress difference (deviator stress) due to the strength of the filter-paper strips exceeds 5 %.

(*1*) For values of axial strain above 2 %, use the following equation to compute the correction:

$$\Delta(\sigma_1 - \sigma_3) = K_{fp}\, P_{fp} / A_c \tag{10}$$

where:

$\Delta(\sigma_1 - \sigma_3)$ = correction to be subtracted from the measured principal stress difference (deviator stress),
K_{fp} = load carried by filter-paper strips per unit length of perimeter covered by filter-paper,
P_{fp} = perimeter covered by filter-paper, and
A_c = cross-sectional area of specimen after consolidation.

(*2*) For values of axial strain of 2 % or less, use the following equation to compute the correction:

$$\Delta(\sigma_1 - \sigma_3) = 50\varepsilon_1 K_{fp} P_{fp} / A_c \tag{11}$$

where:

ε_1 = axial strain (decimal form) and other terms are the same as those defined in Subparagraph (*1*) of 10.4.3.1.

NOTE 25—For filter-paper generally used in triaxial testing, K_{fp} is approximately 0.19 kN/m (1.1 lb/in.).

10.4.3.2 *Correction for Rubber Membrane*— Use the following equation to correct the principal stress difference (deviator stress) for the effect of the rubber membrane if the error in principal stress difference (deviator stress) due to the strength of the membrane exceeds 5 %:

$$\Delta(\sigma_1 - \sigma_3) = (4E_m t_m \varepsilon) / D_c \tag{12}$$

where:

$\Delta(\sigma_1 - \sigma_3)$ = correction to be subtracted from the measured principal stress difference (deviator stress),
D_c = $\sqrt{4A_c/\pi}$ = diameter of specimen after consolidation,
E_m = Young's modulus for the membrane material,
t_m = thickness of the membrane, and
ε_1 = axial strain (decimal form).

(*1*) The Young's modulus of the membrane material may be determined by hanging a 15-mm (0.5-in.) circumferential strip of membrane using a thin rod, placing another rod through the bottom of the hanging membrane, and measuring the force per unit strain obtained by stretching the membrane. The modulus value may be computed using the following equation:

$$E_m = (F / A_m) / (\Delta L / L) \tag{13}$$

where:

E_m = Young's modulus of the membrane material,
F = force applied to stretch the membrane,
L = unstretched length of the membrane,
ΔL = change in length of the membrane due to the force, F, and
A_m = area of the membrane = $2\, t_m W_s$

where:

t_m = thickness of the membrane, and
W_s = width of circumferential strip, 0.5 in. (15 mm).

NOTE 26—A typical value of E_m for latex membranes is 1400 kPa (200 lb/in.).

NOTE 27—The corrections for filter-paper strips and membranes are based on simplified assumptions concerning their behavior during shear. Their actual behavior is complex, and there is not a consensus on more exact corrections.

10.4.4 Calculate the effective minor principal stress, σ'_3 for a given applied axial load as follows:

$$\sigma'_3 = \sigma_3 - \Delta u \tag{14}$$

where:

σ_3 = effective consolidation stress, and
Δu = induced pore-water pressure at the given axial load (total pore-water pressure minus the total back pressure).

10.5 *Principal Stress Difference (Deviator Stress) and Induced Pore-Water Pressure versus Strain Curves*—Prepare graphs showing relationships between principal stress difference (deviator stress) and induced pore-water pressure with axial strain, plotting deviator stress and induced pore-water pressure as ordinates and axial strain as abscissa. Select the principal stress difference (deviator stress) and axial strain at failure in accordance with 3.2.3.

10.6 *$p' - q$ Diagram*— Prepare a graph showing the relationship between p', $(\sigma'_1+\sigma'_3)/2$ and q, $(\sigma_1-\sigma_3)/2$, plotting q as ordinate and p' as abscissa using the same scale. The value of p' for a given axial load may be computed as follows:

$$p' = ((\sigma_1 - \sigma_3) + 2\sigma'_3) / 2 \tag{15}$$

where:

$\sigma_1 - \sigma_3$ = principal stress difference (deviator stress), and
σ'_3 = effective minor principal stress.

10.7 Determine the major and minor principal stresses at failure based on total stresses, σ_{1f} and σ_{3f} respectively, and on effective stresses, σ'_{1f} and σ'_{3f} respectively, as follows:

$$\sigma_{3f} = \text{effective consolidation stress,} \tag{16}$$

$$\sigma_{1f} = (\sigma_1 - \sigma_3)\ \text{at failure}\ + \sigma_{3f}, \tag{17}$$

$$\sigma'_3 f = \sigma_{3f} - \Delta u_f\text{, and} \tag{18}$$

$$\sigma'_{1f} = (\sigma_1 - \sigma_3)\ \text{at failure} + \sigma'_{3f} \tag{19}$$

where Δu_f is the induced pore-water pressure at failure.

10.8 *Mohr Stress Circles*—If desired, construct Mohr stress circles at failure based on total and effective stresses on an arithmetic plot with shear stress as ordinate and normal stress as abscissa using the same scales. The circle based on total stresses is drawn with a radius of one half the principal stress difference (deviator stress) at failure with its center at a value equal to one half the sum of the major and minor total principal stresses. The Mohr stress circle based on effective stresses is drawn in a similar manner except that its center is at a value equal to one half the sum of the major and minor effective principal stresses.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the data sheet(s)/form(s), as given below, is covered in 7.2.1.3.

11.2 Record as a minimum the following general information (data):

11.2.1 Identification data and visual description of specimen, including soil classification and whether the specimen is undisturbed, compacted, or otherwise prepared, 11.2.2 Values of plastic limit and liquid limit, if determined in accordance with Test Method D4318,

11.2.3 Value of specific gravity of solids and notation if the value was determined in accordance with Test Method D854 or assumed,

11.2.4 Particle-size analysis, if determined in accordance with Test Method D422,

11.2.5 Initial specimen dry unit weight, void ratio, water content, and percent saturation, (specify if the water content specimen was obtained from cuttings or the entire specimen),

NOTE 28—The specific gravity determined in accordance with Test Method D854 is required for calculation of the saturation. An assumed specific gravity may be used provided it is noted in the test report that an assumed value was used.

11.2.6 Initial height and diameter of specimen,

11.2.7 Method followed for specimen saturation (that is, dry or wet method),

11.2.8 Total back pressure,

11.2.9 The pore pressure Parameter B at the end of saturation,

11.2.10 Effective consolidation stress,

11.2.11 Time to 50 % primary consolidation,

11.2.12 Specimen dry unit weight, void ratio, water content, and percent saturation after consolidation,

11.2.13 Specimen cross-sectional area after consolidation and method used for determination,

11.2.14 Failure criterion used,

11.2.15 The value of the principal stress difference (deviator stress) at failure and the values of the effective minor and major principal stresses at failure, (indicate when values have been corrected for effects due to membrane or filter strips, or both),

11.2.16 Axial strain at failure, percent,

11.2.17 Rate of strain, percent per minute,

11.2.18 Principal stress difference (deviator stress) and induced pore-water pressure versus axial strain curves as described in 10.5,

11.2.19 The $p' - q$ diagram as described in 10.6,

11.2.20 Mohr stress circles based on total and effective stresses, (optional),

11.2.21 Slope of angle of the failure surface (optional),

11.2.22 Failure sketch or photograph of the specimen, and

11.2.23 Remarks and notations regarding any unusual conditions such as slickensides, stratification, shells, pebbles, roots, and so forth, or other information necessary to properly interpret the results obtained, including any departures from the procedure outlined.

12. Precision and Bias

12.1 *Precision*—Test data on precision is not presented due to the nature of the soil materials tested by this procedure. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Subcommittee D18.05 is seeking any data from users of this test method that might be used to make a limited statement on precision.

12.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

13. Keywords

13.1 back pressure saturation; cohesive soil; consolidated undrained strength; strain-controlled loading; stress-strain relationships; total and effective stresses

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes made to this standard since the last edition (2002) that may impact the use of this standard.

(1) The cap connection was changed to be a requirement of the chamber equipment in 5.4, rather than a requirement specific to the baseplate.

(2) Pressure/volume controller were added as acceptable vacuum control devices in 5.6.

(3) A requirement was added for isolating air/water interfaces (if used) from the pressure systems in 5.6.

(4) Note 15 was made 7.2.1.3, making wetting of filter paper disks mandatory when using the wet mounting method.

(5) In section 8.2.3, references concerning back pressure saturation were provided. An associated figure was added as Figure 3. subsequent sections, notes, and figures were renumbered.

(6) Footnotes 3, 4, and 5 were added.

ANSI/ANS-16.1-2003

American Nuclear Society

REAFFIRMED
August 4, 2008
ANSI/ANS-16.1-2003 (R2008)

measurement of the leachability of solidified low-level radioactive wastes by a short-term test procedure

an American National Standard

This standard has been reviewed and reaffirmed by the ANS Nuclear Facilities Standards Committee (NFSC) with the recognition that it may reference other standards and documents that may have been superceded or withdrawn. The requirements of this document will be met by using the version of the standards and documents referenced herein. It is the responsibility of the user to review each of the references and to determine whether the use of the original references or more recent versions is appropriate for the facility. Variations from the standards and documents referenced in this standard should be evaluated and documented. This standard does not necessarily reflect recent industry initiatives for risk informed decision-making or a graded approach to quality assurance. Users should consider the use of these industry initiatives in the application of this standard.

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American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure

Secretariat
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American National Standard

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Foreword

(This Foreword is not part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003.)

The characteristics of radioactive wastes from the nuclear industry are dependent on many diverse factors, most of which do not lend themselves to simple definition and standardization. In this standard, low-level wastes are considered to be those radioactive wastes that are defined as low-level in Title 10, *Code of Federal Regulations*, Part 61, "Licensing Requirements for Land Disposal of Radioactive Waste" (2003). In general, Section 61.2 of Part 61 defines low-level wastes as those containing source, special nuclear, or by-product materials that are not classified as high-level radioactive waste, transuranic waste, spent nuclear fuel, or uranium or thorium tailings and waste. Resistance to leaching of radionuclides is not specifically mentioned in Part 61 nor is containment of radionuclides called out as an express requirement for low-level radioactive waste packages. Minimization of contact of waste by water is a fundamental concern of Part 61, however, as evidenced by the statement in Section 61.7 that ". . . a cornerstone of the system is stability . . . so that . . . **access of water to the waste can be minimized**. Migration of radionuclides is thus minimized . . ." (bold emphasis added).

In addition, there are several statements in Section 61.57 that address minimization of contact of water with waste. These statements are in recognition of the fact that contact of waste with water is an initial step in a potentially major pathway for radionuclide release and migration off-site. "Leaching," or the release of radionuclides from a waste form through contact with water, is thus a major factor in the subsequent migration of the radionuclides from the waste, through groundwater, and off the site. It follows, therefore, that leaching is a phenomenon that is of fundamental interest in low-level radioactive waste disposal and that the measurement of the leach resistance of potential waste forms is important in low-level waste management.

Low-level radioactive waste accrues in the form of combustible, noncombustible, compactible, and noncompactible solids (cloth, metal, paper, wood), liquids (evaporator bottoms, decontamination solutions), slurries (filter sludges, ion-exchange resins), and powders (incinerator ash, salts). The ANS-16.1 standard was designed principally for one type of low-level radioactive waste: low-level, non-self-heating, radioactive fluids (liquids, slurries, and free-flowing powders). However, it can be used to measure the leach resistance of any waste solidified into a well-defined geometric shape.

An accepted method for managing these liquids, slurries, and powders is solidification, packaging, and subsequent shipment for disposal by shallow-land burial. Solidification can restrict dispersal during handling and transportation and can provide a nonchanging volume during the residence time of the waste in the burial trench.

At present, generators of low-level radioactive wastes (e.g., nuclear power plants, laboratories, and hospitals) need a common basis for evaluating the alternatives for packaging, handling, storing, and shipping their radioactive wastes. Vendors of solidification systems need a common basis for evaluating the leachability of the waste forms made by their solidification processes. Burial ground operators need leaching information to improve the efficiency of their handling, disposal, and site maintenance operations. The 5-day test provides a measure of leach resistance performance that can be done in the field in a reasonable time period to provide confidence to generators, vendors, and operators that the material intended for shallow burial meets minimum requirements for leach resistance (for example, the U.S. Nuclear Regulatory Commission requires a minimum leachability index of 6.0 for radioisotopes).

Leaching, which can occur when water contacts a solidified waste form, is an important mechanism for the dispersal of radioactivity. Leach testing has thus been recognized as a primary technique for the evaluation and comparison of solidified waste forms.1),2) Even so, the situation remains complex for several reasons:

(1) leaching can proceed by several concurrent mechanisms such as diffusion, dissolution, and erosion, the relative importance of which can change with time, and temperature, substances dissolved in the water, matrix material, the radionuclides of interest, pH, and other variables;

(2) the actual leaching conditions that a solidified waste form will encounter during its sound life (i.e., the time during which the waste form meets the specifications for all applicable parameters) are imprecisely known, with postulated conditions varying widely;

(3) investigators of waste forms have tended to use leach testing procedures unique to their own studies, which makes comparisons difficult.

As a first step toward rectifying the last situation, the International Atomic Energy Agency (IAEA) published a suggested standard leach test in 1971.1) This suggested test met with consent in principle but was not put into practice. Instead, much of the leach testing being performed used procedures described as "modified" IAEA tests. The "modifications" were unique to individual laboratories, so that standardization and comparability of results was still lacking. The test presented in the ANSI/ANS-16.1-2003 standard has much in common with the original IAEA test.

Working Group ANS-16.1 of the Standards Committee of the American Nuclear Society had the following membership at the time it approved this standard:

R. D. Spence, Chair, *Oak Ridge National Laboratory*

O. U. Anders, *Individual*
H. W. Godbee, *Individual*
A. Icenhour, *Oak Ridge National Laboratory*
R. M. Neilson, *Idaho National Engineering and Environmental Laboratory*

This standard was processed and approved for submittal to ANSI by the American Nuclear Society's Nuclear Facilities Standards Committee (NFSC) on ANSI/ANS-16.1-2003, "Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure." Committee approval of the standard does not necessarily imply that all committee members voted for its

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1)"Leach Testing of Immobilized Radioactive Waste Solids, A Proposal for a Standard Method," E. D. HESPE, Ed., International Atomic Energy Agency, *At. Energy Rev.*, **9**, 1 (1971).

2) "Long-Term Leach Testing of Solidified Radioactive Waste Forms," ISO 6961-1982(E), International Organization for Standardization.

approval. At the time it approved this standard, the NFSC committee had the following members:

D. J. Spellman, Chair, *Oak Ridge National Laboratory*
S. Ahmad, Standards Administrator, *American Nuclear Society*

C. K. Brown, *Southern Nuclear Operating Company*
R. H. Bryan, Jr., *Tennessee Valley Authority*
H. Chander, *U.S. Department of Energy*
M. T. Cross, *Westinghouse Electric Company*
J. Dewes, *Westinghouse Savannah River Site*
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R. A. Hill, *GE Nuclear Energy*
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M. J. Wright, *Entergy Operations*

Contents

Annexes

Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure

1 Scope, Purpose, and Application

1.1 Scope

This standard, ANSI/ANS-16.1-2003,[1] provides a uniform procedure to measure and index the release of radionuclides from waste forms as a result of leaching in demineralized water for 5 days.[2] The results of this procedure do not apply to any specific environmental situation except through correlative studies of actual disposal site conditions. The test presented in this standard has much in common with the original International Atomic Energy Agency proposal and has by now become familiar to those working in the radioactive waste-form development field. It contains the provisions published in the original version of this standard in 1986.

1.2 Purpose

The quantification of the leaching characteristics of solidified wastes requires a standardized, practical method to measure the ability of the solids to impede the release of radioisotopes when water comes into contact with them. The purpose of this standard is to establish such a test, define a material parameter, and provide a mathematical procedure for calculating a "Leachability Index" value for the test data collected over the time period of the test.

This standard is intended to serve as a basis for indexing radionuclide release from solidified low-level radioactive waste forms in a short-term (5-day) test under controlled conditions in a well-defined leachant. It is not intended to serve as a definition of the long-term (several hundred to thousands of years) leaching behavior of these forms at conditions representing actual disposal conditions.

Under actual leaching conditions, mechanisms other than diffusion (e.g., chemical reaction, surface layers and films, cracking, etc.) are important considerations. Also, the interplay of retardation mechanisms (filtration, ion exchange, coprecipitation, etc.) and enhancement mechanisms (chelation, desorption, dissolution, etc.) for radionuclide migration are important considerations.

1.3 Application

The mechanisms involved in leaching can differ from one type of material to another, from one leachant to another, and from one set of leaching conditions to another. However, if they are known through generic studies, predictions for the release of radioactivity as a function of time can be made. In spite of the differences in materials, leachants, and conditions, a procedure applicable to all products of low-level radioactive waste solidification processes can be devised for purposes of quantitative assessment. The test set forth in this standard is short-term, simple, and emphasizes reproducible conditions that can be readily achieved. The essential test parameters are fixed in detail.

The test consists of a procedure in which the leachant is replaced at designated intervals to generate seven data points over 5 days and ten data points if the test is extended to 90 days. The procedure permits the accumulation of sufficient data in a reasonably short time for quantitative assessment purposes. In its "extended" form, the laboratory leaching can be extended

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[1] ANSI/ANS-16.1-2003 is hereafter referred to as "this standard."

[2] Periods of time with the units of days or hours are spelled out since the letters "d" or "h" are used to represent other quantities.

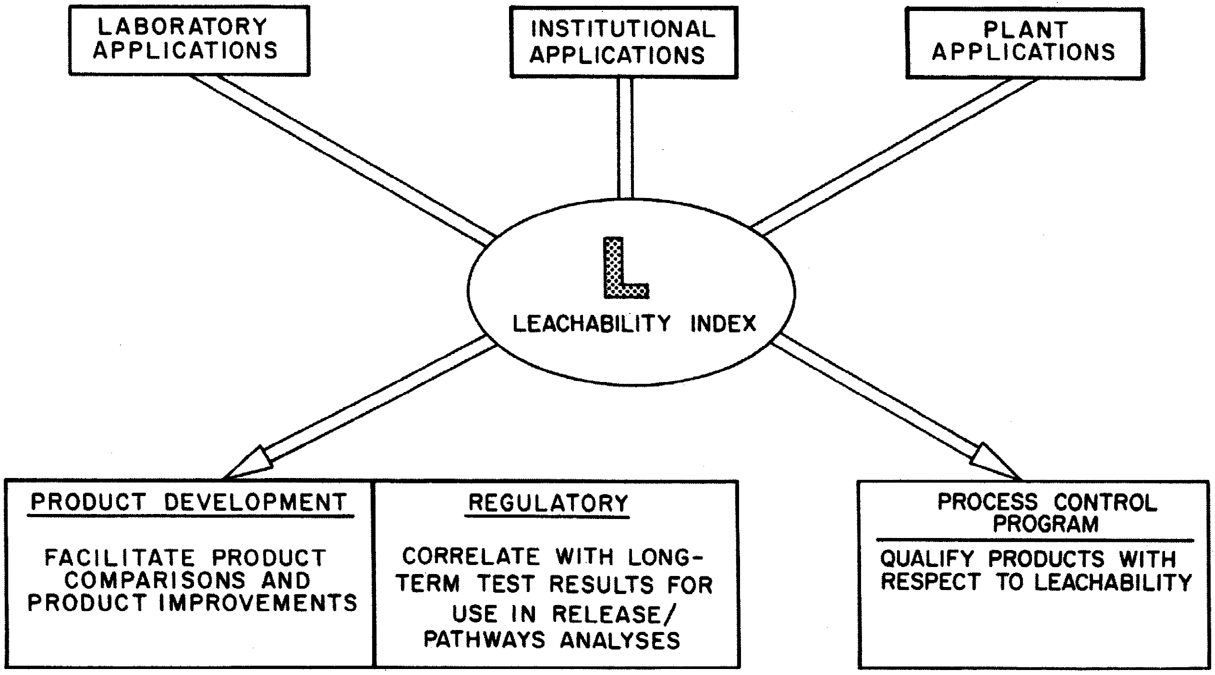

Figure 1.

to 3 months as part of the overall related generic studies.[3]

The data obtained by the procedure of this standard are expressed as a material parameter of the leachability of each leached species, called the "Leachability Index" (L). Some of the anticipated uses for the Leachability Index are presented in Fig. 1.

2 Glossary of Terms

aliquot: A known fractional part of a defined quantity.

binder: Solidification agent.

cumulative fraction leached: The sum of the fractions leached during all previous leaching intervals, plus the fraction leached during the last leaching interval, using the initial amount of the species of interest present in the specimen as unity (100%) and assuming no radioactive decay.

fraction leached: A portion of a constituent of a specimen or waste form that has been released from the specimen or waste form during the leaching process, using the quantity present initially as unity (100%).

leachability: A rate constant (or a combination of several rate constants) that describes the leaching of a nuclide from a material under a given set of conditions.

Leachability Index: An index value related to the leaching characteristics of solidified waste materials as measured by the leach test defined in this standard. In this standard, the Leachability Index has an exact theoretical meaning only for homogeneous, chemically inert materials, for which bulk diffusion is the predominant rate-determining process during leaching.

leachate: Leachant after use.

leachant: The liquid that contacts the specimen during the course of a leaching test or contacts a solid waste form at a disposal site.

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[3] See Annexes B and E.

leaching interval: The length of time during which a given volume of leachant is in contact with a specimen or solid waste form.

leaching rate/leach rate: The amount of a constituent of the specimen or solid waste form that is leached during one time unit (e.g., g/day or μCi/s). It is frequently expressed per unit of exposed surface area [e.g., g cm^{-2} (day)$^{-1}$].

leach test specimen/leach specimen: The solid body that is immersed into the leachant during the leach test. This body must be representative of the solid that is formed by the combination of waste with a solidification agent.

leach test/leaching test: Procedure to be followed for the determination of the Leachability Index.

may: Denotes permission, neither a requirement nor a recommendation.

packaging: Any material or structure covering the surface of a waste such as a plastic bag, drum, concrete cask, etc., but exclusive of a coating or surface treatment.

remaining fraction: Unity minus cumulative fraction leached (i.e., the fraction still remaining with the specimen or waste form after leaching) assuming no decay of the radioactivity.

residual liquid: Free liquid present in the specimen container at the time the specimen is removed from the container.

semi-infinite medium: A body of which the outer boundary is considered to be effectively at an infinite distance from the inner region.

shall: Denotes a requirement.

should: Denotes a recommendation.

wash-off: Liquid containing the mobile surface contamination removed from the specimen by immersing it in demineralized water for 30 s.

3 Test Procedure

Many different testing methods with various leachant compositions, leachant-renewal frequencies, and test conditions (e.g., temperatures and pressures) have been used in the past to determine the amount of a radionuclide leached from a solidified waste form as a function of time[4] [1–6][5]. Since these factors interact to influence the leach test results, this standard's test specifies a defined leachant, a set leachant-renewal schedule, a fixed leachant temperature, and other specified test conditions. The procedure can be extended to other leachants[6] and different leachant-renewal frequencies (as well as other temperatures and test conditions) so that it more nearly represents anticipated conditions under which a solidified waste form may be stored, transported, or disposed[6] but are not part of this standard.

3.1 Specimen Preparation

The method for preparing proper test specimens is specific for each waste type and solidification process. A specific procedure by which specimens that meet the following specifications can be prepared shall be developed for each given type of solid under consideration.

Precautions shall be taken to ensure that the specimen is representative of the solidified waste (see "Glossary of Terms," Section 2) and that the homogeneity of the test specimen is the same as that of the material in the actual solidified waste form. While a small specimen is desirable to limit the radiation field, it shall not be so small that it compromises the specimen homogeneity, requires unattainable analytical sensitivities, or provides substantial difficulties for specimen preparation. The test specimen shall be prepared in the same or similar manner as that established for the solidification process. If specimens are obtained by core drilling of waste forms (which should be avoided), the procedure shall be described, and the specimen shall have surfaces representative of the actual waste form. Using cast samples rather than samples core drilled from a cast monolith is recommended because the pro-

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[4] See Annex D.

[5] Numbers in brackets refer to corresponding numbers in Section 4, "References."

[6] See Annex E.

cess of cutting a sample from a monolith alters the surface of the waste form and may possibly result in microcracks in a sample obtained in this manner. In some cases, core drilling is the only way to accomplish the desired testing, but the possible alterations in the sample matrix structure should be kept in mind.

In addition to being representative of the solidified waste, the specimen shall have a well-defined shape, mass, and volume. The surface condition of the specimen should be representative of the surface condition of the actual full-scale waste product. Where possible, the specimen shall be a monolithic cylinder, parallelepiped, or sphere, the dimensions of which are reported. Cylinders shall have a length-to-diameter ratio in the range of 0.2 to 5. Parallelepipeds shall have a length-to-minimum-thickness ratio in the range of 0.2 to 5. The test specimen geometry should be cylindrical, if possible. The minimum specimen dimension recommended should be 1 cm, unless a need to employ a smaller specimen (e.g., to minimize personnel radiation exposure) is demonstrated.

The representative specimen of the product of a given solidification process shall be prepared, or cast, so that the casting conforms to the sides of the specimen preparation container (to provide a smooth surface), voids within it are eliminated, and homogeneity (as uniform as the character of the material permits) is attained. For glasslike or thermosetting mixtures, this container should be heated to provide a thermal history representative of that which the actual solidified waste form undergoes. The thermal history of the test specimen shall be reported. Immediately after preparation the specimen shall be placed into the specimen container, which may be the same as the specimen preparation container.

The specimen container shall remain sealed during the storage period between preparation and leach testing. The specimen container(s) shall be constructed of material(s) known to be chemically unreactive toward the specimen (e.g., polyethylene, polypropylene, stainless steel, ceramic, and glass). No single container material appears to be superior for all solidified waste products.

3.2 Leach Test Vessel

The vessel in which leaching takes place should be constructed of an “unreactive” material. A material is considered unreactive if

(1) it does not react chemically with the leachant or the specimen;

(2) it does not sorb chemical species extracted from the specimen or those in the leachant itself. This requirement applies to the species of interest extracted from the specimen during leaching. It also applies to those major species extracted during leaching, which influence the composition of the leachant. Sorption shall be determined by a blank test run. A leach vessel made of a material that is sorbent toward the extracted species of interest may be used, provided that either the extent of sorption is small ($<5\%$ of the incremental fraction leached) or the sorbed species are removed from the container and analyzed at the same frequency as the leachate is sampled and replaced;

(3) it does not release interfering species that alter the composition of the leachant during the leaching process;

(4) it can withstand the conditions involved in leaching.

The leach test vessel shall be constructed so that excessive evaporation of the leachant ($>2\%$ over 24 hours) is prevented. The dimensions of the vessel shall permit the entire external geometric surface area ($>98\%$) of the immersed specimen to be in contact with the leachant during the entire leaching interval. The dimensions of the leach test vessel shall also be sufficient to hold the leachant while leaving some free volume for convenience in manipulation of both specimen and leachant.

3.3 Leachant

The leachate shall be sampled and entirely replaced at designated time intervals. The leachant shall be demineralized water with an electrical conductivity of <5 μmho/cm at 25°C and a total organic carbon content (TOC) of <3 ppm. The temperature of the leachant shall be maintained between 17.5 and 27.5°C during the test.

3.4 Leach Test Method

After removal from the specimen container and prior to the initiation of the leach test, the test specimen shall be rinsed by immersion in demineralized water for 30 s. The rinse water volume shall be the same as the required leachant volume (described below).

The container used to store the specimen before leaching shall then be rinsed with an amount of water equal to its volume, to recover any radioactivity present in residual liquid or retained on the container walls. This "container rinse" and the specimen rinse water ("wash-off") are then to be combined and analyzed to determine the quantity present of each radionuclide of interest. The latter are expressed as fractions $(a_r/A_O)_i$, defined as the ratio of activity of the radionuclide i that is present in the combined rinse $(a_r)_i$ to the activity of the same radionuclide in the specimen $(A_o)_i$ at the time it is immersed in the first portion of the leachant (i.e., the beginning of the first leaching interval). This fraction shall be reported for each radionuclide of interest. All radioactivities measured shall be recorded for a common reference time, for example, the beginning of the first leaching interval.

The specimen shall be supported in the leachant by any convenient device, made from unreactive material (as defined above), that does not interfere with the leachate removal and replacement; does not impede leaching; does not damage the surface of the specimen; and, as mentioned above, does not preclude more than a small fraction of the specimen's external surface ($<2\%$) from exposure to the leachant. Examples of suitable specimen supports include wires for suspension, rigid support stands, or coarse-weave wire-mesh baskets. The specimen shall be located within the leachant so that it is surrounded on all sides by a leachant layer.

Sufficient leachant shall be used so that the ratio of the leachant volume, V_L, and the specimen external geometric surface area, S, is maintained within fixed bounds during the leaching interval as given by

$$\frac{V_L\ (\mathrm{cm}^3)}{S(\mathrm{cm}^2)} = 10 \pm 0.2\ (\mathrm{cm}) \ .$$

For example, a 1-cm-diameter, 1-cm-long right circular cylinder (surface area 4.71 cm^2) would need 47.1 ml of leachant. A ratio of leachant volume to specimen external geometric surface area of 10 cm is usually sufficient to minimize leachant-composition changes during reasonably short leaching intervals, while providing a sufficient concentration of extracted species for analysis. In cases where the minimum leachant layer requirement and the leachant volume to specimen surface area ratio requirement cannot be met concurrently, the latter requirement takes precedence, and all specimen surfaces shall be as uniformly covered with leachant as practicality permits. The leachant shall not be stirred during the leaching interval.

At the end of each leaching interval, the leachate shall be removed from the specimen. Quickly removing the specimen from the used leachant and placing it into fresh leachant in a new leach test vessel is an acceptable and widely employed procedure. After leachate removal, the leach test vessel shall be rinsed with demineralized water to remove residual leachate and contained radioactivity. The specimen may also be momentarily rinsed (<5 s) in demineralized water after leachate removal but before leachant renewal. The radioactivity in these rinses shall be measured and included with that of the leachate just removed. During leachant renewal the specimen should be exposed to the air for as short a time as reasonably achievable. In no case shall its surface be allowed to dry completely. The specimen shall then be contacted with fresh leachant solution for the specified time of the next leaching interval.

The rate of radioactivity release, and hence the calculated Leachability Index value, can be a function of the leachant-renewal frequency. A standardized uniform leachant-renewal schedule is thus required. The leachate shall be sampled, and the leachate completely replaced after cumulative leach times of 2, 7, and 24 hours from the initiation of the test, as illustrated in Fig. 2. Subsequent leachate sampling and leachant replacements shall be made at 24-hour intervals for the next 4 days, for a total of 5 days for the standard test. In the "extended" option, three additional leach intervals of 14, 28, and 43 days each extend the entire test to 90 (5 + 14 + 28 + 43) days, as illustrated in Fig. 2. As noted on Fig. 2, the leachant change for the optional last three renewal periods of the test may

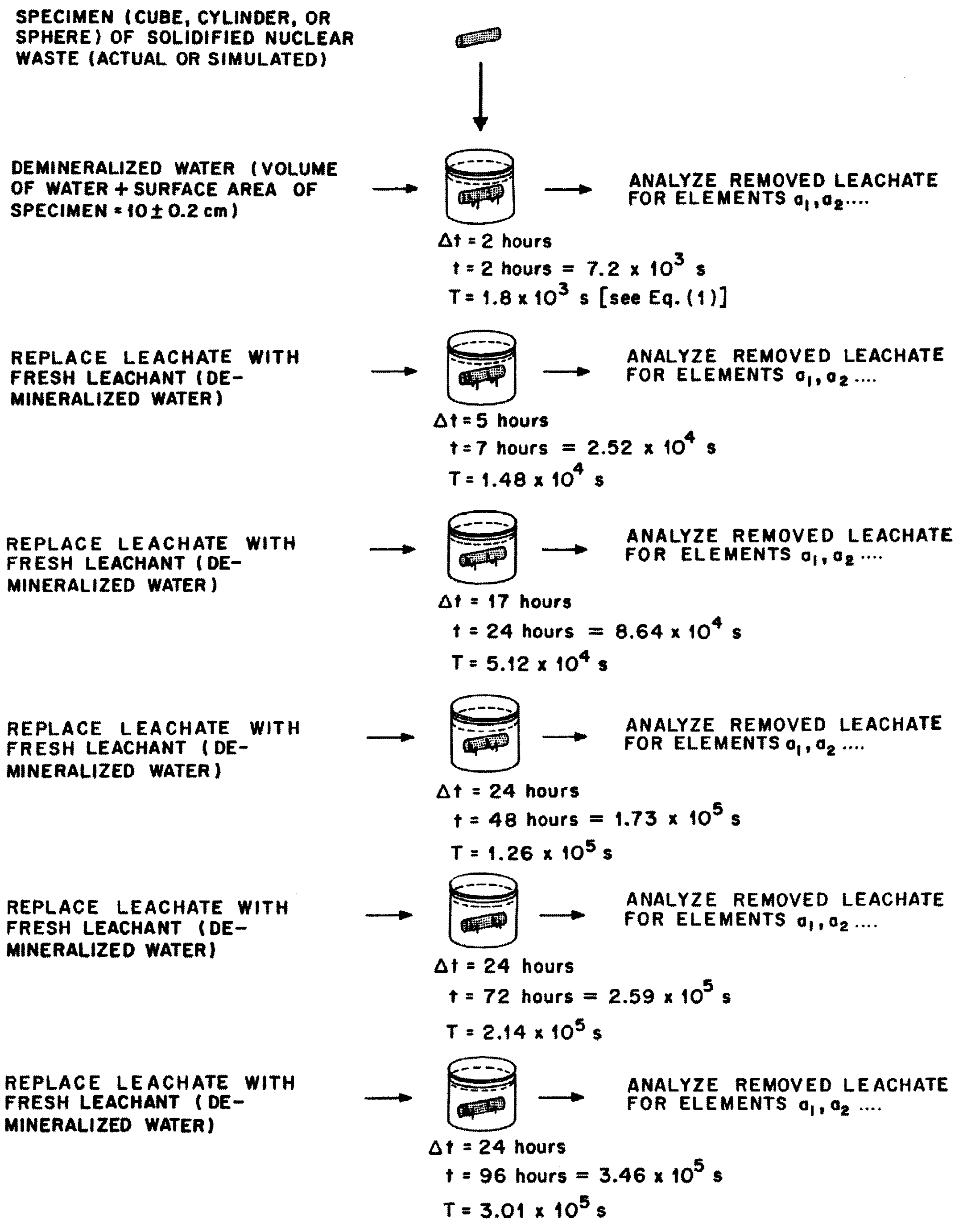

Figure 2.—Continues on next page.

be shifted by 1 day. This permits a leachant change scheduled on Saturday (or Sunday) to be shifted to Friday (or Monday) for these last three optional renewal periods. The reasoning behind this is that with the protracted leachant renewal periods, the boundary conditions of the model[7] are not satisfied and a day under or a day over will not make any significant differ-

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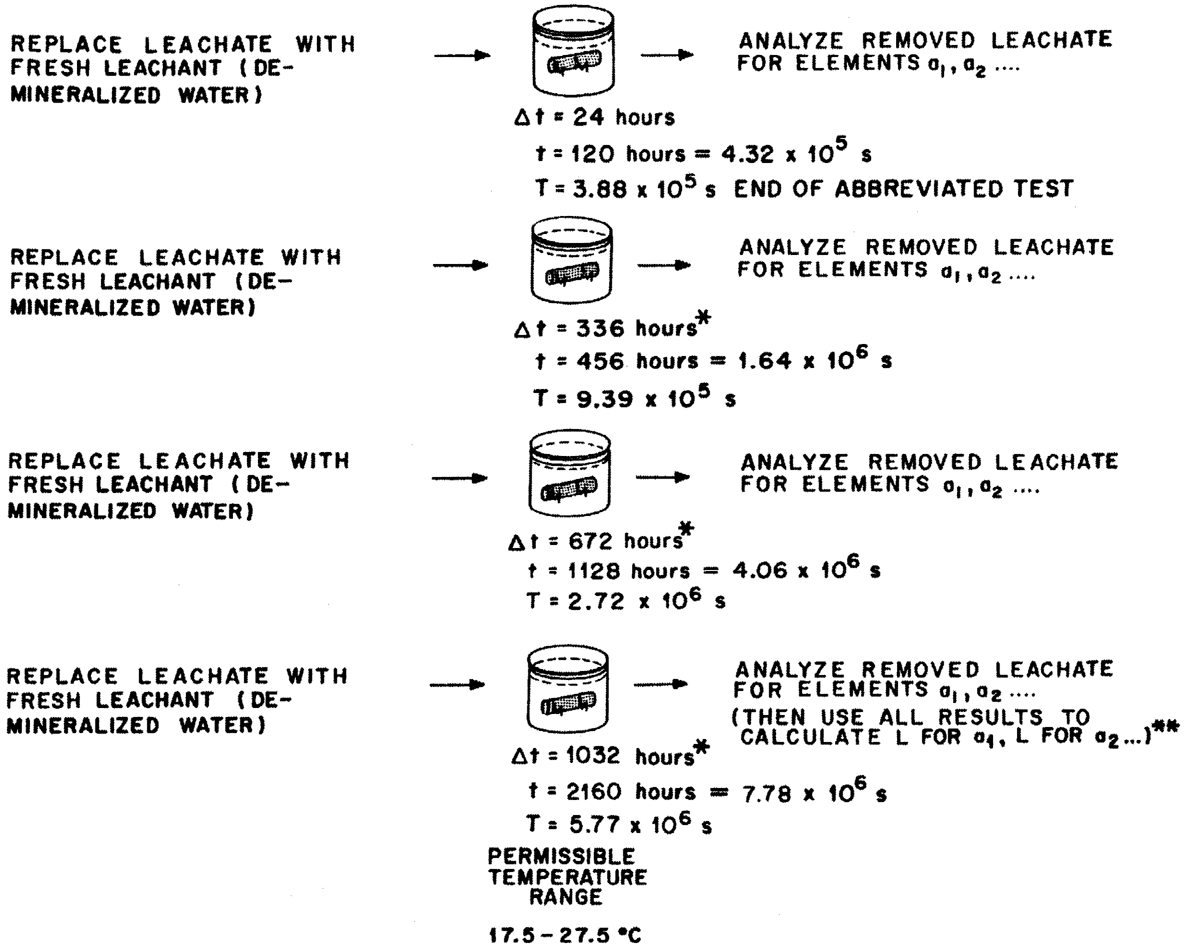
[7] See Annex B.



*THE LONGER LEACHING INTERVALS OF 2, 4, AND 6 WEEKS MAY VARY BY ±1 DAY.

**L = LEACHABILITY INDEX. A HIGH L FOR a_1 INDICATES GOOD RESISTANCE TO LEACHING OF ELEMENT a_1.

Figure 2.—Continued.

ence in the results after such extended time periods. This standard requires that the leachant changeout time be short and insignificant relative to the duration of a leaching interval.

For purposes of extended leaching times or evaluating the effects of longer immersions, a longer-time "extended test" is defined by the same sequence, but with the three additional longer leaching intervals. This extended test requires 90 days of testing.

3.5 Leachate Analysis

An aliquot of the leachate shall be taken at the end of each leaching interval to determine, by a suitable method, the amounts $(a_n)_i$ of the species of interest present in the leachate volume. Any generally accepted state-of-the-art analytical procedure may be employed. The release of the species of interest will always be determined by measuring the quantity present in the leachate rather than the residual in the specimen.

Leachate aliquots taken for analysis shall be representative of the leachate from which they are taken. The intent is to provide aliquots that will permit a determination of all the radioactivity that has been removed from the test specimen during the leaching interval. This includes any radioactivity associated with particulate solids in the leachate. Stirring of the leachate to suspend particulate solids prior to taking the leachate aliquot or dissolution of such solids by the addition of chemicals to the leachate before sampling may be performed, if standard. If precipitation occurs in the leachant during the leaching interval, the amount of the extracted species of interest associated with the precipitate shall be determined and added to the amount of the dissolved species. Under no circumstances shall the leachate be filtered and the filter media with the residue be discarded without analysis.

4 Presentation and Analysis of Data

The following experimental information[8] shall be recorded:

(1) the type of waste and the proportion (by weight and volume) of waste and binder incorporated into the waste-binder mixture prior to solidification;

(2) the type and composition of the immobilizing material (binder), including additives. Commercial names shall be used;

(3) the radionuclides and/or the nonradioactive tracers and the total amount of each in the leaching specimen both as prepared $(A_p)_i$ and after the 30-s rinse $(A_o)_i$.

In the following description, the subscripts, i, are omitted to simplify the presentation. The value of A_o can be obtained by subtracting the contaminant present in the combined initial rinse (a_r) described in 2.4, from that in the specimen as prepared (namely, $A_o = A_p - a_r$). The values of A_p and a_r are corrected for radioactive decay and reported as of a reference time (e.g., the time representing the first exposure of the specimen to the leachant, namely, the beginning of the first leaching interval). The amount of contamination (radionuclide or stable nuclide) may be reported in units of microcuries (μCi) or disintegrations per second (Bq) or mass [e.g., micrograms (μg)]. It may also be reported as a fraction of the quantity in a reference source of the same contaminant;

(4) any departures from the specified methods of specimen preparation;

(5) shape, mass, and dimensions of the leach test specimen;

(6) the history of the specimen between preparation and leaching, including time and temperature, as well as any other relevant information;

(7) beginning and end of each leaching interval (i.e., date, hour, and minute);

(8) electrical conductivity of leachant (μmho/cm at 25°C) and total organic carbon;

(9) volume of leachant used during each leaching interval;

(10) temperature of the leachate at the end of each leaching interval;

(11) volume of leachate aliquot used for analysis;

(12) type and amount of contaminant present in the leachate aliquots at the end of each leaching interval. The amount may be reported in units of microcuries (μCi) or disintegrations per second (Bq) or mass (μg). If the half-life of a radionuclide of concern does not exceed the time elapsed since the beginning of the test by a factor of 20, the data shall be corrected for radioactive decay to the reference time. Although any convenient time may be used as the reference time, the time of the beginning of the first leaching interval (see 4.2) should be used.

The amount of a radionuclide or nonradionuclide should be reported as the fraction of the radioactivity or mass of a reference source of the same radionuclide or nonradionuclide. If this method is chosen, all measurements of a given nuclide shall be reported relative to the same reference source. If the leach test is performed with nonradioactive tracers, the analytical method used shall be sensitive enough to provide meaningful data.

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[8] This information is recorded in Parts A, B, and C of forms such as those illustrated in Annex A.

In this case, A_o represents the total amount of tracer present in the specimen after the initial rinse, and the leaching data (a_n, etc.) are presented in units of micrograms (μg) or some other parameter specific for the analytical procedure. Whichever way is chosen, the units shall be consistent;

(13) surface appearance of specimen before and after leaching;

(14) observed changes in shape and dimensions of specimen during test;

(15) whether the leachate contained undissolved solids.

4.1 Presentation of Test Data

The results of the combined rinse-water determination are entered as "Experimental Data." If the relative amount of radioactivity is significantly greater than that obtained in previous generic studies or significantly exceeds the specifications for the waste form, the leach test shall be terminated, the reasons for the discrepancy ascertained, and the test repeated. Significant discrepancies from previous generic testing or from specifications can indicate inadequate performance of leach testing procedures, a nonrepresentative specimen, a waste form that was not properly prepared, and other less obvious reasons. If no significant discrepancies exist, the collection of data shall continue through the stated testing period.

The incremental fraction leached (a_n/A_O), the incremental leaching rate $[(a_n/A_O)/(\Delta t)_n]$, and the cumulative fraction leached ($\sum a_n/A_O$) shall be recorded as functions of time,[9] where:

a_n is the quantity of a nuclide released from the specimen during leaching interval n, corrected for radioactive decay (see 3.2);

$\sum a_n$ is the cumulative quantity of a nuclide released from the specimen from the beginning of the first leaching interval to the end of the leaching interval of concern, corrected for radioactive decay;

A_o is the total quantity of a given contaminant in the specimen at the beginning of the first leaching interval (i.e., after the initial 30-s rinse);

$\sum(\Delta t)_n = t_n = t$ is the cumulative leaching time, since the beginning of the first leaching interval (s);

$(\Delta t)_n = t_n - t_{n-l}$ is the duration, of the n'th leaching interval (s).

As required in 2.3, the leachate shall be replaced with fresh leachant very quickly, so that change-out time is insignificant when compared to the time of a leaching interval. Thus, for all practical purposes, "elapsed clock time," t_c, at the end of a leaching interval is equal to the sum of all the leaching intervals up to that time; that is, $t_c \cong \sum(\Delta t)_n$.

4.2 Analysis of Results

After initial rapid removal of mobile surface contamination (wash-off), the early leach rates observed with solidified waste forms are most often explained by diffusion.[10] If other mechanisms (e.g., corrosion, erosion, dissolution, etc.) are important, their control is generally discernible only after longer leaching [4].

If <20% of a leachable species is leached from a uniform, regularly shaped solid, its leaching behavior (if diffusion controlled) approximates that of a semi-infinite medium. In many leaching tests the isotope being followed is stable, or its half-life is sufficiently long, relative to the duration of the test, that it may be considered stable. Under these conditions the mass-transport equations permit the calculation [4] of an "effective diffusivity" by the following expression:

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[9] See Part C of Annex A.

[10] See Annex B.

$$D = \pi \left[\frac{a_n / A_o}{(\Delta t)_n} \right]^2 \left[\frac{V}{S} \right]^2 T \ , \tag{1}$$

where:

D is the effective diffusivity (cm^2/s);

V is the volume of specimen (cm^3);

S is the geometric surface area of the specimen as calculated from measured dimensions (cm^2);

T is the leaching time representing the “mean time” of the leaching interval (s) as follows:

$$T = [\tfrac{1}{2}(t_n^{1/2} + t_{n-1}^{1/2})]^2 \ . \tag{2}$$

The D in Eq. (1) is based on incremental (~ differential) data.[11] Using the incremental fraction leached has the advantage that each data point is independent; that is, any error or bias is not carried into subsequent data points (important for the initial points).[12]

If more than 20% of a leachable species has been removed by the time, t, the effective diffusivity can only be calculated from a shape specific solution of the mass transport equations [5,7]. Graphs to evaluate D (diffusion control) for cylinders are available [5]. The 20% value is an approximation but is sufficiently accurate for the purposes of this standard. More precise estimates of D from the data require more complicated calculations.

Table A.1 offers a convenient tabular way to calculate D from the experimental data for several types of cylinders. Integral data are used in this method and not incremental data as in Eq. (1). This is not a disadvantage at the advanced stage of the leaching process (i.e., after >20% has been leached). To use Table A.1, the cumulative fraction leached ($F = \sum a_n / A_O$) is calculated.[13] With this value of F, the corresponding G value for the type of cylinder represented by the specimen, identified by its length-over-diameter (ℓ / d) ratio, is obtained from Table A.1. This value is then used with the other known quantities on the right side of Eq. (3) to arrive at D, namely,

$$D = \frac{Gd^2}{t} \ , \tag{3}$$

where:

G is a time factor for the cylinder, dimensionless;

d is the diameter of the cylinder (cm);

t is the elapsed leaching time since the beginning of the first leaching interval (s).

As a specific example, consider a cylindrical specimen with length $\ell = 6$ cm and diameter $d = 2$ cm. After four leaching intervals (i.e., 2 + 5 + 17 + 24 = 48 hours or 1.73×10^5 s), 50% ($F = 0.5$) of the initial amount of a nuclide is leached out by a diffusion-controlled process. The G value in the column labeled $\ell / d = 3.0$ is found to be 0.0126. This is substituted into Eq. (3) together with the other quantities to arrive at the “effective diffusivity,” D_4, from the data of the fourth leaching interval, as follows:

$$D_4 = \frac{(0.0126)(2)^2}{1.73 \times 10^5} = 2.91 \times 10^{-7} \text{ cm}^2/\text{s} \ .$$

In Fig. 3 a convenient graphical method is provided for evaluating D (diffusion control) for several geometries [8]. Figure 3 also serves as a vivid illustration of the significant deviation of the leaching of finite bodies from the predictions of the semi-infinite medium model, after >20% of the initial radioactivity has been leached. For various geometries, the graph gives cumulative fraction leached versus a dimensionless time factor:

$$Z = \left[(Dt) \left(\frac{S}{V} \right)^2 \right]^{1/2} \tag{4}$$

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[11] In this connection see also Annex C.

[12] All the quantities appearing on the right of Eq. (1) are required entries on the forms given in Annex A.

[13] Last column of Part C of the forms given in Annex A.

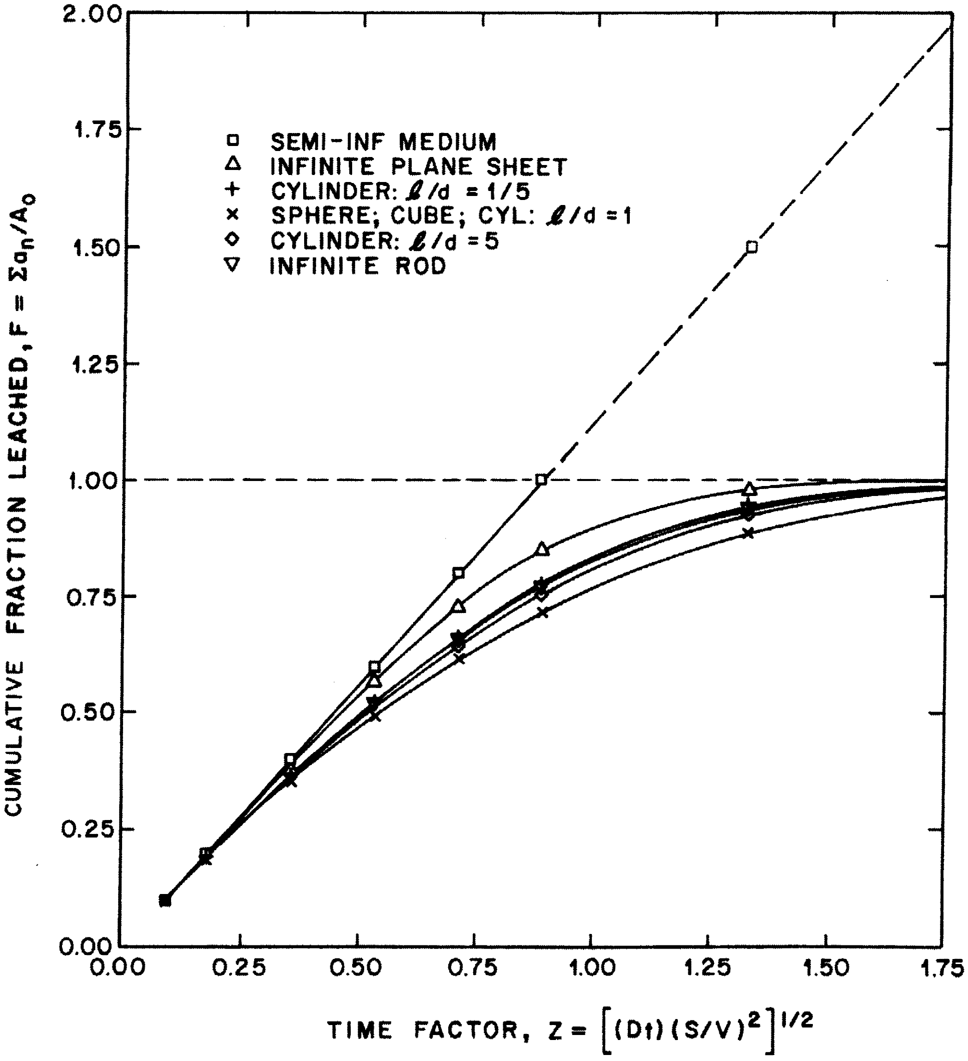

Figure 3.

with specimen geometry as a third parameter. As an illustration of the use of Fig. 3, consider the example given above for Table A.1. From the graph with a cumulative fraction leached of 0.5 and a cylinder with an ℓ/d of 3 (by interpolation between the curves for $\ell/d = 1$ and $\ell/d = 5$), the value of Z is found to be 0.52. For a specimen with the dimensions as above,

$$S = \pi d(\ell + d/2) = 43.98 \text{ cm}^2 \quad ;$$

$$V = (\pi d^2 \ell)/4 = 18.85 \text{ cm}^3 \quad .$$

These values along with $t = 1.73 \times 10^5$ s and $Z = 0.52$ are used in Eq. (4) to obtain D as follows:

$$Z = 0.52 = \left[D(1.73 \times 10^5)\left(\frac{43.98}{18.85}\right)^2 \right]^{1/2} \quad ,$$

from which

$$D_4 = 2.87 \times 10^{-7} \text{ cm}^2\text{/s} \quad ,$$

in close agreement with the value obtained above by the tabular method. For both the tabular and graphic methods, to obtain D, linear interpolation will suffice for values between those given in Table A.1 or Fig. 3.

Conversely, if a reasonable estimate of D is available, a specimen can be sized to approximate a semi-infinite medium for the time of the test using Table A.1 or Fig. 3.

In the foregoing considerations, no need to correct the data for radioactive decay was assumed; that is,

$$(a_n)_\tau = b_n\left(\frac{V_\ell}{v_a}\right)e^{\lambda\tau} \to b_n\left(\frac{V_\ell}{v_a}\right)(1) \equiv a_n \quad , \qquad (5)$$

where:

$(a_n)_\tau$ is the amount of radioactive species present in the aliquot of leachate used for analysis corrected for radioactive decay to the reference time;

b_n is the amount of radioactive species present in the aliquot of leachate used for analysis at the time of analysis;

v_a is the volume of leachate used for analysis (ml);

V_l is the volume of leachate from which v_a was taken (ml);

λ is the radioactive decay constant = ln 2/ $t_{1/2}$ = $0.693/t_{1/2}$ (s^{-1});

$t_{1/2}$ is the half-life of the radionuclide under consideration (s);

τ is the elapsed clock time from the reference time to the counting of the aliquot (s).

If the reference-source method is not used to report the data and the half-life of the radionuclide of interest is short ($t_{1/2} < 20\tau$), the counting data shall be corrected to the reference time. The beginning of the first leaching interval is frequently taken as the reference time. The correction is accomplished by multiplying b_n by its corresponding $e^{\lambda\tau}$ as shown in Eq. (5).[14)]

4.3 Determination of the Leachability Index

The Leachability Index of a nuclide, i, of concern in a material is defined as

$$L_i = \frac{1}{7}\sum_{1}^{7}[\log(\beta/D_i)]_n \quad , \qquad (6)$$

where β is a defined constant (1.0 cm^2/s) and D_i is the effective diffusivity of nuclide i calculated from the test data. These values of D_i are to be calculated by use of Eq. (1) if $\sum a_n/A_O <$ 0.2; otherwise, the tabular method and Eq. (3), or the graphical method using Fig. 3, or their equivalents for other shapes, shall be used. The mean [Eq. (6)] of the seven determinations (L_n) for each radionuclide shall be recorded as its Leachability Index (L_i). This is the single value used (in conjunction with the results of available pertaining generic studies) to classify or rank the material.

A given matrix material might have different Leachability Index values for different radionuclides if they are present as different chemical species that are more or less readily mobilized. Different Leachability Indexes for the same matrix are reported with the particular nuclide as subscript (e.g., $L_{\text{Co}}^{60} = 10.7$).

To illustrate the Leachability Index calculation, the data in Annex A, Table A.2 are used. For example 1, the mean value [Eq. (6)] of the seven log $(\beta/D_i)_n$ values presented in Table A.2 yield

$$L_i = \frac{7.2 + 6.8 + 5.4 + 5.7 + 5.6 + 7.1 + 6.7}{7}$$

$$= 6.4 \quad .$$

For example 2, the result is

$$L_i = \frac{6.4 + 6.6 + 6.1 + 5.9 + 6.1 + 6.5 + 5.6}{7}$$

$$= 6.2 \quad .$$

If the "extended test" is performed, the Leaching Index is given the symbol $\overline{\text{L}}$ and is calculated from the $\log(\beta/D_i)$ values of the first seven leaching intervals plus the added three intervals by the following expression:

$$\overline{L_i} = \frac{1}{10}\sum_{1}^{10}[\log(\beta/D_i)]_n \quad . \qquad (7)$$

The standard leach test is intended to serve for quantitative assessment purposes and as a ba-

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[14)] An example of this correction with Eq. (1) used to calculate D is presented in Annex C.

sis for comparing the leachability of solidified waste forms under one set of prescribed conditions. The extended test permits observation of the leach behavior and the durability of the material tested under extended "stagnant pool" leaching conditions. Ninety-day immersion is still relatively short compared to the length of time of interest for waste disposal and can hardly be considered a guarantor of durability for the material tested. However, some materials are known to physically change and/or deteriorate under immersion conditions over such time periods. For example, ion-exchange resins are known to swell inside cement waste forms, potentially causing problems with cracking and crumbling of the waste form. The extended test can better serve to help screen and identify such behavior by extending immersion well beyond the initial 5 days.

The Leachability Index shall be reported to one decimal position only.

If all the initial and boundary conditions for the diffusion model used in this standard[15] are met, the Leachability Index would be independent of time; that is, each L_n would be equal to L_i. However, despite the best-laid plans of an investigator (even with a material in which diffusion is the only mechanism for mass transport), the measurements of L can include the effects of unknown, and perhaps intermittent, variations that might be insignificant for all practical purposes (see 3.1). This standard uses the confidence range and the correlation coefficient as measures of these discrepancies.

The 99.9% confidence range of the Leachability Index calculated from the first seven leach intervals is defined by the following expression:

$$C = L_i \pm 5.959\sigma_L n^{-1/2} = L_i \pm 2.25\sigma_L \quad , \qquad (8)$$

where:

C is the 99.9% confidence range of L_i, dimensionless;

L_i is the mean of the seven values of L_n [i.e., Eq. (6)], dimensionless;

L_n is the value of L as calculated from the data of the n'th leaching interval, dimensionless;

σ_L is the standard deviation of the seven values of L_n, dimensionless, as follows:

$$\sigma_L = \frac{1}{\sqrt{6}}\left[\sum_1^7 (L_n - L_i)^2\right]^{1/2} \quad . \qquad (9)$$

The correlation coefficient between L and t is defined by the following relationship:

$$r = \frac{\sigma_{Lt}}{\sigma_L \sigma_t} \quad , \qquad (10)$$

where:

r is the correlation coefficient, dimensionless,

$$\sigma_{Lt} = \frac{1}{6}\sum_1^7 (L_n - L_i)(t_n - t_m) \quad , \qquad (11)$$

the covariance of the seven sets of L and t, s;

t_n is the value of t at the end of the n'th leaching interval (s)

$$t_m = \frac{1}{7}\sum_1^7 t_n \quad , \qquad (12)$$

the mean of the seven values of t_n, s;

$$\sigma_t = \frac{1}{\sqrt{6}}\left[\sum_1^7 (t_n - t_m)^2\right]^{1/2} \quad ,$$

the standard deviation of the seven values of t_n (s).

The correlation coefficient varies from −1 to +1. The sign indicates whether L_n is tending to increase ($+r$) or to decrease ($-r$) as t_n increases.

These statistical parameters can be derived or described in any standard text on statistics [9].

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[15] See Annex B.

For the results shown in Table A.2, the confidence range (C) and the correlation coefficient (r) are calculated [Eqs. (8) and (10)] to be as follows:

Parameter	Example 1	Example 2
Standard 5-day Test		
Confidence range Correlation coefficient	4.6 to 8.1 0.09	5.4 to 7.0 -0.65
Extended 90-day Test		
Confidence range Correlation coefficient	5.4 to 7.9 0.56	5.5 to 6.6 -0.59

The calculations made to obtain these statistical parameters shall be carried out to at least two more significant figures than the original values of L_n and t_n. No rounding-off shall be done until the final results are obtained. The confidence interval shall be reported to one decimal position only, and the correlation coefficient to two decimal positions.

The confidence range and correlation coefficient shall be reported with the Leachability Index.

4.4 Limitations of Results

To be practicable, a standard leach test must permit accumulation of sufficient data to determine material parameters (e.g., L or $\bar{L}$) in a relatively short time. To be meaningful, however, these L (or $\bar{L}$) values must be related to long-term leaching studies carried out with similar-type materials (i.e., generic studies) under a wide range of conditions, which determine the actual mass transport mechanisms. These provide the background for interpreting the results of a short-term standard leach test.

If the only leaching mechanism occurring in the waste form is diffusion-controlled leaching, the Leachability Index (L) from the standard 5-day test has an exact theoretical interpretation. The interpretation applies for the conditions of the test for any size and shape waste form made of the material [4,5,8]. This is true as long as the initial and boundary conditions are satisfied.[16]

The Leachability Index, as defined in this standard (L or $\bar{L}$), is a material parameter. It is limited to the material for which it was determined. It is meaningful only if the specimen itself did not undergo significant changes during the leach test and if the actual waste form to which it applies does not undergo changes in its material characteristics.

The logarithmic character of the Leachability Index allows a relatively wide range of experimental error and deviation from the ideal to be tolerated without a significant effect on the index value or its intended use. This is advantageous because minor perturbations that would limit the applicability of the test and the interpretation of its results are eliminated. For example, in 3.2, two methods were given for the calculation of the effective diffusivity, D_4, from a set of data. By one method, a value of 2.91×10^{-7} cm^2/s was obtained; by the other, a value of 2.87×10^{-7} cm^2/s was obtained. The L_n value derived from either would be 6.54. Since the Leachability Index shall be reported to one decimal position only, both methods usually arrive at the same value. When interpolation (or the less precise graphical method) is necessary to determine the value of D, there may be slight variations in the decimal value. Similarly, only a drastic change of the dominant leaching mechanism from diffusion-controlled leaching during the early part of the test will seriously affect the index value obtained. Any such observation shall be reported in the manner described in 3.3.

5 References

[1] "Leach Testing of Immobilized Radioactive Waste Solids, A Proposal for a Standard Method," International Atomic Energy Agency, E. D. Hespe, Ed., *At. Energy Rev.*, **9**, 1 (1971).

[2] "Long-Term Leach Testing of Solidified Radioactive Waste Forms," ISO 6961-1982(E), International Organization for Standardization.

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[16] See Annex B.

[3] J. E. MENDEL et al., “A State-of-the-Art Review of Materials Properties of Nuclear Waste Forms,” PNL-3802, Pacific Northwest National Laboratory (1981).

[4] H. W. GODBEE and D. S. JOY, “Assessment of the Loss of Radioactive Isotopes from Waste Solids to the Environment, Part 1: Background and Theory,” ORNL-TM-4333, Oak Ridge National Laboratory (1974).

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[6] “Materials Characterization Center, Materials Characterization Center Test Methods Preliminary Version,” PNL-3990, Pacific Northwest National Laboratory (1981); see also “Nuclear Waste Materials, Handbook, Waste Form Test Methods,” Materials Characterization Center, DOE/TIC-11400, U.S. Department of Energy (1981).

[7] C. W. NESTOR, Jr., “Diffusion from Solid Cylinders,” ORNL/CSD/TM-84, Oak Ridge National Laboratory (Jan. 1980).

[8] H. W. GODBEE and E. L. COMPERE, “A Figure-of-Merit Determined from the ANS Leachability Standard,” *Trans. Am. Nucl. Soc.*, **33**, 205 (1979).

[9] W. VOLK, *Applied Statistics for Engineers*, 2nd ed., McGraw-Hill Book Company, New York (1969).

Annex A

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Data forms, time factor table, and example calculations of the leachability index

A.1 Description of leach specimen and leachant

Page___ of ___

LEACH TEST RESULTS

LEACH TEST IDENTIFICATION NUMBER ____________________
LABORATORY WHERE TESTS PERFORMED ____________________
ANALYST ____________________
DATE RESULTS REPORTED ____________________

Part A. Description of Leach Specimen

Specimen Identification Number ____________________
Proportion of Waste Incorporated in mixture ____________________ Weight %
Volume % ____________________

(*Based on initial volumes*)

Type of Waste, Chemical and Radioisotopic Composition, and Specific Activity of the Waste ____________________

Type and Composition of the Solidification Agent ____________________

Preparation of Specimen ____________________

Shape and Dimension[a] of Specimen
Sphere, diameter, d (cm) = ____________________
Cylinder, diameter, d (cm) = ____________________ &
length, l (cm) = ____________________
Parallelepiped, length, l (cm) = ____________________ ,
width, w (cm) = ____________________ &
height, h (cm) = ____________________
Other, Shape ____________________

Dimensions ____________________
Initial Weight of Specimen, W (g) = ____________________
Volume of Specimen[a], V (cm^3) = ____________________
Surface Area of Specimen[a], S (cm^2) = ____________________
Storage Conditions ____________________

Appearance

Description of Leachant

Leach Interval (n)	Temp. (°C)	Electrical Conductivity (mho/cm)	Volume, V_L (mL)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Calculated from dimensions of specimen.

A.2 Description of leach test procedure

Page ___ of ___

LEACH TEST RESULTS

LEACH TEST IDENTIFICATION NUMBER ____
LABORATORY WHERE TESTS PERFORMED ____
ANALYST ____
DATE RESULTS REPORTED ____
Part B. Description of Leach Test Procedure

Specimen Preparation[a] ____

Diagram of Leach Apparatus:

Leachate Sampling Procedure ____

Analytical Techniques:
Counting Instrument Identification and Calibration ____

Constituent a_1, Analytical Procedure, Standard Deviation of Method ____

Constituent a_2, Analytical Procedure, Standard Deviation of Method ____

Constituent a_i, Analytical Procedure, Standard Deviation of Method ____

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[a] If different from "Preparation of Specimen" in Part B.

LEACH TEST RESULTS

LEACH TEST IDENTIFICATION NUMBER ____________________
LABORATORY WHERE TESTS PERFORMED ____________________
ANALYST ____________________
DATE RESULTS REPORTED ____________________

Part C. Experimental Data

Constituent Analyzed[a] ____________________

Free Standing Water in Leach Specimen Container: ___Yes ___No
If Yes, Volume (mL) = _____ ; Radioactivity (μCi) = _____ ; and % of A_O = _____
Specimen Rinse Before Initiation of Leaching: *Do not include with results in the table below.*
Volume [a] *(mL) = _____ ; Radioactivity (μCi) = _____ ; and % of A_O = _____*
Initial Amount in Specimen, A_O (μCi) [after 30-s rinse] = ____________________

Leach Interval (n)	Temp. (°C)	Time and Date[b]		V_L^c (mL)	$(\Delta t)_n$ (s)	$t = \sum(\Delta t)_n$ (s)	As Analyzed[d]	Blank[d]	Corrected Conc.[d]	a_n[e]	a_n/A_O	$[a_n/A_O][1/(\Delta t)_n]$ (fraction/s)	$F = \sum a_n/A_O$
		In	Out										
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													

[a] Determined at end of rinse operation.
[b] Date, hour, and minute.
[c] At the end of the leaching interval.
[d] Concentration, show units.
[e] a_n = corrected concentration × V_L × factor to convert to same units as A_O.
The value of a_n must include any radioactivity rinsed from the specimen and the leach apparatus at the end of the leaching interval.

A.3 Experimental Data

Table A.1 – Time factors for leaching of finite cylinders[1),2)]

Cumulative Fraction Leached (F)	Dimensionless Time Factor ($G = Dt/d^2$)						
	ℓ/d = 0.3	ℓ/d = 0.5	ℓ/d = 1.0	ℓ/d = 1.5	ℓ/d = 2.0	ℓ/d= 3.0	ℓ/d = 5.0
0.20	3.10D-4	5.59D-4	9.98D-4	1.26D-3	1.42D-3	1.62D-3	1.80D-3
0.21	3.43D-4	6.21D-4	1.11D-3	1.40D-3	1.58D-3	1.80D-3	2.00D-3
0.22	3.79D-4	6.86D-4	1.23D-3	1.54D-3	1.74D-3	1.98D-3	2.21D-3
0.23	4.17D-4	7.55D-4	1.35D-3	1.70D-3	1.92D-3	2.18D-3	2.43D-3
0.24	4.57D-4	8.28D-4	1.48D-3	1.86D-3	2.11D-3	2.39D-3	2.66D-3
0.25	4.98D-4	9.05D-4	1.62D-3	2.04D-3	2.30D-3	2.61D-3	2.90D-3
0.26	5.42D-4	9.86D-4	1.77D-3	2.22D-3	2.51D-3	2.84D-3	3.16D-3
0.27	5.89D-4	1.07D-3	1.92D-3	2.41D-3	2.72D-3	3.09D-3	3.43D-3
0.28	6.37D-4	1.16D-3	2.08D-3	2.61D-3	2.95D-3	3.34D-3	3.71D-3
0.29	6.88D-4	1.26D-3	2.25D-3	2.82D-3	3.19D-3	3.6lD-3	4.00D-3
0.30	7.40D-4	1.35D-3	2.43D-3	3.05D-3	3.43D-3	3.89D-3	4.31D-3
0.31	7.96D-4	1.46D-3	2.61D-3	3.28D-3	3.69D-3	4.18D-3	4.63D-3
0.32	8.54D-4	1.56D-3	2.81D-3	3.52D-3	3.97D-3	4.49D-3	4.97D-3
0.33	9.14D-4	1.68D-3	3.01D-3	3.77D-3	4.25D-3	4.81D-3	5.32D-3
0.34	9.77D-4	1.80D-3	3.22D-3	4.04D-3	4.55D-3	5.14D-3	5.68D-3
0.35	1.04D-3	1.92D-3	3.44D-3	4.31D-3	4.86D-3	5.48D-3	6.06D-3
0.36	1.11D-3	2.05D-3	3.68D-3	4.60D-3	5.18D-3	5.85D-3	6.45D-3
0.37	1.18D-3	2.18D-3	3.92D-3	4.90D-3	5.51D-3	6.22D-3	6.86D-3
0.38	1.25D-3	2.32D-3	4.17D-3	5.21D-3	5.86D-3	6.61D-3	7.29D-3
0.39	1.33D-3	2.46D-3	4.43D-3	5.54D-3	6.23D-3	7.02D-3	7.73D-3
0.40	1.41D-3	2.61D-3	4.70D-3	5.88D-3	6.60D-3	7.44D-3	8.19D-3
0.41	1.49D-3	2.77D-3	4.99D-3	6.23D-3	7.00D-3	7.87D-3	8.67D-3
0.42	1.58D-3	2.93D-3	5.28D-3	6.60D-3	7.40D-3	8.33D-3	9.16D-3
0.43	1.67D-3	3.10D-3	5.59D-3	6.98D-3	7.83D-3	8.80D-3	9.67D-3
0.44	1.76D-3	3.28D-3	5.91D-3	7.37D-3	8.27D-3	9.29D-3	1.02D-2
0.45	1.85D-3	3.46D-3	6.24D-3	7.78D-3	8.72D-3	9.79D-3	1.07D-2
0.46	1.95D-3	3.65D-3	6.58D-3	8.21D-3	9.19D-3	1.03D-2	1.13D-2
0.47	2.05D-3	3.84D-3	6.94D-3	8.65D-3	9.69D-3	1.09D-2	1.19D-2
0.48	2.16D-3	4.05D-3	7.31D-3	9.11D-3	1.02D-2	1.14D-2	1.25D-2
0.49	2.27D-3	4.26D-3	7.70D-3	9.58D-3	1.07D-2	1.20D-2	1.31D-2
0.50	2.38D-3	4.48D-3	8.10D-3	1.01D-2	1.13D-2	1.26D-2	1.38D-2
0.51	2.49D-3	4.71D-3	8.52D-3	l.06D-2	1.18D-2	1.32D-2	1.45D-2
0.52	2.61D-3	4.94D-3	8.95D-3	1.11D-2	1.24D-2	1.39D-2	1.52D-2
0.53	2.74D-3	5.19D-3	9.40D-3	1.17D-2	1.30D-2	1.46D-2	1.59D-2
0.54	2.87D-3	5.44D-3	9.86D-3	1.22D-2	1.37D-2	1.52D-2	1.66D-2
0.55	3.00D-3	5.70D-3	1.03D-2	1.28D-2	1.43D-2	1.60D-2	1.74D-2
0.56	3.13D-3	5.98D-3	1.09D-2	1.35D-2	1.50D-2	1.67D-2	1.82D-2
0.57	3.28D-3	6.26D-3	1.14D-2	1.41D-2	1.57D-2	1.75D-2	1.90D-2
0.58	3.42D-3	6.55D-3	l.19D-2	1.48D-2	1.64D-2	1.83D-2	l.99D-2
0.59	3.57D-3	6.86D-3	1.25D-2	1.54D-2	1.72D-2	1.91D-2	2.08D-2
0.60	3.73D-3	7.17D-3	1.31D-2	1.62D-2	1.80D-2	2.00D-2	2.17D-2
0.61	3.89D-3	7.50D-3	1.37D-2	1.69D-2	1.88D-2	2.08D-2	2.26D-2
0.62	4.06D-3	7.84D-3	1.43D-2	1.77D-2	1.96D-2	2.18D-2	2.36D-2
0.63	4.23D-3	8.19D-3	1.50D-2	1.85D-2	2.05D-2	2.27D-2	2.46D-2
0.64	4.41D-3	8.56D-3	1.56D-2	1.93D-2	2.14D-2	2.37D-2	2.56D-2

(Continued)

Table A.1 – Continued

Cumulative Fraction Leached (F)	Dimensionless Time Factor ($G = Dt/d^2$)						
	$\ell/d = 0.3$	$\ell/d = 0.5$	$\ell/d = 1.0$	$\ell/d = 1.5$	$\ell/d = 2.0$	$\ell/d = 3.0$	$\ell/d = 5.0$
0.65	4.59D-3	8.94D-3	1.64D-2	2.02D-2	2.24D-2	2.47D-2	2.67D-2
0.66	4.78D-3	9.33D-3	1.71D-2	2.11D-2	2.33D-2	2.58D-2	2.78D-2
0.67	4.98D-3	9.75D-3	1.79D-2	2.20D-2	2.44D-2	2.69D-2	2.90D-2
0.68	5.18D-3	1.02D-2	1.87D-2	2.30D-2	2.54D-2	2.80D-2	3.02D-2
0.69	5.40D-3	1.06D-2	1.95D-2	2.40D-2	2.65D-2	2.92D-2	3.15D-2
0.70	5.62D-3	1.11D-2	2.04D-2	2.50D-2	2.77D-2	3.04D-2	3.27D-2
0.71	5.84D-3	1.16D-2	2.13D-2	2.61D-2	2.89D-2	3.17D-2	3.41D-2
0.72	6.08D-3	1.21D-2	2.23D-2	2.73D-2	3.01D-2	3.30D-2	3.55D-2
0.73	6.33D-3	1.26D-2	2.33D-2	2.85D-2	3.14D-2	3.44D-2	3.70D-2
0.74	6.59D-3	1.31D-2	2.43D-2	2.97D-2	3.27D-2	3.59D-2	3.85D-2
0.75	6.86D-3	1.37D-2	2.54D-2	3.11D-2	3.42D-2	3.74D-2	4.010-2
0.76	7.14D-3	1.43D-2	2.65D-2	3.24D-2	3.56D-2	3.90D-2	4.17D-2
0.77	7.44D-3	1.49D-2	2.77D-2	3.39D-2	3.72D-2	4.06D-2	4.34D-2
0.78	7.75D-3	1.56D-2	2.90D-2	3.54D-2	3.88D-2	4.24D-2	4.52D-2
0.79	8.08D-3	1.63D-2	3.03D-2	3.70D-2	4.05D-2	4.42D-2	4.71D-2
0.80	8.42D-3	1.70D-2	3.18D-2	3.87D-2	4.24D-2	4.61D-2	4.91D-2
0.81	8.78D-3	1.78D-2	3.32D-2	4.04D-2	4.42D-2	4.81D-2	5.12D-2
0.82	9.16D-3	1.86D-2	3.48D-2	4.23D-2	4.63D-2	5.03D-2	5.35D-2
0.83	9.56D-3	1.95D-2	3.65D-2	4.43D-2	4.84D-2	5.25D-2	5.58D-2
0.84	9.99D-3	2.04D-2	3.83D-2	4.64D-2	5.07D-2	5.49D-2	5.83D-2
0.85	1.05D-2	2.14D-2	4.02D-2	4.87D-2	5.31D-2	5.75D-2	6.10D-2
0.86	1.09D-2	2.25D-2	4.23D-2	5.11D-2	5.57D-2	6.03D-2	6.38D-2
0.87	1.15D-2	2.36D-2	4.45D-2	5.38D-2	5.85D-2	6.32D-2	6.69D-2
0.88	1.21D-2	2.49D-2	4.69D-2	5.66D-2	6.15D-2	6.64D-2	7.02D-2
0.89	1.27D-2	2.62D-2	4.95D-2	5.97D-2	6.49D-2	6.99D-2	7.38D-2
0.90	1.34D-2	2.77D-2	5.23D-2	6.31D-2	6.85D-2	7.37D-2	7.78D-2
0.91	1.41D-2	2.94D-2	5.55D-2	6.69D-2	7.25D-2	7.80D-2	8.22D-2
0.92	1.50D-2	3.12D-2	5.90D-2	7.11D-2	7.70D-2	8.27D-2	8.71D-2
0.93	1.60D-2	3.33D-2	6.31D-2	7.59D-2	8.21D-2	8.81D-2	9.26D-2
0.94	1.71D-2	3.58D-2	6.77D-2	8.14D-2	8.81D-2	9.43D-2	9.91D-2
0.95	1.84D-2	3.87D-2	7.33D-2	8.80D-2	9.51D-2	1.02D-1	1.07D-1
0.96	2.01D-2	4.22D-2	8.00D-2	9.61D-2	1.04D-1	1.11D-1	1.16D-1
0.97	2.22D-2	4.68D-2	8.87D-2	1.06D-1	1.15D-1	1.22D-1	1.28D-1
0.98	2.52D-2	5.33D-2	1.01D-1	1.21D-1	1.313D-1	1.39D-1	1.45D-1
0.99	3.04D-2	6.43D-2	1.22D-1	1.46D-1	1.57D-1	1.67D-1	1.74D-1

[1] Adapted from C. W. Nestor, Jr., "Diffusion from Solid Cylinders," ORNL/CSD/TM-84, Oak Ridge National Laboratory (Jan. 1980).

[2] Values for $G = Dt/d^2$ are presented in the form given on a computer readout. Conventional mathematical form would be expressed in powers of 10. For example, 3.10D-4 is equivalent to 3.10×10^{-4}.

Table A.2 – Examples for the computation of the Leachability Index from leaching data[1]

Leach Interval (n)	Leaching Time (s)		Fraction Released During Interval a_n/A_O	Release Rate (fraction/s) $[a_n/A_o l[1/(\Delta t)_n]$	Diffusivity D (cm^2/s)	Leachability Index $\log(\beta/D)$
	Incremental $(\Delta t)_n$	Cumulative $t = \sum(\Delta t)_n$				
Example 1[2]						
1	7.78×10^{3}	7.78×10^{3}	2.81×10^{-3}	3.61×10^{-7}	6.01×10^{-8}	7.2
2	1.04×10^{4}	1.81×10^{4}	2.43×10^{-3}	2.34×10^{-7}	1.63×10^{-7}	6.8
3	6.57×10^{4}	8.38×10^{4}	4.18×10^{-2}	6.35×10^{-7}	4.31×10^{-6}	5.4
4	8.64×10^{4}	1.70×10^{5}	2.27×10^{-2}	2.63×10^{-7}	2.02×10^{-6}	5.7
5	8.64×10^{4}	2.57×10^{5}	1.90×10^{-2}	2.20×10^{-7}	2.38×10^{-6}	5.6
6	8.64×10^{4}	3.43×10^{5}	2.98×10^{-3}	3.45×10^{-8}	8.49×10^{-8}	7.1
7	2.59×10^{5}	6.02×10^{5}	1.13×10^{-2}	4.36×10^{-8}	2.09×10^{-7}	6.7
	End of Standard Test and Beginning of Extended Test					
8	1.30×10^{6}	1.90×10^{6}	1.36×10^{-2}	1.05×10^{-8}	3.02×10^{-8}	7.5
9	2.42×10^{6}	4.32×10^{6}	2.10×10^{-2}	8.68×10^{-9}	5.33×10^{-8}	7.3
10	4.23×10^{6}	8.55×10^{6}	1.70×10^{-2}	4.02×10^{-9}	2.39×10^{-8}	7.6
Example 2[3]						
1	7.78×10^{3}	7.78×10^{3}	6.86×10^{-3}	8.82×10^{-7}	3.40×10^{-7}	6.4
2	1.04×10^{4}	1.81×10^{4}	2.94×10^{-3}	2.82×10^{-7}	2.66×10^{-7}	6.6
3	6.57×10^{4}	8.38×10^{4}	1.76×10^{-2}	2.67×10^{-7}	8.52×10^{-7}	6.1
4	8.64×10^{4}	1.70×10^{5}	1.74×10^{-2}	2.01×10^{-7}	1.33×10^{-6}	5.9
5	8.64×10^{4}	2.57×10^{5}	1.02×10^{-2}	1.18×10^{-7}	7.67×10^{-7}	6.1
6	8.64×10^{4}	3.43×10^{5}	5.58×10^{-3}	6.46×10^{-8}	3.32×10^{-7}	6.5
7	2.59×10^{5}	6.02×10^{5}	3.60×10^{-2}	1.39×10^{-8}	2.37×10^{-6}	5.6
	End of Standard Test and Beginning of Extended Test					
8	1.30×10^{6}	1.90×10^{6}	1.00×10^{-1}	7.72×10^{-8}	2.75×10^{-6}	5.6
9	2.42×10^{6}	4.32×10^{6}	1.08×10^{-1}	4.46×10^{-8}	2.61×10^{-6}	5.6[4]
10	4.23×10^{6}	8.55×10^{6}	1.06×10^{-1}	2.51×10^{-8}	2.39×10^{-6}	5.6[4]

Results of Computations for the Standard Test

Parameter	Example 1	Example 2
Leachability Index [Eq. (6)]	6.4	6.2
Confidence range [Eq. (8)]	4.6 to 8.1	5.4 to 7.0
Correlation coefficient [Eq. (10)]	0.09	−0.65

[1] Adapted from data given in R. M. Neilson, Jr., P. D. Kalb, and P. Colombo, “Lysimeter Study of Commercial Reactor Waste Forms: Waste Form Acquisition, Characterization and Full-Scale Leaching,” BNL-51613, Brookhaven National Laboratory (Sep. 1982).

[2] Specimen is a right circular cylinder with a diameter of 41.3 cm (16.3 in.) and a length of 109.3 cm (43.0 in.). The radionuclide leached is ^{137}Cs (half-life of 30 years).

[3] Specimen is a right circular cylinder with a diameter of 55.25 cm (21.75 in.) and a length of 54.6 cm (21.5 in.). The radionuclide leached is ^{137}Cs (half-life of 30 yr).

[4] Obtained using Table A.1 since $F > 0.20$.

Annex B

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Mass transport considerations

B.1 Initial and boundary conditions

A considerable amount of experimental data obtained from samples that maintained their dimensional integrity during leaching indicates that internal bulk diffusion is the most likely rate-determining mechanism during the initial phases of the leaching process [3–5].[1] Although additional mechanisms probably do occur to some degree, they are more likely to become rate-determining only during later stages of leaching. Thus, the recommended data-handling procedure of this standard is a deliberate, albeit permissible, simplification of mass transport theory for the purpose of classifying and ranking solidified radioactive wastes according to leachability.

The equations presented in this standard (see Section 4, "Presentation and Analysis of Data," and Annex C) are derived from simple diffusion theory and hold exactly only for cases where:

(1) after leaching commences, the concentration of the species being leached is zero at the surface of the waste form;

(2) the leachant is continuously moving and never changes significantly in its composition, pH, etc. (nor builds up significant concentrations of the leached species);

(3) the leached material is homogeneous and remains essentially unchanged chemically and physically;

(4) the specimen surface is smooth and does not deteriorate with time (e.g., craze, spall, or form a protective corrosion product layer);

(5) the leachable species is mobilized by the leachant so that bulk diffusion is the limiting process;

(6) there are no time-dependent interactions between the leachable species and the leachant, the matrix, or other leached constituents of the matrix;

(7) the radionuclide (or nuclide) of interest is present as but one chemical species.

Leaching may be enhanced by surface irregularities and roughness as well as swelling, fissuring, surface deterioration, and chemical or physical breakdown of the matrix material. Leaching may be retarded by intermittent and stagnant leaching conditions if the leachant becomes significantly concentrated with the leached species. It may also be inhibited by the buildup of solid surface deposits, by curing, and by other chemical changes that reduce the bulk diffusivity within the matrix. The presence of nonhomogeneous regions of the matrix, where the species of interest is preferentially sorbed or immobilized by precipitation, will also retard leaching.

Reasons such as these, as well as others mentioned in this standard, bring out that a generic leaching program is needed for the product from each type of waste and solidification process. Such a program should establish what can be expected from a particular waste form in the long term and under different leaching conditions (see Annex E). The Leachability Index is then only a normalization factor to prove that a given material behaves like the class of materials to which it supposedly belongs and that has been tested extensively by the generic studies.

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[1] The numeric citations in this Annex can be found in Section 5 of the main text.

Other mass transfer equations may be derived for a given type of waste form from the results of such long-term studies. It is likely that their early-leaching predictions will closely approximate diffusion control and the behavior predicted by the simplified interpretation of the Leachability Index. The latter can thus be used to normalize such generic mass transfer equations to the specimen at hand.

The generic mass transfer equations may account for other leaching mechanisms such as the leaching after chemical transformation(s) of the species of interest, leaching from a chemically and physically changing (aging) matrix, dissolution of the surface of the waste form, corrosion, and chemical as well as physical changes of the surface during prolonged exposure to the leachant (e.g., fissuring, hydroxide gel formation, etc.). The generic mass transfer equations may also account for the effect of slower leachant renewal rates (e.g., "stagnant pool" conditions). In addition, they may apply to leachants and conditions that simulate actual leaching. These would include the groundwater at a disposal site and changes in the chemistry of the leachant with time (e.g., the buildup of the species of interest).

B.2 Interpolation and extrapolation

If (a) single-parameter diffusion is the only leaching mechanism, (b) the material is homogeneous, (c) long-term stability has been proven, (d) the radiation fields are insignificant, and (e) the material is leached continuously with demineralized water at ambient temperature (see 3.3), the Leachability Index values have specific meaning. Namely, the Leachability Index is the negative logarithm of the effective diffusivity of the radionuclide.

In the examples presented below, the waste form is assumed to be leached continuously; thus, t, T, and T_m represent elapsed clock time since the initiation of leaching. Time T_m is the cumulative leaching time representing the "mean time" of the n'th leaching interval for any geometry.

If radioactive decay is negligible, the residual or unleached fraction $(1-F)$ remaining in the waste form at a given time t can be calculated using L with Table A.1 and Fig. 3 of this standard or with available analytical solutions [5,7,8].

If radioactive decay is not negligible, the actual fraction of the radionuclide remaining with the waste form $[(1-F)_\lambda]$ is calculated by application of the radioactive decay correction to $(1-F)$. For the case in which a radionuclide is not being generated by other decay chains, this amounts to

$$(1-F)_\lambda = (1-F)e^{-\lambda t} \quad .$$

The amount of radioactivity that has leached from the waste form and is still in the environment $\left(\sum a_n\right)_\lambda$ at time t is given by

$$\left(\sum a_n\right)_\lambda = FA_O e^{-\lambda t} \quad .$$

The total amount of the radionuclide that has entered the environment since the waste form was placed into the leachant up to the time of interest t is given by

$$\sum(a_n e^{-\lambda t_n})$$

with the sum taken for all n for which $t_n < t$.

The foregoing relationships may be used to calculate the amounts of radionuclide inside and outside a waste form of given dimensions at time t if the form is leached in demineralized water and the Leachability Index of the material has been determined. Tables or charts that give this information for the radionuclides of interest may be prepared [5].

The difference of the amounts leached $[(F_n - F_{n-1})A_o]$ calculated for t_n and t_{n-1} divided by $(t_n - t_{n-1})$ or $(\Delta t)_n$ represents the leaching rate R at time T_m. The actual rate (i.e., with radioactivity taken into account) at which the radionuclide is released into the environment at that time is given by

$$R = \frac{\Delta F}{(\Delta t)_n} A_o e^{-\lambda T_m} \quad .$$

This expression is applicable to any geometry (with the constraints given in Section B.1 and above). Equations for the mean leaching time of the interval (T_m) are not readily available for most geometries. However, this is not usually a disadvantage since if $(\Delta t)_n < 0.2\, t_n$, the average leaching time of the interval $[\frac{1}{2}(t_n + t_{n-1})]$ is a close approximation to T_m for any geometry. Alternatively, R could be determined for a semi-infinite medium by solving Eq. (1) for a_n/A_O and then correcting for radioactive decay (i.e., multiplying the right side of the expression obtained by $e^{-\lambda T}$). The relationship arrived at is

$$R = \left(\frac{D}{\pi}\right)^{1/2} \left(\frac{S}{V}\right) T^{-1/2} A_o e^{-\lambda T} \quad .$$

The results of generic studies make possible estimates, such as those described earlier, for different leachants and for leaching under conditions where leaching is controlled by several different mass transport mechanisms.

Annex C

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Calculations with correction for radioactive decay

Within the constraints given in Annex B, the solution of the mass transport equations for a specimen that may be considered a semi-infinite medium permits the effective diffusivity to be computed by

$$D = \pi \left[\frac{a_n / A_o}{(\Delta t)_n} \right]^2 \left[\frac{V}{S} \right]^2 T \tag{C.1}$$

for a "stable" nuclide (or leached species) with half-life $t_{1/2} > 20\tau$. It is also applicable if the data are presented in terms of a reference source (see 3.2).

If the half-life of the radionuclide is short, a correction for the radioactive decay during the time between the reference time and the time of the counting of the respective aliquot is to be applied by multiplying each of the counting rates by its respective decay correction factor $e^{\lambda\tau}$. For example,

$$(a_n)_\tau = b_n \left(\frac{V\ell}{v_a} \right) e^{\lambda\tau} = (AA - B) \left(\frac{V\ell}{v_a} \right) e^{\lambda\tau} \quad , \tag{C.2}$$

where AA is the amount of the radionuclide in the aliquot as analyzed at time τ and B is the amount, if any, of the radionuclide in a blank having the same volume as the aliquot analyzed at time τ.

For the purpose of illustration, assume that the information given in Table C.1 has been collected for a radionuclide with a half-life of 65 days (5.62×10^6 s). Further, assume that the specimen is a right circular cylinder with $\ell = 1.0$ cm and $d = 1.0$ cm ($S = 4.71$ cm^2 and $V = 0.785$ cm^3). Also, assume that the reference time is 1 day before the first leaching interval begins and that at this reference time the specimen contains 15 mCi of ^{85}Sr $[(A_O)_\tau]$. The individual aliquots are assumed to be counted at the times (τ_n) and to have the values (b_n) shown in Table C.1. Table C.1 presents all the pertinent facts as well as the values of a_n derived from them. These values are used in Eq. (C.1) [or Eq. (1)] to determine D, as illustrated below for the seventh leaching interval:

$$D_7 = \left(\frac{3.14\ldots}{4} \right) \left(\frac{7.14 \times 10^{-2}}{15} \right)^2 \left(\frac{0.785}{4.71} \right)^2 \left[\frac{(4.32 \times 10^5)^{1/2} + (3.46 \times 10^5)^{1/2}}{4.32 \times 10^5 - 3.46 \times 10^5} \right]^2$$

or

$$D_7 = 1.0 \times 10^{-10} \text{ cm}^2/\text{s} \quad .$$

Thus, $\log(\beta/D_7) = L_7 = 10.0$.

In this illustration, the total activity leached (corrected to the beginning of the leach test) amounts to 18.8% $[(\sum a/A_O)(100)]$. Thus, Eq. (C.1) is applicable over the ten leach intervals. However, it must be kept in mind that when the total amount leached exceeds 20%, other methods to calculate D must be used for this and subsequent intervals (see 3.3). For example, if the amount leached in each interval were five times more than shown in Table C.1 and all other parameters $[(A_O)_\tau$, d, P,

t_n, τ_n, V_L, and v_a] were the same, the numbers given in Columns 1, 2, 3, 5, and 6 would be the same. However, the numbers given in Columns, 4, 7, and 8 would be five times larger. In other words, the total amount leached (corrected for radioactive decay) would exceed 20% after the sixth leaching interval. That is, the total amount leached at the end of the seventh leaching interval would be $(5)(4.44 \times 10^{-2})(100) = 22.2\%$. Thus, for the seventh and subsequent intervals in this case, Table A.1, Fig. 3, or an equivalent solution for a finite specimen must be used to determine a value for D. As a specific illustration (first with Table A.1 and then with Fig. 3), consider the tenth leaching interval for which the cumulative fraction leached is $F_{10} = (5)(1.88 \times 10^{-1}) = 0 94$.

From Table A.1 with $F_{10} = 0.94$ and $\ell/d = 1.0$, the value read for G is 6.77×10^{-2}. Therefore,

$$D_{10} = \frac{Gd^2}{t} = \frac{(6.77 \times 10^{-2})(1)^2}{7.69 \times 10^6}$$

or

$$D_{10} = 8.80 \times 10^{-9} \text{ cm}^2/\text{s} \quad .$$

Thus, $\log(\beta/D_{10}) = L_{10} = 8.1$.

From Fig. 3 with $F_{10} = 0.94$ and $\ell/d = 1.0$, the value read for Z is ~1.6. Therefore,

$$D_{10} = Z^2 \left(\frac{S}{V}\right)^2 t^{-1} \quad ,$$

$$D_{10} = (1.6)^2 \left(\frac{0.785}{4.71}\right)^2 (7.69 \times 10^6)^{-1} \quad ,$$

or

$$D_{10} = 9.25 \times 10^{-9} \text{ cm}^2/\text{s} \quad .$$

Thus, $\log(\beta/D_{10}) = L_{10} = 8.0$. As mentioned in 3.4 when the graphical method (or interpolation) is necessary to determine the value of D, there may be a slight variation in the decimal value of L (see 8.1 and 8.0).

Table C.1 – Illustrative leach results

1	2	3	4	5	6	7[1]	8[2]
Leach Interval (n)	$t_n = \sum(\Delta t)_n$ (s)	τ_n (s)	b_n (mCi)	$\frac{V_l}{v_a}$	$e^{-\lambda\tau}$	$(a_n)_\tau = b_n\left(\frac{V_\ell}{v_a}\right)e^{\lambda\tau}$ (mCi)	$\sum a_n$ $F = A_O$
1	7.20×10^3	9.36×10^4	9.06×10^{-3}	9.42	1.01	8.62×10^{-2}	5.75×10^{-3}
2	2.52×10^4	1.12×10^5	7.86×10^{-3}	9.42	1.01	7.48×10^{-2}	1.07×10^{-2}
3	8.64×10^4	1.72×10^5	1.43×10^{-2}	9.42	1.02	1.37×10^{-1}	1.99×10^{-2}
4	1.73×10^5	2.59×10^5	1.27×10^{-2}	9.42	1.03	1.23×10^{-1}	2.81×10^{-2}
5	2.59×10^5	3.45×10^5	9.66×10^{-3}	9.42	1.04	9.46×10^{-2}	3.44×10^{-2}
6	3.46×10^5	4.32×10^5	8.04×10^{-3}	9.42	1.05	7.95×10^{-2}	3.97×10^{-2}
7	4.30×10^5	5.18×10^5	7.08×10^{-3}	9.42	1.07	7.14×10^{-2}	4.44×10^{-2}
8	1.64×10^6	1.73×10^6	5.43×10^{-2}	9.42	1.24	6.34×10^{-1}	8.67×10^{-2}
9	4.06×10^6	4.15×10^6	4.72×10^{-2}	9.42	1.67	7.43×10^{-1}	1.36×10^{-1}
10	7.69×10^6	7.78×10^6	3.13×10^{-2}	9.42	2.61	7.70×10^{-1}	1.88×10^{-1}

NOTE: Specimen is assumed to be a right circular cylinder with $\ell = 1.0$ cm and $d = 1.0$ cm. The radionuclide being leached is taken to be ^{85}Sr with a half-life of 65 days. The total amount of ^{85}Sr in the specimen at the reference time is 15 mCi $[(A_O)_\tau]$. For each leach interval, the value of V_ℓ is 47.1 cm^3, and the value of v_a is 5 cm^3. The radioactivity in the 30-s rinse is negligible [i.e., the B in Eq. (C.2) is nil].

[1] Algebraic manipulation of the exponents of e for any consistent time correction (namely, of the aliquots counted and of the activity originally in the specimen) will lead to the relationship

$$\frac{(a_n)_\tau}{(A_o)_\tau} = \frac{a_n}{A_o} \; .$$

Thus, either may be used in Eq. (C.1) [or Eq. (1)].

[2] Treatment of the exponents of e as mentioned in footnote 1 will lead to the relationship

$$\frac{\sum(a_n)_\tau}{(A_o)_\tau} = \frac{\sum a_n}{A_o} \; .$$

Thus, either may be used with Table A.1 or Fig. 3.

Annex D

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Formulas for simulated seawater

Formulas for simulated seawater recommended by the International Organization for Standardization (ISO) Standard "Long-Term Leach Testing of Radioactive Waste Forms," identified as ISO 1691 1982(E), are listed in Table D.1.

Table D.1 – Relative compositions for simulated seawater

Compound	Grams of Compound[1]		
	I	II	III
NaCl	23.497	23.538	23.538
$MgCl_2$	4.981	4.985	5.934
Na_2SO_4	3.917	4.087	4.087
$CaCl_2$	1.102	1.108	—
KCl	0.664	0.665	0.666
$NaHCO_3$	0.192	—	—
KBr	0.096	0.096	0.096
Subtotal	34.449	34.479	34.321
H_2O	965.551	965.521	965.679
Total	1000.000	1000.000	1000.000

[1] Masses are listed to three decimal places to ensure summing to 1000 grams in Table D.1. Practically, weighing to three significant figures is usually considered acceptable.

Annex E

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Generic studies

In this annex certain recommendations for generic studies involving solidified low-level radioactive wastes are presented not only to bring out some of the limitations inherent in any simple, short-term test but also to bring out some of the factors that must be considered in the planning of long-term generic studies. The Leachability Index defined in this standard is related to the leachability of the material of the waste form. It can serve both as a quality indicator and as a factor to relate limited, short-term results with results from exhaustive long-term generic studies obtained with the same type of solidified waste material. The standard test is extended to 3 months since several researchers have observed that some low-level waste forms (namely, some wastes incorporated in cement or asphalt) can undergo a dramatic increase in leachability after a few weeks to a couple of months. Apparently, this is caused by effects such as the swelling of spent resin beads and the hydration of salts (e.g., Na_2SO_4). The extended test requires leaching for 3 months in an effort to go beyond the point in time when such effects will be manifest (i.e., the sample will crumble or swell). The effects are to be reported if found (see Section 4, "Presentation and Analysis of Data"). In the absence of generic long-term data, the Leachability Index (L_i) may thus serve alone for intermediate-term estimates (see Annex B) relative to the leaching behavior of the material tested.

E.1 Generic studies considerations

Generic studies involve the investigation of specimens made of materials similar to that of the solid waste form to which the results are to be applied.

These materials are typically characterized by the type of waste solidified, the binder used, the relative amounts of waste and binder employed, and the curing process. Different radioactive wastes (e.g., ion-exchange resins, filter sludges, evaporator bottoms, decontamination liquors, organic solutions, etc.) as well as some high-specific radioactivity wastes, result in characteristically different solidified waste materials, even though the binder and the binder-to-waste ratios employed for solidification are the same.

Leachability is a material parameter, like porosity or color; it is specific for the material of the specimen tested, the chemical (or radiochemical) species leached, the leachant, the leaching conditions, and the leachant renewal schedule. Leachability studies therefore consider all these variables, and the results are strictly applicable only to cases where all these factors are the same within certain error ranges.

E.2 Objectives of generic studies

The primary objective of a generic studies program is to provide the backup information for estimating the long-term performance of a given type of solidified low-level radioactive waste material prepared by the process under consideration. This includes the development of detailed procedures for the preparation of specimens, procedures for the analysis of the leachates, and detailed procedures to carry out the standard leach test within the specifications given in this standard (e.g., leaching vessel design, method of leachant replacement, temperature control, leachant certification, etc.).

For the interpretation of the data obtained from a specific material by the standard leach test as summarized by the Leachability Index, the generic studies must provide information on the leaching characteristics of the type of solidified waste product over the long term and, in the process, elucidate the effective leaching mechanisms. They also must provide procedures for normalizing the results of the generic studies to the data from the standard leach test. Estimates can then be made as to the leaching behavior of the solidified waste material for which the Leachability Index values have been obtained. Procedures to extrapolate to different size and shape waste forms, based on the mass transfer mechanisms identified by the generic studies, should be developed.

In addition, however, the generic studies should investigate the long-term stability of the type of solidified waste prepared, so that estimates can be made for the “sound life” of the waste form under anticipated environmental conditions. The “sound life” is the period of time during which the solid waste form meets the specifications for all the applicable parameters. The parameters affecting long-term stability of a specific type product should be identified and the levels at which they become significant for the performance of the material determined.

A generic studies program dealing with materials prepared by a specific waste solidification process should thus include investigations of

(1) physical changes, due to exposure to the standard leach test conditions and typical disposal-site environments. These changes include such things as swelling, warping, crumbling, blistering, shrinking, fissuring, and cracking;

(2) chemical changes, due to continued curing, aging, air oxidation, hydrolysis, chemical interaction with leachants of different types (e.g., groundwater, seawater, or various brines, etc.), and exposure to radiation fields, and other factors;

(3) biodegradability, due to attack by bacteria, fungi, insects, rodents, etc.

Annex F

(This Annex is not a part of American National Standard for the Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS-16.1-2003, but is included for information purposes only.)

Chemical interpretation of results

F.1 Dissolution versus reaction or saturation

The most important question in deciding how to chemically interpret the results of this leach test is the following: "Is a chemical reaction required before the radioactive element is released into solution?" If no reaction is required except ionic dissociation of a molecule or salt upon dissolution, the constituent dissolving is a small part of the solid mass, and saturation is not reached, then the criteria of the mathematical model are met, and the effective diffusivity interpretation from Fick's law for the rate constant is satisfactory.

If a reaction is required for release, then the mathematical criteria are not met. The kinetics may appear to follow the same square root of time pattern, but the coefficient is no longer an effective diffusivity but is a diffusion-reaction constant (D/R). Such D/R constants will depend on the concentration of the reacting species in the leachant and upon the changed porosity and tortuosity of the solid after reaction. The concentration profile in the specimen is distorted also. There is a reaction front that moves into the specimen from its surface. Inside the front there is no release because there is no reaction.

The greatest confusion arises because most radioactive elements assume different forms in typical binder systems. Some of the forms of even one element are soluble and thus meet the mathematical criteria, some are reactable and thus do not meet the mathematical criteria, and yet other forms are totally inert to both solution and reaction in a particular leachant. This gives rise to abrupt transitions in the slope of the cumulative release versus square root of time graphs or to rising and falling patterns on release rate versus square root of time plots. Such plots become the starting point for generic studies and provide initial long-term release predictions for a specific radioactive species and binder system. For this reason, L from 7 days leaching may be used for quality assurance, but only L from 90 days or longer should be used for overall performance evaluation.

In addition, solution and reaction may be occurring simultaneously in any given test, and the results then represent a superposition of both effects. This is specifically the case where cementitious binders are used. Alkaline binders react with water to use up the available hydrogen ion and develop a layer of more porous structure on the surface. That reaction slows down as the hydrogen ion concentration disappears (pH rises) and ceases when the available acidity is used up. Meanwhile, the nonreactive dissolution processes continue unchanged until saturation is reached in the interior of the sample. This gives rise to leach rates that are sensitive to frequency of renewal of leachant (new hydrogen ion) and binder chemistry (acid neutralization capacity per unit volume of sample).

F.2 Physical changes

Physical disruption of the sample structure also modifies the interpretation of the rate constant from the test results. The disruption may be caused by porosity and tortuosity changes produced by a chemical reaction in a surface layer of the sample, or the disruption may be caused by an expansion of structure by osmotic pressures inside the sample structure, or the disruption may even be punctures of coatings either on the sample surface or on solid particles inside the sample. These disruptions may give rise to changes, sometimes abrupt, in the rate of release to solution of radioactive materials. Such rate changes may represent tortuosity decreases due to cracking. Separation of these rate changes from chemical mechanism rate changes requires careful microstructural investigations.

F.3 Leachant selection

If it is desirable to compare laboratory leach data to field performance, then the leachant used in the laboratory should have characteristics of chemical reaction and solubility similar to the leachant that is present in the field. Leachants reported in the literature in various research efforts are listed in Table G.1.

Table G.1 – Leachants reported by researchers using ANSI/ANS-16.1-2003 configuration

	Leachant	Simulation of What Field Condition?	Reference
d	Deionized water	Pure rain	ANSI/ANS-16.1-2003
e	Seawater[1)]	Deep sea disposal	ANSI/ANS-16.1-1986
f	Dilute nitric and sulfuric acids	Acid rain	
g	Acetic acid	Failed codisposal with domestic waste	U.S. Environmental Protection Agency research and Toxicity Characteristic Leach Procedure
h	Sulfate water	Sulfate groundwater	

[1)] See seawater recipe in Annex D.

Standard Test Method for Electronic Friction Cone and Piezocone Penetration Testing of Soils[1]

This standard is issued under the fixed designation D 5778; 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 procedure for determining the point resistance during penetration of a conical-shaped penetrometer as it is advanced into subsurface soils at a steady rate.

1.2 This test method is also used to determine the frictional resistance of a cylindrical sleeve located behind the conical point as it is advanced through subsurface soils at a steady rate.

1.3 This test method applies to friction-cone penetrometers of the electric and electronic type. Field tests using mechanical-type penetrometers are covered elsewhere by Test Method D 3441.

1.4 This test method can be used to determine porewater pressures developed during the penetration, thus termed piezocone. Porewater pressure dissipation, after a push, can also be monitored for correlation to time rate of consolidation and permeability.

1.5 Additional sensors, such as inclinometer, seismic geophones, resistivity, electrical conductivity, dielectric, and temperature sensors, may be included in the penetrometer to provide useful information. The use of an inclinometer is highly recommended since it will provide information on potentially damaging situations during the sounding process.

1.6 Cone penetration test data can be used to interpret subsurface stratigraphy, and through use of site specific correlations, they can provide data on engineering properties of soils intended for use in design and construction of earthworks and foundations for structures.

1.7 The values stated in SI units are to be regarded as standard. Within Section 13 on Calculations, SI units are considered the standard. Other commonly used units such as the inch-pound system are shown in brackets. The various data reported should be displayed in mutually compatible units as agreed to by the client or user. Cone tip projected area is commonly referred to in square centimetres for convenience. The values stated in each system are not equivalents; therefore, each system must be used independently of the other.

NOTE 1—This test method does not include hydraulic or pneumatic penetrometers. However, many of the procedural requirements herein could apply to those penetrometers. Also, offshore/marine CPT systems may have procedural differences because of the difficulties of testing in those environments (for example, tidal variations, salt water, waves). Mechanical CPT systems are covered under Test Method D 3441.

1.8 *This standard does not purport to address all of the safety concerns, 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:* [2]

- D 653 Terminology Relating to Soil, Rock, and Contained Fluids
- D 3441 Test Method for Mechanical Cone Penetration Tests of Soil
- D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
- E 4 Practices for Force Verification of Testing Machines

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions are in accordance with Terminology Convention (D 653).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent load transfer*—apparent resistance measured on either the cone or friction sleeve of an electronic cone penetrometer while that element is in a no-load condition but the other element is loaded. Apparent load transfer is the sum of cross talk, subtraction error, and mechanical load transfer.

3.2.2 *baseline*—a set of zero load readings, expressed in terms of apparent resistance, that are used as reference values during performance of testing and calibration.

3.2.3 *cone tip*—the conical point of a cone penetrometer on which the end bearing component of penetration resistance is developed. The cone has a 60° apex angle, a diameter of 35.7

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

Current edition approved Nov. 1, 2007. Published December 2007. Originally approved in 1995. Last previous edition approved in 2000 as D 5778 – 95 (2000).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

mm, and a corresponding projected (horizontal plane) surface area or cone base area of 10 cm^2. Also, enlarged cones of 43.7 mm diameter (base area = 15 cm^2) are utilized.

3.2.4 *cone penetration test*—a series of penetration readings performed at one location over the entire vertical depth when using a cone penetrometer. Also referred to as a cone sounding.

3.2.5 *cone penetrometer*—a penetrometer in which the leading end of the penetrometer tip is a conical point designed for penetrating soil and for measuring the end-bearing component of penetration resistance.

3.2.6 *cone resistance,* q_c—the measured end-bearing component of penetration resistance. The resistance to penetration developed on the cone is equal to the vertical force applied to the cone divided by the cone base area.

3.2.7 *corrected total cone resistance,* q_t—tip resistance corrected for water pressure acting behind the tip (see 13.2.1). Correction for water pressure requires measuring water pressures with a piezocone element positioned behind the tip at location u_2 (See section 3.2.26). The correction results in estimated total tip resistance, q_t.

3.2.8 *cross talk*—an apparent load transfer between the cone and the friction sleeve caused by interference between the separate signal channels.

3.2.9 *electronic cone penetrometer*—a friction cone penetrometer that uses force transducers, such as strain gauge load cells, built into a non-telescoping penetrometer tip for measuring, within the penetrometer tip, the components of penetration resistance.

3.2.10 *electronic piezocone penetrometer*—an electronic cone penetrometer equipped with a low volume fluid chamber, porous element, and pressure transducer for determination of porewater pressure at the porous element soil interface.

3.2.11 *end bearing resistance*—same as cone resistance or tip resistance, q_c.

3.2.12 *equilibrium pore water pressure,* u_0—at rest water pressure at depth of interest. Same as hydrostatic pressure (see Terminology D 653).

3.2.13 *excess pore water pressure,* Δu—the difference between porewater pressure measured as the penetration occurs (u), and estimated equilibrium porewater pressure (u_0), or: $\Delta u = (u - u_0)$. Excess porewater pressure can either be positive or negative for shoulder position filters.

3.2.14 *friction cone penetrometer*—a cone penetrometer with the capability of measuring the friction component of penetration resistance.

3.2.15 *friction ratio,* R_f—the ratio of friction sleeve resistance, f_s, to cone resistance, q_c, measured at where the middle of the friction sleeve and cone point are at the same depth, expressed as a percentage.

3.2.16 *friction reducer*—a narrow local protuberance on the outside of the push rod surface, placed at a certain distance above the penetrometer tip, that is provided to reduce the total side friction on the push rods and allow for greater penetration depths for a given push capacity.

3.2.17 *friction sleeve*—an isolated cylindrical sleeve section on a penetrometer tip upon which the friction component of penetration resistance develops. The friction sleeve has a surface area of 150 cm^2 for 10-cm^2 cone tips or 225 cm^2 for 15-cm^2 tips.

3.2.18 *friction sleeve resistance,* f_s—the friction component of penetration resistance developed on a friction sleeve, equal to the shear force applied to the friction sleeve divided by its surface area.

3.2.19 *FSO*—abbreviation for full-scale output. The output of an electronic force transducer when loaded to 100 % rated capacity.

3.2.20 *local side friction*—same as friction sleeve resistance, f_s (see 3.2.18).

3.2.21 *penetration resistance measuring system*—a measuring system that provides the means for transmitting information from the penetrometer tip and displaying the data at the surface where it can be seen or recorded.

3.2.22 *penetrometer*—an apparatus consisting of a series of cylindrical push rods with a terminal body (end section), called the penetrometer tip, and measuring devices for determination of the components of penetration resistance.

3.2.23 *penetrometer tip*—the terminal body (end section) of the penetrometer which contains the active elements that sense the components of penetration resistance. The penetrometer tip may include additional electronic instrumentation for signal conditioning and amplification.

3.2.24 *piezocone*—same as *electronic piezocone penetrometer* (see 3.2.10).

3.2.25 *piezocone porewater pressure, u*—fluid pressure measured using the piezocone penetration test.

3.2.26 *piezocone porewater pressure measurement location:* u_1, u_2, u_3—fluid pressure measured by the piezocone penetrometer at specific locations on the penetrometer as follows (**1**):[3] u_1—porous filter location on the midface or tip of the cone, u_2—porous filter location at the shoulder position behind the cone tip (standard location) and, u_3—porous filter location behind the friction sleeve.

3.2.27 *porewater pressure*—total porewater pressure magnitude measured during penetration (same as 3.2.25 above).

3.2.28 *porewater pressure ratio parameter,* B_q—the ratio of excess porewater pressure at the standard measurement location Δu_2, to corrected total cone resistance q_t, minus the total vertical overburden stress, σ_{vo} (see Eq 10).

3.2.29 *push rods*—the thick-walled tubes or rods used to advance the penetrometer tip.

3.2.30 *sleeve friction, sleeve, and friction resistance*—same as friction sleeve resistance.

3.2.31 *subtraction error*—an apparent load transfer from the cone to the friction sleeve of a subtraction type electronic cone penetrometer caused by minor voltage differences in response to load between the two strain element cells.

3.3 *Abbreviations:*

3.3.1 *CPT*—abbreviation for the cone penetration test.

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[3] The boldface numbers given in parentheses refer to a list of references at the end of the text.

3.3.2 *PCPT or CPTu*—abbreviation for piezocone penetration test (note: symbol “u” added for porewater pressure measurements).

3.3.3 *CPTù*—abbreviation for the piezocone penetration test with dissipation phases of porewater pressures (ù).

3.3.4 *SCPTu*—abbreviation for seismic piezocone test (includes one or more geophones to allow downhole geophysical wave velocity measurements).

3.3.5 *RCPTu*—abbreviation for resistivity piezocone (includes electrical conductivity or resistivity module).

4. Summary of Test Method

4.1 A penetrometer tip with a conical point having a 60° apex angle and a cone base area of 10 or 15 cm^2 is advanced through the soil at a constant rate of 20 mm/s. The force on the conical point (cone) required to penetrate the soil is measured by electrical methods, at a minimum of every 50 mm of penetration. Improved resolution may often be obtained at 20- or 10-mm interval readings. Stress is calculated by dividing the measured force (total cone force) by the cone base area to obtain cone resistance, q_c.

4.2 A friction sleeve is present on the penetrometer immediately behind the cone tip, and the force exerted on the friction sleeve is measured by electrical methods at a minimum of every 50 mm of penetration. Stress is calculated by dividing the measured axial force by the surface area of the friction sleeve to determine sleeve resistance, f_s.

4.3 Most modern penetrometers are capable of registering pore water pressure induced during advancement of the penetrometer tip using an electronic pressure transducer. These penetrometers are called “piezocones.” The piezocone is advanced at a rate of 20 mm/s, and readings are taken at a minimum of every 50 mm of penetration. The dissipation of either positive or negative excess porewater pressure can be monitored by stopping penetration, unloading the push rod, and recording porewater pressure as a function of time. When porewater pressure becomes constant it is measuring the equilibrium value (designated u_0) or piezometric level at that depth.

5. Significance and Use

5.1 Tests performed using this test method provide a detailed record of cone resistance which is useful for evaluation of site stratigraphy, homogeneity and depth to firm layers, voids or cavities, and other discontinuities. The use of a friction sleeve and porewater pressure element can provide an estimate of soil classification, and correlations with engineering properties of soils. When properly performed at suitable sites, the test provides a rapid means for determining subsurface conditions.

5.2 This test method provides data used for estimating engineering properties of soil intended to help with the design and construction of earthworks, the foundations for structures, and the behavior of soils under static and dynamic loads.

5.3 This method tests the soil in-situ and soil samples are not obtained. The interpretation of the results from this test method provides estimates of the types of soil penetrated. Engineers may obtain soil samples from parallel borings for correlation purposes but prior information or experience may preclude the need for borings.

6. Interferences

6.1 Refusal, deflection, or damage to the penetrometer may occur in coarse grained soil deposits with maximum particle sizes that approach or exceed the diameter of the cone.

6.2 Partially lithified and lithified deposits may cause refusal, deflection, or damage to the penetrometer.

6.3 Standard push rods can be damaged or broken under extreme loadings. The amount of force that push rods are able to sustain is a function of the unrestrained length of the rods and the weak links in the push rod-penetrometer tip string such as push rod joints and push rod-penetrometer tip connections. The force at which rods may break is a function of the equipment configuration and ground conditions during penetration. Excessive rod deflection is the most common cause for rod breakage.

7. Apparatus

7.1 *Friction Cone Penetrometer*—The penetrometer tip should meet requirements as given below and in 10.1. In a conventional friction-type cone penetrometer, the forces at the cone tip and friction sleeve are measured by two load cells within the penetrometer. Either independent load cells or subtraction-type penetrometers are acceptable for use (Fig. 1).

7.1.1 In the subtraction-type penetrometer, the cone and sleeve both produce compressive forces on the load cells. The load cells are joined together in such a manner that the cell nearest the cone (the “C” cell in Fig. 1*b*) measures the compressive force on the cone while the second cell (the “C + S” cell in Fig. 1*b*) measures the sum of the compressive forces on both the cone and friction sleeve. The compressive force from the friction sleeve portion is computed then by subtraction. This cone design is common in industry because of its rugged design. This design forms the basis for minimum performance requirements for electronic penetrometers.

7.1.1.1 Alternative designs have separate and non-dependent load cells separate for tip and sleeve. For instance, in Fig. 1*a*, the cone penetrometer tip produces a compression force on the cone load cell (the “C” cell in Fig. 1*a*) while the friction sleeve produces a tensile force on the independent friction sleeve load cell (the “S” cell). Designs are also available where both the tip and sleeve load cells are independent and operate in compression **(2)**. These penetrometer designs result in a higher degree of accuracy in friction sleeve measurement, however, may be more susceptible to damage under extreme loading conditions.

7.1.1.2 Typical general purpose cone penetrometers are manufactured to full scale outputs (FSO) equivalent to net loads of 10 to 20 tons. Often, weak soils are the most critical in an investigation program, and in some cases, very accurate friction sleeve data may be required. To gain better resolution, the FSO can be lowered or the independent type penetrometer design can be selected. A low FSO subtraction cone may provide more accurate data than a standard FSO independent type cone depending on such factors as system design and thermal compensation. If the FSO is lowered, this may place electrical components at risk if overloaded in stronger soils.

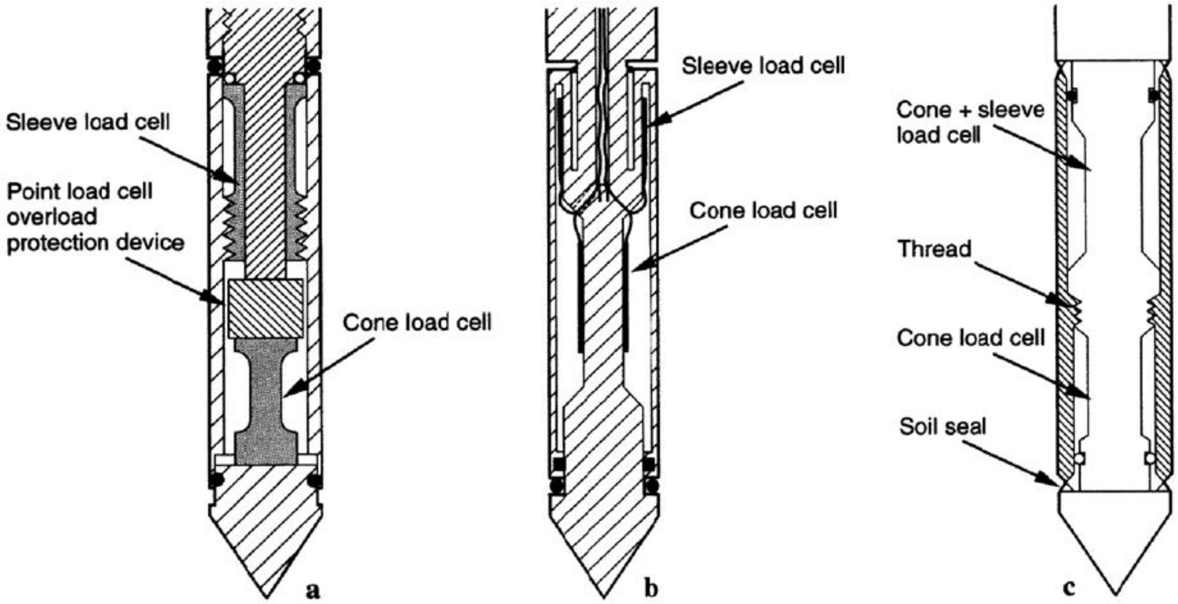

FIG. 1 Common Configurations for Electric Friction-Cone Penetrometers (2) Showing: (a) Compression-type Tip and Sleeve Load Cells, (b) Tension-type Sleeve Design, and (c) Subtraction-type Penetrometer

Expensive preboring efforts may be required to avoid damage in these cases. The selection of penetrometer type and resolution should consider such factors as practicality, availability, calibration requirements, cost, risk of damage, and preboring requirements.

7.1.1.3 The user or client should select the cone design requirements by consulting with experienced users or manufacturers. The need for a specific cone design depends on the design data requirements outlined in the exploration program.

7.1.1.4 Regardless of penetrometer type, the friction sleeve load cell system must operate in such a way that the system is sensitive to only shear stresses applied to the friction sleeve and not to normal stresses.

7.1.2 *Cone*—Nominal dimensions, with manufacturing and operating tolerances, for the cone are shown on Fig. 2. The cone has a diameter d = 35.7 mm, projected base area $A_c = 1000$ mm^2, + 2 %–5 % with an apex angle of 60°. A cylindrical extension, h_e, of 5 mm should be located behind the base of the cone to protect the outer edges of the cone base from excessive wear. The 10 cm^2 cone is considered the reference standard for which results of other penetrometers with proportionally scaled dimensions can be compared.

7.1.2.1 In certain cases, it may be desirable to increase the cone diameter in order to add room for sensors or increase ruggedness of the penetrometer. The standard increase is to a base diameter of 43.7 mm which provides a projected cone base area of 1500 mm^2 while maintaining a 60° apex angle. Nominal dimensions, with manufacturing and operating tolerances for the 15 cm^2 cone, are shown in Fig. 2, based on the international guides **(3)**.

7.1.2.2 The cone is made of high strength steel of a type and hardness suitable to resist wear due to abrasion by soil. Cone tips which have worn to the operating tolerance shown in Fig. 2 should be replaced. Piezocone tips should be replaced when the tip has worn appreciably (as shown) and the height of the cylindrical extension has reduced considerably (as shown).

NOTE 2—In some applications it may be desirable to scale the cone diameter down to a smaller projected area. Cone penetrometers with 5 cm^2 projected area find use in the field applications and even smaller sizes (1 cm^2) are used in the laboratory for research purposes. These cones should be designed with dimensions scaled in direct proportion to standard 10-cm^2 penetrometers. In thinly layered soils, the diameter affects how accurately the layers may be sensed. Smaller diameter cones may sense thinner layers more accurately than larger cones. If there are questions as to the effect of scaling the penetrometer to either larger or smaller size, results can be compared in the field to the 10-cm^2 penetrometer for soils under consideration. This is because the 10-cm^2 cone is considered the reference penetrometer for field testing.

7.1.3 *Friction Sleeve*—The outside diameter of the manufactured friction sleeve and the operating diameter are equal to the diameter of the base of the cone with a tolerance of +0.35 mm and −0.0 mm. The friction sleeve is made from high strength steel of a type and hardness to resist wear due to abrasion by soil. Chrome-plated steel is not recommended due to differing frictional behavior. The surface area of the friction sleeve is 150 cm^2 ± 2 %, for a 10-cm^2 cone. If the cone base area is increased to 15 cm^2, as provided for in 7.1.2.1, the surface area of the friction sleeve should be adjusted proportionally, with the same length to diameter ratio as the 10-cm^2 cone. With the 15-cm^2 tip, a sleeve area of 225 cm^2 is similar in scale.

7.1.3.1 The top diameter of the sleeve must not be smaller than the bottom diameter or significantly lower sleeve resistance will occur. During testing, the top and bottom of the sleeve should be periodically checked for wear with a micrometer. Normally, the top of the sleeve will wear faster than the bottom.

7.1.3.2 Friction sleeves must be designed with equal end areas which are exposed to water pressures **(2, 3, 4, 5, 6)**. This will remove the tendency for unbalanced end forces to act on the sleeve. Sleeve design must be checked in accordance with A1.7 to ensure proper response.

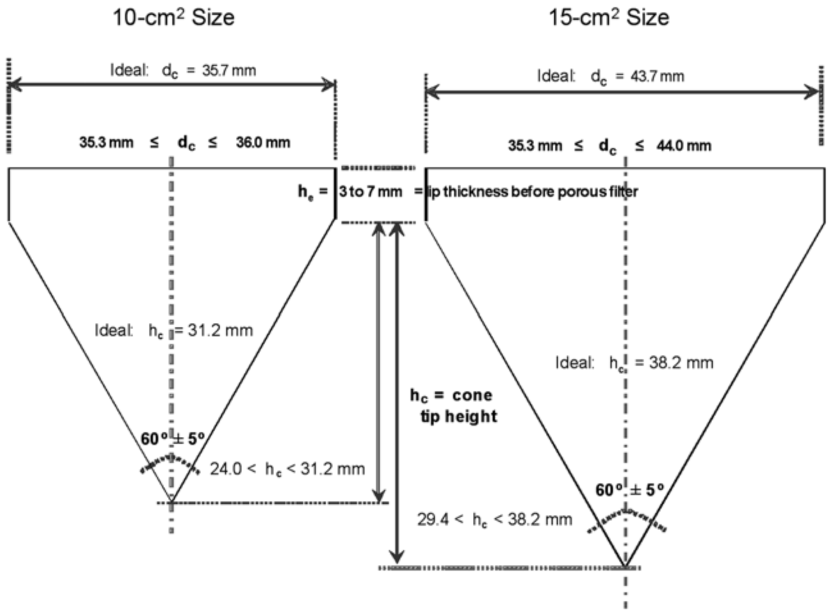

FIG. 2 Manufacturing and Operating Tolerances of Cones (3)

7.1.4 *Gap*—The gap (annular space) between the cylindrical extension of the cone base and the other elements of the penetrometer tip should be kept to the minimum necessary for operation of the sensing devices and should be designed and constructed in such a way to prevent the entry of soil particles. Gap requirements apply to the gaps at either end of the friction sleeve and to other elements of the penetrometer tip.

7.1.4.1 The gap between the cylindrical extension of the cone base and other elements of the penetrometer tip, e_c, must not be larger than 5 mm for the friction cone penetrometer.

7.1.4.2 If a seal is placed in the gap, it should be properly designed and manufactured to prevent entry of soil particles into the penetrometer tip. It must have a deformability at least two orders of magnitude greater than the material comprising the load transferring components of the sensing devices in order to prevent load transfer from the tip to the sleeve.

7.1.4.3 *Filter Element in the Gap*—If a filter element for a piezocone is placed in the gap between cone and sleeve the sum of the height of cylindrical extension, h_e, plus element thickness filling the gap, e_c, can range from 8 to 20 mm (see 7.1.8 for explanation).

7.1.5 *Diameter Requirements*—The friction sleeve should be situated within 5 to 15 mm behind the base of the cone tip. The annular spaces and seals between the friction sleeve and other portions of the penetrometer tip must conform to the same specifications as described in 7.1.4. Changes in the diameter of the penetrometer body above the friction sleeve should be such that tip or sleeve measurements are not influenced by increases in diameter. International reference test procedures require that the penetrometer body have the same diameter as the cone for the complete length of the penetrometer body **(3, 7, 8)**.

7.1.5.1 For some penetrometer designs, it may be desirable to increase the diameter of the penetrometer body to house additional sensors or reduce friction along push rods. These diameter changes are acceptable if they do not have significant influence on tip and sleeve data. If there is question regarding a specific design with diameter increases, comparison studies can be made to a penetrometer with constant diameter. Information on diameters of the complete penetrometer body should be reported.

NOTE 3—The effects caused by diameter changes of the penetrometer on tip and sleeve resistance are dependent on the magnitude of diameter increase and location on the penetrometer body. Most practitioners feel that diameter increases equivalent to addition of a friction reducer with area increases of 15 to 20 % should be restricted to a location at least eight to ten cone diameters behind the friction sleeve.

7.1.6 The axis of the cone, the friction sleeve (if included), and the body of the penetrometer tip must be coincident.

7.1.7 *Force Sensing Devices*—The typical force sensing device is a strain gauge load cell that contains temperature compensated bonded strain gages. The configuration and location of strain gages should be such that measurements are not influenced by possible eccentricity of loading.

7.1.8 *Electronic Piezocone Penetrometer*—A piezocone penetrometer can contain porous filter element(s), pressure transducer(s), and fluid filled ports connecting the elements to the transducer to measure pore water pressure. Fig. 3 shows the common design types used in practice including: 10-cm^2 friction-type, type 1 and type 2 piezocone, and 15-cm^2 size. The standard penetrometer should be the type 2 piezocone with filter located at the shoulder (both 10-cm^2 and 15-cm^2) to allow correction of tip resistances. The electric friction penetrometer without porewater transducers can be used in soils with minor porewater pressure development, such as clean sands, granular soils, as well as soils and fills well above the groundwater table. The type 1 with face filter element finds use in fissured geomaterials and materials prone to desaturation, as well as dissipation readings. Numerous design and configuration aspects can affect the measurement of dynamic water pressures. Variables such as the element location, design and volume of ports, and the type and degree of saturation of the fluids,

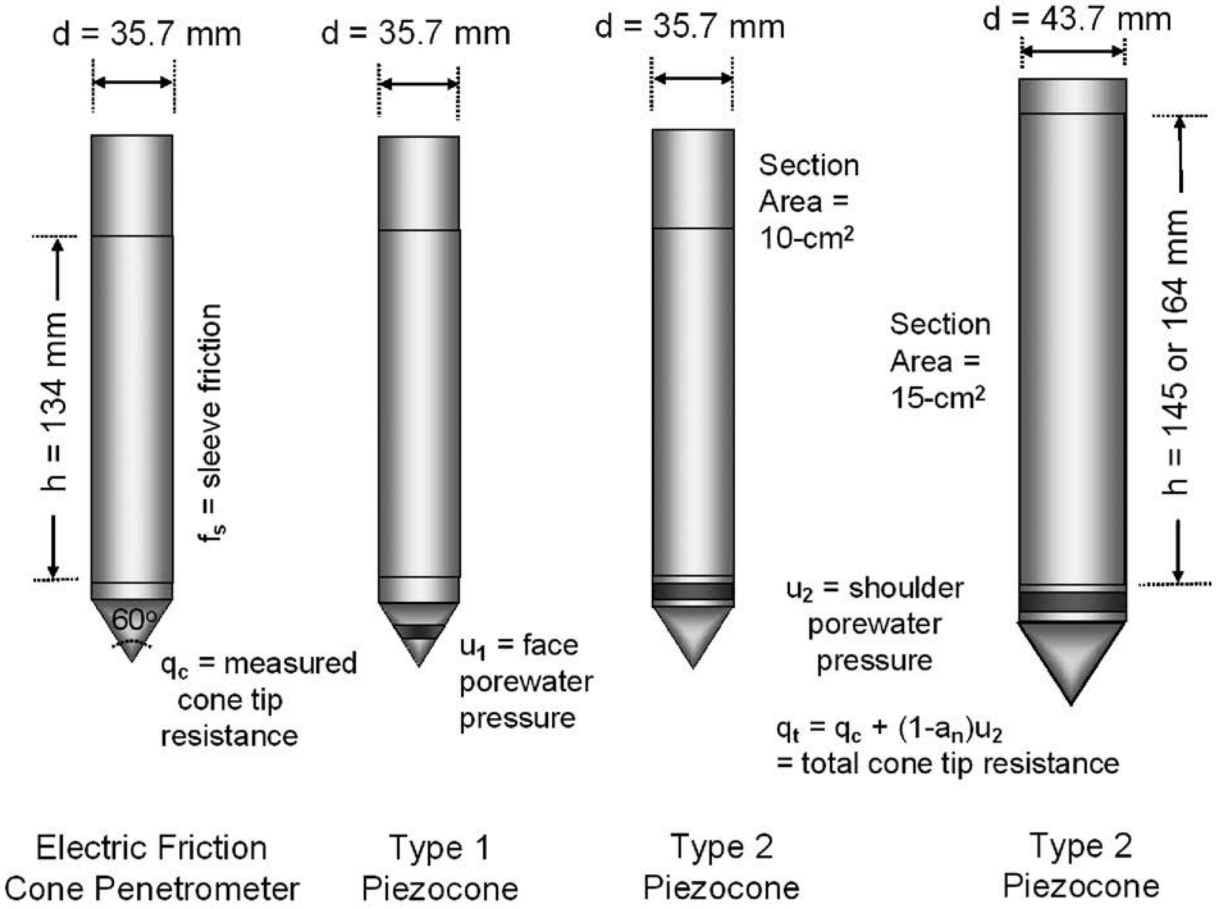

FIG. 3 Penetrometer Design Configurations: (a) Electronic Friction-type, (b) Type 1 Piezocone, (c) Standard 10-cm^2 Type 2 Piezocone, and (d) 15-cm^2 Type 2 Version (5)

cavitation of the element fluid system and resaturation lag time, depth and saturation of soil during testing all affect the dynamic porewater pressure measured during testing and dissipation tests of dynamic pressures (**1**, **6**). It is beyond the scope of the procedure to address all of these variables. As a minimum, complete information should be reported as to the design, configuration, and the preparation of the piezocone system that is used for the particular sounding.

7.1.8.1 Measurement of hydrostatic water pressures during pauses in testing are more straightforward. The presence of air entrained in the system only affects dynamic response. In high permeability soils (that is, clean sands), hydrostatic pressures will equalize within seconds or minutes. In low permeability materials such as high plasticity clays, equalization can take many hours. If the goal of the exploration program is only to acquire hydrostatic pressures in sands, some of the preparation procedures for dynamic pressure measuring can be relaxed, such as deairing fluids.

7.1.8.2 The porewater pressure measurement locations of the porous element are limited to the face or tip of the cone, u_1, directly behind the cylindrical extension of the base of the cone, u_2, or behind the sleeve, u_3. Some penetrometers used for research purposes may have multiple measurement locations.

7.1.8.3 There are several advantages to locating the porous element immediately behind the tip of the cone in location u_2, primarily the required correction of measured q_c to total tip stress, q_t, as detailed extensively (**1-6**). Also, the element is less subject to damage and abrasion, as well as fewer compressibility effects (**1**, **6**). Elements located in the u_2 location may be subject to cavitation at shallow depths in dense sands because the zone behind the height of cylindrical extension is a zone of dilation in drained soils. Similar response can occur in stiff fissured clays and crusts (**1**). Porewater pressure measurements obtained at the u_1 face location are more effective for compressibility determinations and layer detection, particularly in fissured soils, but are more subject to wear (**9**). At the u_2 location, a minimum 2-mm cylindrical extension of the cone tip (h_e) should be maintained for protection of the cone. Typical filter element thickness at all locations in the horizontal plane ranges from 5 to 10 mm.

7.1.8.4 The miniature diaphragm-type electronic pressure transducer is normally housed near the tip of the cone. For dynamic pressure measurements, the filter and ports are filled with deaired fluid to measure dynamic porewater pressure response. The volume of connecting ports to the transducer should be minimized to facilitate dynamic pressure response. These electronic transducers are normally very reliable, accurate, and linear in response. The transducer shall have a precision of at least ±14 kPa (±2 psi). The porewater pressure transducer must meet requirements given in 10.2.

7.1.8.5 *Element*—The element is a fine porous filter made from plastic, sintered steel or bronze, or ceramic. Typical pore size is between 20 to 200 microns **(6, 9)**. Different materials have different advantages. Smearing of metallic element openings by hard soil grains may reduce dynamic response of the system, thus normally not used for face elements but best suited for shoulder filter positions. Ceramic elements are very brittle and may crack when loaded, but perform well on the cone face as they reduce compressibility concerns. Polypropylene plastic elements are most commonly used in practice, particularly at the shoulder. Plastic filters (as high-density polyethylene, HDPE, or high-density polypropylene, HDPP) may be inappropriate for environmental type CPTs where contaminant detection is sought. Typically, the filter element is wedged at the tip or midface (u_1) location, or located at the shoulder in the gap immediately above the cone extension (designated u_2) location. At these locations, it is important to design the penetrometer such that compression of the filter elements is minimized.

7.1.8.6 *Fluids for Saturation*—Glycerine, or alternatively silicone oil, is most often used for deairing elements for dynamic response. These stiff viscous oils have less tendency to cavitate, although cavitation may be controlled by the effective pore size of the element mounting surfaces. Water can be used for the fluid if the entire sounding will be submerged, or if dynamic response is not important. The fluids are deaired using procedures described in 11.2.

7.2 *Measuring System*—The signals from the penetrometer transducers are to be displayed at the surface during testing as a continuously updated plot against depth. The data are also to be recorded electronically for subsequent processing. Electronic recording shall be digital and use at least twelve bit (one part in 4096) resolution in the analog to digital conversion, although 16-bit resolution and higher may be preferable in very soft ground. Either magnetic (disk or tape) or optical (disk) non-volatile storage may be used. In analog systems, the temperature stability and accuracy of the A-to-D converter shall be such that the overall cone-transmission-recording system complies with calibration requirements set forth in the annex.

7.2.1 Use of analog systems is acceptable but the system resolution may be lower than requirements in the annex and Section 10. Use of an analog recorder as a supplement to digital system is advantageous because it can provide system backup.

NOTE 4—Depending upon the equipment, data stored digitally on magnetic drives, tapes, floppy disks, or other media are often used. The data files should include project, location, operator, and data format information (for example, channel, units, corrected or uncorrected, etc.) so that the data can be understood when reading the file with a text editor.

7.3 *Push Rods*—Steel rods are required having a cross sectional area adequate to sustain, without buckling, the thrust required to advance the penetrometer tip. For penetrometers using electrical cables, the cable is prestrung through the rods prior to testing. Push rods are supplied in 1-meter lengths. The push rods must be secured together to bear against each other at the joints and form a rigid-jointed string of push rods. The deviation of push rod alignment from a straight axis should be held to a minimum, especially in the push rods near the penetrometer tip, to avoid excessive directional penetrometer drift. Generally, when a 1-m long push rod is subjected to a permanent circular bending resulting in 1 to 2 mm of center axis rod shortening, the push rod should be discarded. This corresponds to a horizontal deflection of 2 to 3 mm at the center of bending. The locations of push rods in the string should be varied periodically to avoid permanent curvature.

7.3.1 For the 10-cm^2 penetrometer, standard 20-metric ton high tensile strength steel push rods are 36-mm outside diameter, 16-mm inside diameter, and have a mass per unit length of 6.65 kg/m. For 15-cm^2 penetrometers, the test may be pushed with 44.5-mm outside diameter rods or with standard rods used for the 10-cm^2 penetrometer.

7.4 *Friction Reducer*—Friction reducers are normally used on the push rods to reduce rod friction. If a friction reducer is used, it should be located on the push rods no closer than 0.5 m behind the base of the cone. Friction reducers, that increase push rod outside diameter by approximately 25 %, are typically used for 10-cm^2 cones. If a 15-cm^2 penetrometer is advanced with 36-mm push rods there may be no need for friction reducers since the penetrometer itself will open a larger hole. The type, size, amount, and location of friction reducer(s) used during testing must be reported.

7.5 *Thrust Machine and Reaction*—The thrust machine will provide a continuous stroke, preferably over a distance greater than 1 m. The thrust machine should be capable of adjusting push direction through the use of a leveling system such that push initiates in a vertical orientation. The machine must advance the penetrometer tip and push rods at a smooth, constant rate (see 12.1.2) while the magnitude of thrust can fluctuate. The thrust machine must be anchored or ballasted, or both, so that it provides the necessary reaction for the penetrometer and does not move relative to the soil surface during thrust.

NOTE 5—Cone penetration soundings usually require thrust capabilities ranging from 100 to 200 kN (11 to 22 tons) for full capacity. High mass ballasted vehicles can cause soil surface deformations which may affect penetrometer resistance(s) measured in near surface layers. Anchored or ballasted vehicles, or both, may induce changes in ground surface reference level. If these conditions are evident, they should be noted in reports.

7.6 *Other Sensing Devices*—Other sensing devices can be included in the penetrometer body to provide additional information during the sounding. These instruments are normally read at the same continuous rate as tip, sleeve, and porewater pressure sensors, or alternatively, during pauses in the push (often at 1-m rod breaks). Typical sensors are inclinometer, temperature, resistivity (or its reciprocal, electrical conductivity), or seismic sensors, such as geophones that can be used to obtain downhole shear wave velocity. These sensors should be calibrated if their use is critical to the investigation program. The use of an inclinometer is highly recommended since it will provide information on potentially damaging situations during the sounding process. An inclinometer can provide a useful depth reliability check because it provides information on verticality. The configuration and methods of operating such sensors should be reported.

8. Reagents and Materials

8.1 *O-Ring Compound*—A petroleum or silicon compound for facilitating seals with O-rings. Use of silicon compounds may impede repair of strain gages if the strain gauge surface is exposed to the compound.

8.2 *Glycerine*, or $CHOH(CH_2OH)_2$, for use in porewater pressure measurement systems. Approximately 95 % pure glycerine can be procured from most drug stores.

8.3 *Silicone Oil (or fluid)*, for use in porewater pressure measurement systems. This material is available in varying viscosities ranging from 1400 to 10 000 CP.

NOTE 6—Detailed comparisons and discussions on the use of these fluids can be found elsewhere (**6**, **9**).

9. Hazards

9.1 *Technical Precautions—General*:

9.1.1 Use of penetrometer components that do not meet required tolerances or show visible signs of non-symmetric wear can result in erroneous penetration resistance data.

9.1.2 The application of thrust in excess of rated capacity of the equipment can result in damage to equipment (see Section 6).

9.1.3 A cone sounding must not be performed any closer than 25 borehole diameters from any existing unbackfilled or uncased bore hole.

9.1.4 When performing cone penetration testing in prebored holes, an estimate of the depth below the prebored depth which is disturbed by drilling, should be made and penetration resistance data obtained in this zone should be noted. Usually, this depth of disturbance is assumed to be equal to at least three borehole diameters.

9.1.5 Significant bending of the push rods can influence penetration resistance data. The use of a tubular rod guide is recommended at the base of the thrust machine and also in prebored holes to help prevent push rod bending.

9.1.6 Push rods not meeting requirements of 7.3 may result in excessive directional penetrometer drift and possibly unreliable penetration resistance values.

9.1.7 Passing through or alongside obstructions may deflect the penetrometer and induce directional drift. Note any indications of encountering such obstructions, such as gravels, and be alert for possible subsequent improper penetrometer tip operation.

9.1.8 If the proper rate of advance of the penetrometer is not maintained for the entire stroke through the measurement interval, penetration resistance data will be erroneous.

9.2 *Technical Precautions—Electronic Friction Cone Penetrometer*:

9.2.1 Failure of O-ring seals can result in damage to or inaccurate readings from electronic transducers. The O-ring seals should be inspected regularly, after each sounding, for overall condition, cleanliness and watertightness.

9.2.2 Soil ingress between different elements of a penetrometer tip can result in unreliable data. Specifically, soil ingress will detrimentally affect sleeve resistance data. Seals should be inspected after each sounding, maintained regularly, and replaced when necessary. If very accurate sleeve resistance data is required, it is recommended to clean all seals after each sounding.

9.2.3 Electronic cone penetrometer tips should be temperature compensated. If extreme temperatures outside of the range established in A1.3.3 are to be encountered, the penetrometer should be checked for the required temperature range to establish they can meet the calibration requirements. Also, harsh environments may severely affect the data acquisition system of power supplies, notebook or field computers, and other electronics.

9.2.4 If the shift in baseline reading after extracting the penetrometer tip from the soil is so large that the conditions of accuracy as defined in 10.1.2.1 are no longer met, penetration resistance data should be noted as unreliable. If baseline readings do not conform to allowable limits established by accuracy requirements in 10.1.2.1, the penetrometer tip must be repaired, and recalibrated or replaced.

9.2.5 Electronic friction cone penetrometers having unequal end areas on their friction sleeves can yield erroneous f_s readings because of dynamic porewater pressures acting unevenly on the sleeve (**2**, **3**, **4**, **6**). Friction sleeve design should be checked in accordance with A1.7 to ensure balanced response. The response is also dependent on location of water seals. If O-ring water seals are damaged during testing, and sleeve data appear affected, the sounding data should be noted as unreliable and the seals should be repaired.

9.3 *Piezocone Penetrometer*—The electronic piezocone penetrometer tip measures pore water pressures on the exterior of the penetrometer tip by transferring the pressure through a de-aired fluid system to a pressure transducer in the interior of the tip. For proper dynamic response, the measurement system (consisting of fluid ports and porous element) must be completely saturated prior to testing. Entrained air must be removed from the fluid-filled system or porewater pressure fluctuation during penetrometer tip advancement will be incorrect due to response lag from compression of air bubbles (see 11.2, 12.3.2, and 12.3.3). For soundings where dynamic response is important, the prepared filter elements should be replaced after every sounding.

10. Calibration and Standardization

10.1 *Electronic Friction Cone Penetrometers*:

10.1.1 The requirements for newly manufactured or repaired cone penetrometers are of importance. Newly manufactured or repaired electronic cone penetrometers are to be checked to meet the minimum calibration requirements described in the annex. These calibrations include load tests, thermal tests, and mechanical tests for effects of imbalanced hydrostatic forces. Calibration procedures and requirements given in the annex are for subtraction-type cone penetrometers. Calibration requirements for independent-type cone penetrometers should equal or exceed those requirements. The calibration records must be certified as correct by a registered professional engineer or other responsible engineer with knowledge and experience in materials testing for quality assurance. Applied forces or masses must be traceable to calibration standard forces or masses retained by the National Institute of Standards and Technology (NIST), formerly the

National Bureau of Standards. For description of calibration terms and methods for calibrating, refer to the annex.

10.1.2 *Baseline Readings*—Baseline or zero-load readings for both cone and friction sleeve load cells and porewater pressure transducers must be taken before and after each sounding. The baseline reading is a reliable indicator of output stability, temperature-induced apparent load, soil ingress, internal friction, threshold sensitivity, and unknown loading during zero setting. Take the initial baseline reading after warming electrical circuits according to the manufacturer's instructions, generally for 15 to 30 min, and in a temperature environment as close as possible to that of the material to be sounded. If temperature is of concern, immerse the penetrometer tip in a bucket of fresh tap water, or insert the penetrometer tip in the ground while electrically warming circuits to stabilize its temperature and then extracted for rapid determination of initial baseline. After a sounding is completed, take a final baseline. The change in initial and final baseline values should not exceed 2 % FSO for the cone tip, sleeve, and pressure transducer.

10.1.2.1 Maintain a continuous record of initial and final baselines during production testing. After each sounding, compare the final baseline to the initial baseline for agreement within the tolerances noted above. In some cases during heavy production testing where the cone is not disassembled and cleaned after each sounding, the initial baseline for the next sounding can serve as the final baseline to the previous sounding as long as agreement is within allowable limits.

10.1.2.2 If the post sounding baseline shift exceeds above criteria, inspect the cone for damage by inspecting the tip and checking to see that the sleeve can be rotated by hand. If there is apparent damage, replace parts as required. Clean the cone and allow temperatures to equalize to presounding conditions, and obtain a new baseline. If this value agrees with the initial baseline within the above criteria, a load range calibration check is not required. If the pre and post baselines are still not within the above criteria then it is likely that the shift was caused by an obstacle or obstruction and linearity should be checked with a load range calibration.

10.1.2.3 If the baseline shift still exceeds the above criteria, perform a load range calibration as described in 10.1.2.1. If the cone load cell baseline shift exceeds 2 % FSO, the cone is likely damaged and will not meet load range criteria in 10.1.2.2. Sleeve load cell baseline shifts for subtraction-type penetrometers usually can exceed 2 % FSO and still meet load range criteria.

10.1.2.4 Report data for the sounding where unacceptable baseline shift occurs as unreliable. In some cases it may be obvious where the damage occurred and data prior to that point may be considered reliable. The location where obvious damage occurred should be clearly noted in reports.

10.1.3 *Penetrometer Wear and Usage*

10.1.3.1 For penetrometers used regularly during production, periodic load range checks should be performed. The inspection period can be based on production footage such as once every lineal 3000 m (approx. 10^4 linear feet) of soundings. If field load range equipment is not available, the penetrometer may be checked in the laboratory at the end of a project.

10.1.3.2 For penetrometers that are used infrequently, a periodic check may be based on time period, such as once every year. If a penetrometer has not been used for a long period of time, checking it before use is advisable.

10.1.3.3 For projects requiring a high level of quality assurance, it may be required to do load range checks before and after the project.

10.1.3.4 Load range calibrations are required if the initial and final baselines for a sounding do not meet requirements given in 10.1.2.1.

10.1.3.5 Records documenting the history of an individual penetrometer should be maintained for evaluation of performance.

10.2 *Porewater Pressure Transducer*—Calibrate newly manufactured or repaired transducers in accordance with requirements in the annex. During production, the transducer should be calibrated at regularly scheduled intervals and whenever linear performance is suspect. The reference gauge can be a Bourdon tube pressure gauge, or electronic pressure transducer that is calibrated annually to NIST traceable loading device (dead weight testing apparatus).

10.2.1 Prior to testing, baseline values or initial zeroing of the transducer is performed on the porewater pressure transducer at ambient air pressures at the surface. Maintain records as to the baseline values for the transducer in similar fashion to those for tip and sleeve resistance. If significant changes in baseline values occur, normally 1 to 2 % FSO, perform load range tests to check for possible damage and nonlinear response.

10.3 *Calibrations of Other Sensing Devices*—Calibration data for other sensors in the penetrometer body may require calibrations using procedures similar to those given in the annex for load cells and pressure transducers. The need for calibration depends on the requirements of the individual investigation program. For noncritical programs, the occurrence of reasonable readings may be sufficient. In critical programs, it may be necessary to load the sensor through the range of interest with reference standards to ensure accurate readings.

11. Conditioning

11.1 Power electronic cone penetrometer and data acquisition systems for a minimum time period to stabilize electric circuits before performing soundings. Power the system to manufacturer's recommendations prior to obtaining reference baselines. For most electronic systems this time period is 15 to 30 min.

11.2 Electronic piezocone penetrometer soundings require special preparation of the transmitting fluid and porous elements such that entrained air is removed from the system. For soundings where dynamic response is important, replace the prepared filter elements and the ports flushed after every sounding. Some of the techniques discussed below have been successful for preparation of elements. Regardless of the techniques used, report the equipment and methods.

11.2.1 Field or laboratory tests can be performed to evaluate assembled system response, if desired. Place the cone tip and element in a pressurized chamber and subject to rapid pressure change. Compare the response of the system to the applied pressure changes and if responses match, the system is properly prepared.

11.2.2 Place elements in a pure glycerine or silicone oil bath under a vacuum of at least 90 % of one atmosphere (–90 kPa). Maintain vacuum until air bubble generation is reduced to a minimum. Application of ultrasonic vibration and low heat ($T < 50°C$) will assist in removal of air. Generally with use of combined vacuum, ultrasonic vibration, and low heat, filter elements can be deaired in about 4 h, although it is best to allow for 24 h to ensure best performance. Results will depend upon the viscosity of the fluid and pore size of the filter element.

11.2.3 Elements can be prepared in water by boiling the elements while submerged in water for at least 4 h, although damage may result from prolongued exposure in this approach **(1)**.

11.2.4 *Other Suitable Means*—Report other techniques, such as commercially-purchased pre-saturated filter elements that are available, or grease-filled slot **(2, 5)**.

11.2.5 *Storage*—Store prepared elements submerged in the prepared fluid until ready for use. Fill the containers and evacuate during storage. Allowable storage length depends on the fluid. If elements are prepared in water they must be deaired again one day after containers are opened and exposed to air. Elements stored in glycerine or silicone may be stored for longer periods, up to several months, after storage containers have been exposed to air.

12. Procedure

12.1 *General Requirements*:

12.1.1 Prior to beginning a sounding, perform site surveys to ensure hazards such as overhead and underground utilities will not be encountered. Position the thrust machine over the location of the sounding, and lower leveling jacks to raise the machine mass off the suspension system. Set the hydraulic rams of the penetrometer thrust system to as near vertical as possible. The axis of the push rods must coincide with the thrust direction.

12.1.2 Set the hydraulic ram feed rate to advance the penetrometer at a rate of 20 ± 5 mm/s for all electronic cone penetrometers. This rate must be maintained during the entire stroke during downward advance of the rods while taking readings.

12.1.3 Check push rods for straightness and permanent bending (See Section 7.3). Push rods are assembled and tightened by hand, but care must be taken and threads may need cleaning to ensure that the shoulders are tightly butted to prevent damage to the push rods. For electronic cone penetrometers using cables, the cable is prestrung through the push rods. Add friction reducer to the string of push rods as required, usually the first push rod behind the penetrometer tip and other rods as required.

12.1.4 Inspect penetrometer tips before and after soundings for damage, soil ingress, and wear. In very soft and sensitive soils where accurate sleeve data is required, dismantle electronic cone penetrometer tips and friction sleeves after each sounding to clean and lubricate as required. If damage is found after a sounding, note and record this information on the sounding data record or report.

12.2 *Friction Cone Penetrometers*:

12.2.1 Power up the penetrometer tip and data acquisition system according to the manufacturer's recommendations, typically 15 to 30 min, prior to use.

12.2.2 Obtain an initial baseline reading for the penetrometer in an unloaded condition at a temperature as close as possible to ground conditions. Obtain baseline readings with the penetrometer tip hanging freely in air or in water, out of direct sunlight. Compare baseline readings with the previous baseline reading for the requirements given in 10.1.2.1. If thermal stability needs to be assured, immerse the penetrometer tip in water at temperature close to ground; or perform an initial short penetration test hole, stop penetration and allow the penetrometer tip to reach soil temperature, and withdraw the penetrometer.

12.2.3 Measure the depth at which readings were taken with an accuracy of at least ±100 mm from the ground surface.

12.2.4 Determine the cone resistance and friction sleeve resistance, continuously with depth, and record the data at intervals of depth not exceeding 50 mm.

12.2.5 During the progress of sounding, monitor tip and sleeve forces continuously for signs of proper operations. It is helpful to monitor other indicators such as ram pressure or inclination to ensure that damage may not occur if highly resistant layers or obstructions are encountered. Inclination is a particularly useful indicator of imminent danger to the system (see 12.4).

12.2.6 At the end of a sounding, extract the penetrometer tip, obtain a final set of baseline readings with the penetrometer tip hanging freely in air or in water, and check them against the initial baseline. Record initial and final baselines on all documents related to the sounding.

12.3 *Electronic Piezocone Penetrometers*:

12.3.1 Power up the penetrometer tip and data acquisition system according to the manufacturer's recommendations, typically 15 to 30 min, prior to use.

12.3.2 Assemble the piezo elements with all fluid chambers submerged in the de-aired medium used to prepare the elements. Flush all confined areas with fluid to remove air bubbles. Tighten the cone tip to effectively seal the flat surfaces. For water fluid systems, protect the assembled system from evaporation by enclosing the porous element inside a fluid-filled plastic bag or cap sealed to the penetrometer tip.

12.3.3 If unsaturated soil is first penetrated and it is desired to obtain accurate dynamic porewater pressure response once below the ground water, it may be necessary to prebore or sound a pilot hole to the water table. In many cases, the piezocone fluid system may cavitate during penetration through unsaturated soil or in dilating sand layers below the water table and this can adversely affect dynamic response. As the cone is advanced deeper, the saturation levels may recover as air bubbles are driven back into solution according to Boyles Law. Evaluation of proper interpretation of dynamic response requires experience **(1, 6)**. Pre-punching or pre-boring with a two-level phase approach to soundings may help alleviate desaturation problems.

12.3.4 Record baseline readings with the penetrometer tip hanging freely in air, or in water, out of direct sunlight. Compare baseline readings with reference baseline readings for requirements given in 10.1.2.1 and 10.2. A baseline for the porewater pressure transducer is obtained immediately after assembly to avoid evaporation effects. If evaporation is a problem, temporarily immerse the penetrometer in a bucket of water until ready for baseline. Do not obtain transducer baselines with protective caps or covers in place as these may induce pressure in the system. Note the pressure from the pressure transducer to see if it is a reasonable value for the equipment and assembly technique used.

12.3.5 Follow procedures similar to electric friction cone in 12.2.4-12.2.6 with the addition of recording porewater pressure readings.

12.3.6 *Dissipation Tests*—If dissipation tests are to be conducted during progress of the sounding, penetration is temporarily stopped at the location of interest. If porewater pressures are measured at the u_2 or u_3 locations, it is common practice to release the force on the push rods. If porewater pressures are measured at the midface location u_1, maintain the force on the push rods. Record porewater pressure versus time during conduct of the dissipation test. Monitor pressures until equilibrium porewater pressure is reached or 50 % of the initial excess porewater pressure has dissipated. In fine grained soils of very low conductivity, very long times may be required to reach the 50 % dissipation. Depending on the requirements of the program, and any concern of friction buildup on the push rods, dissipation testing may be terminated prior to reaching the 50 % level. Report dissipation test data as a record of porewater pressure versus time, or more commonly, u versus logarithm of time.

12.3.7 *Hydrostatic Porewater Condition*:

If full dissipations are carried out, then the porewater transducer will eventually record the hydrostatic condition, thus providing an evaluation of the position of the groundwater table or phreatic surface.

12.4 *Penetrometer Operation and Data Interpretation-Guidelines*:

12.4.1 *Directional Drift of Penetrometer*:

12.4.1.1 The penetrometer may drift directionally from vertical alignment. Large deviations in inclination can create nonuniform loading and result in unreliable penetration resistance data. Reduce drift by accurately setting thrust alignment and using push rods which meet tolerances given in 7.3.

12.4.1.2 Passing through or alongside obstructions such as boulders, cobbles, coarse gravel, soil concretions, thin rock layers, or inclined dense layers will deflect the penetrometer tip and induce drifting. Note and record any indication of encountering such obstructions, and be alert for possible subsequent improper penetrometer tip operations as a sign of serious directional drift.

12.4.1.3 Penetrometer inclination is typically monitored in cone penetrometers. Impose limitations on inclination in the system to prevent damage to push rods and non-symmetric loading of the penetrometer tip. Generally, a 5° change in inclination over 1 m of penetration can impose detrimental push rod bending. Total drift of over 12° in 10 m of penetration imposes non-symetric loading and possible unreliable penetration resistance data.

12.4.2 *Push Rod Addition Interruptions*—Short duration interruptions in the penetration rate during addition of each new push rod can affect initial cone and friction sleeve readings at the beginning of the next push. If necessary, note and record the depths at which push rods are added and where long pauses may have affected initial startup resistances.

12.4.3 *Piezocone Porewater Pressure Dissipation Interruptions*—Porewater pressure dissipation studies, for which soundings are stopped and rod load is released for varying time durations, can affect the initial cone, friction sleeve, and dynamic porewater pressure readings at resumptions of cone penetration. If dissipation tests are performed, be aware of possible rebound effects on initial excess porewater pressures. Note and record the depth and duration for which dissipation values are taken.

12.4.4 *Interruptions Due to Obstructions*—If obstructions are encountered and normal advance of the sounding is stopped to bore through the obstructions, obtain further penetration resistance data only after the penetrometer tip has passed through the estimated zone of disturbance due to drilling. As an alternative, readings may be continued without first making the additional penetration and the disturbed zone evaluated from these data. Note and record the depth and thickness of obstructions and disturbed zones in areas where obstructions are drilled through.

12.4.5 *Excessive Thrust Capacity*—If excessive thrust pressure begins to impede the progress of the sounding, it may be necessary to withdraw and change friction reducers. Alternately, sometimes friction may be reduced by withdrawing the penetrometer and rods up to one third to one half of the penetration depth and then repushing to depth at which the friction caused stopping. Continue collection of sounding data from the point of stopping. Note and record the delay time and depths to which the penetrometer was moved. Long delays and pauses may cause buildup of friction on the rods. Hold delays to the minimum required to perform dissipation tests or equipment repairs.

12.4.5.1 If a high resistance layer is encountered, and the hydraulic thrust machine is physically moved during penetration, terminate the sounding. Another indicator of reaching thrust capacity is the rebound of rods after they are released. The magnitude of rebound depends on the flexibility of the thrust machine and the push rods. An operator must become familiar with the safe deflection of the system and decide when excessive deflections are being reached.

12.4.6 *Unusual Occurrences*—As data are recorded, it is important to note unusual occurrences in testing. When penetrating gravels, it is important to note “crunching” sounds that may occur when particle size and percentage of coarse particles begin to influence penetration. Note and report all occurrences of coarse gravels.

12.5 *Withdrawal*:

12.5.1 Withdraw the push rods and penetrometer tip as soon as possible after attaining complete sounding depth.

12.5.2 Upon complete withdrawal of the penetrometer, inspect the penetrometer tip for proper operation. The friction sleeve should be able to be rotated through 360° by hand without detectable binding.

12.5.3 Record baseline readings with the penetrometer tip hanging freely in air, or in water, out of direct sunlight. Compare baseline readings with initial baseline reading for requirements given in 10.1.2.1.

12.6 *Hole Closure*—In certain cases, it may be prudent or required by state law or specificiations, that the cone hole be filled, sealed, or grouted and closed after the sounding is completed. For example, in complex groundwater regimes, hole closure should be made to protect the water aquifer. Details on various methods for hole closure are provided elsewhere **(10)**.

13. Calculation

13.1 *Friction Cone Penetrometers*—Most electronic cone penetrometers in use at the present time measure a change in voltage across a strain gauge element to determine change in length of the strain element. Using known constitutive relationships between stress and strain for the strain element, the applied force may be determined for the cone or friction sleeve. The applied force may then be converted to stresses using the basic equations given in 13.2 and 13.3. Since there are a wide variety of additional, optional measurements currently being obtained with electronic cone penetrometers and new ones being continually developed, it is beyond the scope of this procedure to detail the makeup, adjustments, and calculations for these optional measurements.

13.2 *Cone Resistance, q_c—Required*:

$$q_c = Q_c/A_c \tag{1}$$

where:

q_c = cone resistance MPa (for example, ton/ft^2, kg$_f$/cm^2, or bar),

Q_c = force on cone kN (for example, ton, or kg$_f$), and

A_c = cone base area, typically 10 cm^2, or 15 cm^2.

13.2.1 *Corrected Total Cone Resistance (Required)*—Calculation of corrected total cone resistance requires measurement of porewater pressures measured at the shoulder in the u_2 position.

$$q_t = q_c + u_2\,(1 - a_n) \tag{2}$$

where:

q_t = corrected total cone resistance, MPa (ton/ft^2, kg$_f$/cm^2, bar, or suitable units for stress),

u_2 = porewater pressure generated immediately behind the cone tip, kPa (for example, tsf, kg$_f$/cm^2, bar, or suitable units for pressure), and

a_n = net area ratio (see A1.7).

13.2.1.1 The correction to total cone resistance is particularly important when porewater pressures are generated during penetration (for example, saturated clays, silts, and soils with appreciable fines). Generally, the correction is not so significant for CPTs in clean sands, dense to hard geomaterials, and dry soils. The correction is due to porewater pressures acting on opposing sides of both the face and joint annulus of the cone tip (**1**, **2**, **4**, **6**).

NOTE 7—In all cases, the total value q_t should be used, substituted for (or both) q_c, wherever possible. In no cases should q_c be backdetermined from q_t for use in equations, charts, formulae, or other purposes. It is always a forward procedure with corrected total q_t to be preferred.

13.2.1.2 Empirical adjustment factors based on select soil types have been developed for some pressure elements in the u_1 position, however these are not reliable. On a site-by-site basis, a relationship between u_1 and u_2 may be possible.

13.3 *Friction Sleeve Resistance, f_s—Required*:

$$f_s = Q_s/A_s \tag{3}$$

where:

f_s = friction sleeve resistance kPa (ton/ft^2, kg$_f$/cm ^{2}, bar, or suitable units for stress),

Q_s = force on friction sleeve kN (ton, kg$_f$, or suitable units for force), and

A_s = area of friction sleeve, typically 150 cm^2 for 10-cm^2 tip, or 200 to 300 cm^2 for larger 15-cm^2 cones.

NOTE 8—A corrected sleeve friction resistance may also be obtained (f_t), yet this requires both u_2 and u_3 measurements simulaneously (**2**, **3**, **4**, **6**). Thus, the raw f_s has been accepted for practical reasons. A simplified correction has been adopted by selected organizations (for example, (**6**)).

13.4 *Friction Ratio, R_f—(Optional)*:

$$R_f = (f_s/q_c)\cdot 100 \tag{4}$$

where:

R_f = friction ratio, %,

f_s = friction sleeve resistance kPa (ton/ft^2, kg$_f$/cm^2, bar, or suitable units for stress),

q_c = cone resistance kPa (ton/ft^2, kg$_f$/cm^2, bar, or suitable units for stress), and

100 = conversion from decimal to percent.

13.4.1 Determination of the friction ratio requires obtaining a cone resistance and friction sleeve resistance at the same point in the soil mass. The point of the cone is taken as the reference depth. Typically, a previous cone tip resistance reading at friction sleeve midpoint depth is used for the calculations. For the 10-cm^2 penetrometer, the standard offset is 100 mm. If an offset other than midheight is used it must be reported.

NOTE 9—In some cases, if readings are compared at the same point in a soil mass which has alternating layers of soft and hard materials erratic friction ratio data will be generated. This is because cone resistance is sensed, to varying degrees, ahead of the cone. The erratic data may not be representative of soils actually present.

NOTE 10—The friction sleeve resistance and friction ratio obtained from the mechanical friction cone penetrometers will differ considerably from values obtained from electronic friction cone penetrometers. When using soil classification charts that use R_f and q_c, it is important to use charts based on correlations for the type of penetrometer being used.

13.5 *Porewater Pressure Data*:

13.5.1 SI metric units for reporting porewater pressure data are kPa.

13.5.2 *Conversion of Measured Porewater Pressures to Equivalent Height of Water—Optional*—If it is desired to display porewater pressure in equivalent height of water, convert the dynamic or static water pressures to height by dividing pressure by the unit weight of freshwater, γ_w= 9.8 kN/m^3(62.4 lb$_f$/ft^3). For salt water, use γ_w= 10.0 kN/m^3(64.0 lbf/ft^3).

13.5.3 *Estimate of Equilibrium Porewater Pressure (Hydrostatic Porewater Pressure)*—Excess porewater pressure can only be calculated by knowing equilibrium pore water pressure, u_o (see 3.2.14). The equilibrium water pressure can be measured by dissipation test or estimated by calculation as follows (see Terminology D 653):

$$u_o = \text{estimated equilibrium water pressure} = h_w \cdot \gamma_w \tag{5}$$

In saturated soils below the groundwater level, the hydrostatic case is obtained from:

$$u_o = (z - z_w)\,\gamma_w \tag{6}$$

For soils above the groundwater table that are saturated due to full capillarity, Eq 6 is also applicable. For dry soils above the groundwater table, it is commonly adopted that $u_o = 0$. In partially-saturated soils (vadose zone), there can be great transient variations and variability in the u_o profile.

where:
h_w = height of water, m (or feet), evaluate from site conditions,
γ_w = unit weight of (fresh) water = 9.8 kN/m^3(or 62.4 lbs/ft^3),
z = depth of interest (m or feet),
z_w = depth to the groundwater table (phreatic surface).

In layered soils with multiple perched aquifers the assumption of a single height of water may be in error.

13.6 *Normalized CPT Measurments* In the latest soil behavioral classification charts and CPT interpretation methods, normalized readings for cone tip resistance, sleeve friction, and porewater pressure are utilized **(2, 4, 11,)**, as defined below.

13.6.1 Normalized cone tip resistance:

$$Q = (q_t - \sigma_{vo}) / \sigma_{vo}' \tag{7}$$

13.6.2 *Normalized Porewater Pressure Parameter, B_q*—This parameter is normally calculated with the shoulder porewater pressure measurement (location immediately behind the cone tip), designated u_2.

$$B_q = \Delta_2/(q - \sigma_{vo}) \tag{8}$$

13.6.3 Normalized friction ratio:

$$F = f_s /(q_t - \sigma_{vo}) \tag{9}$$

where:
Δu = excess pore water pressure ($u_2 - u_o$) (see 3.2.13),
u_o = estimated equilibrium water pressure, or hydrostatic porewater (see 13.5.3),
σ_{vo} = total vertical overburden stress, and
σ_{vo}' = effective overburden stress = $\sigma_{vo} - u_o$

The total overburden stress is calculated:

$$\sigma_{vo} = \Sigma\,(\gamma_{ti}\,\Delta z_i) \tag{10}$$

where:
Δz_i = layer thickness, and
γ_{ti} = total soil unit weight for layer.

14. Report

14.1 Report the following information:

14.1.1 General—Each sounding log should provide as a minimum:

14.1.1.1 Operator name,

14.1.1.2 Project information,

14.1.1.3 Feature notes,

14.1.1.4 Ground surface elevation and water surface elevation (if available),

14.1.1.5 Sounding location, including coordinates

14.1.1.6 Sounding number, and

14.1.1.7 Sounding date.

14.1.2 Reports should contain information concerning:

14.1.2.1 *Equipment Used*—Design drawings and data on all sensors,

14.1.2.2 Graphical data,

14.1.2.3 Electronic digital data or tabular data (optional),

14.1.2.4 Procedures followed, and

14.1.2.5 *Calibration Information*—For all sensors, information required in Section 10.

14.1.3 The report should contain a text that discusses items required in 14.2 and 14.3. Each sounding should be documented with:

14.1.3.1 Sounding plot.

14.1.3.2 *Accompanying Tabular Output*—Tabular output is considered optional due to its bulk. It is optional as long as computer data files are preserved and archived for later use.

14.1.3.3 *Computer Data Files*—Provide in ASCII format, spreadsheet file, or text, or other common file format. Computer data files must contain header as required in 14.1, sounding log information. Certain interpretation programs require data to be in a particular format. It is the responsibility of the user to determine acceptable formats.

14.1.3.4 The comments should contain notes on equipment and procedures, particular to the individual sounding.

14.2 *Equipment*—The report should include notes concerning:

14.2.1 Penetrometer manufacturer,

14.2.2 Types of penetrometer tips used,

14.2.3 Penetrometer details such as net area ratio, friction sleeve end areas, location and types of sensors, location and type of friction reducers,

14.2.4 Offset between tip and sleeve resistance used for friction ratio determination,

14.2.5 Serial numbers of penetrometer tips,

14.2.6 Type of thrust machine,

14.2.7 Method used to provide reaction force—with notes as to possible surface deformations,

14.2.8 Location and type of friction reduction system (if any),

14.2.9 Method of recording data,

14.2.10 Condition of push rods and penetrometer tip after withdrawal,

14.2.11 Any special difficulties or other observations concerning performance of the equipment,

14.2.12 Details on piezocone design, filter elements, and fluid conditioning procedures, and

14.2.13 Information on other sensing devices used during the sounding.

14.3 *Calibration Certifications*—For each project the report should include the load range calibrations of the cones used that were performed in accordance with Section 10. The report should include the initial and final baseline readings for each sounding. Calibration records for the porewater pressure transducers are required as given in 10.2. If the project requires calibrations of other sensors they should also be submitted in final reports.

14.4 *Graphs*—Every report of friction cone penetration sounding is to include a cone tip resistance plot, q_c MPa, or preferably total cone tip resistance, q_t MPa (or ton/ft^2, kg$_f$/cm^2, bar, or other acceptable unit of stress) with depth below ground surface m (ft), friction sleeve resistance, f_s, kPa (ton/ft^2, kg$_f$/cm^2, bar, or other acceptable units of stress), and friction ratio, R_f (%), on the same plot. (See Fig. 4 and Fig. 5 for example plots.) As a minimum, the plot should provide general information as outlined in 14.1. Electronic piezocone penetrometer soundings should provide an additional plot of porewater pressure kPa (or lb$_f$/in.^{2}, kg$_f$/cm^2, bar, or other acceptable units of pressure) versus depth, m (ft). Porewater readings can be plotted as pressures, or alternatively, the pressure may be converted to equivalent heights of water (that is, $h_w = u_2/\gamma_w$).

14.4.1 Symbols q_t and f_s for tip and sleeve resistance are accepted by the International Society for Soil Mechanics and Geotechnical Engineering (**1**, **2**, **3**, **7**).

14.4.2 For uniform presentation of data, the vertical axis (ordinate) should display depth and the horizontal axis (abscissa) should display the test values. There are many preferences in plotting such that uniform plotting scales and presentation will not be required.

15. Precision and Bias

15.1 *Precision*—There are little direct data on the precision of this test method, in particular because of the natural variability of the ground. Committee D-18 is actively seeking comparative studies. Judging from observed repeatability in approximate uniform deposits, persons familiar with this test estimate its precision as follows:

15.1.1 *Cone Resistance*—Provided that compensation is made for unequal area effects as described in 13.2.1, a standard deviation of approximately 2 % FSO (that is, comparable to the basic electromechanical combined accuracy, nonlinearity, and hysteresis).

15.1.2 *Sleeve Friction—Subtraction Cones*—Standard deviation of 15 % FSO.

15.1.3 *Sleeve Friction—Independent Cones*—Standard deviation of 5 % FSO.

15.1.4 *Dynamic Porewater Pressure*—Strongly dependent upon operational procedures and adequacy of saturation as described in 11.2. When carefully carried out a standard deviation of 2 % FSO can be obtained.

15.2 *Bias*—This test method has no bias because the values determined can be defined only in terms of this test method.

NOTE 11—Jefferies and Davies (**11**) report q_t repeatability of the two different soundings in compact clean sand using two different cones by the same manufacturer. Approximately 50 % of the data lay within 8 % of the average of the two tests, and 90 % of the data lay within 15 % of the average. In this trial the transducers (that conformed to the requirements in A1.5) were loaded to between one tenth and one fifth of their rated FSO, so confirming a standard deviation of better than 2 % FSO.

16. Keywords

16.1 cone penetration test; cone penetrometer; explorations; field test; friction resistance; geotechnical test; in-situ testing; penetration tests; penetrometer; piezocone; point resistance; porewater pressures; resistance; sleeve friction; soil investigations

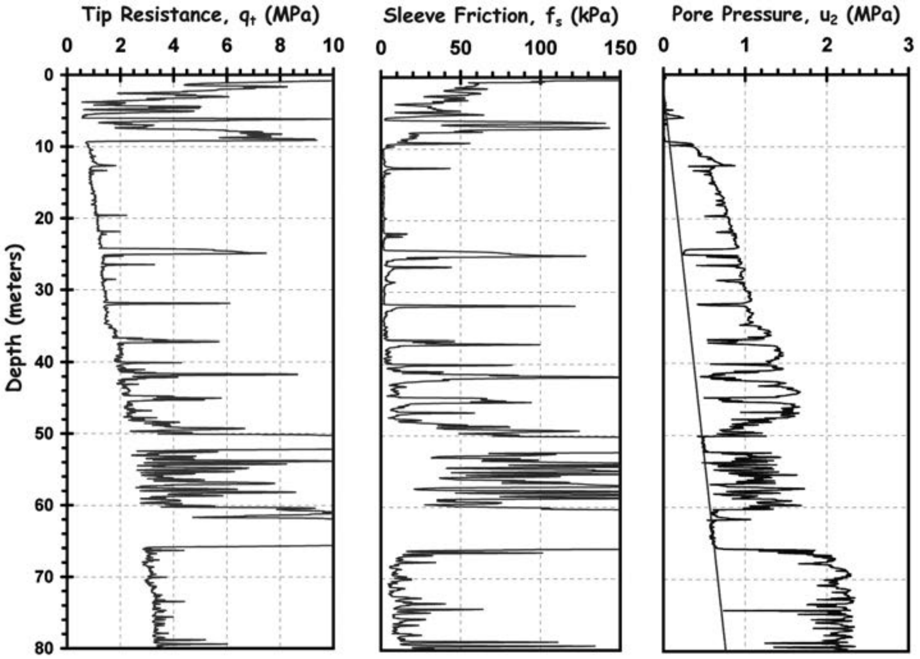

FIG. 4 Example Graph Presentation Results from a Conventional Piezocone Penetration Test

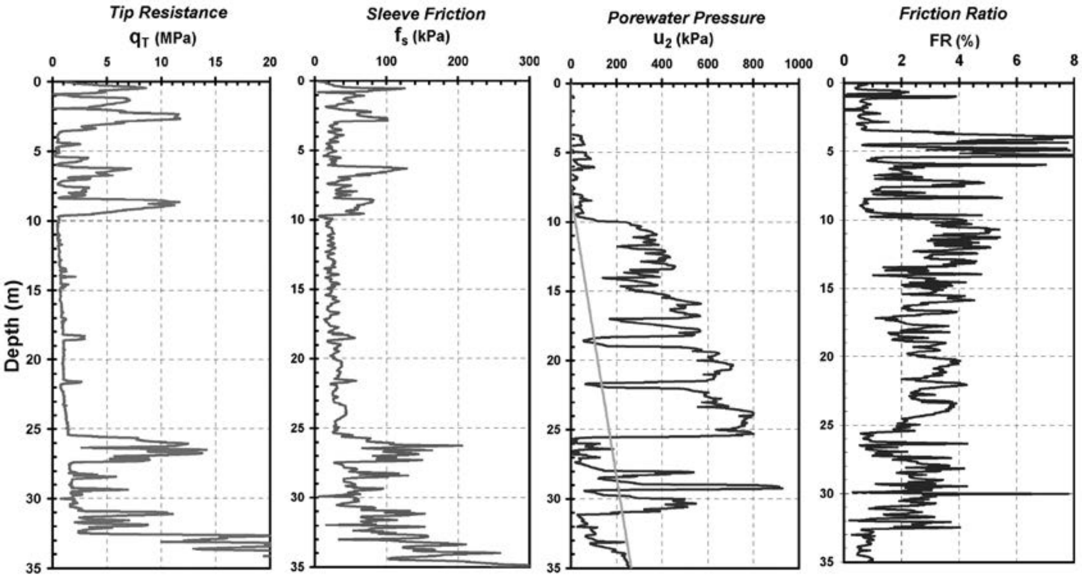

FIG. 5 Illustrative Piezocone Graph Showing Tip Resistance, Sleeve Friction, Penetration Porewater Pressure, and Friction Ratio

ANNEX

(Mandatory Information)

A1. CALIBRATION REQUIREMENTS ON NEWLY MANUFACTURED OR REPAIRED ELECTRONIC FRICTION CONE AND PIEZOCONE PENETROMETERS

A1.1 *Introduction:*

A1.1.1 This annex describes procedures and requirements for calibrating electronic cone penetrometers. The evaluation of cone penetrometer calibration as described in this annex is a quality assurance standard for newly manufactured and repaired penetrometer tips. Many of the standards may be impractical to evaluate under field operating conditions. Therefore, determination of these calibration errors for any individual penetrometer tip should be performed in a laboratory environment under ideal conditions by the manufacturer or other qualified personnel with necessary knowledge, experience, and facilities.

A1.1.2 The electronic cone penetrometer is a delicate instrument subjected to severe field conditions. Proper use of such an instrument requires detailed calibration after manufacture and continuous field calibrations. Years of cone penetrometer design and performance experience have resulted in refined cone designs and calibration procedures which make the electronic cone penetrometer a highly reliable instrument. Reports of these experiences form the basis for requirements in this annex (**1**, **2**, **3**, **9**).

A1.1.3 The required calibration tolerances developed in this annex are based on subtraction type electronic cone penetrometers. These penetrometers are more robust than electronic cone penetrometers with independent tip and sleeve load cells and are the most widely used design. The subtraction type penetrometer, however, has less precision due to the subtraction process (**3**, **9**). As a result, calibration tolerances given here are considered maximum values and requirements for more sensitive cone penetrometers imply smaller tolerances having greater precision. The calibration process consists of loading the penetrometer tip with reference forces and pressures and then comparing measured output to the reference.

A1.1.4 Calibrations in the laboratory environment should be performed with the complete penetrometer system to be used in the field. The same make and model computer, cable, signal conditioning system, and penetrometer to be used in the field shall be calibrated in the laboratory. Depending on the components of the system some components may be substituted with acceptable replacements. Each individual penetrometer must be tested over a range of loads to assure adequate performance.

A1.2 *Terms Related to Force Transducer Calibrations:*

A1.2.1 Fig. A1.1 is a graphical depiction of terms related to transducer calibrations and defines the concepts of zero-load error, nonlinearity, hysteresis, and calibration error (**2**, **8**).

A1.2.2 To evaluate several of these values, the FSO (full scale output) of the penetrometer tip is needed. The manufacturer shall provide full scale output information for the system. Cone penetrometer tips usually are available in nominal

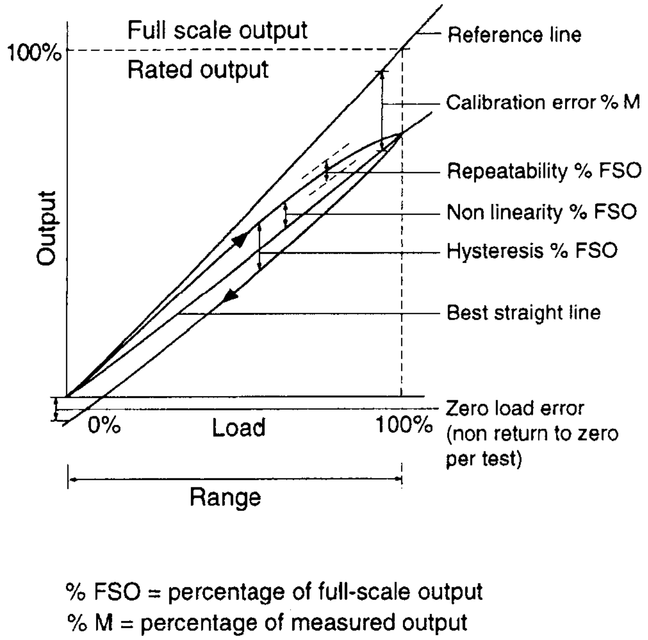

FIG. A1.1 Definition of Calibration Terms for Load Cells and Transducers (2, 8)

capacities of 2, 5, 10, and 15 metric tons. Typical full-scale outputs for these penetrometer tip ranges as follows:

Nominal Capacity	Full-Scale Output of Cone, q_c		Full-Scale Output of Friction Sleeve, f_s	
metric tons	ton/ft^2	MPa	ton/ft^2	kPa
2	200	20	2	200
5	500	50	5	500
10	1000	100	10	1000
15	1000	100	10	1000

A1.2.3

It is important to check with the manufacturer on the full scale output of electronic cone penetrometer tips to avoid overloading and damaging penetrometer tips.

A1.3 *Zero Load Baseline Values:*

A1.3.1 Zero-load output variation of the cone penetrometer during testing and calibration is a reliable indicator of output stability, internal O-ring friction, and temperature-induced apparent load. The variation in zero load output is affected by temperature fluctuation because temperature compensated strain gages do not compensate for material effects and system component effects (**1**, **2**, **3**, **8**).

A1.3.2 Systems with microprocessors provide "reference baseline" values for the transducers that are not equal to zero but are measured positive or negative values depending on the electronics of the system. For the particular penetrometer and penetrometer system used, the baseline values should remain relatively constant through the life of the penetrometer. As testing is performed in the field, the baseline resistances are monitored for changes. If large changes are noted the penetrometer should be loaded to check for linearity and possible damage. Evaluate the zero-load error during load range calibration by taking the difference between initial and final baseline values.

A1.3.3 *Thermal Stability*—For ensurance of thermal stability, evaluate a particular design of a newly manufactured cone under a range of temperature conditions. Newly manufactured penetrometer tips are first cycled to a minimum of 80 % of FSO five times at room temperature, to remove any residual nonlinearity. After cycling, establish an initial reference baseline value at room temperature after the cone has been electrically powered for about 30 min. To evaluate thermal stability, stabilize the penetrometer tip at temperatures of 10 and 30°C and new baseline values are obtained. The change in baseline values must be $\leq$ 1.0 % FSO of either cone or friction sleeve resistances.

A1.4 *Load Range Calibration:*

A1.4.1 Calibrate newly manufactured or repaired cone penetrometers over a range of loads after production or repair. Load test the cone penetrometer system in a universal testing machine or specially designed cone penetrometer calibration device capable of independently loading the cone and friction sleeve. If a universal testing machine is used, a calibration certificate (current within the last year) in accordance with Practice E 4 must be available. If a cone calibration apparatus is used, it should also have a calibration document current within the last year. The calibration document shows that applied forces or masses are traceable to standard forces or masses retained by the National Institute of Standards and Technology. The universal testing machine or cone calibration devices must be capable of loading the penetrometer tip to 100 % FSO.

A1.4.2 Selection of loading steps and maximum loading varies depending on need and application. Select the load steps and maximum load to cover the range of interest and not necessarily the maximum capacity of the cone. Some calibrations stress more frequent load steps at lower loads to evaluate weaker materials. Selection of more frequent lower load steps may result in higher levels of calibration error since the best fit line is more influenced by the low range data.

A1.4.3 Perform the loading after the cone is subjected to five cycles of compressive loading and reference baselines, or internal zeroing, have been obtained at room temperature. The penetrometer is loaded in a minimum of six increments at forces equivalent to 0, 2, 5, 10, 25, 50, and 75 % FSO. At each increment of force, record both cone and sleeve resistances. Compute the actual cone tip resistance by dividing the applied force by the cone base area. The friction sleeve resistance is taken as the corresponding axial force over the sleeve area. Determine the "best fit straight line" by linear regression of applied force and measured output. The linearity is the difference between measured cone resistance and best-straight line cone resistance divided by the cone FSO. Evaluate hysteresis by comparing the difference between cone resistance at the same level of applied force in loading and unloading and dividing by cone FSO. Calculate calibration error by taking the difference between the best-fit-straight line cone resistance and actual cone resistance and dividing by the actual cone resistance. Calibration error can become larger with smaller measured outputs and, therefore, it is not evaluated at loadings equivalent to less than 20 % of cone FSO.

A1.4.3.1 When calibrating the penetrometer, monitor the friction sleeve resistance to evaluate apparent load transfer. With a subtraction-type electronic cone penetrometer tip, the apparent friction sleeve resistance is caused by electrical subtraction error, crosstalk, and any load transferred mechanically to the sleeve. With a cone, that provides for independent cone and sleeve measurements, apparent friction sleeve resistances are caused by electrical crosstalk and mechanical load transfer. Apparent load transfer must be less than 1.5 % of FSO of the friction sleeve (1000 kPa).

A1.4.3.2 Maximum nonlinearity should be 0.2 %, maximum calibration error should be 0.5 %, and maximum apparent load transfer should be 1.2 %. For this calibration, the zero load error was zero. Hysteresis was not evaluated in this example because the testing machine was incapable of producing the exact same force on the loading and unloading steps.

A1.4.4 For calibration of the friction sleeve element, apply the forces in seven increments at 0, 2, 5, 10, 25, 50, and 75 % of FSO. Nonlinearity, hysteresis, and calibration error are evaluated in the same manner as calibrations for the cone tip reading. During friction sleeve calibration, monitor cone tip resistance to evaluate apparent load transfer that was not apparent in this calibration.

A1.5 *Force Transducer Calibration Requirements:*

A1.5.1 Calibration requirements developed for electronic cone penetrometers are based on past experience with subtraction-type electronic cone penetrometers and, as a result of this experience, represent the minimum precision requirement of electronic cone penetrometers. In cases where a higher level of precision is required, stricter calibration requirements would be required. Newly manufactured or repaired electronic cone penetrometers are required to meet the following requirements:

Calibration Parameter	Element	Requirement
Zero-load error	Tip and sleeve	≤ ± 0.5 % FSO
Zero-load thermal stability	Cone tip and sleeve	≤ ± 1.0 % FSO
Nonlinearity	Cone tip	≤ ± 0.5 % FSO
	Sleeve	≤ ± 1.0 % FSO
Hysteresis	Tip and sleeve	≤ ± 1.0 % FSO
Calibration error	Cone tip	≤ ± 1.5 % MO at >20 % FSO
	Sleeve	≤ ± 1.0 % MO at >20 % FSO
Apparent load	While loading cone tip	≤ ± 1.5 % FSO of sleeve transfer
	While loading sleeve	≤ ± 0.5 % FSO of cone tip

A1.6 *Pressure Transducer Calibrations:*

A1.6.1 Newly manufactured or repaired pressure transducers shall be supplied with a load range calibration provided by the manufacturer. The load range calibration shall consist of a minimum of six points of loading to at least 75 % of FSO. The applied pressures shall be traceable to reference forces maintained by NIST. The calibration shall meet the manufacturer's stated tolerances. Minimum requirements are linearity better than 1 % of FSO and zero load error less than ±7 kPa (±1.0 lb/in.^{2}).

A1.6.2 The transducer shall be subjected to regular periodic inspection meeting requirements in A1.6.1.

A1.7 Correction of Tip and Sleeve Areas:

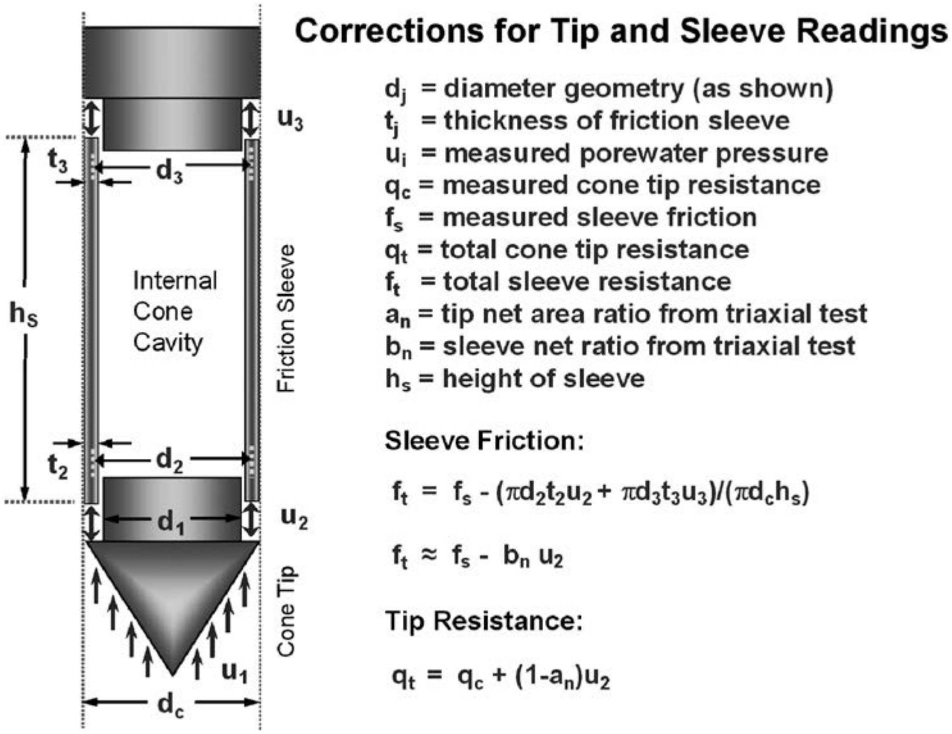

FIG. A1.2 Determination of Net Area Ratio (a_n) for Corrections of Cone Tip Resistances (4)

A1.7.1 The conceptual regions where water pressures can act on the cone tip and sleeve elements are shown in Fig. A1.2. Water pressure that acts behind the cone tip will reduce measured cone resistance, q_c, by the magnitude of water pressure acting on unequal areas of the tip geometry. It is therefore advantageous to use a penetrometer having a net area ratio $a_n = 0.80$ in order to minimize the effect of the correction (**1**, **2**). Water pressure may also act on both ends of the sleeve, resulting in an imbalance of forces if the sleeve is not designed with equal effective end areas. The water pressures acting on the ends of the sleeve are not just a function of geometry, they are also a function of the location of water seals. Water pressures during penetration are not often measured at both ends of the sleeve (that is, simultaneous u_2 and u_3) so a correction is not normally made for f_s (**3**).

A1.7.2 Equal end area friction sleeves should be required for use and should be designed by the manufacturer. The best method for evaluating sleeve imbalance is to seal the penetrometer in a pressure chamber and apply forces to measure the sleeve resistance after zeroing the system. Manufacturers should perform this check for a particular design to assure minimal imbalance.

A1.7.3 In order to calculate the corrected total cone resistance, q_t, as shown in 13.2.1, it will be necessary to determine the area ratio of the cone. The penetrometer can be enclosed in a sealed pressure vessel (for example, triaxial cell) and water pressures should be applied as shown in the example in Fig. A1.3. The net area ratio is then used in computing the corrected total tip resistance.

A1.8 Other Calibrations—Other sensors such as inclination, temperature, etc. may require calibration depending on the requirements of the investigation. Perform such calibrations using similar techniques given in this annex or by other reference procedures. Report such calibrations when required.

A1.9 *Documentation of Calibrations:*

A1.9.1 Laboratory calibration documents consisting of a short report on the equipment and methods of testing, along with tables and figures similar to those in this annex, are required for the following occurrences:

A1.9.1.1 When new penetrometer tips are received, and

A1.9.1.2 When damaged penetrometer tips are repaired.

A1.9.2 The report must be certified by a registered professional engineer or other responsible engineer with knowledge and experience in materials testing for quality assurance. Calibration documents are retained on file by the offices responsible for performing soundings and should be updated at required intervals. For contract soundings, calibration documents should be obtained prior to contract acceptance and after testing on unaltered equipment.

A1.9.3 If the electronic cone penetrometer meets the field calibration requirements given in 10.1.3, it is only necessary to

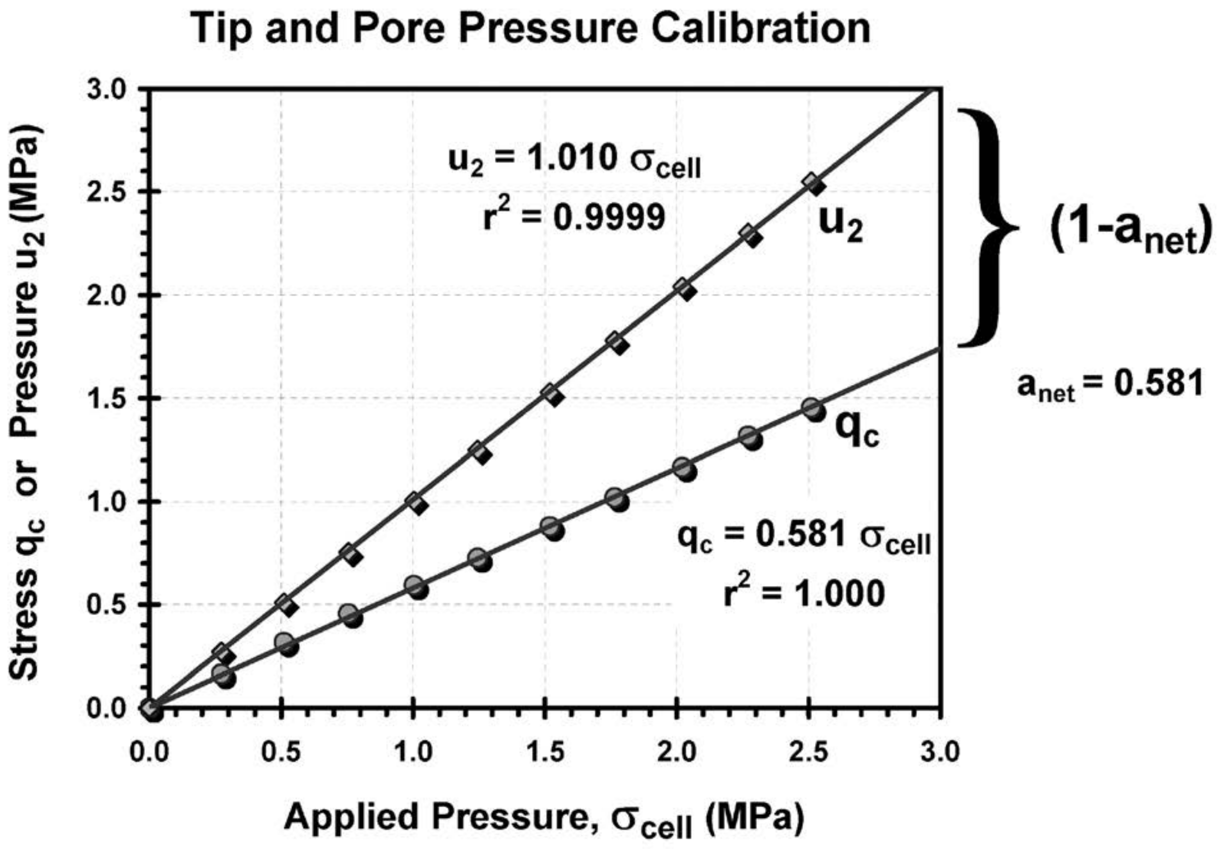

FIG. A1.3 Illustrative Example Determination of Unequal End Area for Correction of Tip Resistances Using Pressurized Triaxial Cell Calibration

adjust the penetrometer tip to the laboratory requirements on a yearly basis. Cone penetrometers should be calibrated using laboratory procedures prior to use on each new project, but they do not need to meet calibration tolerances as required for newly manufactured cones.

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SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 5778 – 95 (2000)) that may impact the use of this standard. (Approved November 1, 2007.)

(*1*) New references added.

(*2*) Excess porewater pressure definition corrected in 3.2.13.

(*3*) Fig. 2 reference citation updated.

(*4*) Revised Fig. 3.

(*5*) Normalized cone tip resistance added to 13.

(*6*) Generally overall improvement in many graphs with newer figures that show better detailing and annotation.

(*7*) Fig. 1 includes three basic cone penetrometer designs (rather than older figure showing only two designs), that is, compression-, tension-, and subtraction-types.

(*8*) Fig. A1.1 and Fig. A1.1 have been replaced with newer figures to show the pressurization calibration.

(*9*) Section 12.6 on hole closure has been added.

(*10*) Use of capital U for porewater pressures is replaced with small lowercase u in 7.1.8.5.

(*11*) Penetrometer gap has now been labeled as e_c in 7.1.4.3.

(*12*) Added reference to Practice D 3740.

(*13*) Common stress and pressure values have been mentioned.

(*14*) Numerous general cleanup and correction of grammatical and spelling errors, too numerous to mention here.

Designation: D3740 – 10

Standard Practice for
Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction[1]

This standard is issued under the fixed designation D3740; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This practice establishes minimum qualifications for agencies engaged in the testing and inspection of soil and rock. Minimum requirements for field and laboratory personnel are defined. The practice also covers the establishment and maintenance of a quality system.

1.2 Criteria are provided for evaluating the capability of an agency to properly perform designated tests on soil and rock, and for establishing essential characteristics pertaining to an agency's organization, personnel, facilities, and quality system. This practice may be supplemented by more specific criteria and requirements for particular projects.

1.3 This practice can be used as a basis to evaluate testing and inspection agencies, or both, and is intended for use for the qualifying or accrediting, or both, of testing or inspection agencies, public or private, engaged in the testing and inspection of soil and rock as used in engineering design and construction.

1.4 This practice is applicable to all standards promulgated by Committee D18 whether or not mentioned in the Referenced Documents Section.

1.5 *This standard does not purport to address all of the safety concerns, 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.*

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1077 Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D5255 Practice for Certification of Personnel Engaged in the Testing of Soil and Rock

E329 Specification for Agencies Engaged in Construction Inspection and/or Testing

E1187 Terminology Relating to Conformity Assessment[3]

E1301 Guide for Proficiency Testing by Interlaboratory Comparisons

2.2 *Other Standards:*

AASHTO R18 Recommended Practice for Establishing and Implementing a Quality System for Construction Materials Testing Laboratories[4]

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions:*

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[1] This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.99 on Quality Control.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

***A Summary of Changes section appears at the end of this standard.**

3.1.1 For definitions of terms used in this practice see Terminologies D653 and E1187.

3.1.2 Soil and Rock — as used in this standard, any test method, practice, specification or guide developed by D18 and listed in volumes 04.08 and 04.09.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *agency*—an organization, or part of an organization, engaged in activities of technically oriented testing or inspection, or both.

3.2.2 *quality manual*—a document stating the quality policy, quality system and quality practices of an organization.

3.2.3 *qualified national authority*—an organization recognized throughout the country, with the capability to assess and monitor the professional and technical activities of an inspection or testing agency, or both.

4. Significance and Use

4.1 This practice provides the basic minimum criteria for use in evaluating the qualifications of a testing or inspection agency, or both, for soil and rock. The criteria may be supplemented by more specific criteria and requirements. An individual user can also use it to judge the qualification of an agency. The existence of a formal accrediting body such as a federal, state, or independent agency is not necessary for the use of this standard.

NOTE 1—Users of this practice should be aware that certain of these requirements may not be achievable and/or applicable to work performed outside of the U.S.A. In such cases, users should ensure that all necessary modifications are made to these requirements such as to render them appropriate to each specific set of circumstances.

4.2 The intent of this practice is to provide a consensus basis for evaluating a testing or inspection agency, or both, with respect to that agency's capability to objectively and competently provide the specific services needed by the user.

4.3 This practice may be used as a basis for accreditation.

4.4 To qualify for accreditation to this standard, the agency must include at least five standards relating to testing methods, inspection methods, or both, from the standards covered under the jurisdictions of Committee D18, in its certificate of accreditation.

4.5 The users of an accredited agency must review the agency's scope of accreditation to ensure the agency has been accredited for its technical competence to perform the tasks requested by the user.

5. Responsibilities and Duties

5.1 The agency shall ensure that only inspections or tests for which it is adequately equipped and staffed are performed.

5.2 The agency shall ensure that personnel perform only inspections and tests for which they are adequately trained, qualified and certified in accordance with applicable specifications.

5.3 The agency shall ensure that all equipment is properly maintained in good operating condition and is calibrated as applicable.

5.4 The agency shall perform all testing and inspection in accordance with appropriate standards and quality control criteria.

6. General Capabilities

6.1 *Laboratory Testing*—The agency performing laboratory testing of soil and rock shall have suitable test equipment and laboratory facilities for storing and testing samples and preparing samples for test.

6.2 *Field Testing and Inspection*—The field services of a soil and rock testing and inspection agency shall include some or all of the following capabilities:

6.2.1 testing of in situ materials,

6.2.2 testing of materials being processed,

6.2.3 checking on adequacy of production equipment or construction equipment used for reworking or processing soil and rock,

6.2.4 observation and inspection of soil or rock placement, and

6.2.5 in-place testing of constructed components.

6.3 *Sampling*—the services of an agency responsible for sampling soil and rock shall include some or all of the following capabilities:

6.3.1 sampling of in situ materials,

6.3.2 sampling of materials which are to be reworked, processed, and reused,

6.3.3 sampling of materials being processed, and

6.3.4 sampling of constructed components.

7. Personnel Qualifications

7.1 *Management and Supervision*—The testing and inspection services of the agency shall be under the direction of a person charged with the engineering managerial or scientific managerial responsibility. The person shall be a licensed registered engineer or other licensed registered professional and a full-time employee of the agency and shall have a minimum of 5 years engineering or scientific experience, as appropriate, in the inspection and testing of soil and rock; or a person with equivalent science-oriented education and experience in having satisfactorily supervised or directed testing or inspection services, or both, of soil and rock is acceptable.

7.2 *Supervising Laboratory Technician*—The supervising laboratory technician shall have at least 3 years experience performing tests on soil and rock.

7.2.1 This person must demonstrate, by written examination(s), the ability to perform the tests in the manner stipulated under ASTM or other governing procedures and shall be capable of evaluating the test results in terms of specification compliance. Current certification by national, regional or state authorities shall be considered as one means of evidence of fulfilling the written examination requirement (Note 2). The certification shall be appropriate to the work required. At a minimum, the written examination(s) shall include at least five test methods listed in ASTM Volumes 4.08 and 4.09.

7.2.2 In addition, a performance evaluation reviewing the technician's competency to perform the test method correctly shall be conducted prior to the technician performing the test independently and at least every 36 months thereafter for each test the person is authorized to perform.

7.3 *Supervising Field Technician*—This person shall have at least 3 years experience in inspecting the kind of work involved in the soil and rock construction project.

7.3.1 This person must demonstrate, by written examination(s), the ability to perform the tests and duties in the manner stipulated under ASTM or other governing procedures and shall be capable of evaluating the test results in terms of specification compliance. Current certification by national, regional or state authorities shall be considered as one means of evidence of fulfilling the written examination requirement (Note 2). The certification shall be appropriate to the work required. At a minimum, the written examination(s) shall include at least five of the testing or inspection methods listed in ASTM Volumes 4.08 and 4.09.

7.3.2 In addition, a performance evaluation reviewing the technician's competency to perform the test method correctly shall be conducted prior to the technician performing the test independently and at least every 36 months thereafter for each test the person is authorized to perform.

7.4 *Inspecting or Testing Technician*—This person shall have a high school diploma or equivalent or trade school training and have had sufficient on-the-job training to properly perform the test or inspection to which the person is assigned.

7.4.1 This person must demonstrate, by written examination(s), the ability to perform the tests in the manner stipulated under ASTM or other governing procedures. Current certification by national, regional or state authorities shall be considered as one means of evidence of fulfilling the written examination requirement (Note 2). The certification shall be appropriate to the work required. At a minimum, the written examination(s) shall include at least five of the testing or inspection methods listed in ASTM Volumes 4.08 and 4.09.

7.4.2 In addition, a performance evaluation reviewing the technician's competency to perform the test method correctly shall be conducted prior to the technician performing the test independently and at least every 24 months thereafter for each test the person is authorized to perform.

7.4.3 A trainee may perform this work while advancing toward certification under the direct physical supervision of a person meeting the requirements above. The trainee cannot independently evaluate test results or sign as responsible for an inspection or testing report.

NOTE 2—Refer to Practice D5255 for other guidance on certification.

7.5 It is satisfactory for a person to fill one or more of the levels of management, supervision, inspector, or technician positions in accordance with 7.1, 7.2, 7.3, and 7.4 provided that person qualifies for the highest level. It is also recognized that frequently a few laboratory control tests or inspections are conducted at small field or peripheral locations. It is not the intent of this practice that the supervisory personnel be directly present at such locations at all times.

8. Quality System Criteria

8.1 The agency shall establish and implement a quality system which meets the following criteria:

8.1.1 *Quality Manual*—The agency shall establish and maintain a quality manual that conforms to the requirements in Section 9, Quality Manual (Requirements). Each document in the quality manual shall indicate its preparation date. If a document is revised, the date of revision shall be indicated on the document. The quality manual shall be available for use by laboratory staff.

8.1.2 *Quality Management*—The agency shall designate a person(s) having responsibility for determining if quality system implementation activities are being conducted by agency staff in the manner specified in the agency's quality manual. This individual(s) shall have direct access to top management (Note 3).

NOTE 3—This individual(s) may have other responsibilities (for example, laboratory manager).

8.1.3 *Laboratory Procedure Manual*—The agency shall establish and maintain a procedures manual, outlining the customary method or inspection procedures for each test or service performed by the laboratory. Copies of current ASTM, AASHTO, or other national standards used need not to be included in the manual. However, for each procedure, the manual shall include specific references to such standards along with any exceptions to them or any special instructions (such as requirement for forms, calculation programs, checking, review, or combinations thereof, etc.) (or both). The referenced standards shall be readily available for use by personnel performing the test or service.

8.1.4 *Equipment Calibration and Verification*—The agency shall calibrate or verify all significant testing equipment associated with tests covered by the scope of this standard which the agency performs. As a minimum, the equipment listed in Table 1 shall be included if it is associated with tests performed by the agency. Applicable equipment shall be calibrated or verified at the intervals specified in the agency's quality manual. The intervals specified in the quality manual shall be no greater than those indicated in Table 1 (Note 4). Newly acquired equipment without manufacturers certification and equipment that has not been calibrated or verified because it has been removed from service shall be calibrated or verified before being placed in service. The agency shall have detailed written procedures for all in-house calibration and verification activities not addressed in standards. These procedures shall indicate the equipment required to perform the calibration or verification.

NOTE 4—When a maximum calibration or verification interval for a specific piece of test equipment is specified in a standard, the maximum interval specified by this document is intended to be the same as the maximum interval specified by the standard.

8.1.5 *Equipment Calibration and Verification Records*—The agency shall maintain calibration and verification records for all equipment specified in the quality manual. Such records shall include:

8.1.5.1 detailed results of the work performed (dimensions, mass, force, frequency, temperature, time, and the like),

8.1.5.2 description of the equipment calibrated or verified including model and serial number or other acceptable identification (Note 10),

8.1.5.3 date the work was done,

8.1.5.4 identification of the individual performing the work,

8.1.5.5 identification of the calibration or verification procedure used,

TABLE 1 Test Equipment Calibration and Verification Requirements

Equipment—Test Method	Requirement	Interval (Month)
Mechanical Shakers	Ck. Sieving Thoroughness	12
Gen. Purpose Balances, Scales & Weights	Verify	12
Compression or Loading Device	Verify Load Indications	12
Mechanical Compactor	Calibrate	12
CA Kneading Compactor	Calibrate	24
Ovens	Verify Temperature Setting(s)	4
Vacuum System	Ck. Pressure	24
Molds	Ck. Critical Dimensions	12
Manual Hammer	Ck. Wt. & Critical Dimensions	12
Sieves	Ck. Physical Condition	6
Liquid Limit Device	Ck. Wear & Critical Dimensions	12
Grooving Tool	Ck. Critical Dimensions	12
Hydrometers	Ck. Critical Dimensions	24
Straightedge	Ck. planeness of edge	6
Weighted Foot Assembly	Ck. weight	12
CBR Annular and Slotted Weights	Ck. weight	12
CBR Penetration Piston	Ck. diameter	12
Standard Metal Specimen	Ck. outside diameter	12
Metal Follower	Ck. diameter	12
Gen. Purpose Balances, Scales, Weights	Verify	12
Compression or Loading Device	Verify Load Indications	12
Ovens	Verify Temperature Settings	4
Sieves	Check Physical Condition	6
Dial Gages, LVDTs, Micrometers	Verify Indications	6
Presure Gages and Transducers	Calibrate	6
Load Cells	Calibrate	12
Flow Meters	Calibrate	12
Thermal Meters and Transducers	Calibrate	12
Sonic Transducers	Verify	6

8.1.5.6 the previous calibration or verification date and the next due date, and

8.1.5.7 identification of any in-house calibration or verification device used.

8.1.6 *Inspection of Facilities*—The agency shall have its facilities inspected at intervals of not more than 3 years by a qualified national authority. The agency shall, within 30 days of the receipt of the evaluation report, submit to the qualified national authority a written report documenting how any deficiencies were corrected.

8.1.7 *Proficiency Sample Testing*—The agency shall participate in a formal proficiency sample program(s) as described in Guide E1301. An inhouse program or a program operated by an independent third party is acceptable. The scope of participation shall be sufficient to validate quality system operation.

8.1.8 *External Audit Records*—The agency shall maintain records of any external audits and documentation describing how the deficiencies were corrected.

8.1.9 *Proficiency Sample Records*—The agency shall retain results of participation in proficiency sample programs including data sheets, summary reports, and documentation describing steps taken to determine the cause of poor results and corrective actions taken.

8.1.10 *Test Methods and Procedures*—The agency shall maintain copies of standard and nonstandard procedures for testing performed which is covered by the scope of this standard and shall ensure that the procedures are the most current and are readily accessible to employees performing the work.

8.1.11 *Test Records*—The agency shall maintain test records which contain sufficient information to permit verification of any test reports. Records pertaining to testing shall include original observations, calculations, derived data and an identification of personnel involved in sampling and testing. The agency shall prepare test reports which clearly, accurately and unambiguously present, but are not limited to, the information specified in Table 2 (Note 5). The procedure for amending reports shall require that the previously existing report be clearly referenced when an amendment is made. The references shall establish a clear audit trail from the latest issuance or deletion to the original report and its supporting data.

NOTE 5—The requirements in Table 2 apply to the record that is used to present the laboratory's test results in their final form. In some cases, a test report or test data sheet is the final form of the data.

8.1.12 *Records Retention*—Records pertaining to testing, equipment calibration and verification, test reports, internal quality system reviews, proficiency sample testing, test technician training and evaluation, and personnel shall be retained by the laboratory in a secure location for a minimum of 3 years.

TABLE 2 Test Report Requirements

A	Name and address of the testing laboratory
B	Identification of the report and the date issued
C	Name and address of the client
D	Identification of the project
E	Description and identification of the test sample
F	Date of receipt of the test sample
G	Date(s) test was performed
H	Identification of the standard test method(s) used and a notation of deviations from the standard
I	Test results and other pertinent data required by the standard test method
J	Identification of any test results obtained from tests performed by a subcontractor
K	The name of the person(s) accepting technical responsibility for the test report
L	Any additional sample and field identification/location information

NOTE 6—There are many circumstances when a longer retention may be advantageous to the agency. Records concerning the calibration and verification of equipment are an example. Retention schedules of this type usually require such records to be held throughout the useful life of the equipment.

9. Quality Manual (Requirements)

9.1 The agency shall establish and maintain a quality manual meeting the following requirements:

NOTE 7—The quality system requirements of AASHTO R18, ISO/IEC 17025, Specification E329, Practice C1077, or Specification D3666, satisfy the requirements of this standard.

9.1.1 *Organization and Organizational Policies*:

9.1.1.1 The quality manual shall contain the legal name and address of the agency and that of the main office or company, if different, and any other information needed to identify the organization.

9.1.1.2 The quality manual shall contain the ownership and management structure of the agency. Names, affiliations and positions of principal officers and directors shall be listed.

9.1.1.3 The quality manual shall contain an organization chart showing relevant internal organizational components.

9.1.1.4 The quality manual shall contain a list showing applicable dates of the qualifications, accreditations and recognition of the agency by others.

9.1.2 *Staff*:

9.1.2.1 The quality manual shall contain an outline or chart showing operational personnel positions and their lines of authority and responsibility.

9.1.2.2 The quality manual shall contain position descriptions for each technical operational position shown on the agency's organization chart in testing areas covered by the scope of this standard. These position descriptions shall identify the position and include a description of the duties associated with the position, required skills, education and experience, and supervision exercised and received. A reference to where the required position descriptions may be found is acceptable if they are not included in the quality manual.

9.1.2.3 The quality manual shall contain a brief biographical sketch, noting the education, work experience, licensure, certifications, and current position of supervisory technical staff involved in testing areas covered by the scope of this standard. Alternatively, the quality manual may contain a reference to the location of the biographical sketches.

9.1.2.4 The quality manual shall contain a document which describes the method(s) used to ensure that all agency technical staff are trained and qualified to perform tests covered by the scope of this standard. In addition to the description of training methods the document shall indicate what position(s) or employee(s) is responsible for the agency training program and maintenance of training records.

NOTE 8—There may be several different methods employed for differing conditions of staff experience and background including (1) on-the-job apprentice training (one on one) for new employees with little or no experience in laboratory or inspection work; (2) formal in-house training sessions for certification, rating, or competency evaluation; and (3) training by external organizations. An individual with prior experience performing a specific test need only have competency confirmed by the agency.

9.1.2.5 The quality manual shall contain a document describing the method(s) used to evaluate staff competency to ensure that each test covered by the scope of this standard is performed in accordance with standard procedures. This description shall include the frequency of evaluations for each technician and indicate what position(s) or employee(s) is responsible for evaluating staff competency and maintaining records. These procedures shall ensure that each technician performing the test method is evaluated.

NOTE 9—Proficiency sample testing may be useful in evaluating staff competency, however, it should be used in conjunction with observation of actual testing performed.

9.1.2.6 The quality manual shall contain a form(s) for recording training and competency evaluation activities summarized under 9.1.2.4 and 9.1.2.5 including the name of the trainee, name of the evaluator, test method evaluated, the dates and results.

9.1.3 *Facilities and Equipment*:

9.1.3.1 *Inventory*—The quality manual shall contain an inventory of major sampling, testing, calibration and verification equipment associated with the test methods covered by the scope of this standard. A reference to where the inventory is located is acceptable if it is not included in the quality manual. The inventory shall include, for each piece of major equipment, the name, manufacturer, model and serial number (Note 10 and Note 11).

NOTE 10—Major equipment includes equipment such as shakers, physical or chemical testing machines, balances, baths, ovens, microscopes, and computing equipment dedicated to testing. Equipment such as chairs, desks and file cabinets may be excluded. Major equipment does not usually include expendable items such as miscellaneous glassware, sieves, molds and viscometers.

NOTE 11—An identification number assigned by the agency or other unique identifying information may be substituted for the model and serial number if this is the practice normally followed by the agency.

9.1.3.2 *Equipment Calibration and Verification*:

(1) The quality manual shall contain a list(s) giving a general description of equipment for performing tests covered by the scope of this standard that require calibration or verification. For each item listed, the list shall include the interval of calibration or verification, a reference to the calibration or verification procedure used (Note 12), and the location of calibration and verification records (Note 13).

NOTE 12—When standard calibration procedures are used, the standard shall be referenced. When the procedure used has been prepared by the agency, the in-house designation shall be referenced. It shall be indicated if the work is performed by an outside agency.

NOTE 13—In addition to being in the quality manual, this information may also be included in the calibration and verification records on each piece of equipment.

(2) The quality manual shall contain a document that describes the agency's method for ensuring that the calibration and verification procedures are performed for all required equipment at the specified intervals. This document shall include the name of individual(s) responsible for ensuring that calibration and verification activities are carried out, and procedures for handling equipment that is new, removed from service, out of calibration or defective.

(*3*) The quality manual shall contain in-house equipment calibration and verification procedures, when they cannot be referenced in applicable standards, or have a reference to their location.

(*4*) The quality manual shall contain certificates or other documents that establish the traceability of in-house equipment or reference standards used for calibration and verification, or have a reference to their location in the agency.

9.1.4 *Test Records and Reports*:

9.1.4.1 The quality manual shall contain a document that describes methods used by the agency to produce test records and to prepare, check and amend test reports.

9.1.4.2 The quality manual shall contain typical test report forms which illustrate the manner in which tests results and supporting information (see 8.1.11) are documented.

NOTE 14—A printout showing a typical test record is acceptable if the laboratory uses electronic media for report storage.

9.1.5 *Sample Management*—The quality manual shall contain a document describing procedure(s) for sample identification, storage, retention, and disposal of samples.

NOTE 15—In this context, the term storage refers to what is done before testing. The term retention refers to what is done after testing.

9.1.6 *Diagnostic and Corrective Action*:

9.1.6.1 The quality manual shall contain a document(s) describing participation in proficiency sample and on-site inspection programs, methods used to identify poor results and procedures followed when poor results occur or deficiencies occur.

9.1.6.2 The quality manual shall contain a document outlining the method(s) used in responding to external technical complaints.

9.1.7 *Internal Quality System Review*—The quality manual shall contain a document describing the scope of internal quality system reviews, establishing the frequency of these reviews, identifying individuals responsible for the review, describing the distribution of reports to management and identifying the location of resulting records.

9.1.8 *Subcontracting*—The quality manual shall contain a document describing the policies that the agency follows relative to subcontracting, if it engages in such activities. A reference to where the policies may be found is acceptable if they are not included in the quality manual. These policies shall include procedures followed by the agency in selecting competent subcontractors who meet the requirements of this practice and reporting the results of testing performed by subcontractors. If the agency does not engage in such activities, the quality manual shall contain a statement to that effect.

10. Records and Reporting Requirements

10.1 The agency shall maintain a system of records that permits verification of any issued report. A record of each report and related records shall be retained for at least three years and shall include the name of the person performing the test(s).

10.2 The agency shall maintain the following records:

10.2.1 Detailed results (for example, worksheets) of all required equipment calibration and verification,

10.2.2 Results of internal audits,

10.2.3 The results of any on-the-job training performed including name of person, date of training, by whom and type of training,

10.2.4 The results of any activities performed to ensure continued competence in performing standard test methods, including name of person, date of competency check, by whom, what type of activity,

10.2.5 The results of audits and inspections of the agency and certifications of agency personnel with applicable dates,

10.2.6 Records of verification of competency of any external organizations used, and

10.2.7 Records or resumes that document the qualifications, work experience, and training history of each person.

10.3 Each report, as a minimum, shall include:

10.3.1 The name and address of the agency,

10.3.2 The date the report was issued and the date the test or inspection was performed,

10.3.3 The name of the client,

10.3.4 Identification of the report, the project, and the name and title of the person technically responsible for the report, and the standard test method(s) used,

10.3.5 Specific identification and description of the test specimen or item inspected that includes field identification and detailed location information, for example, applicable horizontal and vertical coordinates of the sample source,

10.3.6 The date the test sample or item inspected was received by the agency, if applicable,

10.3.7 The standard test method(s) used with a notation of all known deviations from the referenced methods or requirements of the method(s), or both, not performed by the agency,

10.3.8 Identification of test results or other data, or both, obtained from subcontractor(s), and,

10.3.9 The results and other pertinent data required by the test or inspection method(s) used.

10.4 Agency test reports shall accurately and clearly present the specified test results and all pertinent data.

10.5 Corrections or additions to reports shall clearly reference the report being amended.

11. Keywords

11.1 construction materials testing; quality assurance; quality control; quality manual; quality system

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D3740 – 08) that may impact the use of this standard. (Approved March 1, 2010.)

(*1*) Changes were made to Section 7 Personnel Qualifications to remove the names of specific certification organizations. Additional certification/qualification criteria were added to provide guidance to the user.

(*2*) The minimum years of experience required for the Supervising Laboratory Technician and Supervising Field Technician was changed from 5 years to 3 years.

(*3*) Several notes which included the name(s) of specific non sole-source organizations were deleted, and the remaining notes were renumbered.

STANDARD OPERATING PROCEDURE

DM-004 Sonic Drilling

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1. Objective

Describe common sonic drilling procedures.

Prior to drilling confirm that utility clearance has been completed and that the drilling rig has been appropriately decontaminated.

2. Execution

- Collect soil cores in runs of 5 to 10 feet. Some sonic rigs can collect a 20 foot sample, but the process generates a significant amount of heat that may degrade sample quality.
- Classify and sample the soil located within the liner.
- Excess soil should be placed in a 55-gallon drum for disposal.
- The core barrel should be cleaned with tap water following each use.
- The core barrel is then advanced within the isolation casing to collect the next soil core interval.
- Add water between the inner core barrel and the outer override casing. This will reduce friction between the casings and adsorb heat.
- Maximize drilling advance rate. The faster the core barrel is advanced, the less likely the core barrel will heat up. Drilling with a 3-inch diameter core barrel and a 5-inch diameter override casing, instead of the standard 4-inch core barrel and 6-inch over-ride casing, may increase advance rates and reduce the potential for soil core heating.
- If a significant decrease in drilling advance rate is observed, stop drilling and remove soil that has accumulated in the core barrel. Resume drilling through the resistant material (gravel, boulder, hard clay, etc.). When the resistant material has been penetrated and the drilling advance rate increases, stop drilling and remove what material has accumulated in the core barrel.
- Wash down the core barrel with cool water to cool the core barrel and associated casing, and resume drilling.
- If a well is to be installed in the borehole, the sandpack and grout are placed as the core-barrel and over-ride casing(s) are selectively vibrated out of the ground. The vibratory action should facilitate settlement of the sandpack and grout. Upon completion, no casing is left in the ground other than the well casing and screen.

3. Limitations

- Disturbance of the soil core is most likely to occur during removal of the soil core from the core barrel. The soil cores are usually vibrated out of the core barrel into plastic bags approximately 5 feet in length. As the plastic bags are a little larger than the soil core itself, fragmentation of the soil core may occur as the core is extruded into the bag or while the bagged core is being moved in an unsupported manner. Soil conditions that are prone to disturbance include wet or dry zones that contain little or no fines, and well graded sands that contain significant volumes of water.

- If integrity of the soil core is of concern, the following procedures should be implemented:
    - Measures should be taken to ensure that the core, from the time it is extruded from the core barrel, is rigidly supported through the use of some type of cradle or carrying device.
    - The core should not be removed from its cradle until all sampling of the core has been completed. Acrylic liners are available for some core sizes and can be used to hold the core together upon removal from the core barrel.
    - If the soil is to be sampled for volatile organic compounds (VOCs), acrylic liners must be used.
    - Sampling of the soil core for VOCs or semi-volatile organic compounds (SVOCs) must be approved on a case by case basis. Proposals for VOC or SVOC soil core sampling must include provisions to minimize core fragmentation and heat generation, such as:
        - Acetate liners in the core barrel so that the soil core does not have to be extruded out of the core barrel.
        - Limit the length of soil core generated during a given downhole run.
        - Implement practices to reduce the residence time of the soil core in the core barrel.
- For the analysis of SVOCs, the use of the acetate liners is not required.
- The large diameter of the core barrel enables ground water sampling equipment to be placed inside the core barrel so that discrete depth groundwater samples can be collected during borehole advancement.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90

5. Contact

Melissa Felter

STANDARD OPERATING PROCEDURE

FD-001 Field Notebook

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1. Objective

Describe methods for documentation of field activities.

Documentation of site activities is a crucial part of the field investigation process. The field notebook serves as the record of field activities performed or observed during the project. It provides a factual basis for preparing field observation reports, if required, and reports to clients and regulatory agencies. Example field notes are provided in Attachment A.

2. Execution

- Use a separate all-weather bound notebook for each site/location/project number. Spiral notebooks should not be used because pages can be easily removed.
- Write neatly using black or blue pen, preferably a waterproof pen. Use of pencil is also acceptable only with approval of the project manager, such as in but not limited to, certain field conditions [e.g., cold or wet weather].
- Write the project name, project number, book number (i.e., 1 of 3), and date on the front cover. On the inside cover, identify the project name, project number, and “Return Book To:” the office address of the project manager.
- Number all of the pages of the field book starting with the first entry.
- Record activities as they occur. Record only facts and observations, regardless of whether they appear to be relevant at that time.
- Identify conditions or events that could affect/impede your ability to observe conditions (e.g. snow-covered ground surface, inability to access areas of interest).
- Neatly cross out mistakes using a single line and initial them. Erasures are not permitted.
    - If an error is made on an entry in the field notebook, the individual who made the entry should make the corrections. The corrections must be initialed and dated by the person making the correction.
- Sign or initial and date the bottom of every page with an entry if the project requires such documentation.
- Place a diagonal line through unused portions of a page.
- Record the following information upon each arrival at the site:
    - Date/time/weather.
    - GEI personnel.
    - Purpose of visit/daily objectives.
    - People (client, contractor, landowners, etc.) present upon GEI arrival.

- Record the following information during the course of the day:
    - Conversations with contractors/subcontractors, clients, visitors, GEI staff, landowners (site or abutters). If possible, record complete names, titles, and affiliations.
    - Time of arrival and departure of individuals.
    - Activities as they occur.

- Additional examples of observations to record may include and are not limited to:
    - Type and quantity of monitoring well construction materials used.
    - Use of field data sheets or electronic logging equipment (e.g. boring logs, monitoring well sampling logs, etc.).
    - Ambient air monitoring data.
    - Field equipment calibration information.
    - Locations and descriptions of sampling points.
    - Contractor/Subcontractor progress.
    - Sample media (soil, sediment, groundwater, etc.).
    - Sample collection method.
    - Number and volume of sample(s) collected and sample bottle preservatives used.
    - Sample identification number (s) and date and time of sample collection.
    - Approximate volume of groundwater removed before sampling.
    - Any field observations made such as pH, temperature, turbidity, conductivity, water level, etc.
    - References for maps and photographs of the sampling site(s).
    - Information pertaining to sample documentation: bottle lot numbers/ dates, method of sample shipments, chain-of custody record numbers, and overnight shipping numbers.
    - Surveying data (including sketches with north arrows).
    - Changes in weather.
    - Rationale for critical field decisions.
    - Recommendations made to the client representative and GEI Project Manager.
    - Site sketch of conditions at the end of the day.
    - Summary of work completed/work remaining.
    - Allow time at the end of the day to complete entries in the notebook.

3. References

New Jersey DEP Field Sampling Procedures Manual, August 2005.

ASFE Daily Field Report for Geotechnical Field Observation, 2nd Edition (2001), ASFE, Inc.

4. Attachments

Attachment A - Example Field Notes

5. Contact

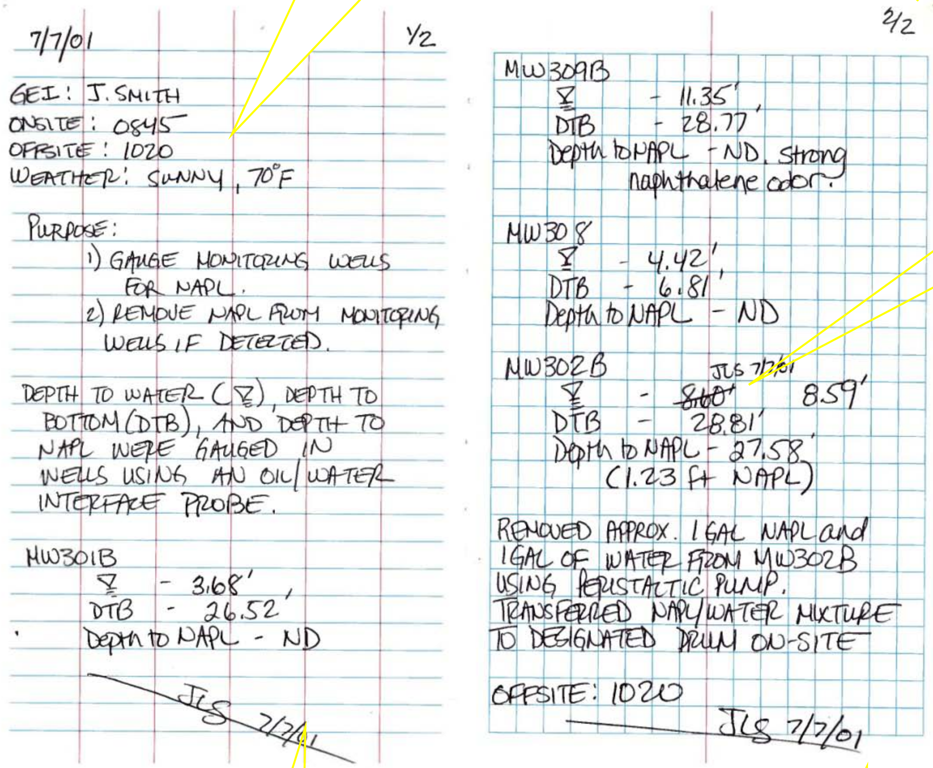
Melissa Felter
Leslie Lombardo

SOP FD-001

Attachment A – Example Field Notes

Start of each day includes:
- **Date**
- **Project Number**
- **People on site**
- **Purpose of Work**
- **Weather Conditions**

Each page is numbered



7/7/01 1/2

GEI: J. SMITH
ONSITE: 0845
OFFSITE: 1020
WEATHER: SUNNY, 70°F

PURPOSE:
1) GAUGE MONITORING WELLS FOR NAPL.
2) REMOVE NAPL FROM MONITORING WELLS IF DETECTED.

DEPTH TO WATER (▽), DEPTH TO BOTTOM (DTB), AND DEPTH TO NAPL WERE GAUGED IN WELLS USING AN OIL/WATER INTERFACE PROBE.

MW301B
▽ - 3.68',
DTB - 26.52'
Depth to NAPL - ND

JLS 7/7/01

2/2

MW309B
▽ - 11.35',
DTB - 28.77'
Depth to NAPL - ND. Strong naphthalene odor.

MW308
▽ - 4.42',
DTB - 6.81'
Depth to NAPL - ND

MW302B
JLS 7/7/01
▽ - ~~8.60'~~ 8.59'
DTB - 28.81',
Depth to NAPL - 27.58'
(1.23 ft NAPL)

REMOVED APPROX. 1 GAL NAPL and 1 GAL OF WATER FROM MW302B USING PERISTALTIC PUMP. TRANSFERRED NAPL/WATER MIXTURE TO DESIGNATED DRUM ON-SITE

OFFSITE: 1020

JLS 7/7/01

Errors are single line crossed out and initialed

Blank Space crossed out and initialed

Bottom of each page signed and dated

STANDARD OPERATING PROCEDURE

FD-002 Field Observation Report

---

1. Objective

Describe methods to generate a Field Observation Report.

The Field Observation Report is used to record a summary of activities, observations, and decisions made during the day's field work. The daily field observation report serves as a permanent record of the day's activity for the Project Manager (PM), In-House Consultant (IHC), and/or client.

2. Execution

- If required, at the close of the day's field work, a Field Observation Report should be prepared by the individual responsible for the field notebook. This report should be completed before leaving work for the day. Contents of the report should include, at a minimum, the following information:
    - A record of person(s) present at the site, time of arrival, departure times (e.g., GEI, contractor(s), client, etc.).
    - A record of the daily objective(s) and the activities performed (e.g., drilled five borings in the overburden).
    - A summary of deviation(s) from the field plan or objectives.
    - A summary of field decisions made, who made them, and the basis for such decisions.
    - A diagram, sketch, and/or map showing the location and extent of the work or other significant observation(s) made during the day.
    - Recommendations that may result from field observations and actions that may result from implementation of those recommendations.
    - A summary listing and field sketch showing location(s) of field activity.
- Submit a draft report to the PM/IHC for review. Complete any editorial changes, sign, date, and submit the report to PM/IHC for approval/signature. Field Observation Reports should be written neatly. They are not required to be typed unless specifically requested by the PM.

3. Limitations

- The Field Observation Report is not a substitute for the field notebook.
- Not all projects require daily Field Observation Reports.
- The Field Observation Report should be based primarily on factual information. Opinions, if necessary, should be identified as such. Any speculation should be clearly noted in the report as such.

- The Field Observation Report should never be released to anyone other than the PM/IHC prior to review and sign-off unless explicitly authorized by the PM/IHC.

4. References

New Jersey DEP Field Sampling Procedures Manual, August 2005

ASFE Daily Field Report for Geotechnical Field Investigations, 2nd Edition (2001), ASFE, Inc.

5. Attachments

Attachment A - Example Field Observation Report

6. Contact

Melissa Felter
Leslie Lombardo

FIELD OBSERVATION REPORT

Project : Guard Booth Upgrades
Client : ACME Industries
Contractor: ABC Contracting
Subcontractor: NA

Date: November 8, 2006
Report No. 1
Page: 1 of 2
GEI Proj. No. 99999-0

SOP FD-002 - Attachment A – Example Field Observation Report

Time of Arrival: 0700 **Departure: 1440** **Weather: Overcast, Raining, 55^0F**

Persons Contacted, Company
Jane Doe, ABC Contracting

GEI Representatives
Bill Smith

Purpose of Site Visit: To observe excavation of soils for new guard booth and sidewalk.

Observations:

1. **Excavation**
    a. Areas for guard booth and sidewalk were laid out by ABC with stakes, string, and spray paint. Locations were between the pavement and wetland area; no excavation occurred in the wetland area.
    b. Staging area for soil stockpile was located to the west of the excavation, along the fenceline; polyethylene sheeting was placed beneath the pile.
    c. HDPE membrane delivered to site; stored in garage area through the inside fence.
    d. ABC crew began hand digging area for sidewalk and guard booth. Sidewalk area measured 22 feet long by 4 feet wide by 4 inches deep. Guard booth area measured 12 feet long by 10 feet wide by 9 inches deep. Utility pole and bollard locations started today.
    e. Rain continued to get worse in the afternoon; ABC covered the entire excavation and soil stockpile with poly sheeting and secured the sheeting with grade stakes.

2. **Subgrade Preparation**
    a. Subgrade preparation for the sidewalk and guard booth areas at the site is complete.

3. **Dewatering**
    a. No dewatering occurred today.

4. **Air Monitoring**
    a. During excavation, I monitored the breathing zone of the workers with an organic vapor meter (OVM). No headspace readings were measured in soil samples S-1 through S-8.

FIELD OBSERVATION REPORT

Project :	Guard Booth Upgrades	**Date:**	November 8, 2006
Client :	ACME Industries	**Report No.**	1
Contractor:	ABC Contracting	**Page:**	2 of 2
Subcontractor:	NA	**GEI Proj. No.**	99999-0

Picture 1: Sidewalk excavation and bollard layout

By: Bill Smith	**Reviewed By:**

STANDARD OPERATING PROCEDURE

FD-003 Sample Management and Chain of Custody

---

1. Objective

Describe methods to label sample containers, manage the samples, and prepare Chain of Custody documentation for the samples. Sample transport is also addressed.

2. Project Setup

When setting up a sampling event, inform the recipients of the samples (laboratories) and recipients of laboratory results (data group and project managers). Discuss with the laboratory the sampling media, turnaround times, and reporting limits for appropriate regulatory criteria for the site. Include the data group on correspondence so that turnaround times, data validation, and project deliverable schedules can be tracked successfully.

- <u>Laboratory</u> - Number of samples, analyses needed: bottle orders and holding times, turnaround times needed, reporting limits needed for regulatory criteria.
- <u>Data group</u> - Number of samples, analyses requested, turnaround times and reporting limits requested, data validation needed, regulatory criteria to use for tabulating results, deliverables needed, and project name and number.
- <u>Schedule</u> - Inform the laboratory and Data Group of schedule delays, changes to analyses, and expediting.

3. Sampling Execution

- Review the work plan prior to sampling to determine the following:
    - Sample matrix and sampling method.
    - Required analysis and sample volumes.
    - Sample container type and preservative requirements.
    - Required analysis methods and/or report formats.
    - The turnaround time required by the project.
    - If the data will be sent directly from the laboratory to the data validator, Project Manager, or Data Group.
    - Holding time restrictions for sampling media and analytical methods.
    - Sample naming convention used for this project site.
- Sample labels should be filled out using a waterproof or permanent marker or pen. Required information includes:
    - Sample ID.
    - Date and time (military time) of sample collection.
    - Project number.
    - Sample preservatives.
    - Sampler’s initials.
    - Laboratory analytical methods.

- Place the label on the jar or bottle, not on the cap. Sample custody begins at this time.
- Record the above information in the field notebook.
- Individually wrap sample jars with packing material, if needed. See SOP SC-002 for guidance on packaging samples for shipment to the laboratory by way of common carrier. Place samples in a cooler with bagged ice or freezer packs (blue ice) immediately after collection. Add sufficient ice or freezer packs to cool samples to approximately 4°C.
- Complete a chain of custody (COC) for the samples as described below. GEI or laboratory COCs may be used as long as they contain fields for all required sample information as described in Section 2.1.

3.1. Chain-of-Custody (COC) Completion

- Fill out COC neatly and in permanent ink. Alternatively, an Excel version of the GEI COC is available and can be filled out electronically.
- Certain analyses (i.e. air analysis by TO-15) require specialized, laboratory issued COCs. Make sure any specialized COCs are available before sample collection.
- Record the project name and number, the sampler's name(s) and the state where the samples were collected.
- For each sample, enter the sample identification number, date and time (military time) collected, the number of sample containers, and any additional information to fulfill project, client or regulatory requirements.
- Record the type of analysis (including laboratory method; e.g. EPA-SW846 Method XX) requested and the preservative (if appropriate) in the vertical boxes.
- Field duplicates should be anonymous to the laboratory, but must be recorded for use by the Data Group. To keep track of this information, link the field duplicate with the proper sample in the field notebook. If required by the Project Manager or Data Group, also document this information on or attach a note to the GEI copy of the COC.
- Trip blanks for large sites should be named similar to the samples they are collected with so that there are not two of the same sample name for the same site. For example, "OU1TB-122509" and "OU3TB-122509" would avoid any mistakes.
- Strike incorrect entries on the COC with a single line, followed by the initials of the person making the correction, the date, and the correct entry.
- When sample custody is ready to be relinquished, complete the bottom of the form with date and time (military time) and signatures of relinquisher and receiver of samples as indicated. The sample collector is always the first signature while the analytical laboratory is the final signature. Theoretically, all individuals handling the samples between collection and laboratory should sign the form; however, if a common carrier (i.e., Federal Express, UPS) is used for shipping, GEI must identify the carrier in the 'Received by' box on the

COC. If the sampler hand delivers the samples to the laboratory, the received box must be signed by the laboratory.

- If the samples are placed in a designated secure area (e.g. GEI sample fridge), note this location in the “Received by” box on the COC.
- GEI uses both single sheet and triplicate COCs. If using the triplicate COCs (white, yellow, and pink copies), the pink copy should be retained by the sampling personnel and provided to the Data Group for proper filing. The white and yellow copies should accompany the samples to the laboratory.
- If you are using the single sheet COC, make a copy of the COC after it has been signed by the lab courier and forward it to the Data Group.
- Prior to sample shipment by common carrier, the COC must be placed inside the cooler in a Ziplock bag or other watertight package.
- If a common carrier such as FedEx is used to transport the samples to the laboratory, include the carrier tracking number and identify the carrier in the “Received by” box on the COC.
- If a courier is used to transport samples to the laboratory (lab courier or GEI personnel), the courier signs the COC in the “Received by” box.
- Place a custody seal on the cooler if shipping via common carrier.
- Transport samples to the laboratory as soon as possible. It is preferable to transport the samples directly to the laboratory from the field. Samples brought back to the office for storage prior to submission to the laboratory must be kept cold (4° C).
- Unused sampling containers/media that are sent back to the lab should be included on a separate COC.
- After the samples are sent to the laboratory, the GEI copy of the COC must be forwarded to the Data Group: datagroup@geiconsultants.com.

4. Limitations

- Keep the number of people involved in handling samples to a minimum.
- Where practical, only allow people associated with the project to handle the samples.
- Always document the transfer of samples from one person to another on the COC.
- The COC should always accompany the samples.
- Give samples positive identification at all times that is legible and written with waterproof or permanent ink.
- When sending samples via a common carrier, use one COC per package.
- Where practical, avoid sending samples from more than one site with separate COCs in a single package.

5. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

6. Attachments

Attachment A - Example Chains of Custody
Attachment B - Shipping Info Pics

7. Contact

Brian Skelly
Leslie Lombardo

Chain of Custody Record

STL Connecticut
128 Long Hill Cross Road
Shelton, CT 06484
Tel: 203-929-8140

Example COC

SEVERN TRENT **STL**
Severn Trent Laboratories, Inc.

STL-4124 (0901)

Client: GEI
Project Manager: Dave Terry
Date: 12-31-07
Chain of Custody Number: 00452
Address: 455 Winding Brook Dr
Telephone Number (Area Code)/Fax Number: 860 368 5300 / 860 368 5307
Lab Number:
City: Glastonbury
State: CT
Zip Code: 06033
Site Contact: M. Felter
Lab Contact: Paul Hobart
Page 1 of 1
Project Name and Location (State): Carroll Gardens NY
Carrier/Waybill Number: FedEx 9383 7603 0879
Contract/Purchase Order/Quote No.:

Analysis (Attach list if more space is needed)

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Matrix: Air	Matrix: Aqueous	Matrix: Sed.	Matrix: Soil	Matrix:	Containers & Preservatives: Unpres.	H2SO4	HNO3	HCl	NaOH	ZnAc/NaOH		VOC 8260B	SVOC 8270C	TO-15 + NAPHTHALENE	Special Instructions/ Conditions of Receipt
CGSB-01 (0-2)	12-31-07	1130				X		X							X	X		
CGSB-02 (3-4)	12-31-07	1250				X		X							X	X		
CGSB-02 (3-4) MS	12-31-07	1250				X		X							X	X		
CGSB-02 (3-4) MSD	12-31-07	1250				X		X							X	X		
CGSB-XX (5-6)	12-31-07	0800				X		X							X	X		
CGTB-123107	12-31-07	1400					TB				X				X			
CGGW-01	12-31-07	1430		X				X			X				X	X		
CGSG-01	12-31-07	0700-1500	X														X	CANISTER# 2013 REGULATOR# 779

~~NEW JERSEY: "CLP data package deliverables"~~

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☒ Unknown

Sample Disposal
☐ Return To Client ☒ Disposal By Lab ☐ Archive For ______ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☒ Other See contract

QC Requirements (Specify) NYS Cat B ASP deliverable send to Lorie Mackinnon Send EDD to datagroup@geiconsultants.com

1. Relinquished By: Melissa J Felter — Date: 12-31-07 — Time: 1600
1. Received By: FedEx WAYBILL 923117-4432 — Date: 12-31-07 — Time:
2. Relinquished By: — Date: — Time:
2. Received By: — Date: — Time:
3. Relinquished By: — Date: — Time:
3. Received By: — Date: — Time:

Comments
USED FLOW CONTROLLER FOR AIR SAMPLE INCLUDED

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

EXAMPLE COC

Chain-of-Custody Record

Laboratory: Accutest

Laboratory Job # (Lab use only)

GEI Consultants
400 Unicorn Park Dr.
Woburn, MA 01801
PH: 781.721.4000
FX: 781.721.4073

Project Information

Project Name: MWRA - Low Service Storage Tank

Project Location: Stoneham, MA

Project Number: 093400

Project Manager: D. Aghjayan

Page _1_ of _1_

Send Report to: rseigener@geiconsultants.com

Send EDD to: labdata@geiconsultants.com

Sample Handling

Samples Field Filtered
YES NO NA

Sampled Shipped With Ice
YES NO

MCP PRESUMPTIVE CERTAINTY REQUIRED – YES NO

If Yes, Are MCP Analytical Methods Required? YES NO NA

If Yes, Are Drinking Water Samples Submitted? YES NO NA

If Yes, Have You Met Minimum Field QC Requirements? YES NO NA

Lab Sample Number	GEI Sample ID	Collection Date	Collection Time	Matrix	No. of Bottles	Sampler(s) Initials	PCBs	VPH Fractions Only	EPH Fractions Only	SVOCs	VOCs, % Solids**	Conductivity	RCRA 8 Metals*	TPH by GC/FID DRO	Sample Specific Remarks
Preservative							None	MeOH	None	None	MeOH	None	None	None	
	093400-LS6-S5(19'-21')	12/29/2009	9:30	SO	3	JMR		x			x				
	093400-LS6-COMP (FILL)	12/29/2009	9:30	SO	1	JMR	x		x	x		x	x	x	
	093400-LS6-COMP (NATIVE)	12/29/2009	15:00	SO	1	JMR	x		x	x		x	x	x	
	093400-LS8-COMP	12/29/2009	14:00	SO	1	JMR	x		x	x		x	x	x	
	093400-LS9-S4 (8'-8'-5")	12/30/2009	14:30	SO	3	JMR		x			x				
	093400-LS9-COMP	12/30/2009	15:00	SO	1	JMR	x		x	x		x	x	x	

MCP Level Needed: GEI requires the most stringent Method 1 MCP standard be met for all analytes whenever possible.

Relinquished by: (signature)	Date :	Time:	Received by: (signature)
1. Joseph M. [illegible]	12/30/09	16:30	1. GEI FRIDGE
2. GEI FRIDGE	1/4/10	1310	2. Naomi [illegible]
3. Naomi [illegible]	1/4/10	1310	3.
4.			4.

Turnaround Time (Business days):

Normal _X_ Other ____
10-Day ____ 7-Day ____
5-Day ____ 3-Day ____

Before submitting rush turnaround samples, you **must** notify the laboratory to confirm that the TAT can be achieved.

Additional Requirements/Comments/Remarks:

Please use MA Landfill List

* Please run TCLP analysis for RCRA 8 Metals results that exceed the 20 times rule.

** Please use % solids sample for VOC and VPH analysis of 093400-LS6-COMP (FILL), 093400-LS6-COMP (NATIVE), 093400-LS8-COMP, and 093400-LS9-COMP

Chain-of-Custody Record

Laboratory:

Laboratory Job #
(Lab use only)

GEI Consultants

455 Winding Brook Drive, Suite 201
Glastonbury, CT 06033
PH: 860.368.5300
FX: 860.368.5307

Project Information

Project Name/Number:	**Project Location:**
Project Contact:	**Phone Number:**

Page __ of __

Send Report to:

Send EDD to: datagroup@geiconsultants.com

Preservative

Analysis

Sample Handling

Samples Field Filtered
YES NO NA

Samples Shipped With Ice
YES NO

Deliverables:

If Yes, Have You Met Minimum Field QC Requirements? YES NO NA

Lab Sample Number	**GEI Sample ID**	**Collection**		**Matrix**	**No. of Bottles**	**Sampler(s) Initials**										**Sample Specific Remarks**
		Date	**Time**													

Relinquished by sampler: (signature) 1.	Date :	Time:	Received by: (signature) 1.
Relinquished by: (signature) 2.	Date :	Time:	Received by: (signature) 2.
Relinquished by: (signature) 3.	Date :	Time:	Received by: (signature) 3.
Relinquished by: (signature) 4.	Date :	Time:	Received by: (signature) 4.

Turnaround Time (Business days):

Normal _____	**Other** _____
10-Day _____	**7-Day** _____
5-Day _____	**3-Day** _____

Before submitting rush turnaround samples, you **must** notify the laboratory to confirm that the TAT can be achieved.

Additional Requirements/Comments/Remarks:

1/17/2011
H:\STAFDATA\Data Management\Procedures\Chains-of-Custody (COCs)\Chain of Custody_Atlantic 2010.xls

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

PACKING SAMPLES FOR SHIPMENT BACK TO THE LABORATORY

A. Line cooler with bubble wrap and large plastic bag. Use absorbent pad inside the bag if bottles contain preservatives.

B. Wipe outside of bottles and put glass in individual bubble bags & seal. Place bottles & the temperature blank into cooler. Leave room for ice in between bottles & on top.

C. Place double bagged or loose ice randomly around bottles throughout the cooler.

D. Place large bag of ice or loose ice on top of the bottles. In warm weather, the cooler should be packed with as much ice as possible.

E. Close outer bag, compress excess air out of bag, twist top and knot. If necessary, use more bubble wrap to fill the dead air spaces. Place chain of custody (COC) and other paperwork in plastic bag and seal. Place on top of cooler.

F. Close cooler, place signed and dated Custody Seals over opening. Tape over the Custody Seal and seal cooler securely. Fill out overnight shipping waybill and attach to the top or handle of the cooler. Attach Saturday delivery stickers if needed. Ship according to DOT regulations.

Rev 011209

PACKING SAMPLES FOR SHIPMENT BACK TO THE LABORATORY

A. Line cooler with bubble wrap and large plastic bag. Use absorbent pad inside the bag if bottles contain preservatives.

B. Wipe outside of bottles and put glass in individual bubble bags & seal. Place bottles & the temperature blank into cooler. Leave room for ice in between bottles & on top.

C. Place double bagged or loose ice randomly around bottles throughout the cooler.

D. Place large bag of ice or loose ice on top of the bottles. In warm weather, the cooler should be packed with as much ice as possible.

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F. Close cooler, place signed and dated Custody Seals over opening. Tape over the Custody Seal and seal cooler securely. Fill out overnight shipping waybill and attach to the top or handle of the cooler. Attach Saturday delivery stickers if needed. Ship according to DOT regulations.

STANDARD OPERATING PROCEDURE

FD-004 Photo Documentation

---

1. Objective

Describe methods to document and retain photographic records.

Keeping a record of photographs taken is crucial to their validity as a representation of existing conditions.

2. Execution

- Photographs of a site, individual samples, or other observations should be taken using a digital camera.
- Set the camera to record the time and date for each photograph.
- All photographic records, along with the following information, should be recorded in the field notebook (SOP FD-001).
    - If applicable, the compass direction describing the direction the photograph was taken (e.g. looking southeast). This may not apply to photographs of individual samples.
    - Brief description of what the photograph is intended to show.
- The field notebook should note who took the photographs.
- The photographs should be electronically backed up on a computer or other data storage device.
- If photographs will be used in a report, memo, or letter, they should be placed on a photograph record template and the relevant information describing the photograph should be inserted into the caption section for each photograph.

3. Limitations

- Some clients and regulatory agencies require photographs of every subsurface soil sample collected. These photographs typically include a “whiteboard” which indicates the site, the boring ID, and the depth of the sample, while logging details are recorded in the field notebook. Under these circumstances, it is not necessary to include compass directions or descriptions.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

5. Attachments

Attachment A – Example of Photo Documentation Template

6. Contact

Melissa Felter
Leslie Lombardo

Attachment A – Example of Photo Documentation Template

GEI Consultants, Inc.

Project: Project Name

Location: Project Location

Photographer:	K. Barber
Date:	10/25/07
Photo No.:	1
Direction:	N

Comments:
Entrance of site with tree mulching operations.

Photographer:	K.Barber
Date:	10/25/07
Photo No.:	2
Direction:	W

Comments:
On-site building built in 1936.

STANDARD OPERATING PROCEDURE

FD-006 Handheld Global Positioning Receiver Operation

---

1. Objective

Use handheld global positioning system (GPS) receivers to locate sample points and site features with "Mapping-Grade" accuracy.

Use handheld GPS receivers to "stake out" proposed sample point locations within the limits of "Mapping Grade" accuracy.

2. Execution

- Handheld GPS receivers provide a low-cost and user-friendly method for locating sample points and site features with a fair degree of horizontal accuracy.
- In simplistic terms, GPS works by measuring the distance from numerous orbiting satellites to a point on the earth surface. Individual satellites broadcast their real-time location in terms of x,y and z coordinates, and the distance from each satellite is measured as a function of the length of time that a time-stamped signal takes to reach the receiver. Built-in GPS software derives new points by intersecting the distances from known orbital locations – in much the same way that points are located by intersecting tape-measured distances from building corners or other pre-existing site features.
- Late-model handheld GPS receivers utilize a real-time differential correction technique called WAAS (Wide Area Augmentation System). This system was designed to provide greater confidence and reliability in using GPS data for commercial aircraft landing approaches, and the additional correction improves all GPS operations.
- Handheld GPS receivers display navigational information on a variety of standard pages. Although each manufacturer uses slightly different formats, all receivers toggle back and forth between the following visual presentations:
- A "satellite" page displays the relative orbital location of all GPS satellites that are currently being tracked by the receiver. The display may include information on the real-time geometrical strength of the solution: satellite intercepts that cross at right angles provide more accurate solutions than intercepts that cross at acute or obtuse angles.
- A "track" page that displays the travel path of the receiver while it is turned on, along with the relative location of recorded points. Many GPS models have a "track-back" function that will guide the user on the same path back to the starting point
- A "navigation" page that displays instantaneous location and the real-time direction and velocity of travel. Some units provide two pages to display this information in different formats. Most units will report the overall "course

made good" (straight-line bearing and distance from the starting point) at any point.

- A "waypoint" page that allows users to "Go To" a created point or previously recorded point by providing a straight-line bearing and distance to the point. The information is instantaneously updated as the user moves along; some units display a pointing arrow that directs the user to the direction of travel. Be careful of go-to lines that lead through swamps or over cliffs – if you will be travelling in difficult terrain have a paper copy of the USGS quadrangle and a compass on hand for navigation.
- Signal strength degrades significantly next to buildings and underneath tree canopy. Most GPS receivers have an "averaging" function to improve the accuracy of shielded locations. GPS users can also improve precision by locating points three times, at different times of the day. Two of the solutions will generally be closer to each other than to the third and can be averaged for a more reliable fix.
- Most GPS receivers default to latitude and longitude, but data is more accurate and easier to input and when expressed in UTM coordinates to the nearest meter. The handheld GPS setup will have a function somewhere to change to UTM. Most of Connecticut is in UTM Zone 18 but the easternmost parts are in Zone 19.
- Consult "Corpscon" the datum translator available from the National Geodetic Survey website. Corpscon translates instantly from latitude/longitude to UTM coordinates to state plane coordinates and provides tools to identify UTM Zones. Also consult the Trimble, Garmin and Magellan websites for technological improvements and discussion of advanced techniques.

3. Limitations

- Handheld GPS receivers operating in unobstructed locations are currently reckoned to provide 2-5 meter accuracy, meaning that the true location of measured points lie within an "error ellipse" with axes of 2-5 meters centered on the measured location. In other words, even under the best of conditions a real-time GPS solution may be as much as 20 feet off the true horizontal location of a point.
- Due to geodetic restrictions, vertical locations (elevations) have less than half the accuracy of horizontal locations, meaning that even under the best of conditions, a surface elevation displayed on a handheld GPS receiver may be off by more than 50 feet.
- Horizontal and vertical data derived from handheld GPS receivers should never be considered more than relatively accurate, and this level of uncertainty should be identified in any discussion of positional tolerance.

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Garmin Website: .garmin.com

Magellan Website: **.magellan**gps.com
National Geodetic Survey: ://www.ngs.noaa.gov/

5. Contact

Doug Bonoff, PLS

IMPROVED TECHNIQUES FOR THE IN-SITU DETERMINATION OF UNDRAINED SHEAR STRENGTH IN SOFT CLAYS

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ABSTRACT

Undrained shear strength results from the T-bar and Ball penetrometers are compared with those obtained from the field vane, piezo cone, and dilatometer at three sites in the Lower Mainland of British Columbia. The sites include deltaic deposits of soft clay silts and moderately to highly sensitive clays. The sites consist primarily of normally consolidated soils. Test procedures and methods of interpretation are described for each in situ test and lab test. The ability of the T-bar and Ball penetration test to accurately and reliably determine undrained shear strength of clayey soils is discussed in relation to the field vane test, piezo cone test, and the flat dilatometer test.

RÉSUMÉ

Les résultats de la résistance au cisaillement non-drainé de la T-barre et les pénétromètres de boules sont comparés avec ceux qui ont été obtenus à partir d'un essai in situ au scissomètre, piézocône, et un essai au dilatomètre, pris de trois emplacements au sud-ouest de la Colombie Britannique. Les sites incluent les dépôts deltaïques de vase d'argile molle et les argiles modéréments à extrêmement sensibles. Les lieus consistent principalements des sols normalement consolidés. Les méthodes d'essais et d'interprétations sont décrits pour chaque essai in situ et pour chaque essai laboratoire. La capacité des essais de la T-barre et les pénétromètres de boule d'être exact pour sûrement déterminer la résistance au cisaillement non-drainé des sols argileux est discuté par rapport aux essais in situ au scissomètre, piézocône et au dilatomètre.

Key words: T-bar, Ball, CPT, CPTU, Dilatometer, Vane, Undrained Shear Strength, In-Situ Testing, Unconsolidated Undrained

1. INTRODUCTION

Geotechnical engineering design in the Lower Mainland of B.C. frequently requires characterization of the undrained shear strength (s_u) profile in saturated soft sediments. Most commonly, s_u is determined from in situ test results, typically comprising electronic piezometer cone penetration testing (CPTU) or field vane shear testing (FVT). The CPTU offers the advantage of a continuous profile of parameters which allow interpretation of stratigraphy as well as estimates of s_u. Site specific correlations are frequently developed by carrying out adjacent CPTU and FVT soundings.

In very soft sediments, errors introduced by large corrections to the raw data, and the resulting uncertainty and variability of correlation factors between s_u and primarily CPTU parameters has led to the development of new in situ tools for use in these soils, especially in the offshore.

The purpose of this paper is to present the results of experience in the Lower Mainland with two new tools, the T-bar and the Ball penetrometers, examples of full-flow penetrometers. Both the size and the shape of the full-flow penetrometers provide advantages over the standard CPTU in soft soils. The increased size results in better measurement resolution and the full-flow movement of soil around the probes result in only minimal influence of overburden pressure on the calculation of shear strength. The rationale behind the development of the tools is provided and guidelines for the selection of equipment, data collection and reduction of the results are presented. Profiles of estimated s_u are provided for three sites and are compared to values interpreted from CPTU and dilatometer (DMT) profiling.

2. BACKGROUND

The s_u profile is typically determined from the CPTU net tip resistance, $q_{net} = q_t - \sigma_{vo}$, using the following relationship:

$$s_u = \frac{q_t - \sigma_{vo}}{N_{kt}} = \frac{q_{net}}{N_{kt}} \qquad (1)$$

where N_{kt} is an empirical factor, q_t is the measured tip resistance, q_c, corrected for unequal end area pore pressure effects on the cone tip.

In very soft, normally to lightly overconsolidated sediments, σ_{vo} can be a significant proportion of q_c and the pore pressure can be similar in magnitude to q_c. These effects introduce uncertainty to the estimated values of s_u and are considered the likely reason for the large scatter in published N_{kt} values. For this reason, Lunne et al. (1997) recommended the use of excess pore pressure instead of q_{net} to derive s_u for very soft soils.

In an effort to reduce the inaccuracies due to these large corrections but to continue to achieve a continuous profile of resistance, the T-bar test was introduced first in the centrifuge (Stewart and Randolph, 1991) and then in the field (Stewart and Randolph, 1994). The T-bar is a cylindrical bar mounted at 90 degrees to the push rods. Since its introduction, field testing has been carried out at well-characterized sites in Australia (Chung and Randolph 2004), Norway (Lunne et al. 2005), Ireland (Long 2005), and the USA (DeJong et al., 2005). The Ball penetrometer, a spherical ball mounted on the end of the push rods, has been mainly assessed in the centrifuge (Watson et al. 1998) or by numerical modelling (Lu et al. 2000).

In full-flow penetrometers, the soil is assumed to flow around the cylinder or ball during penetration and so the overburden pressure is equilibrated above and below, except at the shaft. The corrections are thus much smaller than for the cone. The analysis procedure is based on the plasticity solution of Randolph and Houlsby (1984), which shows that the undrained strength is determined by:

$$s_u = \frac{q_{net}}{N} \qquad (2)$$

where q_{net} is the net resistance and N is a bearing capacity factor. The general equation for net resistance for push in tools is as follows:

$$q_{net} = q_c - [\sigma_{vo} - u_2(1-a)]\frac{A_s}{A_p} \qquad (3)$$

where q_c is the measured resistance, *a* is the area ratio, u_2 is the pore pressure measured at the standard location, just above the shoulder of a standard cone or just behind the joint between the T-bar or Ball and the push rods, A_s is the cross sectional area of the cone shaft, and A_p the projected area of the tip. For the CPTU, the area ratio A_s/A_p is unity and Equation 3 reduces to the standard expression $q_{net} = q_t - \sigma_{vo}$. For the 100 cm^2 flow penetrometers A_s/A_p is 0.1, resulting in a much smaller difference between q_c and q_{net} than is typical for the CPTU.

3. TESTING PROGRAM AND TEST SITES

3.1 Testing Equipment and Procedures

The following insitu testing tools were used at the test sites:

- Standard 10cm^2 CPTUs with full scale tip capacities of 25 and 100 MPa
- Nilcon Field Vane (FVT)
- Flat Plate Dilatometer (DMT)
- Two T-bars and a Ball with dimensions given in Table 1.

The flow penetrometer tips were deployed on a 10cm^2 CPTU module replacing the regular cone tips, as shown in Figure 1. When a 100 MPa capacity cone is used, the maximum capacities are a function of the size of tip used.

Tip	Projected Area (cm^2)	Capacity (MPa)
Standard cone	10	100
Small T-bar 160mm span x 35.7mm diameter	57.1	17.6
Large T-bar 250mm span x 35.7mm diameter	89.3	11.2
Ball 113 mm diameter	100	10.0

Table 1. Details of Penetrometers

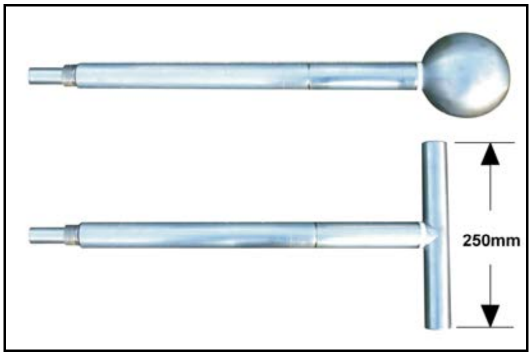

Figure 1. T-bar and Ball Penetrometer Tips

CPTU, T-bar, and Ball penetration tests were carried out at the standard rate of 2 cm/s, tip, friction and u_2 pore pressure data being recorded at 5 cm depth intervals. Sleeve and pore pressure recorded in T-bar and Ball soundings give an indication of stratigraphy but cannot be used with conventional CPTU correlations to soil behaviour type. End resistance of the T-bar and Ball penetrometers was also measured during retraction of the probes. Previous researchers have roughened the surface of the T-bar by sandblasting as theory suggests that N factors vary with surface roughness. This was not done in this testing program.

Field vane testing was carried out using a Nilcon field vane. In some cases, the field vane was advanced from surface, and at other times, the vane testing was performed in conjunction with mud rotary boreholes. The field vane testing was carried out in accordance with ASTM D 2573-01 (2001). The Nilcon field vane allows separation of the rod friction from the torque required to turn the shear vane, which is a significant correction when advancing the vane from surface.

The flat dilatometer test was carried out in accordance with the procedures outlined by Marchetti et al (2001). A special soft membrane was used due to the low strength of the soil being tested.

Piston samples were also obtained from mud-rotary boreholes using a hydraulically actuated sampler. Conventional 3 inch diameter galvanized sample tubes conforming to ASTM 1587-00 (2000) were used. Samples were used for classification testing and undrained shear strengths were measured by unconsolidated undrained (UU) triaxial compression tests.

3.2 Test Sites

Field testing was carried out at three sites in the Lower Mainland of BC, two in the Serpentine River lowland and one in the Fraser River delta. The geological history suggests that surficial soils at all three should be normally consolidated although water level and climatic variations may have resulted in some light overconsolidation. Details of the sites are given below.

3.2.1 Colebrook

The Colebrook site is located under the Highway 99A overpass over Colebrook Road and the adjacent BC Railway (BCR) line, in South Surrey, B.C. It is in the northwest corner of the Serpentine River Lowland, 2.5 km east of the sea at Mud Bay. The subsoils in the western region of the Serpentine River Lowland are Salish Sediments, which are post-glacial deposits of the Quaternary period that were laid down between 10,000 and 5,000 years ago, and include both terrestrial and marine sediments (Armstrong, 1984).

The ground surface at the site lies below sea level, varying between –1.1 and –1.3 m elevation. The test site is covered with a 0.5 to 0.7 m thickness of fill material overlying 0.2 to 0.3 m thickness of peat, which formed the original ground surface. The peat is underlain by a layer of clayey silt interbedded with seams of fine sand to sandy silt which extends to about 2 m depth. These surficial soils are underlain by an extensive deposit of marine clayey silt to silty clay, which extends to a depth of about 25 metres. More details are provided in Weech and Howie (2001).

3.2.2 Mud Bay

The Mud Bay site is located south east of the Colebrook site in the same geological sequence as the Colebrook site but further to the south-east. Here the organic and marine sediments overlying the Vashon Drift are about 15m thick.

3.2.3 Richmond (Vulcan Way)

This site is located in the Fraser River Delta. Soil conditions consist of overbank deposits (clayey silt) with fine sand layers overlying deltaic distributary channel fill sediments.

4. TESTING RESULTS

4.1 Effects of corrections

Figure 2 shows typical profiles of uncorrected and net resistance calculated using Equation 2 for T-bar and standard cone at the Colebrook site. The reduced importance of the correction in the T-bar test is clear when compared to the CPTU profiles.

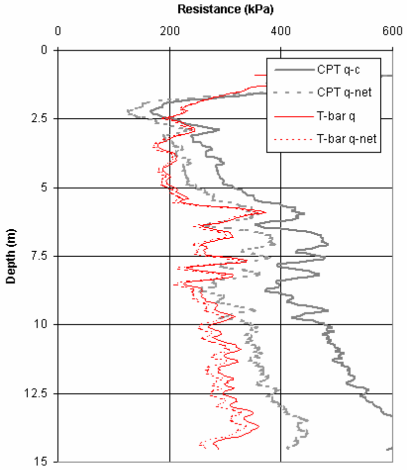

Figure 2. Measured and net resistance for CPTU and T-bar, Colebrook site.

4.2 Comparisons of q_{net} and derivation of N factors

Figure 3 shows q_{net} versus depth for the cone, Ball and small and large T-bars, also at Colebrook. For all three sites, it was observed that q_{net} was similar for the various full flow tips, while for the CPT, the net resistance was higher by about 10 to 15%. For each site, N factors were calculated from the q_{net} profiles using Equation 2 and the vane shear strengths, $(s_u)_{FV}$, as a reference. The values obtained and their standard deviations are shown in Table 2. For the T-bars and Ball, N varies from 10.0 to 12.0 and for the CPT from 11.8 to 15.0. These numbers are in the same range as those obtained elsewhere and predicted by theory.

Test	Colebrook	Mud Bay	Richmond
CPT	13.4 (2.9)	15.0 (4.2)	11.8 (2.7)
Large T-bar	10.7 (2.0)	12.0 (3.0)	10.2 (2.8)
Small T-bar	10.8 (2.1)	11.6 (3.4)	10.0 (2.8)
Ball	10.3 (1.8)		10.0 (2.0)

Table 2. Summary of calculated N Factors – standard deviations shown in brackets

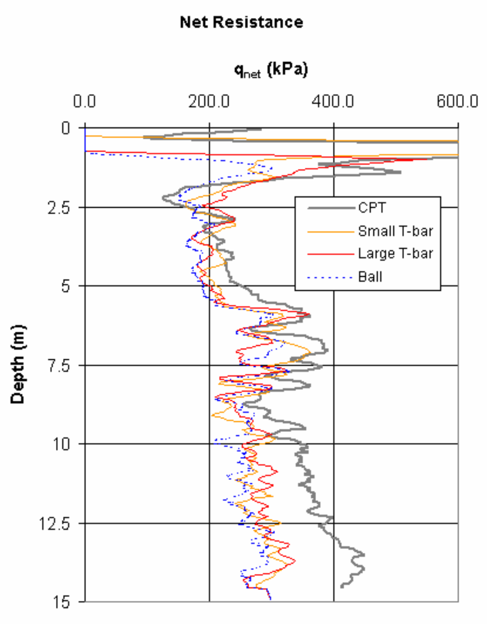

Figure 3. Comparison of q_{net} profiles - Colebrook

4.3 Undrained Shear Strength Profiles

Figures 4, 5 and 6 show profiles of predicted $(s_u)_{FV}$ at the three sites based on the back-calculated N values in Table 2. The measured field vane shear strengths and the results of triaxial UU tests are also shown. Values of s_u interpreted from DMT profiling using the standard Marchetti method are shown for the Mud Bay site.

As site specific N factors were used, agreement between the estimated and measured $(s_u)_{FV}$ values is excellent. Use of a standard N_{kt}=15 with CPTU data at all sites would have underestimated strengths at Colebrook and Richmond. Conversely, if the recommended average value of N=10.5 had been used with the T-bar resistances (Chung and Randolph, 2004), the shear strengths would have been overestimated at Mud Bay but would have been accurate at the other sites.

The s_u predictions by DMT using standard correlations underestimate shear strength at Mud Bay in the regions for which vane shear data are available. The data suggests that a different N value may be appropriate for the zone below 10 m depth.

Shear strengths measured in UU triaxial tests are comparable to $(s_u)_{FV}$ at shallow depths but are considerably lower at greater depths.

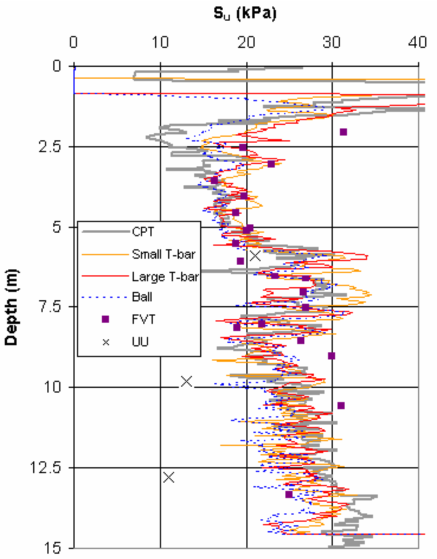

Figure 4. Colebrook site undrained strength profile from CPT, FVT, UU, and full flow penetration tests

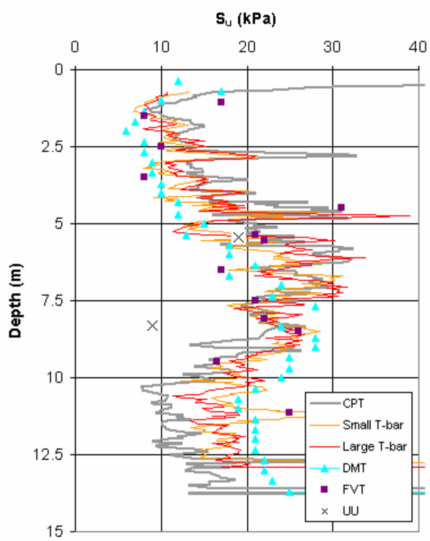

Figure 5. Mud Bay site undrained strength profile from CPT, DMT, FVT, UU, and full flow penetration tests

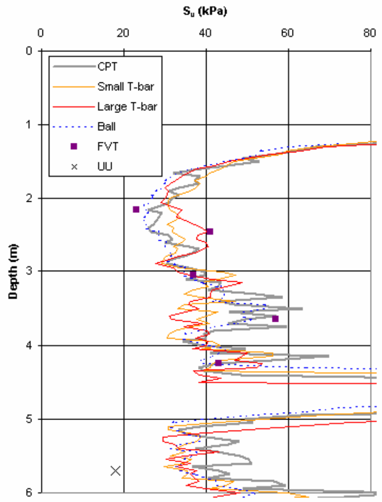

Figure 6. Richmond site undrained strength profile from CPT, FVT, and full flow penetration tests

4.4 Post Peak and Remoulded Strengths

The geological history and index properties at Colebrook and Mud Bay suggest that the soils may have been leached and sensitive. They would thus be expected to exhibit brittle behaviour in the field vane test. The sensitivity, S_t, is determined from the field vane shear test as the ratio of the peak to remoulded s_u. Figure 7 shows S_t vs depth for Colebrook. The test results indicate the soils at Colebrook and Mud Bay to have a FVT sensitivity of about 5 to 10, with S_t increasing with depth. Weech (2002) and Crawford and Campanella (1991) observed S_t values ranging from 6 to 26 in the soils at Colebrook

Lunne et al. (1997) suggest that the CPT friction sleeve, f_s, is close to the remoulded s_u and so S_t can also be estimated to a first approximation using $(s_u)_{peak}$ estimated from q_{net} divided by f_s. As an alternative, Newson et al (2004) have used the ratio of q_{net} measured while pushing to q_{net} measured during retraction to make an estimate of sensitivity. Chung and Randolph (2004) suggest that the ratio of the peak q_{net} to the ultimate resistance after cyclic extraction-penetration tests will ensure complete remoulding and thus may also give a better estimate of S_t. Measuring the resistance during retraction of the full flow probes adds very little time to the test procedure, and provides a continuous profile. However, it may not result in full remoulding of the soil.

Plots of sensitivity calculated from FVT, CPTU, T-Bar and Ball are presented in Figure 7 for Colebrook and in Figure 8 for Richmond. The CPT sensitivity is lower than the FVT values at the Colebrook but is a reasonable first estimate. The ratio of peak q_{net} to retraction q_{net} appears to be more representative of the ratio of peak to post peak s_u from field vane testing.

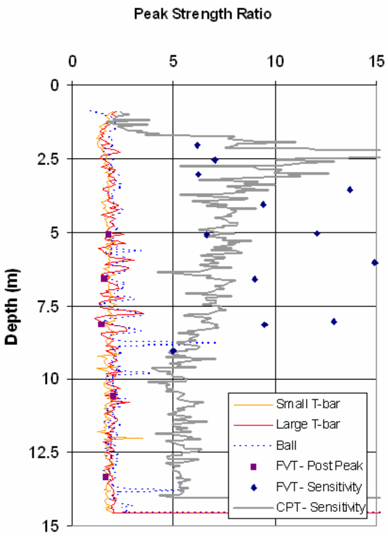

Figure 7. Ratio of peak undrained shear strength to post peak and remoulded strength, Colebrook site.

At both the Colebrook site (Figure 7) and the Mud Bay site, the ratio of net resistance during insertion divided by the net resistance during retraction was similar for all of the full flow probes used. At the Richmond site, the results from the two sizes of T-bar were similar but the pull out resistance of the ball penetrometer was much lower than that of the T-bar. The pull-out resistance of the ball was also much less than CPTU sleeve friction. A possible explanation for this observation is that the soil is not sufficiently plastic to flow back into the cavity created during penetration of the Ball. This would mean that full-flow did not exist around the Ball at this site

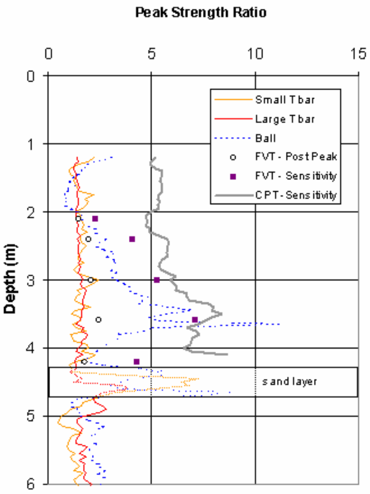

Figure 8. Ratio of peak undrained shear strength to post peak and remoulded strength, Richmond site.

While the sleeve friction measurement is not presented in flow penetrometer results it does provide some indication if full flow occurs around these tools. In the case of the soft soils at the Colebrook and Mud Bay site the sleeve friction measured by the CPTU and during full flow probe soundings were not too different. This was also the case with the CPTU and the T-bar at the Richmond site. The low sleeve frictions measured during the Ball penetration test in Richmond suggests that at low overburden stresses full flow was not occurring, and that the Ball penetrometer should be use with caution in similar soil conditions.

5. DISCUSSION AND CONCLUSION

The test data indicate that in soft sediments, full-flow penetrometers provide values of q_{net} that are smaller than those from CPTU profiles. This is despite the much greater plan area of the flow tools compared to the standard 10 cm^2 cone. The q_{net} values from two different sized T-Bars and the Ball are very similar to each other and require less adjustment for overburden stress and unequal end area pore pressure effects than conventional CPTs. Because of their large size and the shadow effect of the tools on the pore pressure element location and friction sleeve, the flow penetrometers are less sensitive to stratigraphic variations than CPTU parameters. It is thus preferable that they should be used for estimating s_u in very soft to soft soils to complement the profiling capability of the CPTU.

The data also show that with good attention to details of calibration and transducer baselines, it is possible to obtain data of similar quality using standard cone equipment. The use of low capacity load cells in the CPTU can also improve its performance and reduce the likelihood of errors being introduced to the data.

For purposes of estimating s_u, it is always preferable to derive site specific values of N to be applied in Equation 2. In the absence of site specific values, the average value of $N_{T\text{-}bar}$=10.5 suggested by others was shown to provide reasonable estimates of s_u for preliminary analyses. Similarly, a value of N_{kt} of from 13 to 15 was shown to provide preliminary profiles of s_u acceptable for preliminary analyses. Again, these values are in agreement with conventional practice.

It is important to be clear on the specific type of shear strength that is obtained from use of Equation 2 and the N factors indicated. In this paper, an estimate of $(s_u)_{FV}$ is obtained. For low plasticity soils, (s_u) in triaxial compression would be expected to be greater than $(s_u)_{FV}$ (Ladd, 1991). Figures 5 and 6 show that values of $(s_u)_{UU}$ from unconsolidated undrained triaxial compression tests are close to peak $(s_u)_{FV}$ values at shallow depth but are closer to post peak values of $(s_u)_{FV}$ at greater depths. These results are consistent with increasing disturbance of samples occurring with depth. The disturbance occurred during sampling, extrusion and sample preparation for testing. In one case, it proved impossible to prepare a sample from a depth of 7.2 m at Colebrook due to its rapid deterioration during handling. From this experience, it is concluded that UU triaxial compression tests are unlikely to provide useful shear strength parameters in routine investigations. In order to achieve useful measurements of $(s_u)_{peak}$ from laboratory testing in these soils, it will be necessary to employ advanced techniques of sampling, sample preparation and testing. Recent advances in field sampling and laboratory testing techniques for Lower Mainland soils are discussed in Sanin and Wijewickreme (2006) and Wijewickreme and Sanin (2006).

Evidence from this study suggests that the ratio of the intact and retraction values of q_{net} were closer to the ratio of s_u peak to post peak than to S_t values from the FVT. Some authors have suggested that cyclic extraction-penetration tests at selected depth intervals can give an estimate of $(s_u)_{remoulded}$ and sensitivity. The procedure is to carry out cyclic extraction and penetration of the probe over a depth range of ±0.5 m during a pause in penetration. Typically, the probe is cycled back and forth six times or until a constant resistance is observed. This procedure is quite possible but is time consuming and would only be utilized to produce semi-continuous profiles. DeJong et al (2004) suggested that such cyclic remoulding using the T-bar may create a cylindrical void space. The limited cyclic T-bar testing performed in this investigation at the Richmond site resulted in negligible resistance after cycling, indicating that a void was forming. This would suggest that for medium to stiff clays at low confining stress the full flow mechanism is not occurring.

The application of $(s_u)_{CPT}/f_s$ as a means of predicting FVT sensitivity was inconclusive, although the results provided a reasonable first estimate of S_t. Use of a CPTU with a low capacity sleeve would be expected to provide better results.

In conclusion, full-flow penetrometers appear to show promise for estimation of s_u in soft sediments, particularly where they can be pushed from surface. The advantage over the CPTU is the greatly reduced correction for q_{net}, a somewhat smaller range of bearing capacity N values, and increased resolution due to the increased pushing area. This also results in smaller potential zero load error corrections as compared to a standard capacity CPTU.

The deployment of full-flow probes can be hindered if drilling out, or casing is required. In these cases, a Ball penetrometer may be easier to deploy. Ball penetrometers appear to give similar net resistances to both sized T-bars. Some data suggested that full flow conditions did not always occur around the Ball penetrometer. Also due to the much larger projected area of the T-Bar and Ball, they will meet refusal at much lower tip resistances than the CPTU.

6. ACKNOWLEDGEMENTS

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STANDARD OPERATING PROCEDURE

QA-001 Equipment Decontamination

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1. Objective

This SOP describes methods used to decontaminate reusable sampling equipment for projects that require collection of organic and inorganic analytical samples. The goal is to minimize cross-contamination between samples. This maximizes confidence that field samples will be representative of specific locations and conditions.

Refer to the work plan or project manager to determine if different decontamination methods are acceptable.

2. Execution

- All contractor-provided equipment (augers, rods, spoons, backhoe buckets) should be decontaminated by steam cleaning or pressure washing prior to coming on site. If there is doubt about cleanliness of drilling tools, they should be decontaminated before use at the site.
- Sampling equipment decontamination is a sequential procedure consisting of the following steps:

    - Alconox-solution wash (or equivalent non-phosphate detergent)
    - Potable water rinse
    - A ten percent reagent grade nitric acid wash should be used to strip potential inorganic contaminants from sampling devices.
    - Laboratory grade 100 percent methanol, should be used to strip potential organic contaminants from sampling devices.
    - Three distilled/deionized water rinses.

- Alconox solution is a mixture of approximately 1 cup of Alconox per 1 gallon of potable water. Alconox solution wash requires scrubbing the equipment with a brush soaked in Alconox solution to remove visible contamination or dirt from sampling devices.
- Split-spoon samplers must be decontaminated prior to collecting each sample. The procedure follows:

    - Overall wash and scrub in a bucket of Alconox solution
    - Potable water rinse.
    - 10% nitric rinse
    - 100% laboratory grade methanol rinse
    - Three distilled-water rinses.

  The same procedure is applied to all devices that may contact soil or groundwater slated for analytical samples - spoons and knifes used to inspect or sample soils; water level indicators; oil/water interface probes.

Equipment used for well development of multiple wells must be decontaminated between wells.

Pumps and tubing should be flushed using a minimum of one gallon of Alconox-solution followed by a gallon of potable water. Some projects may require methanol (in much lower quantities) and distilled water instead of or in addition to the Alconox-solution and potable water.

For pumps and tubing, a final rinse of the sampling equipment may be performed with the water being sampled.

Equipment blanks measure the effectiveness of the decontamination procedures. Blanks should be collected per guidance provided in QA-002, Field Quality Control Samples.

3. Limitations

- Do not store the deionized/distilled water in polyethylene bottles, use Nalgene, glass, or Teflon. Polyethylene may leach phthalates.
- Do not attempt to decontaminate string or rope - replace it.
- Due to eye and skin absorption hazards, safety glasses and gloves must be worn when handling decontamination solvents.
- Decontamination procedures may also require modification based on state or federal requirements.
- Steam cleaning or pressure washing with potable water is generally an acceptable decontamination method for drilling equipment (i.e., augers). Check with the work plan.
- Dedicated equipment need not be decontaminated beyond initial decontamination prior to field use.

4. References

Environmental Response Team (ERT), US EPA. Sampling Equipment Decontamination, SOP No. 2006, Revision 0.0. August 11, 1994.

US EPA Region 9. Sampling Equipment Decontamination, SOP No. 1230, Revision 1.September 1999.

5. Contacts

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STANDARD OPERATING PROCEDURE

QA-002 Field Quality Control Samples

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1. Objective

Field Quality Control (QC) samples are used to monitor the reproducibility and representativeness of field sampling. The QC samples are handled, transported, and analyzed in the same manner as the associated field samples. QC samples may include trip blanks, equipment blanks, and field duplicates.

2. Execution

2.1. Trip blanks

- Used to monitor possible sources of contamination from transport, storage, inadequate bottle cleaning, or laboratory methodologies.
- Sample containers filled at the laboratory with analyte-free water are transported to and from the site, and are not opened until time of analysis.
- Trip blanks are stored with the sample containers prior to and after field activities and remain with the collected samples until analyzed.
- Generally, one trip blank per volatiles analysis (e.g. volatile organic compounds) shipment.
- Consider submitting a trip blank when sample shipment is by Fed Ex or other large carrier, or laboratory courier.
- Trip blanks should be recorded in the field notebook and on the chain-of-custody that same as all other samples.

2.2. Equipment blanks

- Equipment blanks (also known as equipment rinsate blanks) are used to monitor possible sources of contamination associated with sample collection. Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions
- Equipment blanks are collected by pouring laboratory supplied or distilled or deionized water over sampling tools that have been decontaminated per the work plan, into sample containers.
- Equipment blanks are stored with the associated field samples until submitted for analysis.
- Generally collected when site conditions indicate site related contamination is a concern. Check project-specific work plan and/or quality assurance project plan for required frequency.
- Prepare equipment blanks immediately after the equipment is cleaned in the field and before leaving the sampling site.
- Prepare equipment blanks by rinsing the decontaminated sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers.

- If a potable water rinse is the typical final step, collect the equipment blank with analyte-free water after the potable water rinse.
- Equipment blanks should be recorded in the field notebook and on the chain-of-custody that same as all other samples.

2.3. Field Duplicates

- Used to evaluate the precision and representativeness of the sampling procedures.
- Field duplicates are two samples collected from the same location using the same procedures. Both samples are submitted to the laboratory as individual samples with different sample identification.
- Field duplicates from groundwater sampling for all analyses except volatiles analysis are collected by alternating filling sample containers from the same sampling device. Field duplicates for volatiles analysis are filled sequentially.
- Soil or sediment field duplicates are collected by homogenizing the sample for all analyses except volatiles. The homogenized sample is then divided into two equal portions and placed in separate sample containers. Field duplicates for volatile analysis are collected at two adjacent sampling locations.
- Each sample is assigned different sample identifications.
- Field duplicates are generally collected at frequency of 1/20 samples. Check project-specific work plan and/or quality assurance project plan for required frequency.
- All field QC samples should be labeled in the field and submitted “blind” to the laboratory – as if they are separate, primary samples.
- Field duplicates should be recorded in the field notebook and on the chain-of-custody that same as all other samples.

2.4. Matrix-Spike samples (MS/MSD)

- Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory or in the field with a known concentration of a target analyte(s) to verify percent recoveries.
- Matrix spike and matrix spike duplicate samples are primarily used to check sample matrix interferences. They can also be used to monitor error due to laboratory bias and poor precision. However, a data set of at least three or more results is necessary to statistically distinguish between laboratory performance and matrix interference.
- Generally, the laboratory is required to extract and analyze MS or MS / MSDs at a minimum frequency of 5% of samples being analyzed for the target analyte(s). If the project or client criteria require an MS or MS/MSD, collect sufficient volume in the appropriate containers, and designate the sample to be used as the MS or MS/MSD on the chain of custody.
- Calculate the percent recovery for all spiked analytes for both the MS and MSD. For MS/MSDs also calculate the relative percent difference (RPD). The

RPD for each spiked analyte is calculated using the amount detected not percent recovery. If your data will be subjected to validation, the % recovery and the RPD will generally be determined by the validator.

2.5. Typical QA/QC Frequency

- QA/QC frequency is determined by project, client or regulatory criteria and should be verified prior to sample collection. Generally, QA/QC samples are collected according to the frequency described below:

Duplicate Samples	One per twenty samples collected, or every two weeks, whichever comes first.
Equipment Blanks	One per sampling event, one per 20 samples, or one every two weeks
Trip Blanks	One per sample delivery group, or in each cooler containing VOCs water samples.
MS or MS / MSDs	One MS or MS/MSD per 20 samples, or every two weeks, unless otherwise required by project, client, or regulatory criteria.

3. Limitations

- Trip blanks must never be opened in the field.
- Trip blanks are usually for VOCs only because less volatile compounds are not likely to cross-contaminate other samples by simply being in close proximity.
- Laboratory-grade water must be used during the collection of equipment blanks.
- Field duplicates must have different sample identifications.

4. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (November 1986), U.S. Environmental Protection Agency Department of Solid Waste, Washington, D.C.

U.S. Environmental Protection Agency Office of Emergency and Remedial Response, 1990, Quality assurance/quality control guidance for removal activities: EPA/540/G-90/004, Sampling QA/QC Plan and Data Validation Procedures Interim Final, April, 1990.

5. Contact

Brian Conte
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STANDARD OPERATING PROCEDURE

SC-001 Environmental Sample Types and Sampling Strategies

1. Objective

Describe types of samples and strategic approaches to sample locations.

Refer to Attachment A for guidance on compatible sampling materials.

2. Sample Types

Grab Samples

A grab (or discrete) sample is a single aliquot (part of the sampled media) collected from a single location at a specific time.

Surface soil samples are typically “grab” samples. Volatile organic samples are always grab samples because the least amount of sample disturbance is necessary.

Composite Samples

Composite samples are non-discrete samples composed of more than one aliquot collected from different sampling locations and/or at different points in time. Analysis of composite samples produces an average value.

Composite samples are frequently collected to characterize waste soil that has been stockpiled for eventual disposal. Several grab samples are collected from the stockpile and are blended together into a single sample.

Screening Samples

Screening samples may be grab or composite in nature. However, they offer potential advantages such as rapid results and low cost. The trade-off is that they may only provide results within a range and/or they may have elevated detection limits. Screening samples are most often used to evaluate presence/absence and/or indications of the potential magnitude of impacts.

3. Sampling Strategies

Generally, there are three sampling strategies: random, systematic, and judgmental sampling.

- Random sampling involves collection of samples in a non-systematic fashion from the entire site or a specific portion of a site.
- Systematic sampling involves collection of samples based on a grid or a pattern which has been previously established.
- Judgmental sampling is the collection of all other samples. This sampling might be from areas most likely to be contaminated, areas most likely to be clean, or areas where information is lacking.

Often, a combination of these strategies is the best approach depending on the type of the suspected/known contamination, the uniformity and size of the site, the level/type of information desired, etc.

4. Attachments

Attachment A - General Guidelines for selecting equipment

5. Contacts

Jerry Zak
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General Guidelines for selecting equipment on the basis of construction material and target analyte(s)

[✔, generally appropriate for use shown; Si, silica; Cr, chromium; Ni, nickel; Fe, iron; Mn, manganese; Mo, molybdenum; CFC, chlorofluorocarbon; B, boron]

Construction material for sampling equipment		**Target analyte(s)**	
Material	**Description**	**Inorganic**	**Organic**
	Plastics[1]		
Fluorocarbon ploymers[2] (other varies available for differing applications)	Chemically inert for most analytes	✔ (potential source of fluoride)	✔ (Sorption of some organics)
Polypropylene	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polypropylene (linear)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polyvinyl chloride (PVC)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Silicone	Very porous. Relatively inert for most inorganic analytes	✔ (potential source of Si)	**Do not use**
	Metals		
Stainless steel 316 (SS 316)	SS-316-metal having the greatest corrosion resistance. Comes in various grades. Used for submersible pump casing.	✔ (Potential source of Cr, Ni, Fe, and possible Mn and Mo) **Do not use** for surface water unless encasted in plastic.	✔ Do not use if corroded[3]
Stainless steel 304	Similar to SS-316, but less corrosion resistant	**Do not use**	✔ Do not use if corroded[3]
Other metals: brass, iron, copper, aluminum, galvanized and carbon steels	Refrigeration-grade copper or aluminum tubing are used routinely for collection of CFC samples	**Do not use**	✔ Routinely used for CFCs Do not use if corroded[3]
	Glass		
Glass, borosilicate (laboratory grade)	Relatively inert. Potential sorption of analytes	✔ **Do not use** for trace element analyses. Potential source of B and Si	✔

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[1] Plastic used in connection with inorganic trace-element sampling should be uncolored or white. Tubing used for trace metal sampling should be cleaned by soaking in 5-10 percent HCl solution for 8-24 hours, rinsing with reagent water (metals free) and allowed to air dry in mercury-free environment. After drying, the tubing is doubled-bagged in clear polyethylene bags, serialized with a unique number, and stored until used.

[2] Fluorocarbon polymers include materials such as Teflon™, Kynar™, and Tefzel™ that are relatively inert for sampling inorganic or organic analytes. Only fluoropolymer should be used for samples that will analyzed for mercury because mercury vapors can diffuse in or out of other materials, resulting in either contaminated or biased results.

[3] Corroded/weathered surfaces are active sorption sites for organic compounds.

STANDARD OPERATING PROCEDURE

SC-002 Environmental Sample Handling

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1. Objective

Describe appropriate environmental sample handling procedures.

The procedures include collection and transport of environmental samples to a laboratory for chemical analysis. Appropriate sample handling should ensure that samples are properly:

- labeled and documented;
- preserved;
- packaged; and
- transported

2. Execution

- Prior to mobilizing to the field, select a shipper or arrange for a courier for sample delivery to the laboratory. If using a shipper (i.e., FedEx or UPS) determine the time constraints for pickup requests, the location and hours of the nearest shipping office, and any size/weight restrictions.
- A waterproof or permanent ink pen should be used for all labels. The label should have an adhesive backing and be placed on the jar or bottle, not on the cap. In addition, clear packing tape can be placed over the sample label to secure it to the bottle as moisture from the samples can loosen the label adhesive.
- Record the following information on the label and in the field notebook (See SOPs FD-001 and FD-003):
    - Project number
    - Sample identification (i.e. MW-201 or SS-2)
    - Date and time (military time) of collection
    - Sampler's initials
    - Analysis methods
    - Preservative, if present
- Pre-preserved laboratory jars are preferable and should be used whenever practicable. If sample jars are not pre-preserved, add preservative as appropriate.
- At each sampling location, samples should be collected in order of volatility, most volatile first. Samples collected for volatile analysis should be placed in sample containers immediately upon retrieval of the sample.
- Aqueous samples for volatile analysis should be collected without air bubbles.
- The collection and preservation method of soil samples for volatile analysis may depend on project, client, or state regulatory requirements. Check with your Project Manager and/or SOPs SM-001 and SM-002 where appropriate.

- Care must be taken to avoid getting soils on the threads of sample jars, which can cause a faulty seal.
- If compositing samples in the field, specify the basis for composite (i.e. volume, weight, spoon recovery, etc.) and record in the field book the procedure for compositing the sample.
- Once samples have been collected and labeled, place samples in a cooler with sufficient bagged ice or freezer packs (blue ice) (if allowed) to chill samples to 4°C. If using ice, use double-bagged ice.
- Complete the chain-of-custody (COC) (SOP FD-003).
- If transporting the samples by way of a shipper:
    i. The sample cooler should have water drains securely sealed with duct tape, both on the inside and outside of the cooler.
    ii. Place a layer of packing material on the bottom of the cooler as a cushion.
    iii. Individually wrap each sample bottle with bubble packing or suitable packing material and place the wrapped bottles upright in the cooler with sufficient packing material between samples to avoid breakage.
    iv. Methanol preserved samples for volatiles analysis should be packed so they remain upright with the soil completely covered by the methanol during transport.
    v. Place a layer of packing material on top of the sample bottles.
    vi. Place bagged ice or freezer packs on top of the packing material. Fill the remaining space in the cooler with packing material to eliminate the possibility of vertical movement of samples.
    vii. Place the completed and signed chain-of-custody form in a sealable plastic bag and place on top of the packing material in the cooler, or tape it to the inside lid of the cooler.
    viii. Fill out the appropriate shipping or courier forms and attach to the top or handle of the cooler. If necessary, place the proper shipping labels on the cooler. Have the courier sign the COC form (or write pickup by FEDEX, UPS, etc. with date and time). Place a signed and dated custody seal on the cooler.
- All samples should be submitted to the laboratory as soon as possible. In many cases, same day shipping will be required by the client or the project manager. Be clear on this before beginning the field work.
- A copy of the waybills should be kept by the field supervisor to track shipments if necessary.

3. Limitations

- If samples are shipped on a Friday, call the laboratory ahead of time to confirm that personnel will be at the laboratory to receive and log-in the samples.
- During warm weather, make sure to use plenty of ice in the shipping container.

- Field personnel should be aware of analyses which have short hold times and schedule sampling events and shipping accordingly. Shipment of samples for analyses with short hold times must be arranged for in advance. Refer to the project work plan, quality assurance project plan, or state/federal regulations for holding time and preservative information. Contact the laboratory ahead of time when shipping samples with short hold time to ensure the lab is prepared for these analyses.
- For glassware containing preservatives (e.g., HCl, HNO_3), take care not to overfill the container, thus flushing the preservative out of the bottle.
- Never composite samples for VOCs in the field. Collect individual aliquots and direct the laboratory to perform compositing, if needed.
- Collection of aqueous samples should not be performed over the opening of a monitoring well. Preservatives from overfilling, a marker pen or other objects could fall into the well.
- If the recharge volume for a monitoring well is low, completely fill all volatile vials and then collect the minimum sample volume required for each remaining analysis.
- During subsurface soil sampling, if the recovery from the split-spoon sample is inadequate, if appropriate, resample the bottom of the borehole to obtain proper sample volume.
- Laboratories will homogenize and test the contents of the sample container, unless directed otherwise. Samples should not contain rocks, twigs, leaves, etc… unless these materials are of interest.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Preservation Techniques for Volatile Organic Compound (VOC) Soil Sample Analyses, WSC#99-415. Massachusetts Department of Environmental Protection.

5. Contacts

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STANDARD OPERATING PROCEDURE

SC-003 Investigation Derived Waste

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1. Objective

Describe characterization and management of Investigation Derived Waste (IDW) resulting from site investigation activities.

IDW is solid and/or aqueous waste generated during environmental site investigations.

2. Execution

- Determine the suspected contamination type and impacted media based on previous investigations, available analytical data, and/or site history.
- Consider the following when selecting IDW management option(s):
  - Anticipated volume of IDW to be generated during on-site activities
  - Potential contaminants and their concentrations
  - Proximity to population centers and the potential for unauthorized site access
  - Potential exposures to workers
  - Potential for environmental impacts
  - Community concerns
  - Potential storage areas
  - Regulatory constraints
  - Potential on-site treatment options
  - Duration of storage
  - Client concerns or requirements
- Review IDW Management Options summarized in Attachment A for each media suspected of contamination.
- Select IDW Management Option(s) prior to the commencement of field activities that will generate waste materials.
- Include the selected IDW Management Option(s) in the Field Plan or other project documents.

Considerations and guidelines for IDW management for specific field tasks are provided below.

2.1. Test Pit Excavation

- Segregate contaminated soil from uncontaminated soil using visual and/or field screening methods.
- Use appropriate barrier (such as two layers of 6-ml plastic sheeting) for temporary stockpiling of contaminated soil adjacent to test pit.

- Backfill test pits with uncontaminated soil, unless otherwise directed by project manager.
- If directed by the Project Manager to return contaminated soil to the test pit, backfill soil in the same order as the soil was excavated from the test pit.

2.2. Boring/Monitoring Well Installation

- For auger borings, segregate contaminated soil (determined by visual and/or field screening methods) from uncontaminated soil during drilling. Segregate residual contaminated soil from split-spoon sampling.
- Auger cuttings or sediment generated by drive and wash may be spread around the ground surface at the boring location if it is acceptable to the client and the governing regulatory agency. If not, IDW may be placed in an appropriate area or container pending characterization and appropriate disposal. (A useful rule of thumb is to assume generation of one 55-gallon drum of cuttings for each 20 feet drilled with 7-¼-inch-I.D. augers).
- Segregate contaminated drilling fluid from uncontaminated fluid for rotary wash borings.
- Drilling fluid management options include pouring the drilling fluid on the ground near the boring location, if acceptable to the client and governing regulatory agency, or containerizing the fluid in drums or tanks.

2.3. Well Development/Sampling

Contaminated groundwater removed from wells by pumping or bailing for the purpose of well development and sampling may be poured on the ground near the well, if it is acceptable to the client and the governing regulatory agency. Otherwise, it should be containerized in drums or tanks.

2.4. Decontamination Fluids

Decontamination fluids may be poured on the ground in the vicinity of the well if approved by the project manager. Alternatively, the fluids may be containerized in drums or tanks.

2.5. Disposable Personal Protective Equipment

Disposable personal protective equipment (PPE) should be managed like any other IDW. However, with the clients' and project manager's approval, it may be removed from the site and disposed of as ordinary rubbish if it has not come into contact with contaminated materials.

3. Limitations

- The simplest IDW management option is to return the IDW to its source location.
- However, the selected IDW management options must meet state/federal regulations and have the client's approval. Consult with state/federal policies for IDW-related matters.

- The client is responsible for the disposal of IDW, should disposal be necessary.

4. References

Guide to Management of Investigation - Derived Wastes (April 1992), United States Environmental Protection Agency, Publication 9345.3-03FS.

Standard References for Monitoring Wells, Massachusetts Department of Environmental Protection, Publication No. WSC-310-91.

5. Attachments

Attachment A - Summary of Investigation Derived Waste Management Options
Attachment B - CTDEP Waste Guidance

6. Contacts

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Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Soil	Boring/monitoring well installation Test pit excavation Soil sampling	Return to source location immediately after generation	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Spread around boring, test pit, or original source location	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Containerize and temporarily store on site	Can temporarily store in stockpiles or covered containers (i.e. drums, roll-off containers). Stockpiles must be underlain by plastic sheeting and covered with plastic sheeting. Plastic sheeting must be secure. Storage consistent with state/federal regulations.
		Send to off-site, treatment or disposal facility within appropriate timeframes	Requires proper shipping documents (i.e. manifest, Bill of Lading, etc.), analytical characterization
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Store temporarily awaiting laboratory analysis.	Storage consistent with state/federal regulations. Can temporarily store in stockpiles or covered containers (i.e. drums, roll-off containers). Stockpiles must be underlain by plastic sheeting and covered with plastic sheeting. Plastic sheeting must be secure.
Sediment/Sludge	Sludge pit sampling Sediment sampling	Return to source immediately after generation	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Store temporarily on site.	Storage consistent with state/federal regulations.
		Send to off-site facility within 90 days	Requires manifests, analytical characterization
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)

Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Aqueous liquids (groundwater, surface water, drilling fluids, other wastewater)	Well installation/development Well purging during sampling Ground water discharge - pump tests Surface water sampling	Pour onto ground close to well	Non-hazardous liquids only. Should not exhibit a sheen or separate phase product. Do not discharge to the ground up-gradient of the source location. Ensure that it is permissible by local, state, and Federal regulations Is acceptable to the client, the governing regulatory agency, and the project manager.
		Store temporarily on site	If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Send to off-site commercial treatment unit within appropriate timeframes	Refer to State regulations for appropriate timeframe. Requires appropriate shipping documents (i.e., manifest, Bill of Lading), analytical characterization
		Send to POTW	Obtain appropriate discharge permit(s)
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. Consistent with final remedial action
		Discharge to surface water	OK if it complies with state and federal regulations. Obtain appropriate discharge permit(s).
Decontamination fluids	Decontamination of PPE and equipment	Store temporarily on site	If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Send to off-site facility within appropriate timeframes	Requires manifests, analytical characterization
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Consistent with final remedial action
Disposable PPE	Sampling, drilling, and test pit excavation observation, other on-site activities	Store temporarily on site	Dispose of appropriately after characterization
		Place in on-site industrial dumpster	Project-specific determination required – must be acceptable to client and project manager
		Send to off-site facility within 90 days	Project-specific determination required
		Store for future treatment and disposal.	Storage consistent with state/federal regulations. Project-specific determination required

Notes:

1) PPE - personal protective equipment
2) POTW - publicly owned treatment works
3) Generation processes listed here are provided as examples.
IDW may also be generated as a result of other site activities.
4) RCRA Container/Waste Pile/Tank requirements:
Containers; 40 CFR 264 Subpart I and 265 Subpart I
Waste Piles; 40 CFR 264 Subpart L and 265 Subpart L
Tanks; 40 CFR 264 Subpart J and 265 Subpart J

Connecticut Department of Environmental Protection

Connecticut's RCRA "Contained-In" Policy

Characterization of Contaminated Soil and Groundwater

Policy
RCRA hazardous waste determinations for contaminated soil and groundwater may compare contaminant concentrations with the characterization criteria below. If the concentrations are below these criteria then the soil and groundwater do not need to be managed in Connecticut as RCRA hazardous waste. If the concentrations are above these levels then the soil/water must be treated, stored, transported, and disposed in the same manner as hazardous waste.

Purpose
To simplify the management of non-hazardous contaminated soil and groundwater and to encourage remediation of contaminated sites.

Applicability
This policy applies to contaminated soil and groundwater managed in Connecticut. It does not establish cleanup criteria. When contaminant concentrations are below the levels described in this policy, but are greater than applicable Connecticut Remediation Standard Regulations ("RSR") criteria, then the soil and groundwater must be handled as non-hazardous contaminated soil and groundwater subject to applicable RSR polluted soil reuse requirements and to Connecticut solid waste requirements.

Contaminant	Soil Characterization Criteria	Groundwater Characterization Criteria
Characteristically hazardous waste "**D codes**"	Non-hazardous if below levels in Toxicity Characteristic Table in 40 CFR 261.24 ("TC Table")[1]	Non-hazardous if below levels in Toxicity Characteristic Table in 40 CFR 261.24 ("TC Table")
Listed hazardous waste **"F,K,P,U codes"** See 40 CFR 261.33 for "**P**" & "**U**" See 40 CFR 261 Appendix VII to identify constituents for which "**F**" & "**K**" wastes are listed.	Non-hazardous if below the lower of A and B: **(A)** Industrial/Commercial Direct Exposure Criteria in RSR[2] **and** --------------- --------------- ----------- [choose one method from B]: **(B) either** TC Table[1] **or** 100 x GA Pollutant Mobility Criteria in RSR[3] **or** 100 x Groundwater Protection Criteria in RSR[4]	Non-hazardous if below 100 x GA Groundwater Protection Criteria in RSR

1. via Toxicity Characteristic Leachate Procedure ("TCLP")
2. via mass analysis
3. via mass analysis or leachate analysis
4. via leachate procedure (eg: TCLP or Synthetic Precipitation Leachate Procedure

STANDARD OPERATING PROCEDURE

SC-004 Headspace VOC Screening

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1. Objective

Describe methods to obtain site-specific measurement of the total volatile organic compound (VOC) concentrations present in the headspace of a jar containing soil.

This information can be used for several purposes:

- Segregate soil based on degree of contamination.
- Identify samples for quantitative analysis of VOCs.
- Evaluate the presence or absence of VOCs in soil.

2. Execution

- A photoionization detector (PID) or flame ionization detector (FID) instrument is used to measure VOCs in jar headspace (JHS) screening.
- Select the appropriate instrument, lamp, and calibration gas for the site-specific contaminants. Calibrate the instrument in accordance with the manufacturer’s instructions before JHS screening begins. Record the type of calibration gas, detector, lamp, and results of calibration in the field notebook.
- Note the highest VOC concentration that the instrument measures in air in the work area before performing JHS screening. Record this as the initial background concentration.
- Half-fill a clean, glass jar with the soil. Quickly cover the open top with one or two sheets of clean, aluminum foil and screw on the cap to tightly seal the jar. Label the jar with the sample location and sample depth.
- Allow headspace development for at least 10 minutes at an ambient temperature of 50°F or greater. Vigorously shake the jar for 15 seconds at the beginning and end of the headspace development period. When ambient temperatures are below 50°F, place the jar in a heated vehicle or building during the headspace development period.
- After headspace development, remove the screw cap to expose the foil seal. Quickly puncture the foil seal with the instrument’s sampling probe and insert it to a point at about one-half of the headspace depth.
- Record the highest VOC concentration that the instrument displays as the JHS concentration. The highest concentration should occur between 2 and 5 seconds after probe insertion.

3. Limitations

- The instruments may work poorly in the rain and in freezing temperatures. Under such conditions, operate the instrument in a heated vehicle or building if possible.

- Prevent water and soil particles from entering the tip of the instrument probe. Use a filter on the instrument's probe.
- Measure background VOC conditions and perform JHS screening away from non-site-related VOC sources, such as vehicle and heavy equipment exhaust.
- The VOC concentration on the instrument's display may vary when the air contains high VOC concentrations or high moisture.
- JHS screening is a guide that helps the screener to segregate soils into broadly defined categories. JHS screening results may differ by orders of magnitude from laboratory testing results.
- Note that states may have specific procedures for field monitoring. In Massachusetts, the Massachusetts Department of Environmental Protection (DEP) requires that screening of gasoline-contaminated soil be performed in accordance with Attachment II of the DEP's policy #WSC-94-400 Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. Consult this procedure or any relevant guidance documents for assistance.

4. References

Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. (April 1994), Massachusetts Department of Environmental Protection, Policy #WSC-94-400.

5. Contacts

Lynn Willey
Leslie Lombardo

STANDARD OPERATING PROCEDURE

SM-001 Soil Sampling Techniques Including Split-Spoon

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1. Objective

Describe standard procedures for the collection of surface and subsurface soil samples, including relevant guidance for collection of sediment samples when working from a floating platform on a water body.

The definition of “surface” soil varies considerably between regulatory organizations. Surface soils may be classified as soils between the ground surface and 2 inches below ground surface, ground surface and 6 inches below ground surface, and even as much as ground surface and 24 inches below ground surface.

The definition of subsurface soil will vary in relation to the definition of surface soil. In general, subsurface soil is everything deeper than surface soil.

Refer to state-specific regulations for the definitions of surface and subsurface soils.

2. Execution

2.1. Surface Soil Sampling

Collection of surface soil samples can be accomplished with tools such as spades, shovels, trowels, scoops, etc. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required.

- Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
- Using a decontaminated stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
- If volatile organic compound (VOC) analysis is to be performed, transfer the sample directly into an appropriate labeled sample container with a stainless steel lab spoon, small diameter core device, or equivalent and secure the cap tightly.
- Place the remainder of the sample into a decontaminated stainless steel, plastic, or other appropriate container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval.
- Either place the sample into appropriate labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the container and mix thoroughly.
- When compositing is complete, place the sample into appropriate labeled containers and secure the caps tightly.

2.2. Sampling with Hand Augers and Thin Wall Tube Samplers

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are generally better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and generally cannot be used below a depth of approximately 3 feet.

2.2.1 Auger Sampling

- Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first 3 to 6 inches of surface soil for an area approximately 6 inches in radius around the drilling location.
- Attach the decontaminated auger bit to a drill rod extension, and attach the "T" handle to the drill rod.
- Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
- After reaching the desired depth, carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole.

2.2.2 Thin-Walled Core Sampling

- Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
- Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.
- Remove the tube sampler, and unscrew the drill rods.
- Remove the cutting tip and the core from the device.
- Discard the top of the core (approximately 1 inch), as this may represent material knocked down from the sides of the boring and not the layer of interest. Place the remaining core into the appropriate labeled sample container.

One type of thin-wall sampler is depicted in Attachment A (this is typically used with a mechanical drill rig).

For either method, If VOC analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, small diameter core sampler, or equivalent and secure the cap tightly. VOC samples should be collected first to minimize the potential for losing volatiles prior to sample collection.

Place the remainder of the sample into a stainless steel, plastic, or other appropriate container and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the container and mix thoroughly.

When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow previous steps, making sure to decontaminate the auger and tube sampler between samples.

Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

2.3. Sampling at Depth with a Split-Spoon (Barrel) Sampler

Split-spoon sampling is generally used with a mechanical drill rig to collect relatively undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split-spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted. A diagram of the split-spoon sampler assembly is provided as Attachment A.

When split-spoon soil sampling is performed to gain geologic information, work should be performed in accordance with ASTM D1586, "Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils". The following procedures are used for collecting soil samples with a split-spoon:

- Select the size (length and diameter) of split-spoon sampler based on the amount of soil that is needed for characterization. The ASTM standard for N-values is 1 3/8 - inch I.D (2-inch O.D.). Specify spoon size and basket type to driller prior to mobilization to the site. Split-spoon samplers are typically available in 1 3/8 – and 3 – inch I.D. sizes. A larger barrel may be necessary to obtain the required sample volume. Note on the boring log where larger split-spoon barrels are used because the ASTM standard penetration test does not apply when driving split spoons larger than 1 3/8 I.D. (2-inch O.D.).

- Select a soft or stiff basket for the spoon (a softer basket generally works better for loose or soft material).
- Prior to hammering the split spoon to collect the sample, verify that the split spoon is seated at the beginning of the desired sample interval. If it is seated above the interval, have driller clean out the hole prior to sampling. Record all depth measurements relative to ground surface.
- Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top. See diagram in Attachment A.
- Place the sampler in a perpendicular position on the sample material.
- For all soil samples, use a 140-lb hammer falling 30 inches to drive the sampler, unless conditions necessitate using a 300-lb hammer.
- Record in the site field book or on field data sheets the length of the tube used to penetrate the material being sampled, the split spoon inside and outside diameters, and the hammer weight.
- Count and record the number of blow counts per 6-inch increments (confirming blow counts with driller if necessary).
- Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The length of recovery and soil type should be recorded on the boring log. If a soil sample is desired, a decontaminated stainless steel knife or spatula should be used to divide the tube contents in half, longitudinally. If possible, avoid collecting soil that has come in contact with the walls of the spoon, and soil at the top of the spoon.
- Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.
- Note any material in the nose (shoe) of the spoon.
- Immediately collect a sample for VOCs (if required by the site-specific field sampling plan) by collecting soil from the entire length of the split spoon, unless otherwise specified by the project manager. When the most impacted interval is sampled for laboratory analysis, screen the spoon with the field instrument first, then collect the soil sample for VOC analysis from the appropriate interval.

2.4. Sampling at Depth from Floating Work Platforms

When performing at-depth sampling from floating work platforms in water bodies that are tidally influenced the following procedures are recommended:

- Use tide gauges that have been surveyed by a state-registered land surveyor and referenced to the vertical datum for the project to measure the elevation of the water body.
- Calculate deck or other work platform reference elevation using the free-board distance and the measured elevation of the water body based on tide gauge readings or predicted tide charts for the project area (adjusted for tidal lag) at time of sampling event.
- Record time and depth of sampling event and convert to an elevation.

3. Limitations

- Weather conditions (e.g., frozen ground) may prevent the collection of samples and should be considered prior to sample collection.
- Tools plated with chrome or other materials should not be used.
- Be aware of local laws regarding subsurface utility clearance prior to conducting subsurface investigations. Contact DigSafe or local utility companies as required.
- Be aware of the length of the drill string, the sample depth, and the required stickup of the drill string to ensure accurate sample interval measurement.
- If drilling with hollow-stem augers, the removal of the drill string from the hole, prior to attaching the split-spoon sampler, may cause soils to be sucked up into the augers (blow-in running sands). Upon recovery, determine if there is blow-in in the split spoon sampler. In general, blow-in is more unconsolidated than the rest of the sample and lacks stratification (do not include blow-in for recovery of sample collection).
- If soils consist of loose sands or soft clay, the drill string and sampler may advance slightly under its own weight, giving a false depth for soil collection.
- Never sample more than two spoons consecutively without advancing the augers unless material is tight. Do not let the split spoon penetrate more than it can hold.
- In many instances, groundwater will fill the auger and the split-spoon.

4. References

ASTM D1586-08a, "Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils". 2008.

United States Environmental Protection Agency, SOP 2012 “Soil Sampling”, Revision 0.0, February 18, 2000.

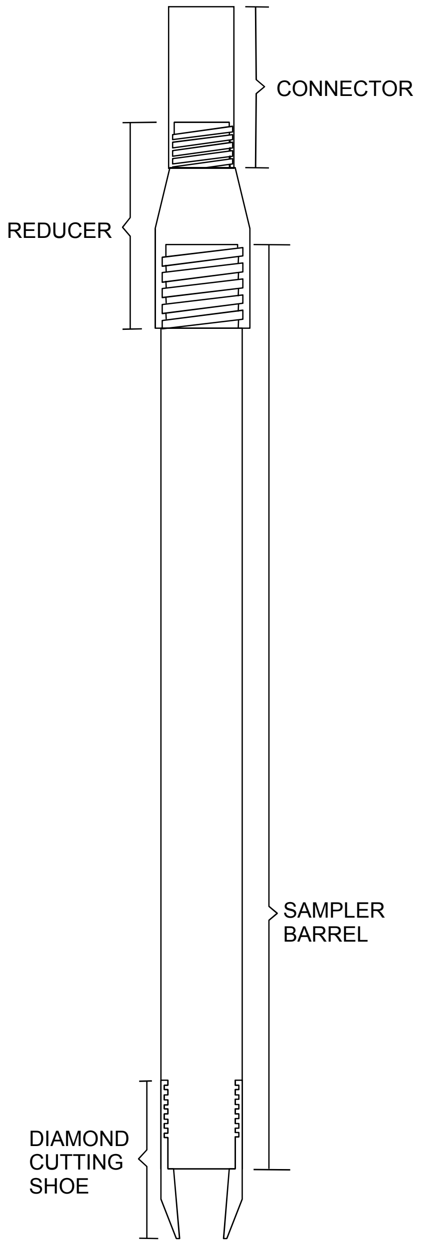
5. Attachments

Attachment A - Sampler Design Assembly

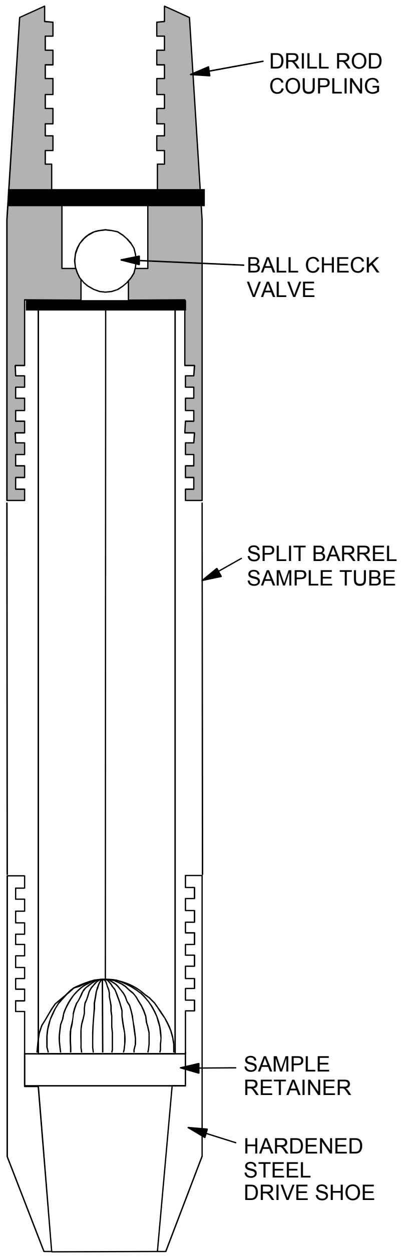
6. Contacts

Gary Fuerstenberg
Mark Ensign

THIN WALL HQ CORE SAMPLER
(2" width x 60" length)



SPLIT-SPOON SAMPLER
(2" width x 24” length)



STANDARD OPERATING PROCEDURES
SM-001

NEW ENGLAND AND ATLANTIC REGIONS

GEI Consultants

June 2011

THIN WALL AND SPLIT-SPOON SAMPLERS

Attachment A

STANDARD OPERATING PROCEDURE

SM-003 Classification of Soil Samples in the Field

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1. Objective

Describe methods to classify soil samples collected in the field in a consistent manner.

2. Execution

- Describe soil samples according to *ASTM D2488-09a, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) and* Attachments A and B*.* This standard is the basis for the Unified oil Classification System.
- Identify and record the soil in terms of the major and minor constituents (i.e., sand gravel, silt, clay), Unified Soil Classification Symbol, sample structure, plasticity and dilatancy for fine-grained soils, color, local or geologic name if known (e.g., Boston Blue Clay or glacial till), odor, presence of iron or other staining, and presence of organic matter, shells, debris, or other unusual characteristics of the same.
- If a soil split-spoon sample contains more than one soil type (for example, the upper portion is silty sand and the lower portion is clay) describe each type separately.
- Record sampler type, blow counts, soil description, etc. on the boring log (see Attachment C).
- GEI consistently applies one modification to the ASTM standard: Use "widely graded" and "narrowly graded" instead of "well-graded" and "poorly graded," respectively.

3. Limitations

Certain projects or clients will require the use of other classification systems. Other classification systems should not be used unless specifically required by the client. If the client requires that we use the Burmister method, obtain the details from the client. An example breakdown is shown below, but some clients (MassDOT, for example) have their own breakdown.

- “and” = 35-50%
- “some” = 20-35%
- “little” = 10-20%
- “trace” = 1-10%

- Describing soil samples is often difficult during cold or wet weather. Make sure your field notes describe these conditions. When possible, collect archive samples and verify sample descriptions in the office.

- The ASTM Standard Practice for Classification of Soils for Engineering Purposes (D2487) may be used in conjunction with the Visual-Manual Method to confirm the soil classification. D2487 includes laboratory testing.

4. References

ASTM D2487-06e1, Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System), ASTM, 2006.

ASTM D2488-09a, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure), ASTM, 2009.

Field Guide for Soil and Stratigraphic Analysis, Midwest Geosciences Group Press, 2001-2005.

Coarse-Grained Soils Visual-Manual Descriptions, GEI Consultants, Soil Description Chart.

Fine-Grained Soils Visual-Manual Descriptions, GEI Consultants, Soil Description Chart.

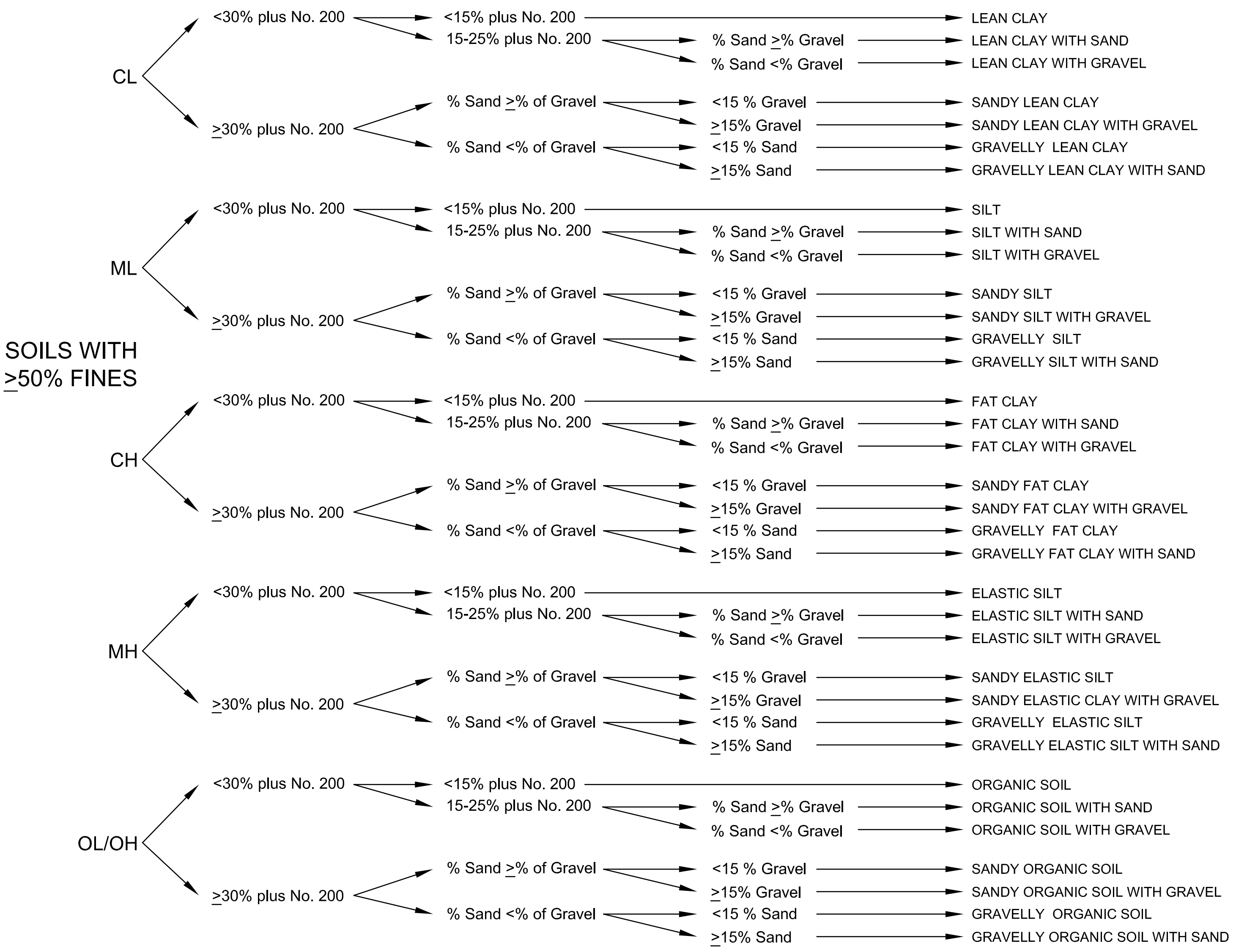
5. Attachments

Attachment A – GEI Soil Description Charts (2007)
Attachment B – Visual Manual Descriptions with example boring log
Attachment C – Describing the Plasticity of Soil Samples

6. Contacts

Lynn Willey
Cathy Johnson

FINE-GRAINED SOILS
VISUAL-MANUAL DESCRIPTIONS



ID OF INORGANIC FINE SOILS FROM MANUAL TESTS

Symbol	Name	Dry Strength	Dilatancy	Toughness*
ML	Silt	None to low	Slow to rapid	Low or thread cannot be formed
CL	Lean Clay	Medium to high	None to slow	Medium
MH	Elastic Silt	Low to medium	None to slow	Low to medium
CH	Fat Clay	High to very high	None	High

1. GROUP NAME and (SYMBOL)
2. Describe fines, sand, and gravel components, in order of predominance. Include plasticity of fines. Include percentages of sand and gravel.
3. Color
4. Sheen, odor, roots, ash, brick, cementation, torvane and penetrometer results, etc.
5. "Fill," local name or geologic name, if known

PEAT

Peat refers to a sample composed primarily of vegetable matter in varying stages of decomposition. The description should begin: PEAT (PT) and need not include percentages of sand, gravel or fines.

CRITERIA FOR DESCRIBING PLASTICITY

Description	Criteria
Nonplastic ML	A 1/8-in. (3 -mm) thread cannot be rolled at any water content
Low Plasticity ML, MH	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit *
Medium Plasticity MH, CL	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High Plasticity CH	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

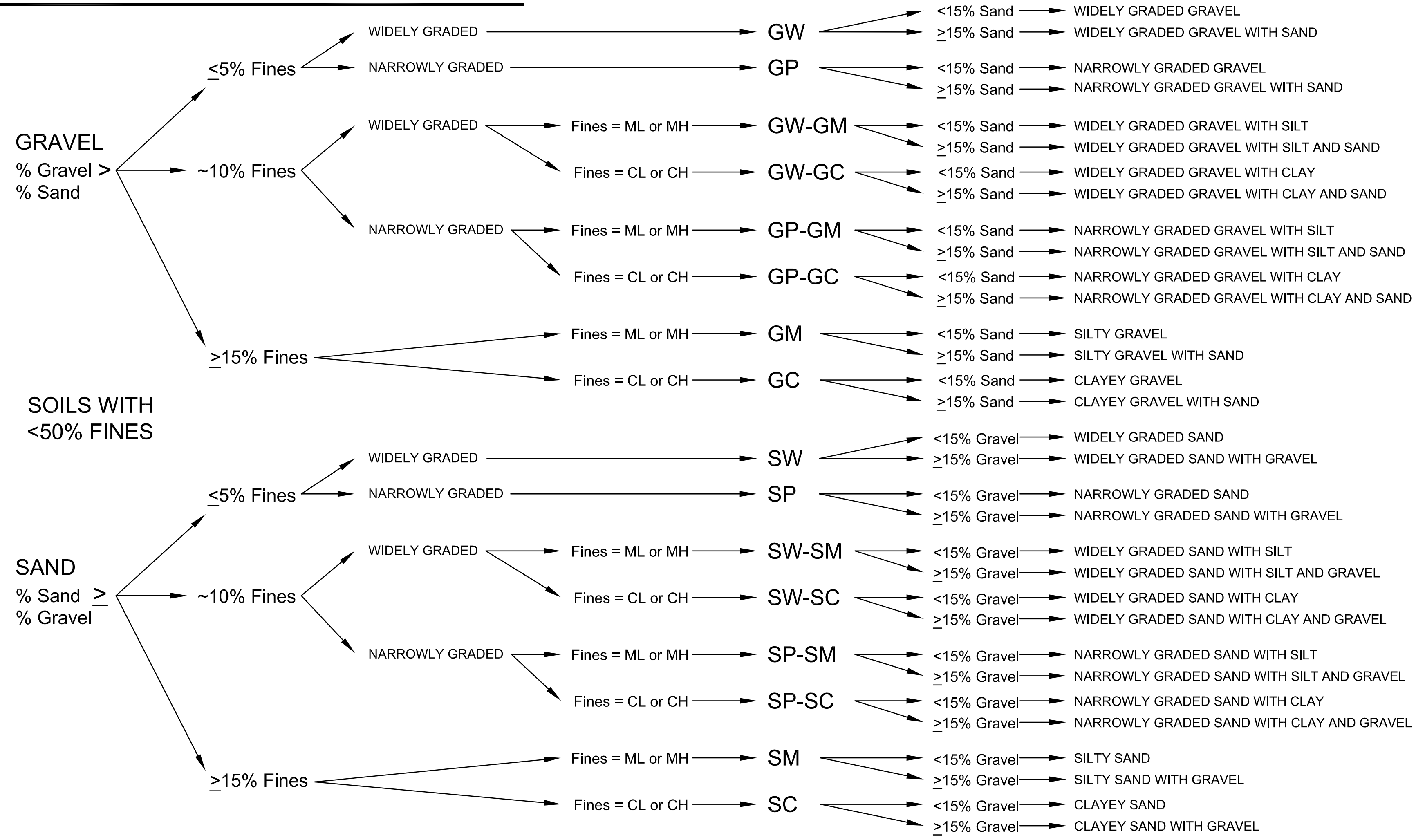
* Toughness refers to the strength of the thread near plastic limit. The lump refers to a lump of soil drier than the plastic, similar to dry strength.

COARSE-GRAINED SOILS
VISUAL-MANUAL DESCRIPTIONS

GEI Consultants

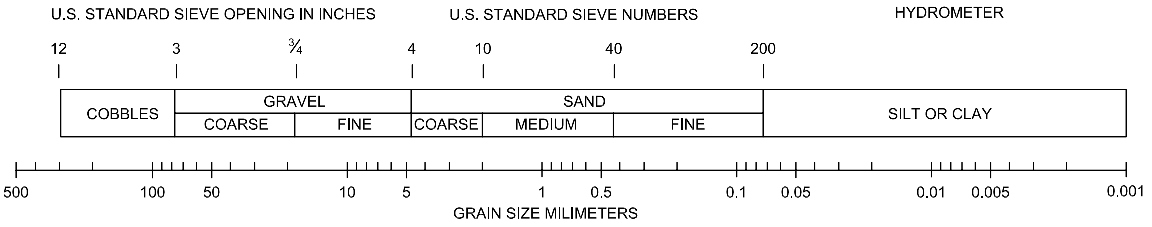
TYPICAL SOIL COLORS

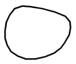
- YELLOW
- OLIVE
- LIGHT BROWN
- BROWN
- REDDISH BROWN
- RED
- GRAY
- BLACK



SOILS WITH <50% FINES

				GROUP SYMBOL		GROUP NAME
GRAVEL % Gravel > % Sand	≤5% Fines	WIDELY GRADED		GW	<15% Sand	WIDELY GRADED GRAVEL
					≥15% Sand	WIDELY GRADED GRAVEL WITH SAND
		NARROWLY GRADED		GP	<15% Sand	NARROWLY GRADED GRAVEL
					≥15% Sand	NARROWLY GRADED GRAVEL WITH SAND
	~10% Fines	WIDELY GRADED	Fines = ML or MH	GW-GM	<15% Sand	WIDELY GRADED GRAVEL WITH SILT
					≥15% Sand	WIDELY GRADED GRAVEL WITH SILT AND SAND
			Fines = CL or CH	GW-GC	<15% Sand	WIDELY GRADED GRAVEL WITH CLAY
					≥15% Sand	WIDELY GRADED GRAVEL WITH CLAY AND SAND
		NARROWLY GRADED	Fines = ML or MH	GP-GM	<15% Sand	NARROWLY GRADED GRAVEL WITH SILT
					≥15% Sand	NARROWLY GRADED GRAVEL WITH SILT AND SAND
			Fines = CL or CH	GP-GC	<15% Sand	NARROWLY GRADED GRAVEL WITH CLAY
					≥15% Sand	NARROWLY GRADED GRAVEL WITH CLAY AND SAND
	≥15% Fines		Fines = ML or MH	GM	<15% Sand	SILTY GRAVEL
					≥15% Sand	SILTY GRAVEL WITH SAND
			Fines = CL or CH	GC	<15% Sand	CLAYEY GRAVEL
					≥15% Sand	CLAYEY GRAVEL WITH SAND
SAND % Sand ≥ % Gravel	≤5% Fines	WIDELY GRADED		SW	<15% Gravel	WIDELY GRADED SAND
					≥15% Gravel	WIDELY GRADED SAND WITH GRAVEL
		NARROWLY GRADED		SP	<15% Gravel	NARROWLY GRADED SAND
					≥15% Gravel	NARROWLY GRADED SAND WITH GRAVEL
	~10% Fines	WIDELY GRADED	Fines = ML or MH	SW-SM	<15% Gravel	WIDELY GRADED SAND WITH SILT
					≥15% Gravel	WIDELY GRADED SAND WITH SILT AND GRAVEL
			Fines = CL or CH	SW-SC	<15% Gravel	WIDELY GRADED SAND WITH CLAY
					≥15% Gravel	WIDELY GRADED SAND WITH CLAY AND GRAVEL
		NARROWLY GRADED	Fines = ML or MH	SP-SM	<15% Gravel	NARROWLY GRADED SAND WITH SILT
					≥15% Gravel	NARROWLY GRADED SAND WITH SILT AND GRAVEL
			Fines = CL or CH	SP-SC	<15% Gravel	NARROWLY GRADED SAND WITH CLAY
					≥15% Gravel	NARROWLY GRADED SAND WITH CLAY AND GRAVEL
	≥15% Fines		Fines = ML or MH	SM	<15% Gravel	SILTY SAND
					≥15% Gravel	SILTY SAND WITH GRAVEL
			Fines = CL or CH	SC	<15% Gravel	CLAYEY SAND
					≥15% Gravel	CLAYEY SAND WITH GRAVEL

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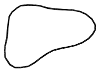
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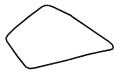
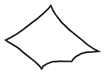

SUBANGULAR


ANGULAR

1. GROUP NAME and (SYMBOL)
2. Structure , if any. (stratified layer thicknesses, lenses, varves, gradational changes)
3. Describe sand, gravel and fines components, with percentages, in order of predominance. Include max gravel size. For test pits give percent cobbles and boulders, by volume, and include max size.
4. Color
5. Sheen, odor, roots, ash, brick, cementation, reaction with HCL, etc.
6. "Fill," local name or geologic name, if known

Describing the Plasticity of Soil Samples

M. Paster – November 2008

References ASTM D 2487 – Soil descriptions – lab
ASTM D 2488 – Soil descriptions – field
ASTM D 4318 – Atterberg limits testing

GEI Practice for Boring and Test Pit Logs

Describe the fines as:

Non-plastic
Low plasticity (The GEI laminated sheets incorrectly use "slightly plastic" for "low plasticity.")
Medium plasticity
High plasticity

Example: ~25% low plasticity fines

Toughness and dry strength:

You should use these tests to help decide how plastic the fines are. Record the results in the remarks column of the field log, but not in the soil description and not necessarily in the typed log.

On final logs, if Atterberg limits tests have been performed:

Do not use the descriptive terms non-plastic, low plasticity, etc. for samples on which Atterberg limits tests have been run. Instead, just give the percentage of fines and then report the actual Atterberg limits at the end of the description.

For example, the end of a silty sand description might be:
. . . ~25% fines, ~10% gravel max size ½ inch, gray. PL=23, LL=35.

(Atterberg limits tests are performed on the fraction of the sample finer than the No. 40 sieve, not just the fines. So the Atterberg limits data applies to the sample, not just to the fines.)

Hints:

High plasticity soils are rare in New England. If you think it's high plasticity, it's probably medium. Some Boston blue clay and some Connecticut River varved clays are high plasticity, but if you think you've found some, check with the project manager.

In New England, if ~10% fines or more, generally stick with GM, SM, ML, and CL. Occasionally GC, SC, CH. Don't use MH unless you have Atterberg limits data.

Estimating plasticity in the field, GEI guidance based on ASTM D 2488:

Plasticity	1/8-inch thread	Dry strength	Toughness
non	Cannot be rolled at any water content.	Dry specimen crumbles when handled.	Only slight pressure needed to roll thread near plastic limit.
low	Thread can barely be rolled.	Dry specimen crumbles with some finger pressure.	Slight to medium pressure needed to roll thread near plastic limit.
medium	Thread is easy to roll. Not much time needed to reach plastic limit.	Dry specimen crumbles with considerable finger pressure.	Medium pressure needed to roll thread near plastic limit.
high	Takes considerable time rolling and kneading to reach plastic limit.	Dry specimen cannot be broken with finger pressure.	Considerable pressure needed to roll thread near plastic limit.

Non-plastic vs. low plasticity:

ASTM D 2488 (soil descriptions - field) defines non-plastic and low plasticity based on the 1/8-inch thread as shown in the table above.

ASTM D 4318 (Atterberg limits testing) indicates that a sample should be called non-plastic for either of the following cases:

- The liquid limit test (dropping the cup) or the plastic limit test (rolling out the thread) cannot be performed because the plasticity is too low.
- The plastic limit is greater than or equal to the liquid limit.

Unfortunately, there are some soils that are low plasticity based on D 2488 (a thread can be rolled), but are non-plastic based on D 4318 (the liquid limit cannot be measured or $PL \geq LL$).

GEI considers these soils to have low plasticity, because that is how they "look" and "feel." We want to document this information so that other people will have a better feel for what the soil looks like and how it behaves. So, if the soil was low plasticity based on D 2488, but non-plastic based on D 4318, that should be explained in the letter or report, and possibly in a note on the log.

BORING LOCATION Maple Ave Sidewalk
GROUND ELEVATION (NGVD)
GROUNDWATER EL. DATE
DATE START/FINISH 2/14/07 - 2/15/07
DRILLED BY Geologic: M. Costigan
LOGGED BY T. Kahl/M. Yako TOTAL DEPTH (FT) 25

B101

PG. 1 OF 1

EL. FT.	DEPTH FT.	SAMPLE TYPE and NO.	BLOWS PER 6 IN.	PEN IN.	REC IN.	PID JAR HS / REMARKS	GRAPHIC LOG	SOIL AND ROCK DESCRIPTIONS
								4" pavement
		S1	13-9 17-14	24	0	0.5 ppm	FILL	S1: Redrove 0.5 to 3.5 ft. Recovery 11": WIDELY GRADED SAND (SW) ~85% sand, ~10% gravel to 1", <5% nonplastic fines, brown. Contains brick fragments and ash. Fill.
	2.5					hard drilling 3 to 4 ft, possible boulder		
	5	S2	7-7 11-13	24	8	2.0 ppm		S2: NARROWLY GRADED SAND WITH SILT AND GRAVEL (SP-SM) ~65% mostly fine sand, ~25% gravel to 3/4 inch ~10% non-plastic fines, brown. Fill.
	7.5	S3	9-10 2-1	24	16	0.0 ppm		S3 (0-10"): Similar to S2.
	10						ORGANICS	S3 (10"-16")": ORGANIC SILT (OL) ~100% slightly plastic fines, dark gray, organic odor, contains white shell fragments.
	12.5	S4	WOH 1-2 1	24	15	0.0 ppm		
	15					hard drilling at 15.5 ft		S4: Similar to S3, bot 6".
	17.5	S5	20-35 50/3"	15	8	Top of rock ~19 ft. Roller bit to 20 ft.	TILL	S5: SILTY SAND WITH GRAVEL (SM) ~60% mostly fine sand, ~25% slightly plastic fines, ~15% gravel to 1/2 inch, olive. Glacial Till.
	20 22.5	C1	RQD 70%	60	54	lost ~10 gallons drill fluid from 23 to 25 ft	ROCK	C1: SCHIST, hard, slight weathering at joint surfaces, joints at ~30 degrees from horizontal and generally parallel to foliation, gray. Marlborough Formation.
	25 27.5 30							Bottom of Boring 25 ft Truck-mounted drill rig. 4-inch casing to 19 ft. Safety-hammer with rope and cathead for SPT. Backfilled with drill cuttings.

BLOWS PER 6 IN.–140 LB. HAMMER FALLING 30 IN. TO DRIVE A 2.0 IN. OD SPLIT SPOON SAMPLER
PEN–PENETRATION LENGTH OF SAMPLER OR CORE BARREL
REC–RECOVERY LENGTH OF SAMPLE
RQD–LENGTH OF SOUND CORES > 4 IN./ LENGTH CORED, %
S–SPLIT SPOON SAMPLE
U–UNDISTURBED SAMPLES,
UF–FIXED PISTON
UO–OSTERBERG
⊻ GROUNDWATER

NOTES:
1: Groundwater at 10 ft depth at start of day 2/15/07.

PROJECT 07999-0
DATE

GEI Consultants

EXAMPLE SOIL DESCRIPTIONS

SANDY SILT (ML) ~60% slightly plastic fines, ~40% mostly fine sand, 1" thick layer of fine to medium sand with <20% fines, gray.

LEAN CLAY (CL) ~90% moderately plastic fines, ~10% fine sand, olive. Boston Blue Clay. Sv = 0.5, 0.5, 0.8 tsf, Qp = 1.0, 1.5, 1.6 tsf

Stratified CLAYEY SAND (SC) and WIDELY GRADED SAND (SW) SC layers 1 to 2 inches thick consist of fine sand with ~30% moderately plastic fines, gray. SW layers 1 to 4 inches thick consist of fine to coarse sand, ~10% gravel to 1/2 inch, <5% fines, brown. Hydraulic Fill.

EXAMPLE ROCK DESCRIPTIONS

(0-9"): GRANITE, hard, one piece, joint surface slightly weathered, pink.
(6-60"): PHYLLITE, joints ~ 45° generally parallel to foliation, 9" to 44" moderate to severe jointing and joint weathering. 44" to 60" single piece, green-gray.

ARGILLITE, medium hard, moderately weathered joints, gray. Cambridge Argillite.

GEOPROBE AND ROTOSONIC

When SPTs are not performed, note sample density (sands) or stiffness (clays) in description.

CRITERIA FOR DESCRIBING DILATANCY OF FINE-GRAINED SOILS

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

SPT: Standard Penetration Test

30-inch drop with 140-lb hammer
1 3/4 to 2 1/4 turns around cathead
2-inch O.D. split spoon sampler

ENV'L TERMINOLOGY FOR SOIL DESCRIPTIONS

- **Ash** - Typically silt-size to medium sand-size.
- Do not use the term "cinders." This is not a technical term. Instead, use "ash," "burnt wood," "burnt material," or a similar term.
- **Coal-like material** - If it looks like coal but you aren't sure.
- **Clinker** - Vitrified (glass-like) or heat-fused material. Often burned impurities in coal. Often looks like pumice, but heavier.
- **Slag** - Similar to clinker, but normally refers to residue from metal ore processing.

- **Sheen** - Iridescent petroleum-like sheen. Not to be used for a "bacterial sheen," which can be distinguished by its tendency to break up on the water surface at angles. Petroleum sheen will be continuous and will not break up.
- **Stained** - Use with a color ("brown-stained") to indicate that the soil is stained a color other than its natural (unimpacted) color.
- **Coated** - Soil grains are coated with NAPL (oil, tar, etc.). There is not enough NAPL to saturate the pore spaces. ("Split spoon sampler coated with brown oil." "Soil grains coated with gray substance with slight gasoline-like odor.")
- **Saturated** - The entire sample pore space is saturated with NAPL. If you use this term, be sure it is not water saturating the pore spaces. Depending on viscosity, the NAPL may drain from a soil sample. ("Sample saturated with green, sticky substance.")
- **Blebs** - Discrete sphericals of NAPL in a soil matrix that was not visibly coated or saturated. ("Occasional blebs of reddish-brown tar.")
- **Oil** - Exhibits a petroleum odor, different from MGP odors.
- **Tar** - Exhibits an MGP odor (e.g. naphthalene-like odor).

- **Odors** - Use terms such as "naphthalene-like odor" or "petroleum-like odor." Use modifiers (strong, moderate, slight) to indicate odor intensity.

STANDARD OPERATING PROCEDURE

SS-002 Sediment Sampling Using Vibracore Equipment

1. Objective

Describe use of Vibracore methods to collect sediment samples.

Fine-grained sediments, such as sands, silts and clays can be collected using Vibracore (VC) equipment. The VC consists of a metal core barrel with a cutting edge, a sample retaining ring, a replaceable plastic liner, and an air powered piston vibrator to drive the core pipe into the unconsolidated sediments. A new plastic liner is used for each sample.

2. Materials

Equipment needed for collection of sediment samples may include (depending on technique chosen):

- Vibracore sampler
- Stainless steel sampling tools
- Laboratory provided sample bottles
- Resealable plastic bags
- Ice
- Coolers, packing material
- Chain of custody records, custody seals
- Decontamination equipment/supplies
- Maps/plot plan
- Safety equipment
- Tape measure
- Camera
- Field data sheets/field notebook/waterproof pen
- Permanent markers
- Sample bottle labels
- Paper towels
- Personal Protection Equipment (PPE)
- Global Positioning System (GPS)

3. Execution

- Sample from downstream to upstream locations so that disturbed sediment will not affect subsequent sampling locations.
- If sediment samples are being collected for laboratory analysis, the sampling equipment (i.e., cutting shoe, retainer, and sampling barrel) shall be decontaminated prior to the collection of samples at each location. Decontamination shall be conducted in accordance with SOP QA-001 –

Equipment Decontamination or according to any requirements that are outlined in the site-specific work plan(s).

- Moor the VC watercraft in a multi-point fashion.
- Measure and record the depth of the water column (depth to top of sediments).
- If possible, record the latitude, longitude, and elevation of the sample location using Global Positioning System (GPS) equipment.
- If GPS is not available, mark the sampling locations with a labeled stake, buoy, flagging, or other device, and document the locations by measuring from known reference points.
- Vibrate the core barrel into the sediments. Penetration rates will vary depending on the sediment type. When the target depth is attained, retrieve the core.
- If sufficient room is available on the VC watercraft, log the core in accordance with SOP SM-003 Soil Classification and collect analytical samples. Note attributes such as cementation, color and mineralogy (if it can be determined). The presence of iron-staining, or other staining, presence of organic matter, shells, debris or detritus will be recorded. Any odors (i.e., tar-like vs. gasoline-like vs. fuel oil-like, etc.) will be recorded. Any visual impacts will be recorded (i.e., sheens vs. non-aqueous phase liquid (NAPL) vs. staining vs. oil blebs).
- Otherwise, ferry core samples to a field representative on shore as soon as practical for logging and sampling.
- Screen for Volatile Organic Compounds (VOCs) throughout the core and record any instrument response. A photoionization detector will be used for this process. When selecting portions of the core for screening, select undisturbed portions if present. Otherwise, disturbed portions may be screened. Screening should be performed in accordance with SOP SC-004 Head Space Screening.
- Analytical samples will be selected based on criteria stipulated in the associated site-specific work plan. Analytical samples shall be collected with stainless steel spatulas (or similar) that have been decontaminated according to procedures that are outlined in SOP QA-001 Equipment Decontamination or the site-specific work plan(s). The samples shall be contained in laboratory provided jars or glassware and kept cool. The sample identification, date, time, and associated details will be recorded. Pertinent information regarding the samples will be recorded on a chain-of-custody form.

4. Limitations

4.1. When marking locations in navigable waterways, inform the appropriate regulatory agencies and take precautions to prevent navigational hazards before, during, and after sampling.

5. References

Annual Book of ASTM Standards (1993), Section 4, v. 4.08 Soil and Rock; Building Stones; Geosynthetics, D2488-90, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure), American Society for Testing and Materials (ASTM).

6. Contacts

Kim Bradley
Ryan Hoffman

Cone Penetration Testing Services

1. The cone penetration testing (CPT) work shall be performed in accordance with ASTM D5778-95, “Standard Test Method for Performing Electonic Friction Cone and Piezocone Penetration Testing of Soils”.

2. During the CPT work, soil parameters including: cone bearing / tip resistance (q_c), sleeve friction (f_s), both of which are used to calculate the friction ratio (Rf), and pore water pressure (U) will be continuously measured, at five centimeter intervals, as the cone penetrometer is advanced into the ground. The recorded measurement units (tsf, ksf, MPa, kPa, bar, psi, ft, m, etc.) shall be established by the subconsultant/owner prior to commencement of field activities.

3. All CPT work involving seismic testing shall involve using a piezocone that is equipped with an oriented geophone, with the X-axis geophone oriented to collect horizontal particle displacement of vertically propagating shear waves (SH). The subconsultant/owner shall specify the seismic test depth intervals, if different than the one meter interval specified in the bid specifications, for each CPT sounding prior to commencement of the work. It is understood that seismic testing shall be charged according on a unit rate per test basis for test depth interval according to rates established at the outset.

   As part of the seismic test procedure, an adequate seismic source generating mechanism, capable of delivering adequate energy to the soil deposits and appropriate for site conditions, shall be used. An appropriate trigger mechanism, consistent with the type of seismic source used, shall also be used. An adequate data acquisition system capable of interfacing with the geophones and trigger signals, and capable of obtaining continuous cone tip resistance and sleeve friction readings and screening out ambient noise due to traffic and other sources, shall be employed.

   The operator will use a computer capable of observing and collecting a sufficient number of data points so as to allow proper evaluation of the response of the shear wave. It is important that the operator recognizes which part of the trace is the response of the shear wave and not that of the much faster compression waves (P-waves). While performing a seismic test, the CPT push rods should be decoupled from the pushing system so as to minimize any vibrations being passed through the CPT push rods.

   When using the beam and hammer method of seismic source production, the ground competency shall be such that clear, higher amplitude shear waves are able to propagate. The operator shall check after each hit to ensure that there is no displacement of the beam during the hammer impact. If there is displacement, it usually results in poor energy transfer and poor wave response.

The geophone in the cone should be aligned with the orientation of the shear waves.

At least two hits should be taken on each side of the beam and consistency and repeatability verified before moving on to the next depth. At each depth, the operator should check that the interval time is reasonable. If not, the data should be collected again and the system verified for proper operation before continuing to the next depth.

As part of the seismic testing data submission, appropriate tables and figures showing as a minimum: the seismic CPT shear wave velocity results as well as a written description of the test method employed (i.e. hammer and beam, auger or buffalo gun, etc.). Optional figures depicting the seismic testing apparatus may also be included.

4. The cone penetration testing (CPT) rig shall be either a unitized, truck or track-mounted, purpose-built vehicle, completely equipped with all associated push rods, tooling and adapters, spare piezocone parts and ancillary equipment necessary to advance a series of CPT's to depths of up to 80 feet below ground surface.

5. The CPT rig shall include a self-contained hydraulic supply system capable of providing a continuous stroke for pushing and pulling the piezocone and push rods into and out of the ground. The operator shall operate the machine so as to advance the cone penetrometer at a constant rate of two centimeters per second while the magnitude of the required thrust fluctuates. The rig shall have sufficient weight and hydraulic capacity so as to advance the CPT tooling to a depth of 150 feet below ground surface in normal, unconsolidated soils (i.e. soft organic deposits, clays, silts and sands). The cone penetrometer, where applicable, shall be fitted with a friction reducer to advance the cone penetrometer to the required depths.

6. The CPT rig shall be equipped with a computerized data acquisition system together with a video monitoring system that displays the CPT data in real time during testing. This system shall include the capability to printout, in graphical form, draft copies of each CPT sounding as they are completed. The draft copies of each CPT plot shall include as a minimum: cone bearing / tip resistance (q_c or q_t), sleeve friction (f_s), pore water pressure (U) and soil behavior type (SBT).

7. The CPT rig shall be mechanically fit, equipped with appropriate and functional safety protection devices, regularly-serviced and be in a clean, orderly and good operating condition and free of any hydraulic leaks. A completely stocked first aid kit and fire extinguisher shall be provided with each vehicle used during the course of the work.

8. The CPT contractor shall furnish and pay for all labor, equipment, materials and fuel necessary for performing and completing the work. A potable water supply

shall be made available to the CPT contractor at each site for decontamination and grout mixing purposes. All time and costs associated with sourcing and procuring potable water from offsite facilities will be charged at pre-established standby rates plus water supply fees and costs.

9. The CPT contractor shall, at the client/owner's request and expense, obtain, fill-out, file and pay for all required test boring permits according to pre-established rates and fees.

10. Prior to commencing the work, the subconsultant/owner shall notify the CPT contractor of any occupational health and safety issues relating to the performance of the work. The CPT contractor shall maintain and abide by its corporate health and safety policies as well as its safe operating procedures at all times.

11. In accordance with pre-established rates, the CPT contractor shall, using specific and detailed information furnished by the subconsultant/owner, make every reasonable effort to ensure that underground utilities are clear in advance of completing each sounding. The CPT contractor will not assume any risk or liability, whatsoever, for damages arising from its activities in connection with miss-located or unmarked buried utilities. The CPT contractor shall not bear any financial obligation or liability arising from damages and losses relating to such unmarked or miss-located underground utilities. It is the responsibility of the subconsultant's/owner's representative to notify the CPT contractor's representative of the location, type and hazards associated with all known overhead and buried utilities in advance of commencing the work.

12. Whether in situ contamination is known to exist at the site, or not, the CPT Contractor, in its activities and work on the site, will not be held liable or responsible for any disturbance and/or redistribution thereof. Instead, we will make every effort to minimize its disturbance and/or redistribution by using perhaps the best and least intrusive techniques available (direct push services) for investigating and gathering geo-environmental data from "soft" subsurface soils.

13. The CPT Contractor's field operators shall be fully-trained, fit and qualified to operate and control the rig and associated equipment. The primary operator shall have at least three years of CPT operating experience. All personnel shall be 40 hour hazardous waste site worker certified in accordance with O.S.H.A. C.F.R. 1910.120.

14. A minimum of two properly calibrated and fully functional electronic piezocones shall be provided with each rig. They shall be manufactured according to established standards, sizes, specifications and tolerances. In addition to having a cone tip and a friction sleeve, together with the associated independent load cells, each piezocone penetrometer shall also be equipped with at least a 500 p.s.i. pore water pressure transducer (with the porous filter element located in the U_2 location immediately behind the tip), a geophone system, an inclinometer and a thermistor.

All calibration test data shall be maintained by the CPT contractor and can be provided to the subconsultant/owner upon request.

15. Before and after each CPT sounding, electronic baseline readings shall be collected and recorded in order to check for any electronic drift that may have occurred during a sounding. All baseline data shall be maintained by the CPT contractor and shall be provided for inspection upon request by the subconsultant/owner.

16. Before and after each CPT sounding, the piezocone shall be visually inspected for any damage or excessive wear and for any soil and/or water ingress. All parts damaged or worn beyond acceptable tolerance, including the cone tip, friction sleeve, wear rings and o-rings, shall be replaced before commencing the next sounding.

17. The number and proposed depths of all planned CPT soundings shall be presented to the CPT contractor in advance of mobilization. The CPT soundings shall be performed in a logical and efficient sequence, unless mutually agreed in advance otherwise.

18. ConeTec makes no guarantees that we will be able to achieve the deep depths that are anticipated. Instead, we will use our best efforts to achieve the depths expected without putting the downhole tooling at undue risk.

19. Time is of the essence for the successful completion of CPT contractor’s services.

20. It shall be assumed that the normal workday shall be nine hours per day from Monday to Friday, excluding crew travel time. Overtime rates shall apply to work performed over and above the normal workday or for work performed on weekends.

21. The CPT operator will locate the CPT rig to a position that is as close as possible to the desired and established test location without compromising the health and safety of the operators, endangering either overhead or marked underground utilities and so as not to damage either the CPT equipment or existing plant facilities. All CPT soundings will be pre-located, named and numbered by the subconsultant/owner. The CPT contractor will assume responsibility for surveying and otherwise locating or establishing elevations for the as-completed CPT locations.

22. Under certain soil conditions where coarse fill or debris (including rock, cobbles and boulders, concrete, wood, asphalt, etc.) or other natural or man-made materials are encountered during the course of the CPT sounding, the sounding may have to be being prematurely terminated. Any subsurface condition that puts undue risk on losing or damaging the downhole tooling or prevents or hinders penetrating safely may result in the sounding being terminated at the sole

discretion of the CPT operator. In the event that a CPT sounding must be terminated or abandoned because of such adverse subsurface ground conditions, it is agreed that the CPT contractor will charge and be paid for all time spent or footage completed up to and including the time/depth of termination. These charges shall include all time spent retracting and extracting equipment from the ground as well as any repair, replacement or refurbishment costs associated with damaged or lost downhole tooling.

23. If it is determined that predrilling is required in order to advance the CPT sounding, all predrilling work shall be performed according to rates and schedules that are negotiated and established in advance of the performance of such services. The CPT contractor shall not be responsible for extracting and properly abandoning that portion of any predrilled borehole that is drilled and/or cased-off by others.

24. Pore water pressure dissipation testing may be required. During these tests, the push rods shall be unweighted and the piezocone shall remain stationary throughout the duration of the test. The data acquisitions system shall be capable of displaying the dissipations test results, in real time and graphical form, as they are being conducted. Except for work involving a daily rate price schedule, payment for dissipation tests shall be according to an hourly rate for the time spent collecting dissipation test data. An adequate supply of either silicone or glycerin pre-saturated porous filter elements shall be maintained in the rig at all times.

25. Unless otherwise directed in writing, upon completion of the CPT soundings, all cone penetrometer test holes shall be abandoned by bentonite grouting in accordance with all applicable laws, established borehole sealing practices and pre-established rates. Re-insertion and/or retraction grouting are the two most common CPT hole sealing methods.

26. The CPT contractor shall maintain a clean, orderly and safe workplace throughout the duration of the contract. A minimum level of traffic protection shall include the placement of traffic cones in the vicinity of the workplace. Other traffic protection equipment can be provided at additional cost. The CPT contractor shall refurbish any disturbed areas using owner-supplied materials and apparatus before demobilizing from the site.

27. Within two weeks of completing the work, a final data report shall be submitted including final copies of all CPT plots, both printed and electronic copies of the tabular data, as well as a description and summary of the equipment and services provided. The data shall be reviewed and checked by an experienced and competent person.

28. The CPT contractor’s invoices are to be paid within 30 days of the invoice date. Charges will be assessed for the services that are rendered according to rates established at the outset of the project and as agreed from time to time throughout the duration of the contract. These charges may include, but are not limited to, all

footage completed and/or all time spent completing the work, all client approved standby time, including time spent awaiting instructions or for subconsultant/owner-supplied services, dissipation testing time, water and soil sampling time, grouting costs, decontamination surcharges, room and board charges, mobilization and demobilization costs as well as pre-approved costs relating to services, materials or supplies provided others.

At no time shall an estimate of costs provided by the CPT contractor be construed to mean that the invoiced amount will be based on the estimate. Invoices will reflect the amount of actual time spent and/or footage completed during the performance of the work.

Final payment for the CPT contractor's services shall be taken to mean that the subconsultant/owner is satisfied with the accuracy and completeness of the work and shall indemnify and save harmless the CPT contractor for any and all claims, damages and losses arising from interpretations, designs and constructions made by the subcontractor/owner or its representatives in respect of or in relation to the CPT contractor's data.

29. The CPT contractor shall maintain in effect throughout the duration of the contract, and for a period of two years following completion of the contract, the following types and amounts of insurance:

Workers' Compensation	Statutory
Employer's Liability	$1,000,000.00
Commercial General Liability	$1,000,000.00
Automobile Liability - Combined	$1,000,000.00
Umbrella/Excess Liability	$3,000,000.00

30. The CPT contractor shall be responsible for maintaining a copy of all records and data relating to the work for a period of five years following project completion.

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COARSE GRAINED SOILS

VISUAL-MANUAL DESCRIPTIONS

FOR SOILS WITH <50% FINES

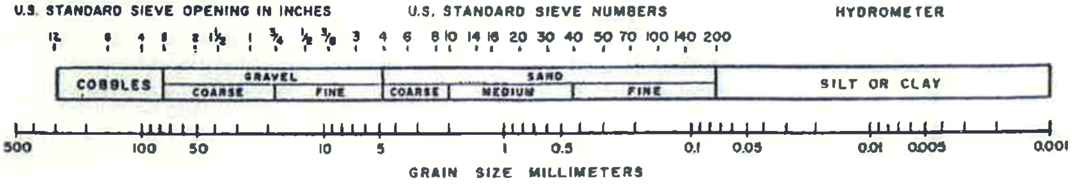
				GROUP SYMBOL		GROUP NAME
GRAVEL % gravel > % sand	≤5% fines	WIDELY GRADED		GW	<15% sand	WIDELY GRADED GRAVEL
					≥15% sand	WIDELY GRADED GRAVEL WITH SAND
		NARROWLY GRADED		GP	<15% sand	NARROWLY GRADED GRAVEL
					≥15% sand	NARROWLY GRADED GRAVEL WITH SAND
	10% fines	WIDELY GRADED	fines=ML or MH	GW-GM	<15% sand	WIDELY GRADED GRAVEL WITH SILT
					≥15% sand	WIDELY GRADED GRAVEL WITH SILT AND SAND
			fines=CL or CH	GW-GC	<15% sand	WIDELY GRADED GRAVEL WITH CLAY
					≥15% sand	WIDELY GRADED GRAVEL WITH CLAY AND SAND
		NARROWLY GRADED	fines=ML or MH	GP-GM	<15% sand	NARROWLY GRADED GRAVEL WITH SILT
					≥15% sand	NARROWLY GRADED GRAVEL WITH SILT AND SAND
			fines=CL or CH	GP-GC	<15% sand	NARROWLY GRADED GRAVEL WITH CLAY
					≥15% sand	NARROWLY GRADED GRAVEL WITH CLAY AND SAND
	≥15% fines		fines=ML or MH	GM	<15% sand	SILTY GRAVEL
					≥15% sand	SILTY GRAVEL WITH SAND
			fines=CL or CH	GC	<15% sand	CLAYEY GRAVEL
					≥15% sand	CLAYEY GRAVEL WITH SAND
SAND % sand ≥ % gravel	≤5% fines	WIDELY GRADED		SW	<15% gravel	WIDELY GRADED SAND
					≥15% gravel	WIDELY GRADED SAND WITH GRAVEL
		NARROWLY GRADED		SP	<15% gravel	NARROWLY GRADED SAND
					≥15% gravel	NARROWLY GRADED SAND WITH GRAVEL
	10% fines	WIDELY GRADED	fines=ML or MH	SW-SM	<15% gravel	WIDELY GRADED SAND WITH SILT
					≥15% gravel	WIDELY GRADED SAND WITH SILT AND GRAVEL
			fines=CL or CH	SW-SC	<15% gravel	WIDELY GRADED SAND WITH CLAY
					≥15% gravel	WIDELY GRADED SAND WITH CLAY AND GRAVEL
		NARROWLY GRADED	fines=ML or MH	SP-SM	<15% gravel	NARROWLY GRADED SAND WITH SILT
					≥15% gravel	NARROWLY GRADED SAND WITH SILT AND GRAVEL
			fines=CL or CH	SP-SC	<15% gravel	NARROWLY GRADED SAND WITH CLAY
					≥15% gravel	NARROWLY GRADED SAND WITH CLAY AND GRAVEL
	≥15% fines		fines=ML or MH	SM	<15% gravel	SILTY SAND
					≥15% gravel	SILTY SAND WITH GRAVEL
			fines=CL or CH	SC	<15% gravel	CLAYEY SAND
					≥15% gravel	CLAYEY SAND WITH GRAVEL

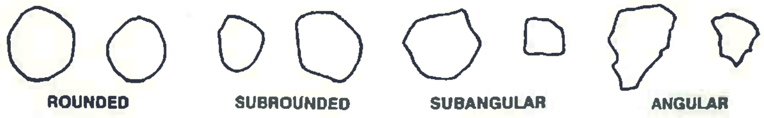
SOIL DESCRIPTION FORMAT

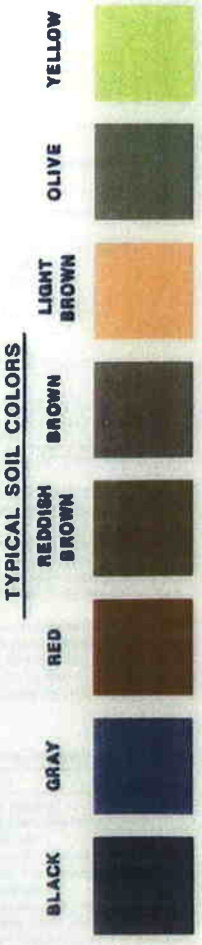
1. GROUP NAME and SYMBOL
2. Structure: stratified, laminated (layers <6 mm thick), lensed, homogeneous
3. Percent gravel, sand, fines (by dry weight), in order of predominance:
   - gravel - fine, coarse, and angularity
   - sand - fine, medium, coarse, and angularity
   - fines - plasticity characteristics
4. Percent cobbles and/or boulders (by volume)
5. Maximum particle size
6. Other - if appropriate - odor, roots, cementation, reaction with HCl, particle shape, moisture condition
7. Color
8. Local or geologic name

EXAMPLES

1. NARROWLY GRADED SAND (SP); mostly fine sand; <5% fines; brown.
2. SILTY SAND WITH GRAVEL (SM); ~60% fine to coarse, subangular sand; ~20% silty fines with low plasticity; ~20% fine, subangular gravel, max. size 10 mm; sample contained ~5% (by volume) subrounded cobbles to 200 mm; gray, Basal Glacial Till.
3. CLAYEY SAND (SC) and WIDELY GRADED SAND (SW); stratified layers ranging from ~6 to 20 mm thick; SC layers consist of fine sand with low plasticity clayey fines ranging from ~20 to 40%; SW layers consist of fine to coarse subrounded sand with <5% fines; SC layers are olive-gray, SW layers are brown; Hydraulic Fill.







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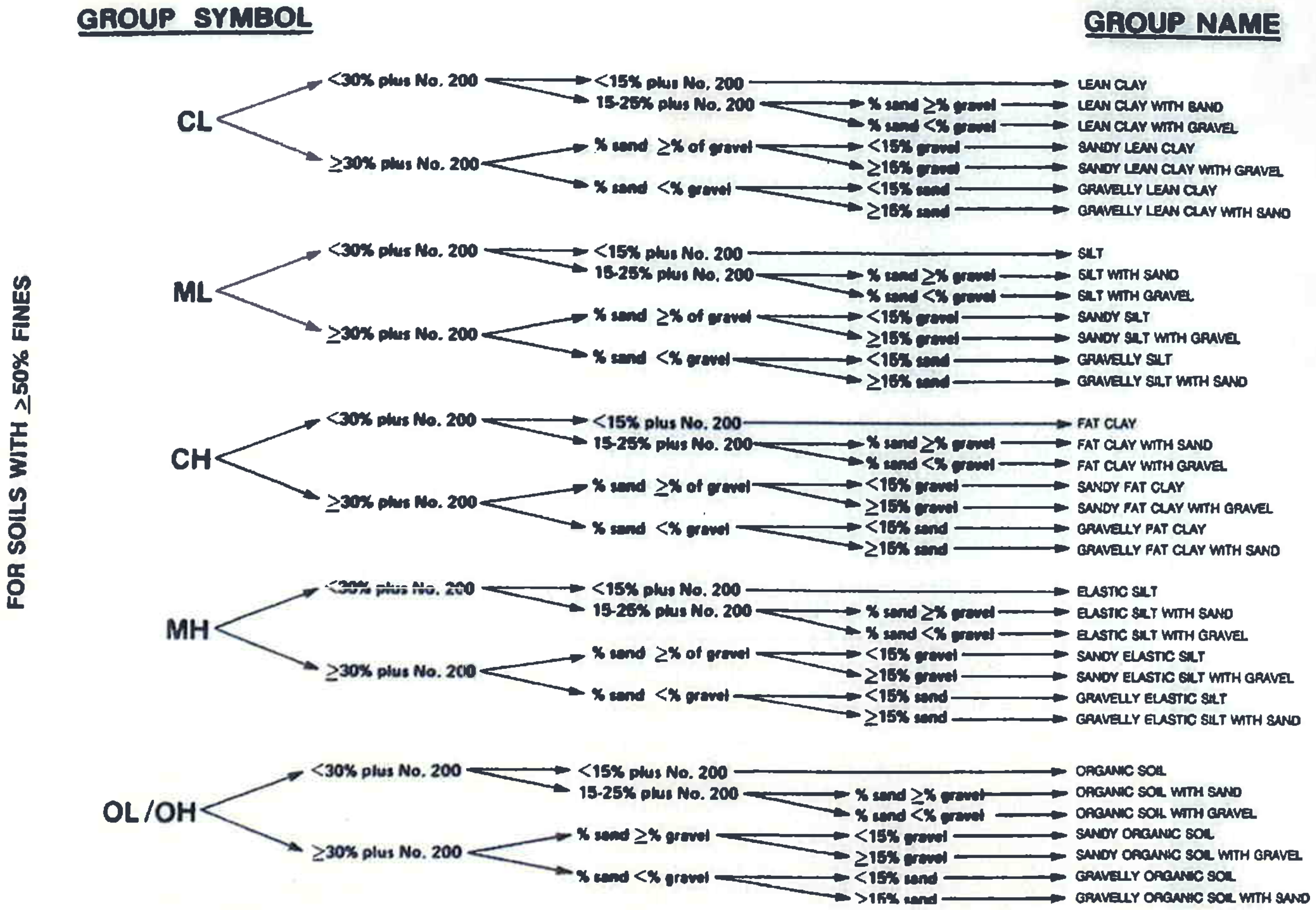
FINE GRAINED SOILS
VISUAL-MANUAL DESCRIPTIONS



SOIL DESCRIPTION FORMAT

1. GROUP NAME and SYMBOL
2. Structure; stratified, laminated, fissured, slickensided, blocky, lensed, homogeneous
3. Plasticity
4. Plasticity characteristics (if performed) - dilatancy, dry strength, toughness at PL
5. Percent gravel, sand; size ranges
6. Other - if appropriate - odor, roots, cementation, reaction with HCl, particle shape, moisture condition
7. Color
8. Local or geologic name
9. Field soil strength measurements:
   Q_p = unconfined compressive strength from pocket penetrometer
   S_v = undrained shear strength from torvane

EXAMPLES

1. LEAN CLAY (CL); homogeneous, medium plasticity, occasional small shell fragments, gray, Boston Blue Clay.
2. SANDY SILT (ML); hetergeneous till structure, nonplastic, ~30% fine to coarse, subangular sand; ~10% angular to subangular fine gravel, max. size 88 mm; brown, Glacial Till.
3. ELASTIC SILT WITH GRAVEL (MH); homogeneous, medium plasticity, medium dry strength, no dilantancy, low toughness; ~20% fine gravel, max. size 10 mm; brown, Q_p = 0.70, 0.75 tsf; S_v = 0.35, 0.40, tsf

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

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