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May 6, 2011

**VIA PDF and FEDERAL EXPRESS**

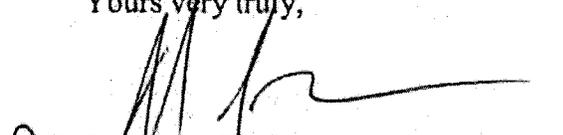
Mr. Rusty Lundberg  
Executive Secretary  
Utah Division of Radiation Control  
Utah Department of Environmental Quality  
195 North 1950 West  
Salt Lake City, UT 84116-3097

**Re: Nitrate Investigation Revised Phase 1 Work Plan -- Nitrate Investigation at the White Mesa Mill Site -- Docket No. UGW09-03**

Dear Mr. Lundberg:

Pursuant to paragraph 2 of the Tolling Agreement, Rev. 1, dated April 28, 2011 between Denison Mines (USA) Corp. and the Co-Executive Secretary of the Utah Water Quality Board, please find enclosed two copies of the *Nitrate Investigation Revised Phase 1 Work Plan, White Mesa Mill Site, Blanding Utah*, dated May 6, 2011.

Yours very truly,

  
Jo Ann S. Tischler  
Director, Compliance and Permitting

Enclosure.

cc Robert D. Baird, URS  
Daniel W. Erskine, Ph.D, INTERA  
David C. Frydenlund  
Ron F. Hochstein  
Harold R. Roberts  
David E. Turk  
Kathy A Weinel



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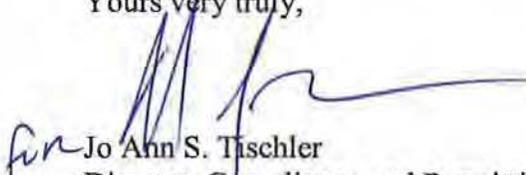
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David E. Turk  
Kathy A. Weinel

# **NITRATE INVESTIGATION REVISED PHASE 1 WORK PLAN**

**White Mesa Mill Site**

**Blanding, Utah**

*Prepared for:*



Denison Mines (USA) Corp.  
1050 17th Street, Suite 950  
Denver, Colorado 80265

*Prepared by:*



6000 Uptown Boulevard NE, Suite 220  
Albuquerque, New Mexico 87110

**MAY 6, 2011**



## TABLE OF CONTENTS

<b>LIST OF FIGURES .....</b>	<b>iii</b>
<b>LIST OF TABLES .....</b>	<b>iii</b>
<b>LIST OF APPENDICES .....</b>	<b>iii</b>
<b>ACRONYMS AND ABBREVIATIONS.....</b>	<b>iv</b>
<b>1.0 INTRODUCTION.....</b>	<b>1</b>
1.1 Problem Definition.....	3
1.1.1 Purpose of the Investigation .....	4
1.1.2 Site Description and Background .....	5
1.1.3 Summary of Previous Investigations.....	8
1.2 Project Description.....	9
1.2.1 Project Objectives.....	13
1.2.2 Project Measurements .....	13
1.3 Quality Objectives .....	14
1.3.1 Precision .....	14
1.3.2 Accuracy.....	14
1.3.3 Representativeness .....	15
1.3.4 Completeness.....	15
1.3.5 Comparability.....	15
1.3.6 Detection and Quantitation Limits .....	15
1.4 Project Organization .....	16
1.5 Special Training and Certification .....	18
1.6 Documents and Records .....	18
1.6.1 Field Documentation .....	18
1.6.2 Reports Generated .....	18
<b>2.0 DATA GENERATION AND ACQUISITION.....</b>	<b>19</b>
2.1 Sampling Design.....	19
2.2 Field Activities and Sampling Methods.....	24
2.3 Sample Handling and Custody.....	27
2.3.1 Sample Identification .....	27
2.3.2 Sample Labeling.....	28
2.3.3 Sample Documentation .....	28
2.3.4 Chain-of-Custody .....	29
2.3.5 Sample Shipment.....	30
2.4 Sample Containers and Holding Times .....	31
2.5 Analytical Methods.....	31
2.5.1 Field Analytical Methods .....	31
2.5.2 Laboratory Analytical Methods.....	32
2.6 Quality Control .....	33
2.6.1 Field Quality Control Methods.....	33
2.6.2 Laboratory Quality Control Methods .....	33
2.6.3 Internal QC Checks .....	34



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2.6.3.1	Field QC Check Procedures.....	34
2.6.3.2	Review of Compliance with Procedures in This Plan .....	34
2.6.3.3	Completeness review .....	34
2.6.3.4	Duplicates .....	34
2.6.3.5	Use of QC Samples to Assess Conformance with This Plan.....	35
2.7	Instrument and Equipment Testing, Inspection and Maintenance.....	35
2.8	Instrument Calibration .....	35
<b>3.0</b>	<b>DATA EVALUATION .....</b>	<b>35</b>
<b>4.0</b>	<b>AUDITS .....</b>	<b>36</b>
4.1	System Audits .....	36
4.2	Performance Audits .....	37
4.3	Follow-Up Actions.....	37
4.4	Audit Records .....	37
<b>5.0</b>	<b>REFERENCES.....</b>	<b>38</b>



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## LIST OF FIGURES

- Figure 1 Phase 1A Natural Nitrate Reservoir and Mill Site Background Geoprobe Locations  
Figure 2 Phase 1B Nitrate Source Areas: Geoprobe Boring Locations  
Figure 3 Phase 1C Potential Geoprobe Boring Locations

## LIST OF TABLES

- Table 1 Geoprobe Boring and Sample Locations  
Table 2 Laboratory Analytical Parameters by Task and Media

## LIST OF APPENDICES

- Appendix A Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)  
Appendix B EPA Method 1312  
Appendix C Soil Boring Log Form  
Appendix D Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations  
Appendix E Nitrate Extraction and Field Test Procedure



## ACRONYMS AND ABBREVIATIONS

ASTM	ASTM International
bgs	below ground surface
CAP	Corrective Action Plan
CCD	counter current decant circuit
CIR	Contaminant Investigation Report
COC	chain-of-custody
CSM	conceptual site model
DOE	U.S. Department of Energy
DQO	Data Quality Objective
DRC	Utah Division of Radiation Control
DUSA	Denison USA
EPA	U.S. Environmental Protection Agency
GPS	global positioning system
ID	identification number
INTERA	INTERA Inc.
MDL	method detection limit
mg/L	milligrams per liter
Mill	White Mesa Mill
msl	mean sea level
NAVLAP	National Voluntary Laboratory Accreditation Program
NELAP	National Environmental Laboratory Accreditation Program
NRC	U.S. Nuclear Regulatory Commission
PG	Professional Geologist
QA	Quality Assurance
QC	Quality Control
RPD	Relative Percent Difference
Site	White Mesa Mill property
SPLP	synthetic precipitation leaching procedure
SX	Solvent Extraction



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UAC	Utah Administrative Code
UCL	Upper Confidence Limit
UDEQ	Utah Department of Environmental Quality
USCS	Unified Soil Classification System
USDA	U.S. Department of Agriculture



## 1.0 INTRODUCTION

Denison Mines (USA) Corp. (DUSA) received a Request for Voluntary Plan and Schedule to Investigate and Remediate Nitrate Contamination at the White Mesa Mill (the "Mill") Site, near Blanding, Utah (the "Request") from the Co-Executive Secretary (the "Co-Executive Secretary") of the Utah Water Quality Board, of the Utah Department of Environmental Quality (UDEQ) on September 30, 2008. In the Request, the Executive Secretary noted that groundwater nitrate levels have exceeded the State water quality standard of 10 milligrams per liter (mg/L) in certain monitoring wells at the Mill Site.

As a result of the Request, DUSA agreed to submit a plan of action and a schedule for Co-Executive Secretary approval for completion of a Contamination Investigation Report ("CIR") to determine the physical cause(s), location(s), transfer mechanism(s) and characteristics of all source(s) of the nitrate contamination in order to form a basis for and facilitate later submittal of a groundwater Corrective Action Plan ("CAP") that meets the requirements of Utah Administrative Code (UAC) R317-6-6.15D, or to demonstrate conclusively that DUSA did not cause or contribute to the nitrate contamination in any manner and that, as a result, such a CAP is not necessary. Subsequently, in a letter dated December 1, 2009, UDEQ noting that elevated chloride concentrations exist, apparently coincident with elevated nitrate concentrations, recommended that DUSA also address and explain the elevated chloride concentrations.

DUSA and the Co-Executive Secretary entered into a Stipulated Consent Agreement Docket No. UGW09-03 dated January 27, 2009 ("Consent Agreement") related to nitrate contamination at the Mill. Pursuant to Item 6.A of the Consent Agreement, DUSA submitted a Nitrate Contamination Investigation Report, White Mesa Uranium Mill Site, Blanding Utah, dated December 30, 2009 (CIR) to the Utah Division of Radiation Control ("DRC"). By a letter dated October 5, 2010, the Co-Executive Secretary notified DUSA of his determination that the CIR is incomplete.

By an email transmitted to the Co-Executive Secretary on October 20, 2010, and pursuant to Item 11 of the Consent Agreement, DUSA requested an amendment to the deadline stipulated in item 7.C of the Consent Agreement DUSA requested item 7.C be amended as follows: a. DUSA representatives would meet with the Co-Executive Secretary and his legal counsel within two weeks from the date of the email to discuss the legal responsibilities of DUSA with respect to the nitrate contamination; b. Once the legal responsibilities of DUSA with respect to the nitrate contamination have been determined, DUSA would, within 30 days after such a determination was made, submit to the Co-Executive Secretary for approval a plan and schedule to perform any further investigations that may be required in order to remedy any such omissions, content



requirements or failures of performance standards, and to submit a revised CIR; and c. DUSA would perform such investigations and submit a revised CIR in accordance with the agreed upon plan and schedule. At an October 26, 2010, meeting with the Co-Executive Secretary, DRC staff and legal counsel DUSA reported that it was premature to submit a schedule for submittal of performance standards and a Corrective Action Plan for the nitrate contamination. In turn, DUSA presented a new theory for a possible source of the nitrate and chloride contamination beneath the Mill, based on DUSA's review of the scientific literature (New Theory), specifically, that the nitrate contamination source is or could be caused by naturally occurring nitrate and chloride salt deposits located in the vadose zone near or beneath the Mill site area, which have been mobilized by natural and/or artificial recharge. The parties agreed that this New Theory warranted additional investigation, along with certain of the other additional studies suggested in the October 5, 2010 DRC Notice. DUSA submitted via email on November 15, 2010, a letter setting out the additional studies to be considered that have been identified to date, including the additional studies suggested in the October 5, 2010 DRC Notice, proposed additional studies relating to the New Theory, and other additional studies that DUSA believes may be relevant. At a November 30, 2010 meeting between DRC Staff and DUSA technical and regulatory staff, DUSA presented a number of additional studies (herein "Additional Studies") to be performed by DUSA in order to complete the CIR. The Co-Executive Secretary and DUSA further agreed that DUSA would prepare a detailed plan and schedule (the "Plan and Schedule") for performing such studies and for submittal of a revised CIR that meets the requirements of all applicable regulations on or before February 15, 2011. During the November 30, 2010 meeting, it was agreed that both the Plan and Schedule and the revised CIR will be subject to Co-Executive Secretary approval. DUSA's commitment to prepare and submit the Plan and Schedule is set out in a Tolling Agreement (the "Tolling Agreement") dated December 15, 2010 between DUSA and the Co-Executive Secretary.

DUSA submitted a draft Work Plan on February 14, 2011. During subsequent discussions with DRC staff, the Co-Executive Secretary and DUSA agreed that the additional studies could require as many as five phases, and the schedule should include points of consultation between phases at which the Co-Executive Secretary and DUSA could evaluate and agree on the redirection, addition, or elimination of subsequent phases.

The Tolling Agreement was revised on April 28, 2011, to allow time for:

- DUSA to prepare and submit a Revised Work Plan for Phase 1 (by May 6, 2011)
- DUSA to prepare and submit a revised Work Plan Revised Phases 2 through 5 (by June 3, 2011), including a Conceptual Site Model of potential nitrate sources,



- The Co-Executive Secretary to review and approve the revised Work Plans, including modifications,
- The Co-executive Secretary and DUSA to agree on a revised or replacement Consent Agreement that incorporates the deliverables and timelines in the approved Phase 2 through 5 Work Plan.

This document is the revised Phase I Work Plan for Phase 1A, B, and C, which is being submitted in accordance with Revised Tolling Agreement and which contains details for the execution of Phase 1 as described in Attachment 1 of the Revised Tolling Agreement. The purpose of this Revised Phase 1 Work Plan is to allow field investigations to begin as early as the week of May 16, 2011, if possible, while the Phase 2 through 5 Work Plan and conceptual site model (CSM) are developed. The Phase 2 through 5 Work Plan will include a schedule for submission of activities and reporting for the remaining phases, including updates of the CSM, to be incorporated into a Revised Consent Agreement.

## 1.1 Problem Definition

Groundwater from Site monitoring wells has been sampled in a number of wells on site since 1978 and nitrate has been measured as nitrate+nitrite and reported as nitrogen. Nitrate (NO<sub>3</sub>-) and nitrite (NO<sub>2</sub>-) are naturally occurring inorganic ions that are part of the nitrogen cycle. Because nitrite is easily oxidized to nitrate, nitrate is the compound predominantly found in groundwater. As mentioned in Section 1, above, nitrate levels in several wells on site have exceeded the Utah Groundwater standard of 10 mg/L.

With respect to the amount and concentration of nitrate in the plume, the highest nitrate level that has been detected at the Site is 69 mg/L in monitor well TWN-2 in a sample collected on June 2, 2010. Nitrate levels in that well have declined and the most recently measured value in a sample of groundwater, taken on February 1, 2011 is 43 mg/L. Typical concentrations in the area of the nitrate plume range from 5 mg/L to 25 mg/L.

Chloride is considered to be a major element that occurs in almost all groundwater (Hem, 1992) and occurs as a chloride ion (Cl<sup>-</sup>). There is no human health standard for chloride in groundwater. The US Environmental Protection Agency (EPA) Standard for chloride in drinking water (250 mg/L) is an aesthetic standard related to the salty taste of water with chloride concentrations in excess of that amount. The recommended EPA criterion for the propagation of wildlife is 1,500 mg/L.

With respect to the amount and concentration of chloride in the plume, the highest chloride level that has been detected at the Site is 1,180 mg/L, in monitor well TW4-24 in a sample collected



on September 10, 2008. Chloride levels in that well have declined and the most recently measured value in a sample of groundwater, taken on February 17, 2011, is 1,100 mg/L. Typical concentrations in the area of the nitrate plume range from 100 mg/L to 300 mg/L.

### **1.1.1 Purpose of the Investigation**

The purpose of the Phase 1 investigation is to determine background concentrations of nitrate and chloride in the alluvial soil column in undisturbed areas in the vicinity of the Mill, to locate a natural nitrate and chloride reservoir in the alluvial soil, and to compare nitrate and chloride concentrations found in soil near potential Mill sources to background concentrations.

There are several potential sources of nitrate and chloride at the Mill and in the vicinity of the Mill. Such sources include septic leach fields, municipal sewage plant discharge water used historically as mill water makeup, livestock activity at the wildlife ponds and the historic pond, the former flyash pond, laboratory chemical spills, and former military uses of the site. Another potential source is a natural nitrate reservoir. Such concentrations or “reservoirs” of nitrate and chloride have been identified in the scientific literature (Walvoord, et al., 2003, Scanlon, et al., 2005 and others). “Unsaturated-zone chloride and nitrate profiles archive changes in recharge related to recent conversion of rangeland to agricultural ecosystems. Increased recharge associated with dryland as well as irrigated agriculture can lead to degradation of groundwater quality because of leaching of salts that have been accumulating in the unsaturated zone for thousands of years prior to cultivation, because of application of fertilizers, and, in irrigated areas, because of evapo-concentration of applied groundwater. In the SHP (southern high plains), median groundwater nitrate-N concentrations increased by 221% beneath irrigated areas and 163% beneath dryland areas, reflecting LU/LC-induced (land use/land cover) contamination of groundwater.” (Scanlon, et al., 2005).

The Phase 1 investigation will use direct push technology (Geoprobe) borings to collect subsurface alluvial soil in and around the Mill which will be analyzed for the presence of nitrate and chloride concentrations. The Investigation is divided into three phases, summarized here. Details of each part of the Phase 1 investigation are provided in Section 1.2, below.

### **Phase 1 A - Geoprobe investigation in Undisturbed Locations**

This phase of the investigation is designed to quantify naturally occurring (i.e. “background”) nitrate and chloride concentrations in alluvial soil at the Site and to explore for a naturally occurring nitrate reservoir. Background concentrations will be established advancing Geoprobe direct push machine (Geoprobe) borings at each of 20 locations spread across the Site (Figure 1), selected to represent areas which have not undergone irrigation or other forms of culturally-induced surface water recharge. This selection process is designed to maximize the opportunity



of finding soil chemistry that reflects only natural cycles of wetting and drying from precipitation and evapotranspiration. The presence of such a nitrate and chloride reservoir would suggest that these concentrations could be present throughout the White Mesa alluvial soil column and could be mobilized to groundwater as the result of increased surface water recharge due to irrigation, surface water impoundment, canal leakage, or other recharge processes. Frequency of borings and sample collection is discussed in detail in Section 2.

### **Phase 1B - Geoprobe Investigation of Potential Nitrate Source Locations**

This phase of the investigation is designed to quantify nitrate and chloride concentrations in alluvial soil at the Site in locations where past or ongoing activities may have contributed nitrate and/or chloride to the soil and/or groundwater. The potential nitrate source locations include up to seven (7) leach fields, as well as other installations such as ammonia tanks, a sewage vault, and Lawzy Lake, a former pond that may have held contaminated water. The potential source locations and details of this phase of the investigation are discussed in Section 2.

### **Phase 1C - Geoprobe Investigation of Other Potential Nitrate Sources**

This phase of the investigation is designed to quantify nitrate and chloride concentrations in alluvial soil at the Site at seven additional locations identified by DUSA for comparison to the background of naturally occurring concentrations established in Phase 1A. At each Geoprobe location, DUSA may choose to collect three (3) discrete one-foot core samples from the bottom one-foot of each of the following intervals: the first 1/3, second 1/3, and third 1/3 depth, based on the total depth of penetration at each site. DUSA may use the Nitrate Field Test to determine whether nitrate concentrations are detectable and whether a sample will be sent to an approved analytical laboratory for analysis. For any boring where soil core samples will be sent for laboratory analysis, DUSA will that the standards of representativeness described under Phase 1A are met. Any such soil laboratory results may then be compared to the site background soil concentrations. The seven additional locations and details of this phase of the investigation are described in Section 2.

#### **1.1.2 Site Description and Background**

The Site is a uranium mill with a vanadium co-product recovery circuit, located within the Colorado Plateau physiographic province approximately 5 miles south of the city of Blanding, Utah. Mill construction began in 1979, and conventionally mined uranium ore was first processed in May 1980. Over its 25 year operating history the Mill has processed over 4 million tons of conventionally mined and alternate feed uranium ores for the recovery of 25 million pounds of U<sub>3</sub>O<sub>8</sub> and 34 million pounds of vanadium to date.



Potential on Site sources of nitrate and chloride addressed in the CIR (INTERA, 2009) include:

- the septic leach fields at the site;
- the municipal sewage plant discharge water used historically as Mill water makeup;
- livestock activities at the wildlife ponds;
- livestock activities at the Historic Pond;
- agricultural activities;
- the former Fly Ash Pond;
- potential historic spills of ammonia-bearing and/or chloride-bearing process chemicals;
- potential breach in the Mill circuit floor drains or tailings transfer lines; and
- a potential leak in the Mill's tailings cells.

Hydrologic considerations presented in the CIR preclude all but the following as candidate potential sources:

- the waters from the Frog Pond, the Upper Wildlife Pond, Lawzy Lake and the Lawzy Sump, including potential livestock activity near the wildlife ponds;
- possible livestock activity near the Historic Pond; and
- possible influences from septic leach fields at the site, in particular the SAG Leach Field and the Main Leach Field.

The other potential sources on Site are downgradient of the nitrate plume, ruling them out. In addition, contamination due to spills would appear to be too small and infrequent to give rise to the identified plumes, and there is no apparent reason to expect a breach in process facilities and drains that would give rise to the plume. Based on the currently available process and historic information, the most plausible source for apparently coincident chloride and nitrate plumes would be a chlorinated sewage-based source north or northwest of the Mill.

### **Site Status**

The Mill was in standby status from November 1999 to April 2002. During the standby period, the Mill received and stockpiled alternate feed materials from the Ashland 1 and Linde formally utilized sites remedial action program sites, as well as other sources of alternate feed materials.

During the period from April 2002 to May 2003, the Mill processed 266,690 tons of alternate feed materials. Subsequently the Mill entered standby mode but continued to stockpile alternate feed materials.



Uranium Mills are licensed to operate either by the US Nuclear regulatory Commission (“NRC”) or by state agencies which have received authorization to implement NRC’s licensing program under the Atomic Energy Act (“Agreement States”). The Mill received its initial source material license from the U.S. Nuclear Regulatory Commission in 1980. The State of Utah became an agreement state in 2005, at which time regulatory authority for the Mill passed to the Utah DRC. The Mill is regulated under Utah Radioactive Materials License UT 1900479. Groundwater quality is regulated by the Mill’s Utah Groundwater Discharge Permit UGW370004.

The Mill is currently operating, having commenced operations in March 2005, with the processing of Cameco alternate feed materials. The Mill has since processed natural ores and alternate feeds. Alternate feeds have been processed both in the main mill circuit and the alternate feed circuit, constructed in 2009.

### **Physical Setting**

The Mill is located near the western edge of the Blanding Basin within the Canyonlands section of the Colorado Plateau physiographic province. Broad, generally horizontal uplift and subsequent erosion have produced topography consisting of high plateaus, mesas, buttes, monuments, and deep canyons incised into the relatively flat-lying Mesozoic and Paleozoic sedimentary rocks.

Northeast of the Mill site, igneous intrusions forming the core of the Abajo Mountains have disturbed the classic flat-lying Colorado Plateau stratigraphy, resulting in uncharacteristic local folding and faulting of sedimentary rocks. The Abajo’s rise to more than 11,000 feet above mean sea level (msl), and have likely provided a source of sediments to the Mill site (5,600 feet above msl) during intrusion and disturbance of older rocks.

Quaternary deposits overlie the sequence of Mesozoic rocks present in the region. The Cretaceous Mancos Shale and Dakota Sandstone represent the local top of the Mesozoic section in the region and are underlain by the Lower Cretaceous Burro Canyon Formation. This unit is underlain in turn by the Jurassic Morrison Formation (includes the Brushy Basin, Westwater Canyon, Recapture, and Salt Wash Members), Summerville Formation, Entrada Sandstone, and the Navajo Sandstone. The Navajo is underlain by the Jurassic Kayenta Formation, which in turn is underlain by Triassic Chinle and Moenkopi Formations. Paleozoic sedimentary rocks underlie these Mesozoic units.

Cretaceous geologic units that stratigraphically overlie the Burro Canyon Formation regionally (Mancos Shale and Dakota Sandstone) have been removed by erosion in the vicinity of the Mill. Thus, the lower Cretaceous Burro Canyon Formation (already present during the Mid-Tertiary



Abajo igneous intrusive event) is directly overlain by Quaternary deposits at the Mill site. The Quaternary colluvial/alluvial sediments are typically coarse-grained deposits that contain little water. The Burro Canyon Formation is described as interbedded conglomerate and grayish-green shale with light-brown sandstone lenses deposited in a fluvial environment (Aubrey, 1989). The average thickness of the unit is approximately 75 feet (US Department of Energy [DOE], 2004).

The Burro Canyon Formation hosts the uppermost occurrence of groundwater at the site. Groundwater in this unit is perched (i.e., isolated from groundwater that occurs in geologic units that underlie the Burro Canyon Formation). Perched water is supported by the relatively impermeable, underlying, fine-grained Brushy Basin Member of the Morrison Formation. The permeability of the Burro Canyon Formation is generally low. Some conglomeratic zones may exist east to northeast of the tailings cells, potentially explaining a relatively continuous zone of higher permeability. The saturated thickness of the perched groundwater zone ranges from approximately 82 feet in the northeast portion of the site to less than 5 feet in the southwest portion of the site (DOE, 2004). Groundwater isopleths based on water level data collected in 2010, indicate that flow in the perched zone is generally from northeast to southwest, although in the eastern portion of the site the gradient has a more southerly component.

Groundwater in the regional Entrada/Navajo aquifer is under artesian pressure (upward flow gradient) providing a hydrologic barrier to any potential seepage from overlying geologic units. Perched groundwater within the Burro Canyon Formation is characterized by low yields and is generally of poor quality, containing moderate to high concentrations of chloride, sulfate, and total dissolved solids (Hunt, 1996).

### **1.1.3 Summary of Previous Investigations**

Previous investigations with respect to the presence of nitrate in groundwater under the Mill include a Nitrate and Chloride Source Review Memo (Tischler, 2009), a Nitrate Contamination Investigation Report (CIR) (INTERA, 2009), Initial Nitrate Monitoring Report (DUSA, 2009), and quarterly nitrate and chloride reporting to the DRC (DUSA, 2010-2011), and ongoing investigations into historic land uses, which have not yet been published.

The Nitrate and Chloride Source Review Memo (Tischler, 2009) identifies and discusses potential nitrate sources at the Mill, including septic leach fields, municipal sewage plant discharge water used historically as mill water makeup, livestock activities at the wildlife ponds and the historic pond, the former fly ash pond, potential historic spills of ammonia-bearing process chemicals, potential breach in the mill circuit floor drains or tailings transfer lines, mill laboratories, and a potential leak in the Mill's tailing cells. The Memo also discusses potential historical sources and off-site sources. The Memo concludes that the most likely source for



nitrate and chloride comes from upgradient of the current plume in the municipal sewage plant discharge water used historically as mill water makeup, possible livestock activity near the historic pond, and possible influences from septic leach fields at the Site, in particular, the SAG leach field and the Main Leach Field. Since the Publication of the Nitrate and Chloride Source Review Memo, DUSA's understanding of historic land uses has continued to be updated through literature, internet, and other land use studies, discussed below.

Land uses proximal to the Mill include farming, ranching, cattle grazing and feed and grain silos. A further evaluation of historical land use in the vicinity of the Site will be performed in order to supplement the source evaluation (the "Source Review Report") that was included in the CIR. This further evaluation is currently under way and will (a) identify areas that have been subject to agricultural activities and (b) evaluate land-use practices that may have led to elevated levels of nitrate and other contaminants in groundwater. Objective (a) is also required to identify areas for sampling of buildup of atmospheric nitrogen, since we seek to sample areas that have not been subject to anthropomorphic activities. This analysis includes evaluation of historical aerial photography, historical Landsat satellite imagery, and an Internet-based search of historic military activities in the region.

The Nitrate CIR (INTERA, 2009) also discusses the potential sources identified in the Source Review Memo. The CIR describes the sampling design and installation of 19 new wells used to characterize the nitrate and chloride plumes. The CIR characterizes the nitrate and chloride plumes with the data collected from existing and new monitoring wells at the Mill. The investigation concludes that the nitrate and chloride appear to originate from the same source, and that source is upgradient of the Mill property more than 1.2 miles from the Mill facilities and is not the result of Mill activities and was not caused or contributed to in any manner by Mill activities.

Beginning with the third quarter of 2010, DUSA performed quarterly sampling and analysis of the new nitrate wells.

## **1.2 Project Description**

This investigation will utilize direct push technology to bore into the subsurface in areas that are undisturbed and areas that are potential sources of nitrate and chloride contributing the groundwater plume at the Mill. The soil borings will be analyzed for the presence of nitrate+nitrite and chloride by an analytical laboratory and field screened using Hach Test Kits for nitrate+nitrite and QuanTab Test Kits for chloride (test kits will be used in Phase 1A Part 2). The nitrate investigation will be divided into three phases. Phase 1A is divided into two parts.



*Part 1 - Geoprobe Investigation to Determine Background Nitrate, Chloride and Ammonia Soil Concentrations in Areas Unimpacted by Human Activities ("undisturbed locations"):*

1. A Geoprobe boring (Boring #1) will be conducted down to bedrock refusal at each of the 20 undisturbed Geoprobe boring locations (per Figure 20 of the February 18, 2011 DUSA Work Plan and Schedule). Per our agreement with DRC in previous meetings, three (3) samples will be collected from each Geoprobe core location. Soil core samples will be collected randomly from the first 1/3, second 1/3, and third 1/3 intervals of the total penetrated depth at each location. Per agreement at previous meetings, DRC or DUSA may collect additional soil samples at additional intervals. Each soil core sample will be sent to the analytical laboratory for analysis of nitrate (as N), chloride, and ammonia nitrogen (as N). Soil analysis will be conducted by an environmental laboratory currently certified by the State of Utah, using EPA approved sample and analysis methods. The Nitrate Extraction and Field Test Procedure, as described in Appendix A of the February 18, 2011 DUSA Work Plan (hereafter Nitrate Field Test) will not be used. Background concentrations for each of the above analytes will be based on the 95% Upper Confidence Limit (UCL) of the 60 samples collected and analyzed. Said UCL will be determined using commonly accepted descriptive statistical methods and will be subject to DRC approval. After DRC approval, said site soil "background" concentration will be used for comparative and determinative purposes in PHASE 1B of the Geoprobe Investigation of Potential Nitrate Source Locations described below.

*Part 2 - Geoprobe Investigation of a Possible Natural Nitrate Salt Reservoir:*

2. A surface soil sample (0 to 0.5 foot below ground surface (bgs)) will be collected by shovel in close vicinity (within 5 horizontal feet) of each of the 20 Geoprobe boring locations from the Phase 1A, Part 1 work, described above. This surface soil sample will be analyzed using the Nitrate Field Test, described above, and will be used as a baseline sample for comparison with laboratory analysis results of the same depth. At DUSA's discretion, this surface sample can be collected instead from the duplicate Geoprobe boring, described below.
3. A duplicate Geoprobe boring (Boring #2) will be installed in close proximity (within 5 horizontal feet) to each of the a) Geoprobe borings (Boring #1) from Phase 1A, Part 1, above) and b) the surface soil samples in Phase 1A, Part 2, above. Each of these duplicate Geoprobe borings (Borings #2) will be driven down to bedrock refusal at each of the 20 undisturbed locations, mentioned in Phase 1A, Part 1, above. Said soil core samples will be collected at each Geoprobe location to



- a. ensure a minimum core recovery of 95%,
  - b. provide discrete and individual 1-foot depth samples,
  - c. be preserved and placed into a sealable plastic bag that is labeled in accordance with EPA approved field methods, and
  - d. Each discrete 1-foot soil sample will be thoroughly mixed and homogenized within the plastic bag before any field or laboratory analysis. Said soil samples will then be considered representative of the individual 1-foot core interval. Per agreement at previous meetings, DRC or DUSA may collect additional soil samples at additional intervals.
4. Thereafter, an aliquot of each discrete 1-foot soil sample will be taken from the bag and analyzed using the Nitrate Field Test described in Appendix A of the February 18, 2011 DUSA Work Plan and Schedule.
  5. Elevated soil samples, i.e. those found with Nitrate Field Test results that are twice the baseline concentration, as determined by field analysis of the 0-0.5-foot bgs sample described above (Phase 1A, Part 2, Item 1) will be sent to the approved analytical laboratory for nitrate (as N) soil analysis. The Nitrate Field Test results from Boring #2 will be used as a tool by DUSA to examine the possibility of a natural nitrate salt deposit in vadose zone soils at the White Mesa Mill site using the 1-foot sample intervals.

### **Phase 1B – Geoprobe Investigation of Potential Nitrate Source Locations**

This investigation is designed to quantify nitrate and chloride concentrations in alluvial soil at the Site in locations where potential Mill-related nitrate and/or chloride sources could have contributed to groundwater. The 24 potential sources that have been identified to date are displayed on Figure 2 and listed below.

1. Main leach field (also known as Leach Field east of Scalehouse, 1985 to present) (High Priority)
2. Sewage vault/lift station (Low Priority)
3. Scale house leach field, (also known as Leach Field south of Scalehouse, 1977-1979) (High Priority)
4. Former office leach field (High Priority)
5. Ammonia tanks (Low Priority)
6. SAG leach field (Leach Field north of mill building, 1998 to 2009) (High Priority)



7. Cell 1 leach field (Leach Field east of Cell #1, up to 1985) (Low Priority)
8. Fly ash pond (Low Priority)
9. Chlorine Tanks (never contained ammonium chlorate)
10. Ammonium sulfate crystal tanks (Low Priority)
11. Lawzy sump (High Priority)
12. Lawzy Lake (High Priority)
13. Former vault/lift station (to Former Office Leach Field) (1992 to 2009) (Low Priority)
14. Truck shop leach field (1979-1985) (Low Priority)
15. New Counter Current Decant/Solvent Extraction (CCD/SX) leach field (Low Priority)
16. Historical Pond (High Priority)
17. Wildlife Pond (High Priority)
18. CCD (Inaccessible)
19. YC Precip Mini Lab (Inaccessible)
20. V2O5 Mini Lab & V2O5 Precip (Inaccessible)
21. SX Mini Lab (Inaccessible)
22. Chem Lab (Inaccessible)
23. Met Lab (Inaccessible)
24. V2O5 Oxidation Tanks (Inaccessible)

These potential sources will be investigated as follows:

Sources 1-8 and 10-17 listed above will be sampled in Phase 1B. Multiple Geoprobe borings will be driven down to bedrock refusal at 16 of the potential on-site Nitrate Sources, shown on the DUSA handout provided in the April 20, 2011 meeting. Of these 16 potential sources, 8 are considered by DUSA as High Priority Locations and will be investigated with four (4) Geoprobe borings at each source in a north-east-west-south relative orientation. For the remaining 8 potential sources, DUSA considers them to be Low Priority Locations and will each receive (2) Geoprobe borings. The location of these borings will be based on best professional judgment, considering the most likely water / waste / wastewater discharge direction.

Source number 9 and numbers 18-24 listed above are inaccessible due to pavement or other construction above the source and will not be sampled. The main leach field and the new CCD/



SX leach field may be associated with special health and safety requirements and may be sampled in a later Phase of the nitrate investigation.

**Phase 1C - Geoprobe Investigation of Other Potential Nitrate Sources:** This phase of the investigation will quantify nitrate and chloride concentrations in alluvial soil at seven additional locations identified by DUSA for the comparison to the background of naturally occurring concentrations established in Phase 1A. These locations, identified in Figure 3, are soil sample locations associated with groundwater wells which have had historically elevated levels of nitrate contamination, but which are not associated with potential Mill site sources.

Soil collected from borings will be field screened for nitrate and chloride, and containerized for shipment to the analytical laboratory for analysis.

### **1.2.1 Project Objectives**

The purpose of this nitrate investigation is to quantify nitrate and chloride in the alluvial soil column in selected locations at the Site with the following goals:

1. To establish background concentrations of nitrate and chloride in the alluvial soil in the vicinity of the Mill
2. The data generated by this investigation will be used to test the “new theory” hypothesis that the nitrate contamination source is or could be caused by naturally occurring nitrate and chloride salt deposits located in the vadose zone near or beneath the Mill site area, which have been mobilized by natural and/or artificial recharge (Walvoord, et al., 2003, Scanlon, et al., 2005 and others).
3. The data will be used to test hypotheses regarding to what extent Mill-related sources contributed, if at all, to the groundwater nitrate plume,
4. The data will be used to test hypotheses regarding to what extent present or historic non-Mill-related sources contributed, if at all, to the groundwater nitrate plume.

The nitrate investigation has been divided into three phases, Geoprobe Investigation in Undisturbed Locations, Geoprobe Investigation of Potential Nitrate Source Locations, and Geoprobe Investigation of Other Potential Nitrate Sources. These phases are described in detail in Section 2.0.

### **1.2.2 Project Measurements**

Project measurements will include laboratory analysis of soil chemistry, field analysis of soil chemistry, and Unified Soil Classification System (USCS) soil type classification made at the time of collection by visual-manual inspection as described in the *Standard Practice for*



*Description and Identification of Soils (Visual-Manual Procedure) ASTM D 2488 – 09a* (ASTM, 2009; Appendix A). Note that the classifications presented in *ASTM D 2488 – 09a* are identical to the USCS classifications presented in the *Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System) ASTM D 2487 – 10* (ASTM, 2010), but derived from field observations rather than laboratory analysis. All soil samples will be submitted to the analytical laboratory for Synthetic Precipitation Leaching Procedure (“SPLP”) using EPA Method 1312 (Appendix B). Method 1312 will produce a leachate of all soil samples which will be analyzed for nitrate, chloride sulfate and nitrogen as ammonia using EPA Method 353.2, EPA method 300.0, and EPA method 350.1 (equivalent to 350.2) respectively. Field nitrate methods include Hach Nitrate and Nitrite Field Strips and QuanTab Chloride Test Strips.

### 1.3 Quality Objectives

Specific quality objectives have been established for each of the data assessment parameters identified. These objectives are expressed as quantitative and qualitative statements concerning the type of data needed to support a decision, based on a specified level of uncertainty. The criteria (predetermined acceptance limits) are expressed as numerical values for laboratory analyses and field tests identified. Further discussion of each parameter and rationale for its use is presented below.

#### 1.3.1 Precision

Precision is defined as the measure of variability that exists between individual sample measurements of the same property under identical conditions. Precision is measured through the analysis of samples containing identical concentrations of the parameters of concern. For duplicate measurements, precision is expressed as the relative percent difference (“RPD”) of a data pair and will be calculated by the following equation:

$$RPD = [(A-B)/\{(A+B)/2\}] \times 100$$

Where A (original) and B (duplicate) are the reported concentration for field duplicate samples analyses (or, in the case of analyses performed by the Analytical Laboratory, the percent recoveries for matrix spike and matrix spike duplicate samples) (EPA SW-846, Chapter 1, Section 5.0, page 28).

#### 1.3.2 Accuracy

Accuracy is defined as a measure of bias in a system or as the degree of agreement between a measured value and a known value. The accuracy of laboratory analyses is evaluated based on analyzing standards of known concentration both before and during analysis. Accuracy will be evaluated by the following equation (EPA SW-846, Chapter 1, Section 5.0, page 24):



$$\% \text{ Recovery} = ( | A-B | / C ) \times 100$$

Where:

A = the concentration of analyte in a sample

B = the concentration of analyte in an unspiked sample

C = the concentration of spike added

### **1.3.3 Representativeness**

Representativeness is defined as the degree to which a set of data accurately represents the characteristics of a population, parameter, conditions at a sampling point, or an environmental condition. Representativeness is controlled by performing all sampling in compliance with this Plan.

### **1.3.4 Completeness**

Completeness refers to the amount of valid data obtained from a measurement system in reference to the amount that could be obtained under ideal conditions. Laboratory completeness is a measure of the number of samples submitted for analysis compared to the number of analyses found acceptable after review of the analytical data. Completeness will be calculated by the following equation:

$$\text{Completeness} = (\text{Number of valid data points} / \text{total number of measurements}) \times 100$$

Where the number of valid data points is the total number of valid analytical measurements based on the precision, accuracy, and holding time evaluation. Completeness is determined at the conclusion of the data validation.

### **1.3.5 Comparability**

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data.

### **1.3.6 Detection and Quantitation Limits**

The method detection limit (MDL) is the minimum concentration of an analyte that can be reliably distinguished from background for a specific analytical method. The quantitation limit represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a sample matrix. Project-required reporting limits are minimum quantitation limits for specific analytical methods and sample matrices that are typically several times the MDL to allow for matrix effects.



## **1.4 Project Organization**

### **Functional Groups**

This Plan specifies roles for a Quality Assurance (QA) Manager as well as representatives of three different functional groups: the data users; the data generators, and the data reviewers/approvers. The roles and responsibilities of these representatives are described below.

### **Overall Responsibility for the QA/QC Program**

The overall responsibility for ensuring that the Quality Assurance/Quality Control (QA/QC) measures are properly employed is the responsibility of the QA Manager. The QA Manager is typically not directly involved in the data generation (i.e., sampling or analysis) activities. The QA Manager a qualified person designated by Denison Mines (USA) Corp. ("DUSA") corporate management.

### **Data Requestors/Users**

The generation of data that meets the objectives of this Plan is necessary for management to make informed decisions to quantify nitrate and chloride in the alluvial soil column in selected locations at the Site with the goal that the data generated by this investigation will be used to test the "new theory" hypothesis that the nitrate contamination source is or could be caused by naturally occurring nitrate and chloride salt deposits located in the vadose zone near or beneath the Mill site area, which have been mobilized by natural and/or artificial recharge. Accordingly, the data requesters/users (the "Data Users") are therefore DUSA's corporate management and regulatory authorities. The data quality objectives ("DQOs") required for any sampling event, such as acceptable minimum detection limits, are specified in this Plan.

### **Data Generators**

The individuals who carry out the sampling and analysis activities at the request of the Data Users are the data generators. This involves sample collection, record keeping and QA/QC activities conducted by one or more sampling and quality control/data monitors (each a "Sampling and QC Monitor"). The Sampling and QC Monitors perform all field sampling activities, collect all field QC samples and perform all data recording and chain of custody activities in accordance with this Plan. Data generation at the contract analytical laboratory (the "Analytical Laboratory") utilized by the Mill to analyze the environmental samples is performed by or under an employee or agent (the "Analysis Monitor") of the Analytical Laboratory, in accordance with specific requirements of the Analytical Laboratory's own QA/QC program.

The responsibilities of the data generators are as follows:



### *Sampling and QC Monitors*

The Sampling and QC Monitors are responsible for field activities. These include:

- a. Ensuring that samples are collected, preserved, and transported as specified in Plan;
- b. Checking that all sample documentation (labels, field data worksheets, chain-of-custody records, packing lists) is correct and transmitting that information, along with the samples, to the Analytical Laboratory in accordance with this Plan;
- c. Maintaining records of all samples, tracking those samples through subsequent processing and analysis, and, ultimately, where applicable, appropriately disposing of those samples at the conclusion of the program;
- d. Preparing quality control samples for field sample collection during the sampling event;
- e. Preparing QC and sample data for review by the QA Manager; and
- f. Preparing QC and sample data for reporting and entry into a computer data base, where appropriate.

### *Analysis Monitor*

The Analysis Monitor is responsible for QA/QC activities at the Analytical Laboratory. These include:

- a. Training and qualifying personnel in specified Analytical Laboratory QC and analytical procedures, prior to receiving samples;
- b. Receiving samples from the field and verifying that incoming samples correspond to the packing list or chain-of-custody sheet; and
- c. Verifying that Analytical Laboratory QC and analytical procedures are being followed as specified in this Plan, by the Analytical Laboratory's QA/QC program, and in accordance with the requirements for maintaining National Environmental Laboratory Accreditation Program ("NELAP") and/or National Voluntary Laboratory Accreditation Program ("NAVLAP") certification.

### *Data Reviewers/Approvers*

The QA Manager has broad authority to approve or disapprove project plans, specific analyses and final reports. In general, the QA Manager is responsible for reviewing and advising on all aspects of QA/QC, including:

- a. Ensuring that the data produced by the data generators meet the specifications set out in this Plan;



- b. Making on-site evaluations and submitting audit samples to assist in reviewing QA/QC procedures;
- c. Determining (with the Sampling and QC Monitor and Analysis Monitor) appropriate sampling equipment and sample containers, in accordance with this Plan, to minimize contamination; and
- d. Supervising all QA/QC measures to assure proper adherence to this Plan and determining corrective measures to be taken when deviations from this Plan occur.

The QA Manager may delegate certain of these responsibilities to one or more Sampling and QC Monitors or to other qualified personnel.

## **1.5 Special Training and Certification**

All soil logging and soil descriptions will be overseen or conducted by a State of Utah Certified Professional Geologist (PG), using the ASTM Standard Practice for Description and Identification of Soils using the visual-manual procedure (Appendix A).

Site-specific training for all field personnel will be completed as required by Mill procedures and will be conducted by Mill personnel.

## **1.6 Documents and Records**

### **1.6.1 Field Documentation**

Field documentation will consist of, but not be limited to, detailed field note books, chain of custody forms, and digital photographs. In addition, the locations of Geoprobe borings and other field activities will be recorded using a hand held global positioning system (GPS) instrument. Soil logging and details from the boring such as sampling intervals and sample location will be recorded on a boring log (Appendix C). DRC requested the use of a boring log that matched WMMW-16. The log that will be used is located in Appendix C and contains the same relevant information fields. The bore log form does not include gamma or neutron logging fields, or well completion fields, since those elements are not part of this investigation.

### **1.6.2 Reports Generated**

Upon completion of the field work and laboratory analysis, a Report describing the results and results of the quality assurance/quality control checks will be generated and submitted to the DRC.



## 2.0 DATA GENERATION AND ACQUISITION

### 2.1 Sampling Design

As described in Section 1.1, the purpose of the nitrate investigation is to quantify nitrate and chloride in the alluvial soil column in selected locations at the Site with two goals:

1. The data generated by this investigation will be used to test the “new theory” hypothesis that the nitrate contamination source is or could be caused by naturally occurring nitrate and chloride salt deposits located in the vadose zone near or beneath the Mill site area, which have been mobilized by natural and/or artificial recharge (Walvoord, et al., 2003, Scanlon, et al., 2005 and others).
2. The data will be used to test hypotheses regarding to what extent Mill-related sources contributed, if at all, to the groundwater nitrate plume.
3. The data will be used to test hypotheses regarding to what extent present or historic non-Mill-related sources contributed, if at all, to the groundwater nitrate plume.

The nitrate investigation has been divided into three phases:

#### 1A. Geoprobe Investigation in Undisturbed Locations

This phase of the investigation is designed to quantify naturally occurring (i.e. “background”) nitrate and chloride concentrations in alluvial soil at the Site and explore for a naturally occurring nitrate reservoir. Background concentrations will be established advancing Geoprobe direct push machine (Geoprobe) borings at each of 20 locations spread across the Site, selected to represent areas which have not undergone irrigation or other forms of culturally-induced surface water recharge. These locations are based on interpretation of aerial photographic imagery, and are presented in Figure 1. Actual locations will be selected in the field by the field team leader in consultation with DUSA management and field personnel. This selection process is designed to maximize the opportunity of finding soil chemistry that reflects only natural cycles of wetting and drying from precipitation and evapotranspiration. The presence of such a nitrate and chloride reservoir would suggest that these concentrations could be present throughout the White Mesa alluvial soil column and could be mobilized to groundwater as the result of increased surface water recharge due to irrigation, surface water impoundment, canal leakage, or other recharge processes. Geoprobe locations and sample locations are summarized in Table 1. As was explained at previous meetings with DUSA and the DRC, there is no requirement for a statistically based sample location selection of Geoprobe locations. They were chosen based on availability of undisturbed sites as identified by historical aerial imagery.



*Part 1 - Geoprobe Investigation to Determine Background Nitrate, Chloride and Ammonia Soil Concentrations in Areas Unimpacted by Human Activities ("undisturbed locations"):*

1. A Geoprobe boring (Boring #1) will be conducted down to bedrock refusal at each of the 20 undisturbed Geoprobe boring locations (per Figure 20 of the February 18, 2011 DUSA Work Plan and Schedule). Three (3) samples will be collected from each Geoprobe core location. Soil core samples will be collected randomly from the first 1/3, second 1/3, and third 1/3 intervals of the total penetrated depth at each location. Each soil core sample will be sent to the analytical laboratory for analysis of nitrate (as N), chloride, and ammonia nitrogen (as N). Soil analysis will be conducted by an environmental laboratory currently certified by the State of Utah, using EPA approved sample and analysis methods. The Nitrate Extraction and Field Test Procedure, as described in Appendix A of the February 18, 2011 DUSA Work Plan (hereafter Nitrate Field Test) will not be used. Background concentrations for each of the above analytes will be based on the 95% UCL of the 60 samples collected and analyzed. Said UCL will be determined using commonly accepted descriptive statistical methods and will be subject to DRC approval. After DRC approval, said site soil "background" concentration will be used for comparative and determinative purposes in PHASE 1B of the Geoprobe Investigation of Potential Nitrate Source Locations described below.

*Part 2 - Geoprobe Investigation of a Possible Natural Nitrate Salt Reservoir:*

1. A surface soil sample (0 to 0.5 foot bgs) will be collected by shovel in close vicinity (within 5 horizontal feet) of each of the 20 Geoprobe boring locations from the Phase 1A, Part 1 work, described above. This surface soil sample will be analyzed using the Nitrate Field Test, described above, and will be used as a baseline sample for comparison with laboratory analysis results of the same depth. At DUSA's discretion, this surface sample can be collected instead from the duplicate Geoprobe boring, described below.
2. A duplicate Geoprobe boring (Boring #2) will be installed in close proximity (within 5 horizontal feet) to each of the a) Geoprobe borings (Boring #1) from Phase 1A, Part 1, above) and b) the surface soil samples in Phase 1A, Part 2, above. Each of these duplicate Geoprobe borings (Borings #2) will be driven down to bedrock refusal at each of the 20 undisturbed locations, mentioned in Phase 1A, Part 1, above. Said soil core samples will be collected at each Geoprobe location to
  - a. ensure a minimum core recovery of 95% Any sample volume required for isotopic analyses will be collected in subsequent phases.
  - b. provide discrete and individual 1-foot depth samples,



- c. be preserved and placed into a sealable plastic bag that is labeled in accordance with EPA approved field methods, and
- d. Each intervals sample will be thoroughly mixed and homogenized within the plastic bag before any field or laboratory analysis. Said soil samples will then be considered representative of the individual 1-foot core interval. Per agreement at previous meetings, DRC or DUSA may collect additional soil samples at additional intervals.

Thereafter, an aliquot of each discrete 1-foot soil sample will be taken from the bag and analyzed using the Nitrate Field Test described in Appendix A of the February 18, 2011 DUSA Work Plan and Schedule.

Elevated soil samples, i.e. those found with Nitrate Field Test results that are twice the baseline concentration, as determined by field analysis of the 0-0.5-foot bgs sample described above (Phase 1A, Part 2, Item 1) will be sent to the approved analytical laboratory for nitrate (as N) soil analysis. The Nitrate Field Test results from Boring #2 will be used as a tool by DUSA to examine the possibility of a natural nitrate salt deposit in vadose zone soils at the White Mesa Mill site using the 1-foot sample intervals.

All of the locations and samples will be documented as described above. If it is determined through discussions with DRC that isotope analyses should be performed on one or more samples from any boring location, the documentation of locations will be used to resample in the same location in a later phase of the investigation. All borings will be plugged with a mixture of bentonite and cement grout prior to abandonment.

### **1B. Geoprobe Investigation of Potential Nitrate Source Locations**

This phase of the investigation is designed to quantify nitrate and chloride concentrations in alluvial soil at the Site in locations where past or ongoing activities may have contributed nitrate and/or chloride to the soil and/or groundwater. .

The potential Mill sources, including leach field locations and dates of operation listed below are provided by DUSA management and are shown on Figure 2:

#### **Potential Nitrate Source Locations:**

1. Main leach field (also known as Leach Field east of Scalehouse, 1985 to present)
2. Sewage vault/lift station (currently active)



3. Scale house leach field, (also known as Leach Field south of Scalehouse, 1977-1979)
4. Former office leach field
5. Ammonia tanks
6. SAG leach field (Leach Field north of mill building, 1998 to 2009)
7. Cell 1 leach field (Leach Field east of Cell #1, up to 1985)
8. Fly ash pond
9. Sodium Chlorate Tanks (as a potential chloride source)
10. Ammonium sulfate crystal tanks
11. Lawzy sump
12. Lawzy Lake
13. Former vault/lift station (to Former Office Leach Field) (1992 to 2009)
14. Truck shop leach field (1979-1985)
15. New Counter Current Decant/Solvent Extraction (CCD/SX) leach field (currently active)
16. Historical Pond
17. Wildlife Pond
18. CCD
19. YC Precip Mini Lab
20. V2O5 Mini Lab & V2O5 Precip
21. SX Mini Lab
22. Chem Lab
23. Met Lab
24. V2O5 Oxidation Tanks

Note that locations 1, 2, and 15 are known to be in use at present. Sampling of these locations will be left for a later phase of the investigation.

The investigation of these potential sources is contingent on access with the Geoprobe rig and subject to approval by DUSA management, based primarily on field team health and safety considerations. The subsurface configuration or design of the leach fields, including the potential



for underground piping, is not known. Any excavation or borings in these leach fields will require prior identification of underground structures, such as piping, septic tanks, or vaults, using techniques such as air knife or equivalent "daylighting" methods. Design drawings and records will be reviewed prior to work and the borings will only be attempted with the full approval of DUSA management. A total of 48 Geoprobe borings will be advanced to bedrock refusal at 16 potential nitrate source locations on Site (Figure 2). Of these 16 potential source locations, eight are considered by DUSA as High Priority Locations and will be investigated with four Geoprobe borings in a north-east-west-south relative orientation bounding each location. DUSA considers the remaining eight potential source locations to be Low Priority Locations, each of which will be investigated with two Geoprobe borings. The location of these borings will be based on best professional judgment, considering the most likely water/waste/wastewater discharge direction.

For all of Phase 1B locations, three (3) core samples from each boring will be collected within from the bottom one-foot of each of the following intervals, based on the total depth of penetration at each site: first 1/3, second 1/3, and third 1/3. At a minimum, all 144 samples (three discrete depths sampled in each of the 32 High Priority Location borings and 16 Low Priority Location borings advanced in Phase 1B) shall be sent to an approved analytical laboratory for soil analysis. Each laboratory result will be compared to the statistically derived site background concentration, to determine if the source could have contributed significant nitrogen and/or chloride mass to cause, in part or in whole, groundwater concentrations found in nearby DUSA wells.

Soil core samples collected from each location will meet the standards of representativeness described for Phase 1A.

The Co-Executive Secretary reserves the right to collect soil samples for laboratory analysis, at DRC cost, from any one-foot core interval, of any boring.

Geoprobe investigation in the area of active leach fields may be conducted in a separate campaign, with a different field crew if determined necessary by DUSA in order to address potential health and safety issues.

All of the locations and samples will be documented as described above. If it is determined through discussions with DRC that isotope analyses should be performed on one or more samples from any boring location, the documentation of locations will be used to resample in the same location in a later phase of the investigation. All borings will be plugged with a mixture of bentonite and cement grout prior to abandonment.



## **1C. Geoprobe Investigation of Other Potential Nitrate Sources**

This phase of the investigation is designed to quantify nitrate and chloride concentrations in alluvial soil at the Site at seven additional locations identified by DUSA (Figure 3) for comparison to the background of naturally occurring concentrations established in Phase 1A. At each Geoprobe location, DUSA may choose to collect three (3) discrete one-foot core samples from the bottom one-foot of each of the following intervals: the first 1/3, second 1/3, and third 1/3 depth, based on the total depth of penetration at each site. DUSA may use the Nitrate Field Test to determine whether nitrate concentrations are detectable and if they are a sample will be sent to an approved analytical laboratory for analysis. For any boring where soil core samples will be sent for laboratory analysis, DUSA will ensure that the standards of representativeness described under Phase 1A are met. Any such soil laboratory results may then be compared to the site background soil concentrations.

All of the locations and samples will be documented as described above. If it is determined through discussions with DRC that isotope analyses should be performed on one or more samples from any boring location, the documentation of locations will be used to resample in the same location in a later phase of the investigation. All borings will be plugged with a mixture of bentonite and cement grout prior to abandonment.

## **2.2 Field Activities and Sampling Methods**

Geoprobe investigations will comply with the Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations ASTM D 6282-98 (Reapproved 2005) (ASTM, 2005). A copy of this guidance is included as Appendix D. Borings will be advanced with a track-mounted Geoprobe rig. The Phase 1 program plans on using a Geoprobe 6620DT direct push machine, or equivalent, using the DT22 Soil Sampling System, or equivalent. The Geoprobe advances a sampler by impacts from a hammer that delivers 35,000 lb of down force and is capable of a retraction force of 47,000 lb. If available, the Phase 1 program will use a dual-tube system that allows continuous 1.125-inch diameter cores to be recovered using five-foot sample barrels.

Dual-tube systems consist of an outer casing and an inner sampler that are advanced simultaneously into the soil. The sampler is removed from the borehole and a new sampler inserted for each five-foot increment of depth. Sample barrels are designed to create a minimum of sample disturbance while gathering high quality specimens.



The proposed system uses 2.25 in. (57 mm) OD probe rods as an outer casing and light-weight center rods for the inner rod string. A cutting shoe is threaded into the leading end of the rod string. When driven into the subsurface, the cutting shoe shears a 1.125 in. (29 mm) OD soil core which is collected inside the casing in a clear PVC liner. The light-weight center rods hold the liner in place while collecting the soil core, and also provide a means of retrieving the liner once the sample is collected. The 2.25 in. probe rods provided a cased hole through which to sample. The main advantage of sampling through a cased hole is that there is no side slough to contend with. In addition, the outer casing effectively seals the probe hole when sampling through perched water tables. These factors mean that sample cross contamination is eliminated.

Geoprobe boring locations for Phases 1A – 1C are shown on Figures 1 – 3, respectively. Locations are approximate and may be changed on judgment of the field team leader in consultation with DUSA personnel. The actual “as built” latitude and longitude of each boring will be measured in the field with a Garmin eTrex GPS instrument using the WGS 84 standard or equivalent. The GPS Wide Area Augmentation System used by the Garmin eTrex provides horizontal accuracy of  $\pm 3$  meters.

Geoprobe boring locations will be named following the convention GP-††X#, where GP stands for Geoprobe, †† is the two-digit number of the location (01, 02...18, 19, 20), X designates the phase of the investigation and # indicates Boring #1 or Boring #2, if applicable. For example, Geoprobe Boring #1 at location 01 in Phase 1A would be named GP-01A1; by contrast, the single boring advanced at location 07 in Phase 1C would be named GP-07C. Note that in this convention, no information is conveyed about the identity or compass orientation of potential nitrate sources in Phase 1B. This information will be noted in the field book by the field team leader.

The naming convention for Geoprobe boring samples is described in Section 2.3.1.

The Geoprobe boring samples will be collected using the following procedure:

1. Set up the Geoprobe in the pre-selected location using a map and GPS. Create a labeled GPS waypoint for the “as built” location. This will be Boring #1.
2. Concurrent with the setup of the Geoprobe at each location, collect a manual soil sample from 0 – 0.5 feet bgs and test for nitrate and chloride according to the field test procedures described below. The total sample volume should fill a one quart sealable plastic bag. This is the “background” or “baseline” sample for this location. This sample will be designated as GP-††-BKG.

3. Advance the Geoprobe sampler to refusal to determine subsurface soil conditions and the depth to the top of bedrock (Dakota Formation or Burro Canyon Formation).
4. Remove the sample barrel after each five-foot flight is advanced to obtain a continuous soil core from surface to total depth in one Geoprobe boring. Note that at least 95% of the soil in every core must be recovered. If this condition is not met, the Geoprobe will be relocated a short distance away and the procedure started over at Step 1.
5. Measure and mark depth in one-foot increments on the boring core sleeve. This is adequate sampling interval resolution to identify elevated nitrate or chloride concentrations on the order of six feet thick (Scanlon, 2005).
6. Open the sleeve to observe and describe the alluvial texture and/or lithology, taking photographs where appropriate. Describe or log the soil texture based on the USCS. Observations will be recorded on soil boring log forms (Appendix C). This task shall be performed by a Utah Licensed Professional Geologist.
7. Place the soil from each one-foot increment into a sealable plastic bag. Mix the soil thoroughly in the plastic bag by gently inverting the bag several times. The purpose of this procedure is to thoroughly blend the soil so that a sample aliquot from the bag will be representative of the entire one-foot interval. Seal the plastic bag, label and store for additional analysis in the event the interval contains elevated nitrate and/or chloride. If multiple soil lithologies are visible in a single one-foot core, the depth and USCS classification of all lithologies present will be noted per Step 6 above. However, by design, all one-foot cores will be homogenized, regardless of lithologic observations. This practice is intended to limit any bias or subjective judgment by field personnel. By the Nyquist Theorem, homogenization of samples at one-foot intervals allows vertical concentration variations at a scale of twice the sampling interval, or two feet.
8. If the Nitrate Field Test is required (see Section 2.1), perform Steps 8a – 8c:
  - a. Select a sample aliquot from the bag and test for nitrate using the nitrate field test kit test strips, as described in Section 2.5.1. Note that soil borings may be advanced by the Geoprobe at a rate that will not allow the Nitrate Field Test to be run simultaneously. As field operations do not depend on the results of the Nitrate Field Test, Steps 8 – 10 may be performed in a controlled setting on the Mill Site or elsewhere.
  - b. Record the test results in the field notebook.
  - c. If any of the soil column analyses indicate the presence of elevated nitrate, select the balance of that interval sample and place in a labeled laboratory approved

container for delivery to the analytical laboratory (per Section 2.3.5) for analysis of nitrate, chloride, sulfate and ammonia by the methods described in Section 2.5.2. “Elevated” concentrations are defined as those one-foot intervals with nitrate concentrations at least twice the average background concentration, based on field analysis of a sample from 0 – 0.5 feet bgs or as determined by field judgment.

9. If laboratory analytical samples are required (see Section 2.1), perform Steps 9a – 9c.
  - a. Identify the interval to be sampled. For samples collected from the basal one-foot interval of the first 1/3 ( $d_1$ ), second 1/3 ( $d_2$ ), and third 1/3 ( $d_3$ ) in a boring of total depth  $TD$ , the intervals are given below, expressed in feet bgs. Note that it is essential to include soil at the interface between alluvial soils and bedrock.

$$d_1 = \left[ \text{round}\left(\frac{TD}{3}\right) - 1 \text{ ft}, \text{round}\left(\frac{TD}{3}\right) \right]$$
$$d_2 = \left[ \text{round}\left(\frac{2TD}{3}\right) - 1 \text{ ft}, \text{round}\left(\frac{2TD}{3}\right) \right]$$
$$d_3 = [\text{round}(TD) - 1 \text{ ft}, TD]$$

- b. Place the soil from each one-foot increment into a sealable plastic bag and label. Mix the soil thoroughly in the plastic bag by gently inverting the bag several times. Once the sample is homogenized, place an aliquot of soil in a labeled laboratory approved container for delivery to the analytical laboratory (per Section 2.3.5) for analysis of nitrate, chloride, sulfate and ammonia by the methods described in Section 2.5.2.
10. Fill the boring with cement/bentonite grout.
11. Move to the next location.

## 2.3 Sample Handling and Custody

### 2.3.1 Sample Identification

Each sample collected at the Site during the nitrate investigation will be identified using a unique sample identification number (ID). The description of the sample type and the point name will be recorded on the chain-of-custody (COC) forms, as well as in the field notes.

Field log books will be used to document field sampling information. Sample IDs will be listed on the sample labels and the COC forms submitted to the laboratory, and will be cross-



referenced to the name in permanently bound field log books, on sample data sheets, and on COC forms.

Geoprobe boring samples will be named according to the boring location and bottom of the depth interval at which they were collected, following the convention GP-††X#-dd, where dd is the bottom of depth interval expressed in feet below ground surface. For example, the sample collected at GP-01A1 in the depth interval between 6 and 7 feet bgs would be named GP-01A1-7; similarly, the sample collected at GP-07C in the depth interval from 21 to 22 feet bgs would be named GP-07C-22.

### **2.3.2 Sample Labeling**

Resealable plastic bags will be labeled with an indelible marker with the:

- sample identification; and
- date.

Laboratory provided sample containers will be labeled with an adhesive label showing the:

- sample identification,
- date,
- the time of collection,
- the project name,
- sampler's initials, and
- analysis required.

Once the label is applied, it will be covered with clear plastic tape to prevent the label from being rendered illegible by water. Containers will be sealed in a plastic bag and placed on ice in a cooler.

### **2.3.3 Sample Documentation**

Documentation during sampling is essential to proper sample identification. All personnel will adhere to the following general guidelines for maintaining field documentation:

- Documentation will be completed in permanent black or blue ink.
- All entries will be legible.
- Errors will be corrected by crossing out the entry with a single line and then dating and initialing the lineout.



- Any serialized documents will be maintained by INTERA and referenced in the site log book.
- Unused portions of pages will be crossed out, and each page will be signed and dated.

The field team leader and sampling personnel are responsible for proper documentation of activities.

#### **2.3.4 Chain-of-Custody**

Standard sample custody procedures will be used to maintain and document sample integrity during collection, transportation, storage, and analysis. A sample will be considered to be in custody if one of the following statements applies:

- It is in a person's physical possession or view.
- It is in a secure area with restricted access.
- It is placed in a container and secured with an official seal in such a way that the sample cannot be reached without breaking the seal.

Chain-of-custody (COC) procedures provide an accurate written record that traces the possession of individual samples from the time of collection in the field to the time of acceptance at the laboratory. The COC form will also be used to document all samples collected and the analyses requested. Information that the field personnel will record on the COC form includes the following:

- Project name and number
- Sampling location
- Name and signature of sampler
- Destination of sample (laboratory name)
- Sample ID
- Date and time of collection
- Number and type of containers filled
- Analyses requested
- Preservatives used (if applicable)
- Filtering (if applicable)
- Signatures of individuals involved in custody transfer, including the date and time of transfer



- Air bill number (if applicable) or courier information
- Project contact and phone number

Unused lines on the COC form will be crossed out. Field personnel will sign COC forms. The COC form will be placed in a waterproof plastic bag and taped to the inside of the shipping container used to transport the samples. Signed air bills will serve as evidence of custody transfer between field personnel and the courier, and between the courier and the laboratory. Copies of the COC form and the air bill will be retained and filed by field personnel before the containers are shipped.

The laboratory sample custodian will receive all incoming samples, sign the accompanying COC forms, and retain copies of the forms as permanent record. The laboratory sample custodian will record all pertinent information concerning the samples, including the persons delivering the samples, the date and time received, sample condition at the time of receipt (e.g., sealed, unsealed, or broken container; temperature; or other relevant remarks), the sample IDs, and any unique laboratory identification numbers for the samples. When the sample transfer process is complete, the custodian is responsible for maintaining internal log books, tracking reports, and other records necessary to maintain custody throughout sample preparation and analysis.

The laboratory will provide a secure storage area for all samples. Access to this area will be restricted to authorized personnel. The custodian will ensure that samples requiring special handling, including samples that are heat- or light-sensitive, radioactive, or have other unusual physical characteristics, are properly stored and maintained pending analysis.

### **2.3.5 Sample Shipment**

The following procedures will be implemented when samples collected during the remediation activities are shipped:

- The cooler will be filled with bubble wrap, sample containers, and packing material. Sufficient packing material will be used to minimize sample container breakage during shipment.
- The COC forms will be placed inside a plastic bag. The bag will be sealed and taped to the inside of the cooler lid. The air bill, if required, will be filled out before the samples are handed over to the carrier. The laboratory will be notified if the sampler suspects that the sample contains any substance that would require laboratory personnel to take safety precautions.
- The cooler will be closed and taped shut with packing tape around both ends. If the cooler has a drain, it will be taped shut both inside and outside of the cooler.



- Signed and dated custody seals will be placed on the front and side of each cooler. Wide clear tape will be placed over the seals.
- The COC form will be transported within the taped, sealed cooler. When the cooler is received at the analytical laboratory, laboratory personnel will open the cooler and sign the COC form to document transfer of samples.
- Multiple coolers may be sent in one shipment to the laboratory. The outsides of the coolers will be marked to indicate the number of coolers in the shipment.

## 2.4 Sample Containers and Holding Times

All sample containers will be supplied by the laboratory and will be certified as new. The type of sample containers to be used for each analysis, the sample volumes required, the preservation requirements, and the holding times for samples prior to extraction and analysis are presented in Table 2.

## 2.5 Analytical Methods

### 2.5.1 Field Analytical Methods

Field analytical methods include the Nitrate Field Test and the Chloride Field Test. Both tests rely on procedures promulgated in the USDA *Soil Quality Test Kit Guide* (USDA, 2001) (Appendix E) using Hach Nitrate and Nitrite Test Strips and QuanTab Chloride Test Strips. The tests require the following equipment:

- 30-mL measuring scoop
  - 120-mL plastic containers with lid
  - Laboratory grade deionized water
  - Filter paper
  - Eye dropper
  - Stopwatch or timer
  - Nitrate/nitrite test strips
  - Chloride test strips
  - Soil data worksheets
1. Extract a soil subsample. Mix the soil thoroughly and measure a level, 30-mL scoop subsample and place it in the plastic container. Weigh the subsample and enter its weight on a soil data worksheet.



2. Add water to Subsample and mix. Add 30 mL of deionized water to the container with the subsample. The resulting mixture equates to a 1:1 soil to water ratio on a volume basis. Put the lid on the container and shake vigorously about 25 times.
3. Fold filter. Fold the filter paper in half and then fold it again, but not quite into a quarter-circle, leaving the edges a little uneven.
4. Insert Filter Paper into Subsample. Open the filter paper into the shape of a cone and push it (pointed part first) quickly in to the jar with the soil/water mixture until it touches the bottom of the jar. Wait until about an eye dropper-full of the solution has seeped through the inside of the filter paper.
5. Place drops on the nitrate/nitrite strips. Using the eye dropper and one nitrate/nitrite strip, place a drop on each of the nitrate/nitrite test strips pads (one for nitrate and the other for nitrite). Note the time.
6. Align the nitrate/nitrite test strip with the color chart on the side of the test strip bottle.
7. After 60 seconds, compare the color of the nitrate and nitrite pads to the corresponding scales to estimate the amount of nitrate and nitrite present based on the color change.
8. Repeat 5 – 7 for the chloride test strips.

Concentrations of each analyte can be estimated by assuming all of the analyte present in the soil sample has gone into solution and by assuming that in situ moisture content of the soil is negligible. Under these assumptions, the soil concentration of (for example) nitrate,  $C_{s-NO_3}$ , can be estimated by:

$$C_{s-NO_3} = \frac{C_{w-NO_3} V_w}{m_{s-dry}}$$

where  $C_{w-NO_3}$  is the aqueous nitrate concentration result from the test strip,  $V_w$  is the volume of water and  $m_{s-dry}$  is the dry mass of soil.

The range of the Nitrate Field Test is 0 – 50 mg/L, that of the Nitrite Field Test is 0 – 3 mg/L, and that of the Chloride Field Test is 30 – 600 mg/L. If reading from the field test exceeds the maximum limit, the test may be rerun by diluting the sample solution.

### 2.5.2 Laboratory Analytical Methods

All soil samples will be submitted to the analytical laboratory for SPLP using EPA Method 1312 using Extraction Fluid #3 (Appendix B). Method 1312 will produce a leachate of all soil



samples which will be analyzed for nitrate, chloride, sulfate and nitrogen as ammonia using EPA Method 353.2, EPA method 300.0, and EPA method 350.1 respectively.

## **2.6 Quality Control**

### **2.6.1 Field Quality Control Methods**

Field quality control measures include complete documentation of all field activities on the appropriate forms. Field QC samples include the collection of field duplicates for analysis by the analytical laboratory. Field duplicates will be collected at a frequency of 1 duplicate per 20 field samples. Duplicates will be collected by mixing the field sample and splitting the sample into 2 containers. The samples will be labeled as separate samples and submitted blind to the laboratory. Duplicate assessment will be completed as described in Section 2.6.3.4 below.

Duplicates for the field measurement of nitrate and chloride will be completed at a frequency of 1 duplicate per 20 samples and will also be assessed as discussed in Section 2.6.3.4 below.

### **2.6.2 Laboratory Quality Control Methods**

Analytical QA/QC will be governed by the QA/QC program of the Analytical Laboratory. Every effort will be made to use Analytical Laboratories that are certified by the State of Utah and by NELAP and/or NAVLAP, is capable of performing the analytical procedures specified in Table 2, and that the QA/QC program of the Analytical Laboratory includes the spikes, blanks and duplicates described below.

#### *Spikes, Blanks and Check Samples*

Analytical Laboratory QC samples will assess the accuracy and precision of the analyses. The following describes the type of QC samples that may be used by the Analytical Laboratory to assess the quality of the data. Analytical QC will be completed as required by the specific method used for analysis. Assessment of analytical laboratory QC samples will be as specified in the method.

#### **a. Matrix Spike/Matrix Spike Duplicate**

A spiked field sample analyzed in duplicate may be analyzed with every analytical batch. Analytes stipulated by the analytical method, by applicable regulations, or by other specific requirements may be spiked into the samples. Selection of the sample to be spiked depends on the information required and the variety of conditions within a typical matrix. The matrix spike sample serves as a check evaluating the effect of the sample matrix on the accuracy of analysis. The matrix spike duplicate serves as a check of the



analytical precision. Assessment of the matrix spike/matrix spike duplicate will be completed using the method and laboratory established limits.

b. Method Blanks

Each analytical batch shall be accompanied by a method blank. The method blank shall be carried through the entire analytical procedure. Contamination detected in analysis of method blanks will be used to evaluate any Analytical Laboratory contamination of environmental samples which may have occurred. Method blank detections will be assessed to determine if there is any affect on the sample data usability. Method blank affects will be discussed and a determination made on a case by case basis.

c. Check Samples

Each analytical batch shall contain a number of check samples. For each method, the Analytical Laboratory will analyze the check samples or their equivalents specified in the analytical method. Check samples may include: a laboratory control sample, calibration checks, laboratory fortified blanks, or sample duplicates. Check samples will be reviewed for compliance with the laboratory and method specified acceptance limits.

### **2.6.3 Internal QC Checks**

#### **2.6.3.1 Field QC Check Procedures**

The QA Manager will perform the QA/QC analysis of field procedures as described below.

#### **2.6.3.2 Review of Compliance with Procedures in This Plan**

Observation of technician performance is monitored by the QA Manager on a periodic basis to ensure compliance with this Plan.

#### **2.6.3.3 Completeness review**

The QA Manager will review all analytical results to confirm that the analytical results are complete (i.e., there is an analytical result for each required constituent). The completeness goal for this project is 95%.

#### **2.6.3.4 Duplicates**

The following analyses will be performed on duplicate field samples:

- a. Relative Percent Difference.



RPDs will be calculated in comparisons of duplicate and original field sample results. Non-conformance will exist when the  $RPD > 35\%$ , unless the measured concentrations are less than 5 times the required detection limit (EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, February 1994, 9240.1-05-01, p. 25).

#### **2.6.3.5 Use of QC Samples to Assess Conformance with This Plan**

QC samples generated during field activities and in the laboratory will be used to assess the usability of the data for meeting project objectives. QC data which do not meet the requirements specified herein may require that the associated sample data be flagged for limited use or be removed from the overall data pool. Data flagging will follow standard EPA guidelines specified in Functional Guidelines as applicable to the analytical method. QC samples will be used to determine if the data meet the project objectives.

### **2.7 Instrument and Equipment Testing, Inspection and Maintenance**

The Analytical Laboratory is responsible for the maintenance of its instruments in accordance with Analytical Laboratory procedures and as required in order to maintain its NELAP and/or NAVLAP certifications. Preventive maintenance will be performed on a scheduled basis to minimize downtime and the potential interruption of analytical work.

Sampling and field equipment shall be tested, inspected and maintained in accordance with manufacturers' recommendations.

### **2.8 Instrument Calibration**

A fundamental requirement for collection of valid data is the proper calibration of all sample collection and analytical instruments. Test kits will use the calibration methods set forth in the instruction manual provided with the test kits. Analytical Laboratory equipment shall be calibrated in accordance with Analytical Laboratory procedures and as described in the analytical methods.

## **3.0 DATA EVALUATION**

Data evaluation will be completed as described throughout this plan. Analytical data will be evaluated as described using laboratory generated QC samples as specified in the analytical methods. Field data will be evaluated against the specific QC samples generated in the field and documentation will be reviewed for completeness and accuracy.



The analytical results of Phase 1A will be used to develop a 95% UCL for the background levels of nitrate, chloride, sulfate and ammonia. Analytical results from Phases 1B and 1C will be compared to the UCL background levels to assess the likelihood of impacts from the source areas and additional locations, where concentrations significantly above background levels will be regarded as indicative of impact.

For the SPLP, Extraction Fluid #3 will be used (Appendix B). Standard extraction requires the addition of nitric acid and sulfuric acid during the leaching process. Since the leachates will be analyzed for nitrate+nitrite and sulfate, the deionized leaching process contemplated by the method (for cyanide containing samples) will be used in lieu of the standard leaching procedure.

As previously described, the soil samples are being leached and analyzed using water methodologies, which will yield concentrations in liquid units (such as mg/L). During the data interpretation and preparation of a revised CSM, the calculations and/or the relationship for converting the results to soil mass units will be provided.

Data usability will be assessed based on compliance with QC standards specified in the analytical method.

## **4.0 AUDITS**

DUSA may perform system and performance audits in order to ensure that data of known and defensible quality are produced during a sampling program. The frequency and timing of system and performance audits shall be as determined by DUSA.

### **4.1 System Audits**

System audits are qualitative evaluations of all components of field and Analytical Laboratory QC measurement systems. They determine if the measurement systems are being used appropriately. System audits will review field and Analytical Laboratory operations, including sampling equipment, laboratory equipment, sampling procedures, and equipment calibrations, to evaluate the effectiveness of the QA program and to identify any weakness that may exist. The audits may be carried out before all systems are operational, during the program, or after the completion of the program. Such audits typically involve a comparison of the activities required under this Plan with those actually scheduled or performed. A special type of systems audit is the data management audit. This audit addresses only data collection and management activities.



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## **4.2 Performance Audits**

The performance audit is a quantitative evaluation of the measurement systems of a program. It requires testing the measurement systems with samples of known composition or behavior to evaluate precision and accuracy. With respect to performance audits of the analytical process, either blind performance evaluation samples may be submitted to the Analytical Laboratory for analysis, or the auditor may request that it provide results of the blind studies that the Analytical Laboratory must provide to its NELAP and/or NAVLAP accreditation agency on an annual basis. The performance audit is carried out without the knowledge of the analysts, to the extent practicable.

## **4.3 Follow-Up Actions**

Response to the system audits and performance audits is required when deviations are found.

## **4.4 Audit Records**

Audit records for all audits conducted will be retained in DUSA Central Files. These records will contain audit reports, written records of completion for corrective actions, and any other documents associated with the audits supporting audit findings or corrective actions.



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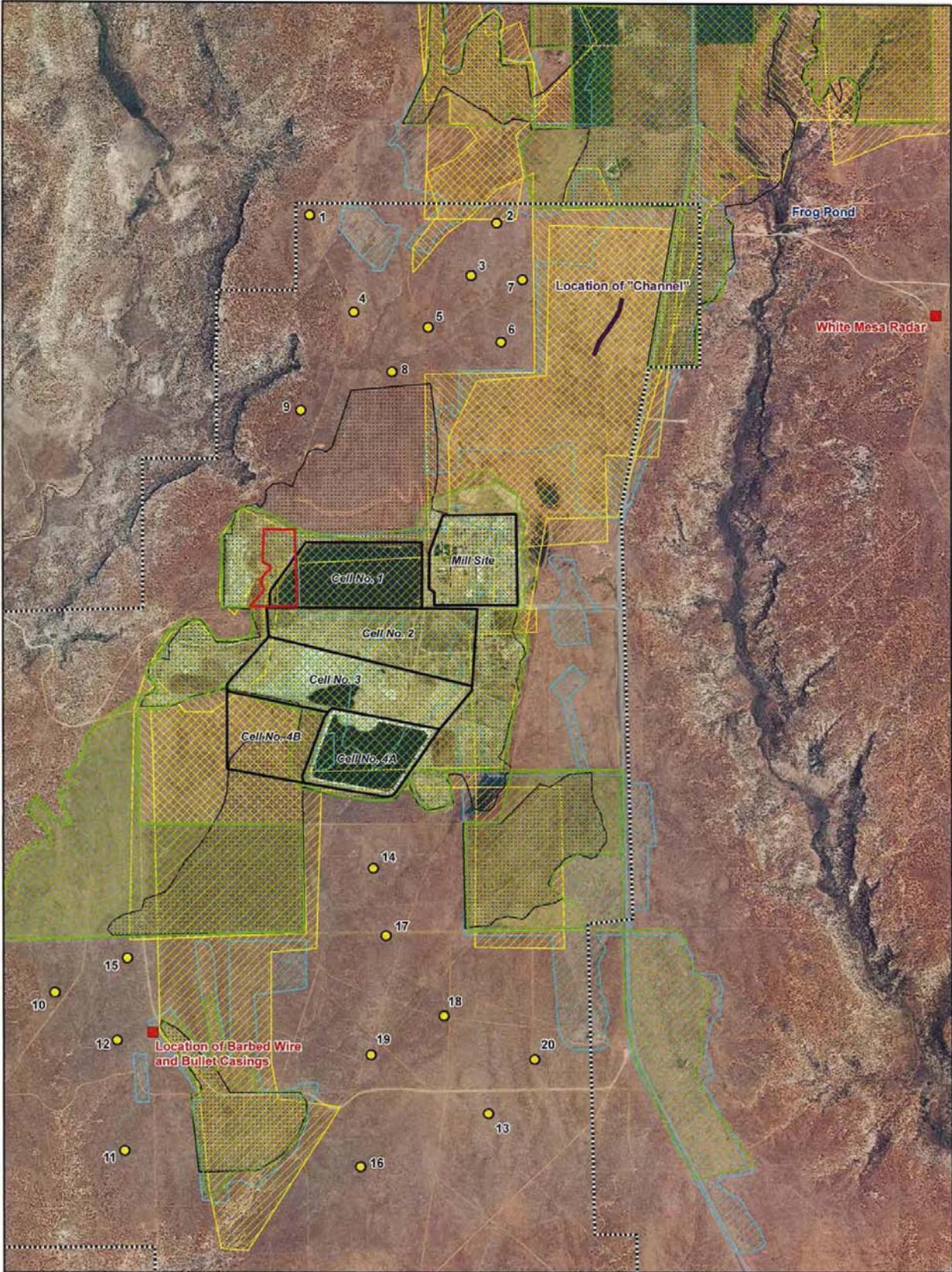
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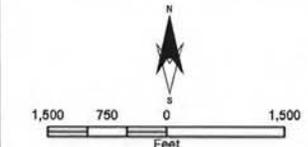
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## Figures



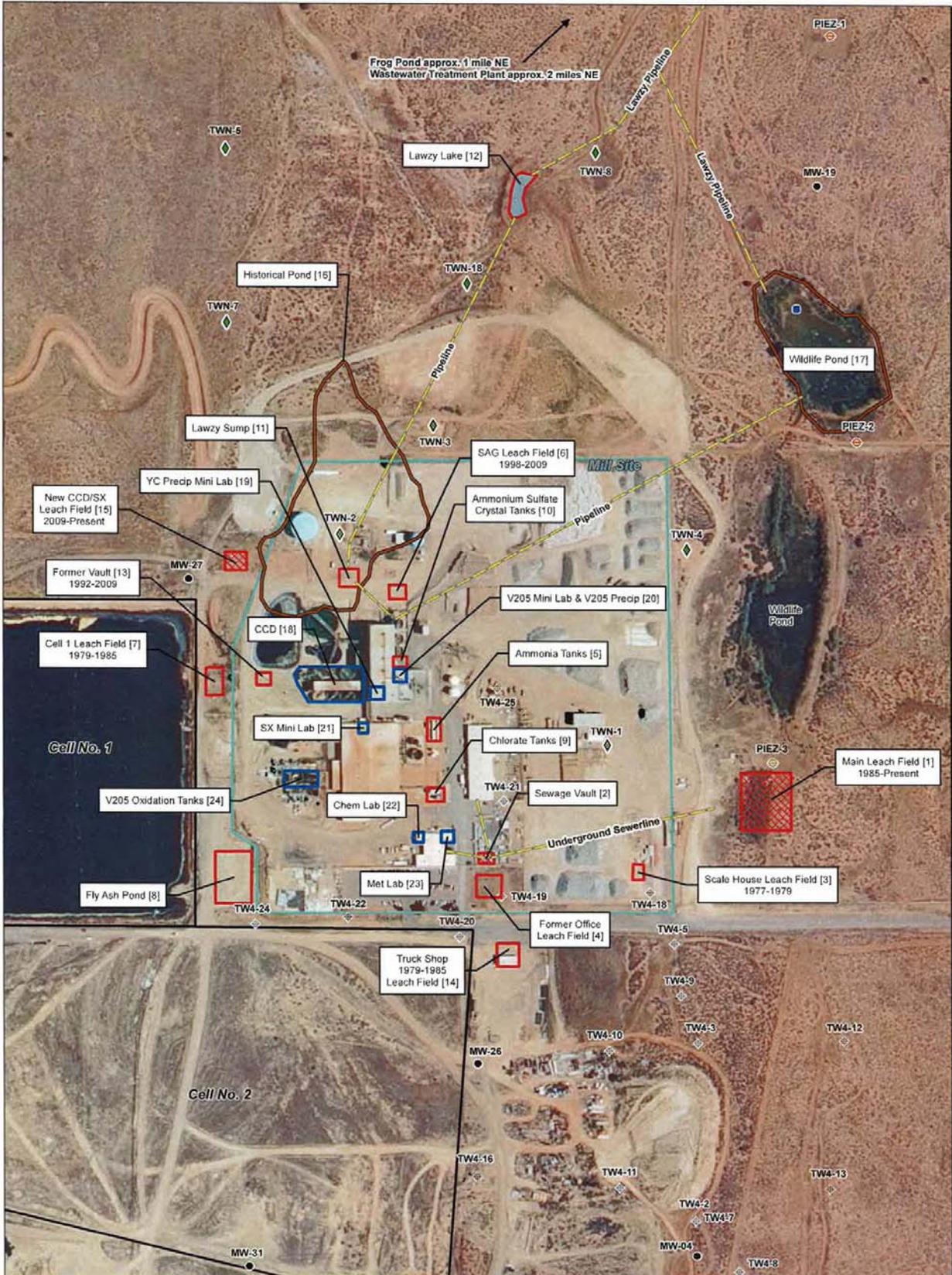
Source(s): Utah GIS Portal website, dated 2009;  
U.S. Geological Survey



Legend	
	Geoprobe Boring Location
	Pershing Missile Staging Area
	DUSA Property Boundary
	Historical Disturbance (From 2009 Imagery)
	Historical Disturbance (From 2006 Imagery)
	Historical Disturbance (From 1997 Imagery)
	Historical Disturbance (From 1984 Imagery)
	Historical Disturbance (From 1973 Imagery)
	Historical Disturbance (From 1955 Imagery)

**Figure 1**  
Phase 1A Natural Nitrate Reservoir  
and Mill Site Background  
Geoprobe Boring Locations  
Nitrate Investigation Revised  
Phase 1 Work Plan



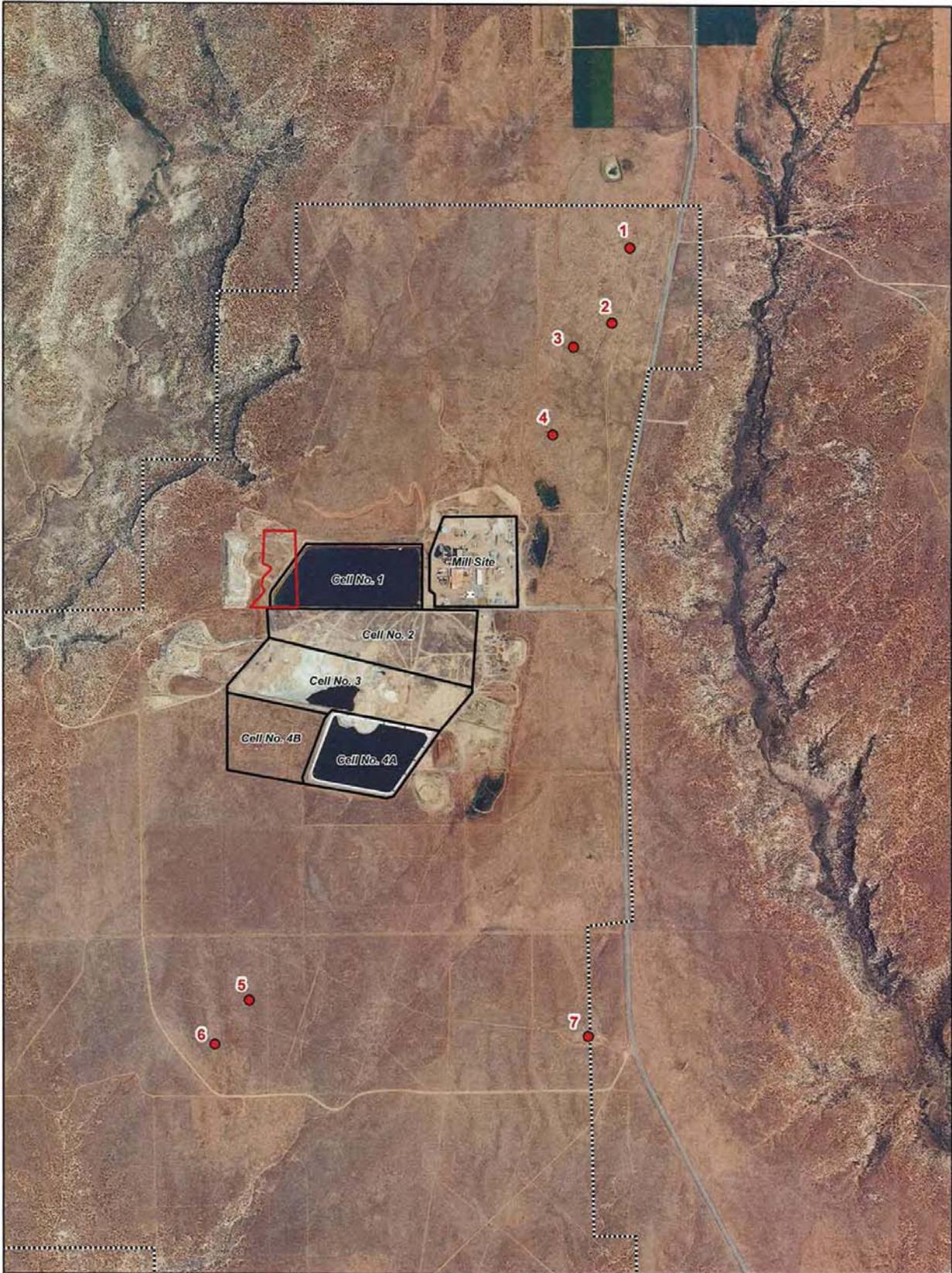


Source(s): Aerial – Utah GIS Portal website, dated 2009.  
Wells – HGC, Inc., May 2008 report.

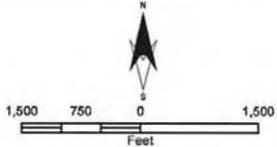
Legend	
	Leach Field (currently in operation) Sampling Method to be Determined
	Potential Mill Site Source – Geoprobe Boring
	Other Potential Nitrate and Chloride Source – Geoprobe Boring
	Inaccessible Potential Mill Site Source – No Geoprobe Boring
	Monitoring Well
	Piezometer
	Spring/Seep
	Surface Water
	Chloroform Monitoring Well
	Nitrate Monitoring Well

**Figure 2**  
Phase 1B Nitrate Source Areas:  
Geoprobe Boring and  
Core Drilling Locations  
Nitrate Investigation Revised  
Phase 1 Work Plan





Source(s): Utah GIS Portal website, dated 2009;  
U.S. Geological Survey



Legend	
<span style="color: red;">●</span>	Upgradient and Downgradient Potential Geoprobe Location
<span style="border: 2px solid red; display: inline-block; width: 15px; height: 10px;"></span>	Pershing Missile Staging Area
<span style="border-top: 1px dashed black; display: inline-block; width: 20px;"></span>	DUSA Property Boundary

**Figure 3**  
Phase 1C Potential Off-Site  
Sources Geoprobe Boring Locations  
Nitrate Investigation Revised  
Phase 1 Work Plan



## Tables

**Table 1: Geoprobe Boring and Sample Locations**

Soil Boring Location	Number of Borings	Number of Samples
<b>Natural Nitrate Reservoir and Background Locations</b>		
Undisturbed Borings 1-20	40	60
<b>On-Site Nitrate Source Area Locations</b>		
1) Main Leach Field	4	12
2) Sewage Vault	2	6
3) Scale House Leach Field	4	12
4) Former Office Leach Field	4	12
5) Ammonia Tanks	2	6
6) SAG Leach Field	4	12
7) Cell 1 Leach Field	2	6
8) Fly Ash Pond	2	6
9) Chlorate Tanks	0	0
10) Ammonium Sulfate Crystal Tanks	2	6
11) Lawzy Sump	4	12
12) Lawzy Lake	4	12
13) Former Vault	2	6
14) Truck Shop Leach Field	2	6
15) New CCD/SX Leach Field	2	6
16) Historical Pond	4	12
17) Wildlife Pond	4	12
18) CCD	0	0
19) YC Precip Mini Lab	0	0
20) V2O5 Mini Lab & V2O5 Precip	0	0
21) SX Mini Lab	0	0
22) Chem Lab	0	0
23) Met Lab	0	0
24) V2O5 Oxidation Tanks	0	0
<b>Potential Off-Site Source Locations</b>		
Borings 1-7	7	Test Strips <sup>a</sup>
<b>Total Borings/ # of Samples</b>	<b>95</b>	<b>204</b>

a= Potential Off-Site Source Location Samples will be field tested for the presence of nitrate and chloride using Hach test kits. If nitrate or chloride are detected in the soil, a sample will be collected and sent to the laboratory for analysis.

**Table 2: Laboratory Analytical Parameters by Task and Media**

Analyte	Analytical Method	Reporting Limit	Holding Time	Preservation	Temperature Requirement
SPLP Leachate	EPA 1312*	NA	28 days	None	Cool to ≤6°C
Nitrate+Nitrite	EPA 353.2	0.01 mg/L	28 days	H <sub>2</sub> SO <sub>4</sub> to a pH <2**	Cool to ≤6°C**
Chloride and Sulfate	EPA 300.0	Chloride – 0.1 mg/L Sulfate – 0.75 mg/L	28 days	None**	Cool to ≤6°C**
Ammonia as N	EPA 350.1	0.05 mg/L	28 days	H <sub>2</sub> SO <sub>4</sub> to a pH <2**	Cool to ≤6°C**

\* Extraction Fluid 3 will be used. Standard extraction requires the addition of nitric acid and sulfuric acid during the leaching process. Since the leachates will be analyzed for nitrate+nitrite and sulfate, the DI leaching process contemplated by the method (for cyanide containing samples) will be used in lieu of the standard leaching procedure.

\*\* Preservation and temperature requirements listed are for the leachates generated from the SPLP leaching procedure 1312.



**Appendix A**  
**Standard Practice for Description and Identification of Soils**



## Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)<sup>1</sup>

This standard is issued under the fixed designation D2488; 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 ( $\epsilon$ ) 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 covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (either intact or disturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.5 *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.* For specific precautionary statements see Section 8.

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:<sup>2</sup>

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1452 Practice for Soil Exploration and Sampling by Auger Borings

D1586 Test Method for Penetration Test (SPT) and Split-Barrel Sampling of Soils

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

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)

### 3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:

*Cobbles*—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved June 15, 2009. Published July 2009. Originally approved in 1966. Last previous edition approved in 2009 as D2488 – 09. DOI: 10.1520/D2488-09A.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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.

**Boulders**—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75- $\mu$ m) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the “A” line (see Fig. 3 of Test Method D2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

*coarse*—passes a 3-in. (75-mm) sieve and is retained on a 3/4-in. (19-mm) sieve.

*fine*—passes a 3/4-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75- $\mu$ m) sieve with the following subdivisions:

*coarse*—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

*medium*—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425- $\mu$ m) sieve.

*fine*—passes a No. 40 (425- $\mu$ m) sieve and is retained on a No. 200 (75- $\mu$ m) sieve.

3.1.7 *silt*—soil passing a No. 200 (75- $\mu$ m) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the “A” line (see Fig. 3 of Test Method D2487).

#### 4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

**NOTE 3**—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

*Dual Symbol*—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

*Borderline Symbol*—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

#### 5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

**NOTE 4**—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

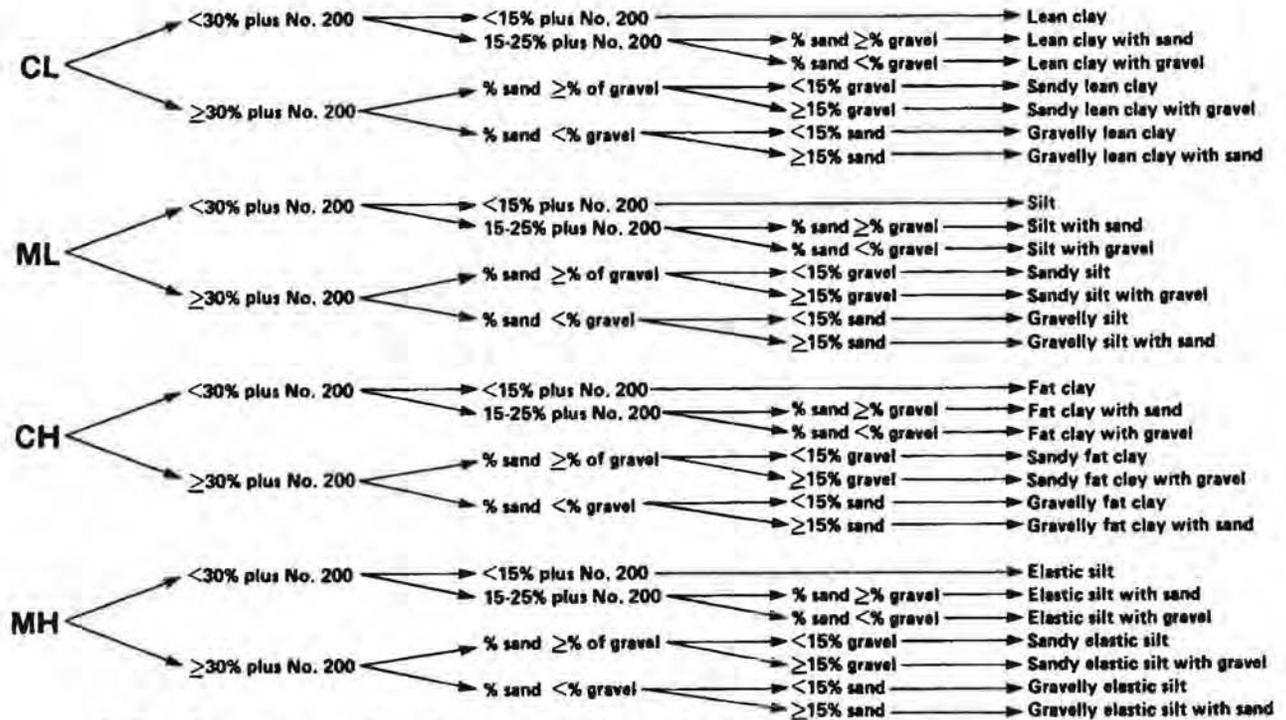
5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D4083 when working with frozen soils.

**NOTE 5**—Notwithstanding the statements on precision and bias contained in this standard: 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 that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D3740 provides a means for evaluating some of those factors.

**GROUP SYMBOL**

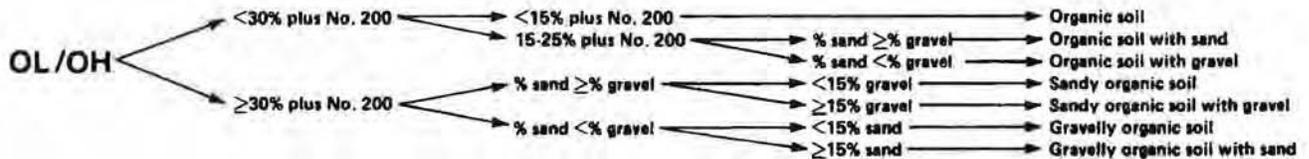
**GROUP NAME**



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.  
 FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)

**GROUP SYMBOL**

**GROUP NAME**



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

**6. Apparatus**

6.1 Required Apparatus:

6.1.1 Pocket Knife or Small Spatula.

6.2 Useful Auxiliary Apparatus:

6.2.1 Test Tube and Stopper (or jar with a lid).

6.2.2 Hand Lens.

**7. Reagents**

7.1 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 Hydrochloric Acid—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 N) to three parts water (This reagent is optional for use with this practice). See Section 8.

**8. Safety Precautions**

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 N) to three parts of distilled

water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 Caution—Do not add water to acid.

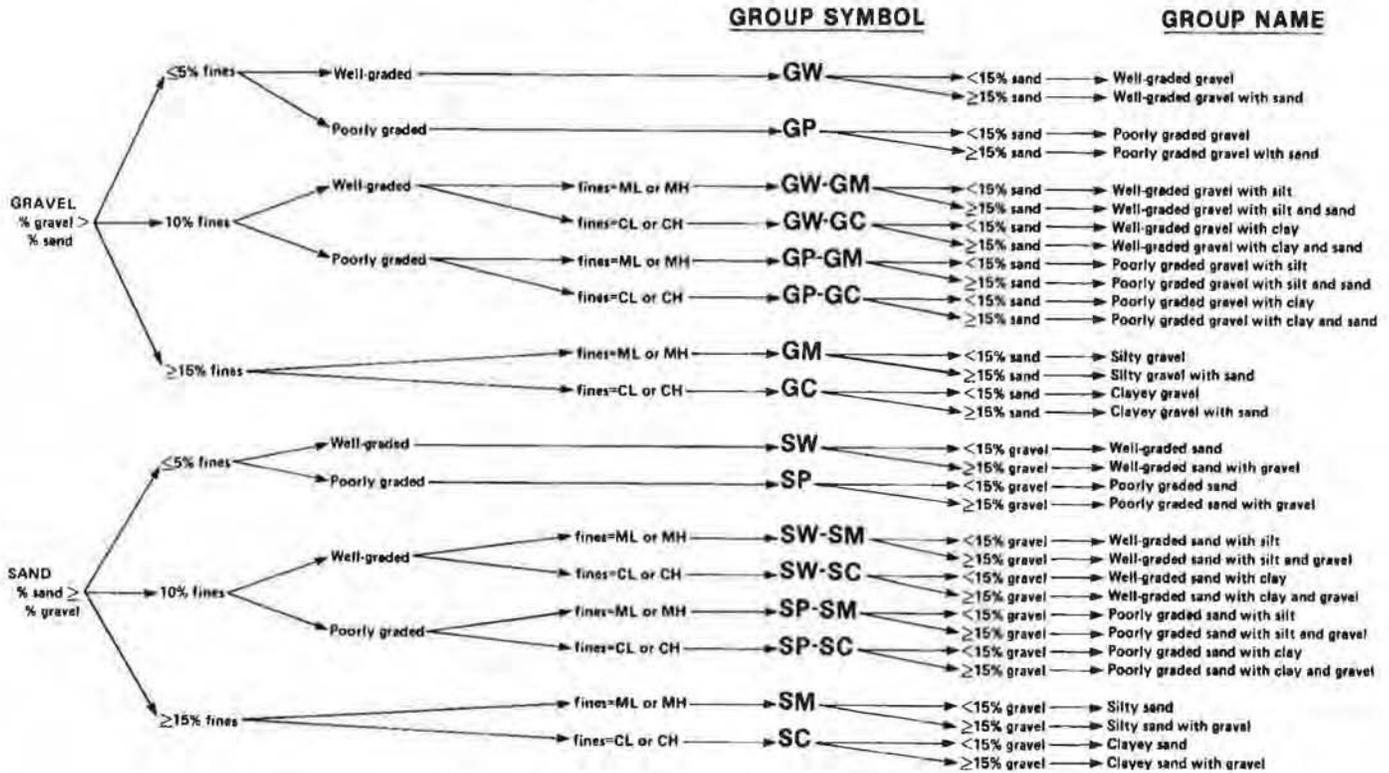
**9. Sampling**

9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D1452, D1587, or D2113, or Test Method D1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (¾ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 Angularity—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 Shape—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.3 Color—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

10.4 Odor—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 Moisture Condition—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 HCl Reaction—Describe the reaction with HCl as none, weak, or strong, in accordance with the criteria in Table



FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

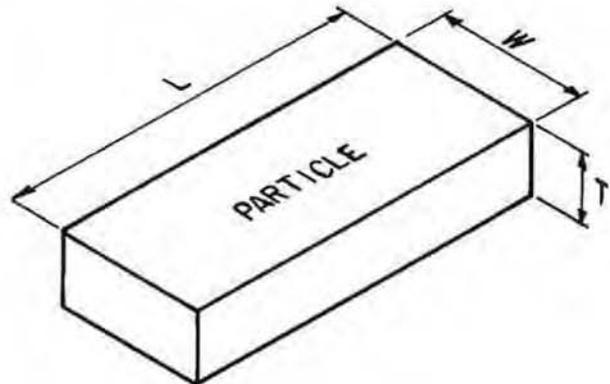
10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maxi-

## PARTICLE SHAPE

W = WIDTH  
T = THICKNESS  
L = LENGTH



FLAT:  $W/T > 3$   
ELONGATED:  $L/W > 3$   
FLAT AND ELONGATED:  
- meets both criteria

FIG. 4 Criteria for Particle Shape

**TABLE 3 Criteria for Describing Moisture Condition**

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

**TABLE 4 Criteria for Describing the Reaction With HCl**

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

**TABLE 5 Criteria for Describing Consistency**

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼ in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

**TABLE 6 Criteria for Describing Cementation**

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

**TABLE 7 Criteria for Describing Structure**

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

imum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. “Hard” means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

## 11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

## 12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

## 13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

## 14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

### 14.2 Dry Strength:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

**NOTE 11**—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accordance with the criteria in **Table 8**. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

### 14.3 Dilatancy:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in **Table 9**. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

### 14.4 Toughness:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose

**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

some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in **Table 10**.

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in **Table 11**.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

### 14.7 Identification of Inorganic Fine-Grained Soils:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see **Table 12**).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see **Table 12**).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see **Table 12**).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see **Table 12**).

**NOTE 12**—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

### 14.8 Identification of Organic Fine-Grained Soils:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and

**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

**TABLE 11 Criteria for Describing Plasticity**

Description	Criteria
Nonplastic	A 1/8-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 12 Identification of Inorganic Fine-Grained Soils from Manual Tests**

Soil Symbol	Dry Strength	Dilatancy	Toughness and Plasticity
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

may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

**NOTE 13**—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

### 15. Procedure for Identifying Coarse-Grained Soils (Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

## 16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

**NOTE 14**—Example: *Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

**NOTE 15**—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

**NOTE 16**—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

*Trace*—Particles are present but estimated to be less than 5 %

*Few*—5 to 10 %

*Little*—15 to 25 %

*Some*—30 to 45 %

*Mostly*—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

**TABLE 13 Checklist for Description of Soils**


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1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
  - Gravel—fine, coarse
  - Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

*For intact samples:*

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

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**17. Precision and Bias**

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

**18. Keywords**

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

**APPENDIXES**
**(Nonmandatory Information)**
**X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS**

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

*In-Place Conditions*—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft<sup>3</sup>; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

## **X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE**

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incorporated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as “Sandy Lean Clay (CL)”; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; “Poorly Graded Sand with Silt (SP-SM)”; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown.

X2.4.3 *Broken Shells*—About 60 % uniformly graded gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % nonplastic fines; “Poorly Graded Gravel with Silt and Sand (GP-GM).”

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; “Poorly Graded Gravel (GP)”; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

## **X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.**

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay  
ML/CL clayey silt  
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

#### X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

**X4.1 Jar Method**—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

**X4.2 Visual Method**—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present.

The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

**X4.3 Wash Test (for relative percentages of sand and fines)**—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

**X4.3.1** While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

#### X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

**X5.1** In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

**X5.2** This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

**X5.3** The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

*Prefix:*

s = sandy  
g = gravelly

*Suffix:*

s = with sand  
g = with gravel  
c = with cobbles  
b = with boulders

**X5.4** The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

<i>Group Symbol and Full Name</i>	<i>Abbreviated</i>
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

#### SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D2488 – 09) that may impact the use of this standard. (Approved June 15, 2009.)

*(I) Revised Section 1.2.3.*

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**Appendix B**  
**EPA Method 1312**

## METHOD 1312

### SYNTHETIC PRECIPITATION LEACHING PROCEDURE

#### 1.0 SCOPE AND APPLICATION

1.1 Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

#### 2.0 SUMMARY OF METHOD

2.1 For liquid samples (*i.e.*, those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter, is defined as the 1312 extract.

2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter.

2.3 If compatible (*i.e.*, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

#### 3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

#### 4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at  $30 \pm 2$  rpm. Suitable devices known to EPA are identified in Table 2.

##### 4.2 Extraction Vessels

4.2.1 Zero Headspace Extraction Vessel (ZHE). This device is for use only when the sample is being tested for the mobility of volatile analytes (*i.e.*, those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device and effectively precludes headspace. This type of vessel allows for initial liquid/solid

separation, extraction, and final extract filtration without opening the vessel (see Step 4.3.1). These vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110 mm filter. The devices contain VITON<sup>®</sup> O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for 1312 analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Step 7.3) refers to pounds-per-square-inch (psig), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.2.2 Bottle Extraction Vessel. When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extraction Vessel (ZHE): When the sample is evaluated for volatiles, the zero-headspace extraction vessel described

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<sup>1</sup>VITON<sup>®</sup> is a trademark of Du Pont.

in Step 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psig).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the sample is evaluated for other than volatile analytes, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psig or more. The type of filter holder used depends on the properties of the material to be filtered (see Step 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10 %) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are listed in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb sample components of interest. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8- $\mu\text{m}$ . Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with reagent water (a minimum of 1-L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to  $\pm 0.05$  units at 25°C.

4.6 ZHE Extract Collection Devices: TEDLAR<sup>®</sup> bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract when using the ZHE device. These devices listed are recommended for use under the following conditions:

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<sup>2</sup>TEDLAR<sup>®</sup> is a registered trademark of Du Pont.

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1 % of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1 % of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100 % solid) or has no significant solid phase (is <0.5% solid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas-tight syringe, pressure filtration unit (see Step 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within  $\pm$  0.01 grams may be used (all weight measurements are to be within  $\pm$  0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

## 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. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at  $90 \pm 5$  degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Sulfuric acid/nitric acid (60/40 weight percent mixture)  $H_2SO_4/HNO_3$ . Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute  $H_2SO_4/HNO_3$  acid mixture may be prepared and used in steps 5.4.1 and 5.4.2 making it easier to adjust the pH of the extraction fluids.

5.4 Extraction fluids.

5.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is  $4.20 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.

NOTE: Solutions are unbuffered and exact pH may not be attained.

5.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is  $5.00 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

5.4.3 Extraction fluid #3: This fluid is reagent water (Step 5.2) and is used to determine cyanide and volatiles leachability.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 There may be requirements on the minimal size of the field sample depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent

solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the sample is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).

6.6 1312 extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Step 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Step 8.0 (Quality Control) for acceptable sample and extract holding times.

## 7.0 PROCEDURE

### 7.1 Preliminary Evaluations

Perform preliminary 1312 evaluations on a minimum 100 gram aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include: (1) determination of the percent solids (Step 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Step 7.1.2); and (3) determination of whether the solid portion of the waste requires particle size reduction (Step 7.1.3).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a representative subsample (100 g minimum) and proceed to Step 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device

discussed in Step 4.3.2, and is outlined in Steps 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration.

NOTE: If sample material (>1 % of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.1.5 to determine the weight of the sample that will be filtered.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid, but even after applying vacuum or pressure filtration, as outlined in Step 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the sample by subtracting the weight of the liquid phase from the weight of the total sample, as determined in Step 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.1.9)}}{\text{Total weight of waste (Step 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Step 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Step 7.1.3 to determine whether the solid material requires particle size reduction or to Step 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Step 7.1.1.9 is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed, and to Step 7.3 with a fresh portion of the waste if the volatile 1312 analysis is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at  $100 \pm 20^\circ\text{C}$  until two successive weighings yield the same value within  $\pm 1\%$ . Record the final weight.

Caution: The drying oven should be vented to a hood or other appropriate device to eliminate the possibility of fumes from the sample escaping into the laboratory. Care should be taken to ensure that the sample will not flash or violently react upon heating.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Weight of dry sample + filter}) - \text{tared weight of filter}}{\text{Initial weight of sample (Step 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed, and to Step 7.3 if the volatile 1312 analysis is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile 1312 analysis is to be performed, return to the beginning of this Step (7.1) and, with a fresh portion of sample, determine whether particle size reduction is necessary (Step 7.1.3).

7.1.3 Determination of whether the sample requires particle-size reduction (particle-size is reduced during this step): Using the solid portion of the sample, evaluate the solid for particle size. Particle-size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm<sup>2</sup>, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Step 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid:

7.1.4.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

7.1.4.2 For wastes and wastewater, extraction fluid #1 should be used.

7.1.4.3 For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.1.5 If the aliquot of the sample used for the preliminary evaluation (Steps 7.1.1 - 7.1.4) was determined to be 100% solid at Step 7.1.1.1, then it can be used for the Step 7.2 extraction (assuming at least 100 grams remain), and the Step 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Step 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Step 7.3. The aliquot of the waste subjected to the procedure in Step 7.1.1.7 might be appropriate for use for the Step 7.2 extraction if an adequate amount of solid (as determined by Step 7.1.1.9)

was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 7.2.10 of the nonvolatile 1312 extraction.

## 7.2 Procedure When Volatiles Are Not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Step 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of 1312 extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single 1312 extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100 % solid, see Step 7.1.1), weigh out a subsample of the sample (100 gram minimum) and proceed to Step 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Step 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the sample (100 gram minimum) and record the weight. If the waste contains <0.5 % dry solids (Step 7.1.2), the liquid portion of the waste, after filtration, is defined as the 1312 extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the 1312 extract. For wastes containing >0.5 % dry solids (Steps 7.1.1 or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the 1312 extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the sample is centrifuged, the liquid should be decanted and filtered followed by

filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the sample (liquid and solid phases) to the filter holder (see Step 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psig (*i.e.*, filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

NOTE: If waste material (>1 % of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.2.5, to determine the weight of the waste sample that will be filtered.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see Step 7.2.12) or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the sample contains <0.5% dry solids (see Step 7.1.2), proceed to Step 7.2.13. If the sample contains >0.5 % dry solids (see Step 7.1.1 or 7.1.2), and if particle-size reduction of the solid was needed in Step 7.1.3, proceed to Step 7.2.10. If the sample as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Step 7.2.11.

7.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Step 7.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \% \text{ solids (Step 7.1.1)} \times \text{weight of waste filtered (Step 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Step 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at  $30 \pm 2$  rpm for  $18 \pm 2$  hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at  $23 \pm 2^\circ\text{C}$  during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the  $18 \pm 2$  hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 7.2.7. For final filtration of the 1312 extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the 1312 extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.2.12 is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 7.2.12 with the initial liquid phase of the sample obtained

in Step 7.2.7. This combined liquid is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Step 7.2.7, is not or may not be compatible with the filtered liquid resulting from Step 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the 1312 extract, and combine the results mathematically, as described in Step 7.2.14.

7.2.14 Following collection of the 1312 extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The 1312 extract shall be prepared and analyzed according to appropriate analytical methods. 1312 extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5 %), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

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

where:

$V_1$  = The volume of the first phase (L).

$C_1$  = The concentration of the analyte of concern in the first phase (mg/L).

$V_2$  = The volume of the second phase (L).

$C_2$  = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

### 7.3 Procedure When Volatiles Are Involved

Use the ZHE device to obtain 1312 extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psig), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (see Step 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Step 4.6 are recommended for use under the conditions stated in Steps 4.6.1-4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Step 7.3, Step 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the sample is 100% solid (see Step 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Step 7.3.5.

7.3.4 If the sample contains <0.5% dry solids (Step 7.1.2), the liquid portion of waste, after filtration, is defined as the 1312 extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For samples containing  $\geq 0.5\%$  dry solids (Steps 7.1.1 and/or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For samples containing <5% solids (see Step 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing >5% solids (see Step 7.1.1), determine the amount of waste to charge into the ZHE as follows:

Weight of waste to charge ZHE =  $\frac{25}{\text{percent solids (Step 7.1.1)}} \times 100$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle-size reduction of the solid portion of the sample was required in Step 7.1.3, proceed to Step 7.3.6. If particle-size reduction was not required in Step 7.1.3, proceed to Step 7.3.7.

7.3.6 Prepare the sample for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Step 7.1.3.1. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle-size reduction. The means used to effect particle-size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle-size has been appropriately altered, proceed to Step 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge samples prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extraction collection device to the top plate.

Note: If sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psig (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the

amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100 % solid (see Step 7.1.1), slowly increase the pressure to a maximum of 50 psig to force most of the headspace out of the device and proceed to Step 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psig to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When liquid flow has ceased such that continued pressure filtration at 50 psig does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the sample and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the 1312 extraction as a solid.

If the original waste contained <0.5 % dry solids (see Step 7.1.2), this filtrate is defined as the 1312 extract and is analyzed directly. Proceed to Step 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (see Steps 7.3.13 through 7.3.15) or stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #3 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \% \text{ solids (Step 7.1.1)} \times \text{weight of waste filtered (Step 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #3 is used in all cases (see Step 5.4.3).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psig (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psig and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate at  $30 \pm 2$  rpm for  $18 \pm 2$  hours. Ambient temperature (*i.e.*, temperature of room in which extraction occurs) shall be maintained at  $23 \pm 2^\circ\text{C}$  during agitation.

7.3.13 Following the  $18 \pm 2$  hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (*i.e.*, no gas release observed), the ZHE is leaking. Check the ZHE for leaking as specified in Step 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (*i.e.*, TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Step 7.3.9. All extracts shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Steps 4.6 and 7.3.1).

**NOTE:** An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured

7.3.14 If the original sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.3.13 is defined as the 1312 extract. If the sample contained an initial liquid phase, the filtered liquid material obtained from Step 7.3.13 and the initial liquid phase (Step 7.3.9) are collectively defined as the 1312 extract.

7.3.15 Following collection of the 1312 extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed. Analyze the 1312 extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (*i.e.*, are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

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

where:

$V_1$  = The volume of the first phases (L).

$C_1$  = The concentration of the analyte of concern in the first phase (mg/L).

$V_2$  = The volume of the second phase (L).

$C_2$  = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Step 8.0 for quality assurance requirements.

## 8.0 QUALITY CONTROL

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) for every 20 extractions that have been conducted in an extraction vessel. Refer to Chapter One for additional quality control protocols.

8.2 A matrix spike shall be performed for each waste type (*e.g.*, wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the 1312 extract and before preservation. Matrix spikes should not be added prior to 1312 extraction of the sample.

8.2.2 In most cases, matrix spike levels should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the

spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of 1312 extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the 1312 extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{ Recovery}) = 100 (X_s - X_u) / K$$

where:

$X_s$  = measured value for the spiked sample  
 $X_u$  = measured value for the unspiked sample, and  
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the 1312 extract is not at least 50% and the concentration does not exceed the appropriate regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standards as the independent variable (x-axis). Solve for the intercept

of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for the unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo 1312 extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES (days)

	From: Field Collection  To: 1312 extrac- tion	From: 1312 extrac- tion  To: Prepara- tive extrac- tion	From: Prepara- tive extrac- tion  To: Determi- native analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi- volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360
NA = Not Applicable				

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

## 9.0 METHOD PERFORMANCE

9.1 Precision results for semi-volatiles and metals: An eastern soil with high organic content and a western soil with low organic content were used for the semi-volatile and metal leaching experiments. Both types of soil were analyzed prior to contaminant spiking. The results are shown in Table 6. The concentration of contaminants leached from the soils were reproducible, as shown

by the moderate relative standard deviations (RSDs) of the recoveries (averaging 29% for the compounds and elements analyzed).

9.2 Precision results for volatiles: Four different soils were spiked and tested for the extraction of volatiles. Soils One and Two were from western and eastern Superfund sites. Soils Three and Four were mixtures of a western soil with low organic content and two different municipal sludges. The results are shown in Table 7. Extract concentrations of volatile organics from the eastern soil were lower than from the western soil. Replicate leachings of Soils Three and Four showed lower precision than the leachates from the Superfund soils.

#### 10.0 REFERENCES

1. Environmental Monitoring Systems Laboratory, "Performance Testing of Method 1312; QA Support for RCRA Testing: Project Report". EPA/600/4-89/022. EPA Contract 68-03-3249 to Lockheed Engineering and Sciences Company, June 1989.
2. Research Triangle Institute, "Interlaboratory Comparison of Methods 1310, 1311, and 1312 for Lead in Soil". U.S. EPA Contract 68-01-7075, November 1988.

Table 1. Volatile Analytes<sup>1</sup>

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

<sup>1</sup> When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

Table 2. Suitable Rotary Agitation Apparatus<sup>1</sup>

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S); 8-vessel extractor (DC20); 12-vessel extractor (DC20B)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2); 4-vessel (3740-4); 6-vessel (3740-6); 8-vessel (3740-8); 12-vessel (3740-12); 24-vessel (3740-24)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 1-liter bottle extractor (YT300RAHW)

<sup>1</sup> Any device that rotates the extraction vessel in an end-over-end fashion at 30 ±2 rpm is acceptable.

Table 3. Suitable Zero-Headspace Extractor Vessels<sup>1</sup>

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing <sup>2</sup>	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device

<sup>1</sup> Any device that meets the specifications listed in Step 4.2.1 of the method is suitable.

<sup>2</sup> This device uses a 110 mm filter.

Table 4. Suitable Filter Holders<sup>1</sup>

Company	Location	Model/ Catalogue #	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

<sup>1</sup> Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5. Suitable Filter Media<sup>1</sup>

Company	Location	Model	Pore Size ( $\mu$ m)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7

<sup>1</sup> Any filter that meets the specifications in Step 4.4 of the Method is suitable.

TABLE 6 - METHOD 1312 PRECISION RESULTS FOR SEMI-VOLATILES AND METALS

	<u>Eastern Soil (pH 4.2)</u>			<u>Western Soil (pH 5.0)</u>	
	<u>Amount Spiked</u> (µg)	<u>Amount Recovered*</u> (µg)	<u>% RSD</u>	<u>Amount Recovered*</u> (µg)	<u>% RSD</u>
<u>FORTIFIED ANALYTES</u>					
bis(2-chloroethyl)- ether	1040	834	12.5	616	14.2
2-Chlorophenol	1620	1010	6.8	525	54.9
1,4-Dichlorobenzene	2000	344	12.3	272	34.6
1,2-Dichlorobenzene	8920	1010	8.0	1520	28.4
2-Methylphenol	3940	1860	7.7	1130	32.6
Nitrobenzene	1010	812	10.0	457	21.3
2,4-Dimethylphenol	1460	200	18.4	18	87.6
Hexachlorobutadiene	6300	95	12.9	280	22.8
Acenaphthene	3640	210	8.1	310**	7.7
2,4-Dinitrophenol	1300	896**	6.1	23**	15.7
2,4-Dinitrotoluene	1900	1150	5.4	585	54.4
Hexachlorobenzene	1840	3.7	12.0	10	173.2
gamma BHC (Lindane)	7440	230	16.3	1240	55.2
beta BHC	640	35	13.3	65.3	51.7
<u>METALS</u>					
Lead	5000	70	4.3	10	51.7
Cadmium	1000	387	2.3	91	71.3

\* = Triplicate analyses.

\*\* = Duplicate analyses; one value was rejected as an outlier at the 90% confidence level using the Dixon Q test.

TABLE 7 - METHOD 1312 PRECISION RESULTS FOR VOLATILES

Compound Name	Soil No. 1		Soil No. 2		Soil No. 3		Soil No. 4	
	(Western)		(Eastern)		(Western and Sludge)		(Western and Sludge)	
	Avg. %Rec.*	%RSD	Avg. %Rec.*	%RSD	Avg. %Rec.**	%RSD	Avg. %Rec.***	%RSD
Acetone	44.0	12.4	43.8	2.25	116.0	11.5	21.3	71.4
Acrylonitrile	52.5	68.4	50.5	70.0	49.3	44.9	51.8	4.6
Benzene	47.8	8.29	34.8	16.3	49.8	36.7	33.4	41.1
n-Butyl Alcohol (1-Butanol)	55.5	2.91	49.2	14.6	65.5	37.2	73.0	13.9
Carbon disulfide	21.4	16.4	12.9	49.5	36.5	51.5	21.3	31.5
Carbon tetrachloride	40.6	18.6	22.3	29.1	36.2	41.4	24.0	34.0
Chlorobenzene	64.4	6.76	41.5	13.1	44.2	32.0	33.0	24.9
Chloroform	61.3	8.04	54.8	16.4	61.8	29.1	45.8	38.6
1,2-Dichloroethane	73.4	4.59	68.7	11.3	58.3	33.3	41.2	37.8
1,1-Dichloroethane	31.4	14.5	22.9	39.3	32.0	54.4	16.8	26.4
Ethyl acetate	76.4	9.65	75.4	4.02	23.0	119.8	11.0	115.5
Ethylbenzene	56.2	9.22	23.2	11.5	37.5	36.1	27.2	28.6
Ethyl ether	48.0	16.4	55.1	9.72	37.3	31.2	42.0	17.6
Isobutanol (4-Methyl -1-propanol)	0.0	ND	0.0	ND	61.8	37.7	76.0	12.2
Methylene chloride	47.5	30.3	42.2	42.9	52.0	37.4	37.3	16.6
Methyl ethyl ketone (2-Butanone)	56.7	5.94	61.9	3.94	73.7	31.3	40.6	39.0
Methyl isobutyl ketone	81.1	10.3	88.9	2.99	58.3	32.6	39.8	40.3
1,1,1,2-Tetrachloro- ethane	69.0	6.73	41.1	11.3	50.8	31.5	36.8	23.8
1,1,2,2-Tetrachloro- ethane	85.3	7.04	58.9	4.15	64.0	25.7	53.6	15.8
Tetrachloroethene	45.1	12.7	15.2	17.4	26.2	44.0	18.6	24.2
Toluene	59.2	8.06	49.3	10.5	45.7	35.2	31.4	37.2
1,1,1-Trichloro- ethane	47.2	16.0	33.8	22.8	40.7	40.6	26.2	38.8
1,1,2-Trichloro- ethane	76.2	5.72	67.3	8.43	61.7	28.0	46.4	25.4
Trichloroethene	54.5	11.1	39.4	19.5	38.8	40.9	25.6	34.1
Trichloro- fluoromethane	20.7	24.5	12.6	60.1	28.5	34.0	19.8	33.9
1,1,2-Trichloro- trifluoroethane	18.1	26.7	6.95	58.0	21.5	67.8	15.3	24.8
Vinyl chloride	10.2	20.3	7.17	72.8	25.0	61.0	11.8	25.4

\* Triplicate analyses

\*\* Six replicate analyses

\*\*\* Five replicate analyses

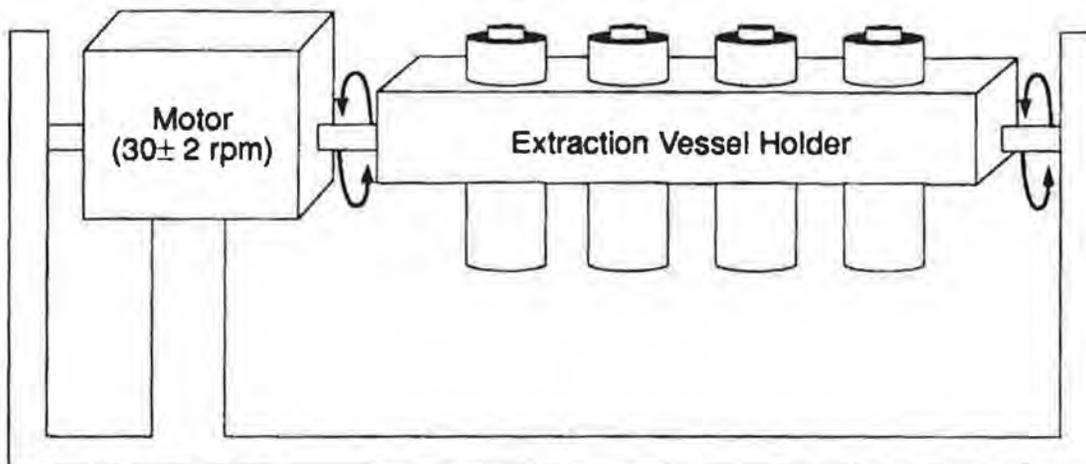


Figure 1. Rotary Agitation Apparatus

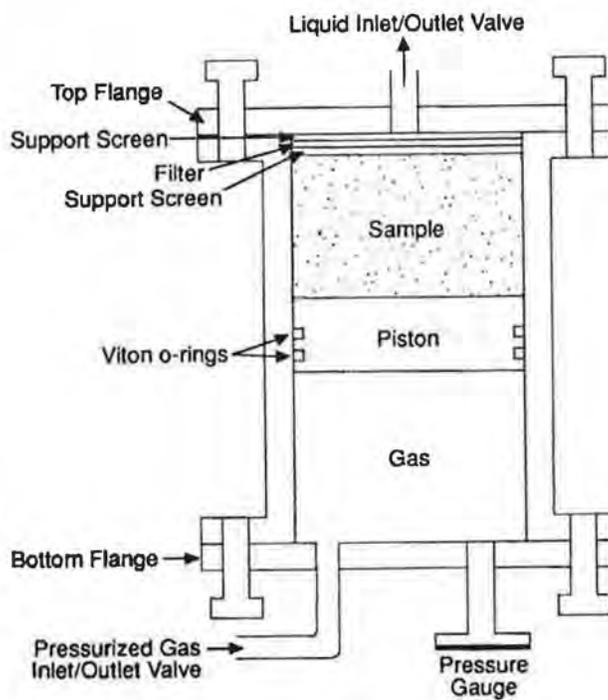
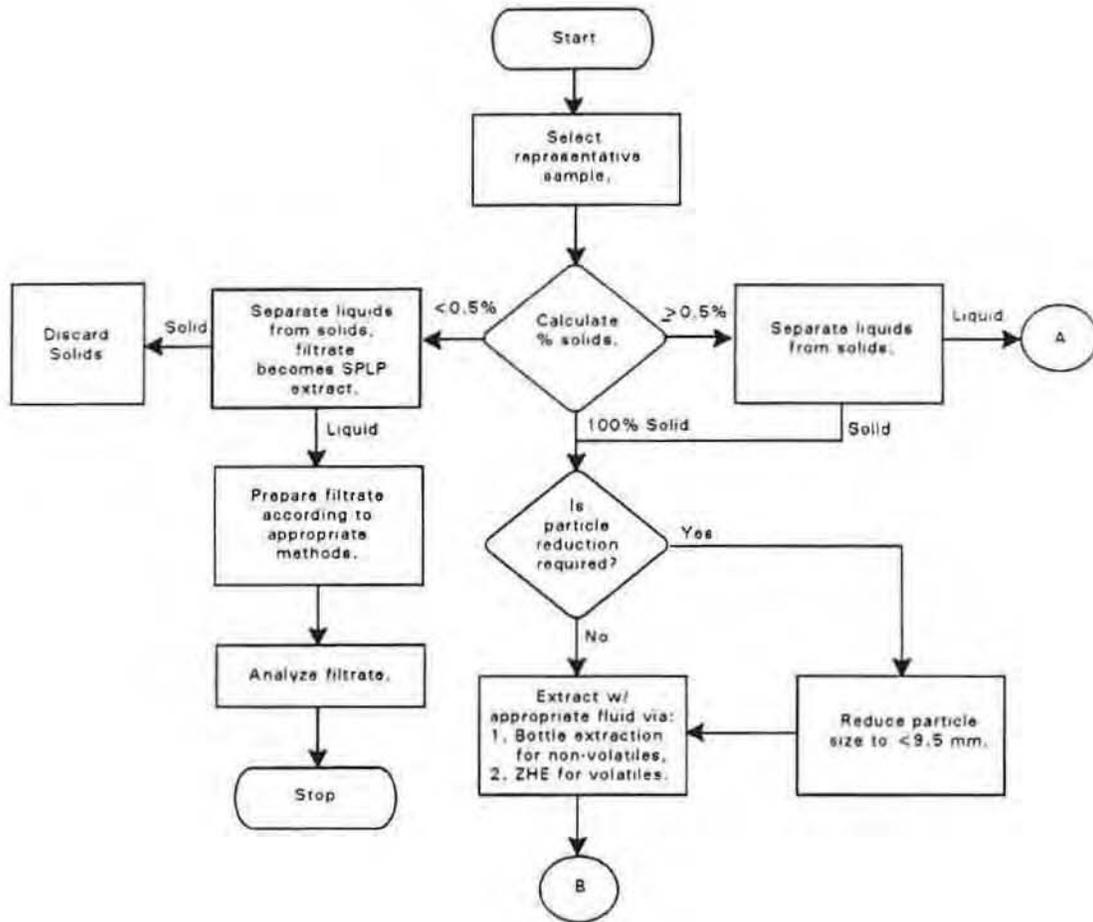


Figure 2. Zero-Headspace Extractor (ZHE)

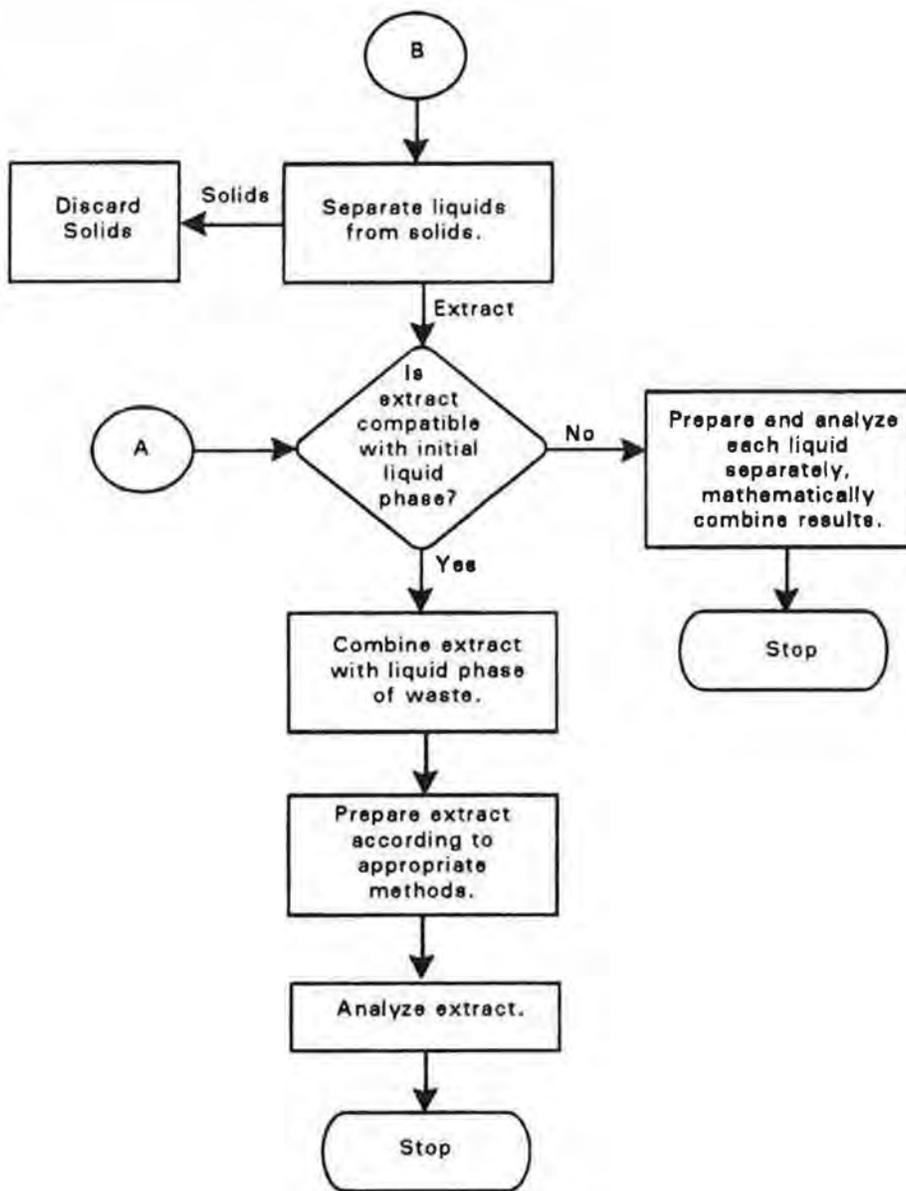
METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE



METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (continued)



**Appendix C**  
**Soil Boring Log Form**



**Appendix D**  
**Standard Guide for Direct Push Soil Sampling for Environmental Site  
Characterizations**



Designation: D 6282 – 98 (Reapproved 2005)

## Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations<sup>1</sup>

This standard is issued under the fixed designation D 6282; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This guide addresses direct push soil samplers, which also may be driven into the ground from the surface or through prebored holes. The samplers can be continuous or discrete interval units. Samplers are advanced by a combination of static push, or impacts from hammers, or vibratory methods, or a combination thereof, to the depth of interest. The guide does not cover open chambered samplers operated by hand such as augers, agricultural samplers operated at shallow depths, or side wall samplers. This guide does not address single sampling events in the immediate base of the drill hole using rotary drilling equipment with incremental drill hole excavation. Other sampling standards, such as Test Methods **D 1586** and **D 1587** and Practice **D 3550** apply to rotary drilling activities. This guide does not address advancement of sampler barrel systems with methods that employ cuttings removal as the sampler is advanced. Other drilling and sampling methods may apply for samples needed for engineering and construction applications.

1.2 Guidance on preservation and transport of samples, as given in Guide **D 4220**, may or may not apply. Samples for chemical analysis often must be subsampled and preserved for chemical analysis using special techniques. Practice **D 3694** provides information on some of the special techniques required. Additional information on environmental sample preservation and transportation is available in other references (**1**, **2**).<sup>2</sup> Samples for classification may be preserved using procedures similar to Class A. In most cases, a direct push sample is considered as Class B in Practice **D 4220** but is protected, representative, and suitable for chemical analysis. The samples taken with this practice do not usually produce Class C and D (with exception of thin wall samples of standard size) samples for testing for engineering properties, such as shear strength and compressibility. Guide **D 4700** has some information on mechanical soil sampling devices similar to direct push tech-

niques, however, it does not address most direct push sampling methods. If sampling is for chemical evaluation in the Vadose Zone, consult Guide **D 4700** for any special considerations.

1.3 Field methods described in this guide, include the use of discreet and continuous sampling tools, split and solid barrel samplers and thin walled tubes with or without fixed piston style apparatus.

1.4 Insertion methods described include static push, impact, percussion, other vibratory/sonic driving, and combinations of these methods using direct push equipment adapted to drilling rigs, cone penetrometer units, and specially designed percussion/direct push combination machines. Hammers providing the force for insertion include drop style, hydraulically activated, air activated and mechanical lift devices.

1.5 Direct push soil sampling is limited to soils and unconsolidated materials that can be penetrated with the available equipment. The ability to penetrate strata is based on hammer energy, carrying vehicle weight, compactness of soil, and consistency of soil. Penetration may be limited or damage to samplers and conveying devices can occur in certain subsurface conditions, some of which are discussed in 5.5. Successful sample recovery also may be limited by the ability to retrieve tools from the borehole. Sufficient retract force must be available when attempting difficult or deep investigations.

1.6 This guide does not address the installation of any temporary or permanent soil, ground water, vapor monitoring, or remediation devices.

1.7 The practicing of direct push techniques may be controlled by local regulations governing subsurface penetration. Certification, or licensing requirements, or both, may need to be considered in establishing criteria for field activities.

1.8 The values stated in SI units are to be regarded as standard; however, dimensions used in the drilling industry are given in inch-pound units by convention. Inch-pound units are used where necessary in this guide.

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.*

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Ground Water and Vadose Zone Investigation.

Current edition approved Jan. 1, 2005. Published February 2005. Originally approved in 1998. Last previous edition approved in 1998 as D 6282-98.

<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

1.10 This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide 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:<sup>3</sup>

- D 653** Terminology Relating to Soil, Rock and Contained Fluids
- D 1586** Test Method for Penetration Test and Split-Barrel Sampling of Soils
- D 1587** Practice for Thin-Wall Tube Sampling of Soils
- D 2488** Practice for Description and Identification of Soils (Visual-Manual Method)
- D 3550** Practice for Ring-Lined Barrel Sampling of Soils
- D 3694** Practices for Preparation of Sample Containers and for Preservation of Organic Constituents
- D 4220** Practices for Preserving and Transporting Soil Samples
- D 4700** Guide for Soil Sampling from the Vadose Zone
- D 5088** Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites
- D 5092** Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers
- D 5299** Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities
- D 5434** Guide for Field Logging of Subsurface Explorations of Soil and Rock
- D 6001** Guide for Direct-Push Water Sampling for Geoenvironmental Investigations

## 3. Terminology

3.1 *Definitions*—General definitions for terminology used in this guide are in accordance with Terminology **D 653**. Definitions for terms related to direct push water sampling for geoenvironmental investigations are in accordance with Guide **D 6001**.

3.1.1 *assembly length, n*—length of sampler body and riser pipes.

3.1.2 *borehole, n*—a hole of circular cross-section made in soil or rock.

3.1.3 *casing, n*—pipe furnished in sections with either threaded connections or bevelled edges to be field-welded, which is installed temporarily or permanently to counteract

caving, to advance the borehole, or to isolate the interval being monitored, or combination thereof.

3.1.4 *caving/sloughing, n*—the inflow of unconsolidated material into an unsupported borehole that occurs when the borehole walls lose their cohesive strength.

3.1.5 *decontamination, n*—the process of removing undesirable physical or chemical constituents, or both, from equipment to reduce the potential for cross-contamination.

3.1.6 *direct push sampling, n*—sampling devices that are advanced into the soil to be sampled without drilling or borehole excavation.

3.1.7 *extension rod, n*—hollow steel rod, threaded, in various lengths, used to advance and remove samplers and other devices during direct pushing boring. Also known as *drive rod*. In some applications, small diameter solid extension rods are used through hollow drive rods to activate closed samples at depth.

3.1.8 *incremental drilling and sampling, n*—insertion method where rotary drilling and sampling events are alternated for incremental sampling. Incremental drilling often is needed to penetrate harder or deeper formations.

3.1.9 *percussion driving, n*—insertion method where rapid hammer impacts are performed to advance the sampling device. The percussion normally is accompanied with the application of a static down-force.

3.1.10 *push depth, n*—the depth below a ground surface datum to which the lower end, or tip, of the direct-push sampling device is inserted.

3.1.11 *sample interval, n*—defined zone within a subsurface strata from which a sample is gathered.

3.1.12 *sample recovery, n*—the length of material recovered divided by the length of sampler advancement and stated as a percentage.

3.1.13 *soil core, n*—cylindrical shaped specimen of sediments or other unconsolidated accumulations of solid particles produced by the physical and chemical disintegration of rocks and which may or may not contain organic matter recovered from a soil sampler.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *closed barrel sampler, n*—a sampling device with a piston or other secured device that is held to block the movement of material into the barrel until the blocking device is removed or released. Liners are required in closed barrel samplers. Also may be referred to as a *protected type sampler*.

3.2.2 *impact heads/drive heads, n*—pieces or assemblies that fit to top of the above ground portion of the direct push tool assembly to receive the impact of the hammering device and transfer the impact energy to sampler extensions.

3.2.3 *open barrel sampler, n*—sampling barrel with open end allowing material to enter at any time or depth. Also may be referred to as an *unprotected type sampler*.

3.2.4 *piston lock, n*—device to lock the sampler piston in place to prevent any entry of a foreign substance into the sampler chamber prior to sampling.

3.2.5 *single tube system, n*—a system whereby single extension/drive rods with samplers attached are advanced into the subsurface strata to collect a soil sample.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.6 *solid barrel sampler, n*—a soil sampling device consisting of a continuous or segmented tube with a wall thickness sufficient to withstand the forces necessary to penetrate the strata desired and gather a sample. A cutting shoe and a connecting head are attached to the barrel.

3.2.7 *split barrel sampler, n*—a soil sampling device consisting of the two half circle tubes manufactured to matching alignment, held together on one end by a shoe and on the other by a connecting head.

3.2.8 *two tube systems, n*—a system whereby inner and outer tubes are advanced simultaneously into the subsurface strata to collect a soil sample. The outer tube is used for borehole stabilization. The inner tube for sampler recovery and insertion.

#### 4. Summary of Guide

4.1 Direct push soil sampling consists of advancing a sampling device into subsurface soils by applying static pressure, by applying impacts, or by applying vibration, or any combination thereof, to the above ground portion of the sampler extensions until the sampler has been advanced to the desired sampling depth. The sampler is recovered from the borehole and the sample removed from the sampler. The sampler is cleaned and the procedure repeated for the next desired sampling interval. Sampling can be continuous for full depth borehole logging or incremental for specific interval sampling. Samplers used can be protected type for controlled specimen gathering or unprotected for general soil specimen collection.

#### 5. Significance and Use

5.1 Direct push methods of soil sampling are used for geologic investigations, soil chemical composition studies, and water quality investigations. Examples of a few types of investigations in which direct push sampling may be used include site assessments, underground storage tank investigations, and hazardous waste site investigations. Continuous sampling is used to provide a lithological detail of the subsurface strata and to gather samples for classification and index or for chemical testing. These investigations frequently are required in the characterization of hazardous waste sites. Samples, gathered by direct push methods, provide specimens necessary to determine the chemical composition of soils, and in most circumstances, contained pore fluids (3).

5.2 Direct push methods can provide accurate information on the characteristics of the soils encountered and of the chemical composition if provisions are made to ensure that discrete samples are collected, that sample recovery is maximized, and that clean decontaminated tools are used in the sample gathering procedure. For purposes of this guide, "soil" shall be defined in accordance with Terminology D 653. Using sealed or protected sampling tools, cased boreholes, and proper advancement techniques can assure good representative samples. Direct push boreholes may be considered as a supplementary part of the overall site investigation or may be used for the full site investigation if site conditions permit. As such, they should be directed by the same procedural review and quality assurance standards that apply to other types of

subsurface borings. A general knowledge of subsurface conditions at the site is beneficial.

5.3 Soil strata profiling to shallow depths may be accomplished over large areas in less time than with conventional drilling methods because of the rapid sample gathering potential of the direct push method. More site time is available for actual productive investigation as the time required for ancillary activities, such as decontamination, rig setup, tool handling, borehole backfill, and site clean-up is reduced over conventional drilling techniques. Direct push soil sampling has benefits of smaller size tooling, smaller diameter boreholes, and minimal investigative derived waste.

5.4 The direct push soil sampling method may be used as a site characterization tool for subsurface investigation and for remedial investigation and corrective action. The initial direct push investigation program can provide good soil stratigraphic information depending on the soil density and particle size, determine ground water depth, and provide samples for field screening and for formal laboratory analysis to determine the chemical composition of soil and contained pore fluids. Use of this method, results in minimum site disturbance and no cuttings are generated.

5.5 This guide may not be the correct method for investigations in all cases. As with all drilling methods, subsurface conditions affect the performance of the sample gathering equipment and methods used. Direct push methods are not effective for solid rock and are marginally effective in partially weathered rock or very dense soils. These methods can be utilized to determine the rock surface depth. The presence or absence of ground water can affect the performance of the sampling tools. Compact gravelly tills containing boulders and cobbles, stiff clay, compacted gravel, and cemented soil may cause refusal to penetration. Certain cohesive soils, depending on their water content, can create friction on the sampling tools which can exceed the static delivery force, or the impact energy applied, or both, resulting in penetration refusal. Some or all of these conditions may complicate removal of the sampling tools from the borehole as well. Sufficient retract force should be available to ensure tool recovery. As with all borehole advancement methods, precautions must be taken to prevent cross contamination of aquifers through migration of contaminants up or down the borehole. Regardless of the tool size, the moving of drilling and sampling tools through contaminated strata carries risks. Minimization of this risk should be a controlling factor in selecting sampling methods and drilling procedures. The user should take into account the possible chemical reaction between the sample and the sampling tool itself, sample liners, or other items that may come into contact with the sample (3, 4).

5.6 In some cases this guide may combine water sampling, or vapor sampling, or both, with soil sampling in the same investigation. Guides D 6001 and D 4700 can provide additional information on procedures to be used in such combined efforts.

#### 6. Criteria for Selection

6.1 Important criteria to consider when selecting sampling tools include the following:

6.1.1 Size of sample.

6.1.2 Sample quality (Class A,B,C,D) for physical testing. Refer to Practice D 4220.

6.1.3 Sample handling requirements, such as containers, preservation requirements.

6.1.4 Soil conditions anticipated.

6.1.5 Ground water depth anticipated.

6.1.6 Boring depth required.

6.1.7 Chemical composition of soil and contained pore fluids.

6.1.8 Probability of cross contamination.

6.1.9 Available funds.

6.1.10 Estimated cost.

6.1.11 Time constraints.

6.1.12 History of tool performance under anticipated conditions (consult experienced users and manufacturers).

6.2 Important criteria to consider when selecting direct push equipment include the following:

6.2.1 Site accessibility.

6.2.2 Site visibility.

6.2.3 Soil conditions anticipated.

6.2.4 Boring depth required.

6.2.5 Borehole sealing requirements.

6.2.6 Equipment performance history.

6.2.7 Personnel requirements.

6.2.8 Decontamination requirements.

6.2.9 Equipment grouting capability.

6.2.10 Local regulatory requirements.

## 7. Apparatus

7.1 *General*—A direct push soil sampling system consists of a sample collection tool, hollow extension rods for advancement, retrieval, and transmission of energy to the sampler, and an energy source to force sampler penetration. Auxiliary tools are required to handle, assemble and disassemble, clean, and repair the sample collection tools and impact surfaces. Necessary expendable supplies are sample containers, sample container caps, sample liners, sample retainers, appropriate lubricants, and personal safety gear.

### 7.2 *Direct Push Tool Systems:*

7.2.1 *Two Tube System*—An outer casing and an inner extension rod with a sampler attached (see Fig. 1) are advanced simultaneously into the soil for the length capacity of the sampler. The sampler is removed from the borehole and a new sampler barrel or plug bit is inserted for each increment of depth. Two-tube sampling systems also may incorporate sample gathering chambers that are fitted into the outer casing shoe. These sample barrels are designed to create a minimum of sample disturbance while gathering high quality specimens (see Fig. 2). Samplers are held in the proper position by different methods, such as extension rods, pneumatic or mechanical packers, spring activated latches, or other devices (see Figs. 1 and 2). Locking devices must be strong enough to hold the sampler while penetrating the sample strata. The outer casing supports the borehole wall. Sample retrieval is expedited by the cased hole and continuous sampling is simplified. Continuous sampling may be a benefit to lithological logging. A cased borehole can be sealed from the bottom up as the casing is extracted (see Section 10). A cased hole may reduce the risk of contamination migration down the borehole and

sample cross contamination. The two-tube system is more susceptible to soil friction because of its larger diameter and may require larger direct push energy than single-tube systems. An oversized drive shoe is sometimes used to reduce friction and buckling but may increase the risk of contamination migration down the borehole.

7.2.2 *Single Tube System*—The single tube system (see Fig. 3), uses a hollow extension/drive rod to advance and retrieve the sampler. The sampler is attached to the bottom of the extension/drive rod. A drive cap is added to the top of the extension/drive rod and the sampler is pushed into the soil. Extension/drive rods generally are smaller in diameter than the sampler. The single tube system minimizes effort for discrete interval sampling under many subsurface conditions. Tool connection time per interval is reduced. Time of removal and reinsertion of samplers into the borehole is affected by soil conditions. Repeated movement of the sampler through contaminated subsurface strata may increase the risk of contamination migration down the borehole. Bottom up borehole sealing may require re-entry in soil formations that collapse (see Section 10).

### 7.3 *Samplers:*

7.3.1 *Split Barrel Samplers*—Split barrel samplers (see Fig. 4) are available for use with direct push drilling methods and are available in various sizes up to 3.0 in. (76.2-mm) inside diameter. The inside tolerance should allow for use of liners. Split barrel sampler shoes used in two tube systems must be of sufficient diameter to prevent the intrusion of soil between the outer diameter of the shoe and the inside wall of the outer tube. Split barrel shoes should be replaced when the leading edge is damaged. Damaged shoes can negatively affect sample recovery. Samplers can be used with or without ball check value fitted split barrel heads. The ball check prevents uphole fluids from flowing down through the sample. Where soil sampling will be performed below the water table, the split barrel head, equipped with a ball check, should be used. The open split barrel is best used with the two tube system because the outer casing protects the borehole against cave-in or sloughing, or in soils in which the borehole wall will not collapse. Split barrel sealing systems are available. Split barrel sections can be joined to create a sampler with a nominal sample length capacity of 48 in. (1.22 m). It is understood that samplers with usable lengths beyond 24 in. (0.61 m) are used to advantage in certain soil types; however, the added weight of the soil sample in the chamber and the added friction within the sampler may prevent loose soils from entering the sampler, affecting sample recovery and representativeness. Split barrel samplers can be fitted with a basket to improve recovery in cohesionless soils. Retainers are available in many styles and materials. Retainers should allow the passage of softer soils. Stiff retainers can reduce specimen recovery in soft soils.

### 7.3.2 *Solid Barrel Samplers:*

7.3.2.1 *Open Solid Barrel Samplers*—Open solid barrel (see Fig. 5) samplers are used with all types of direct push sampling systems. Solid barrels can have inside diameters ranging up to 3 in. (76.2 mm). Barrel lengths range from 6 in. (152.4 mm) to 5 ft (1.53 m). Solid barrel samplers may be one piece or segmented. Sample liners should be used to facilitate removal

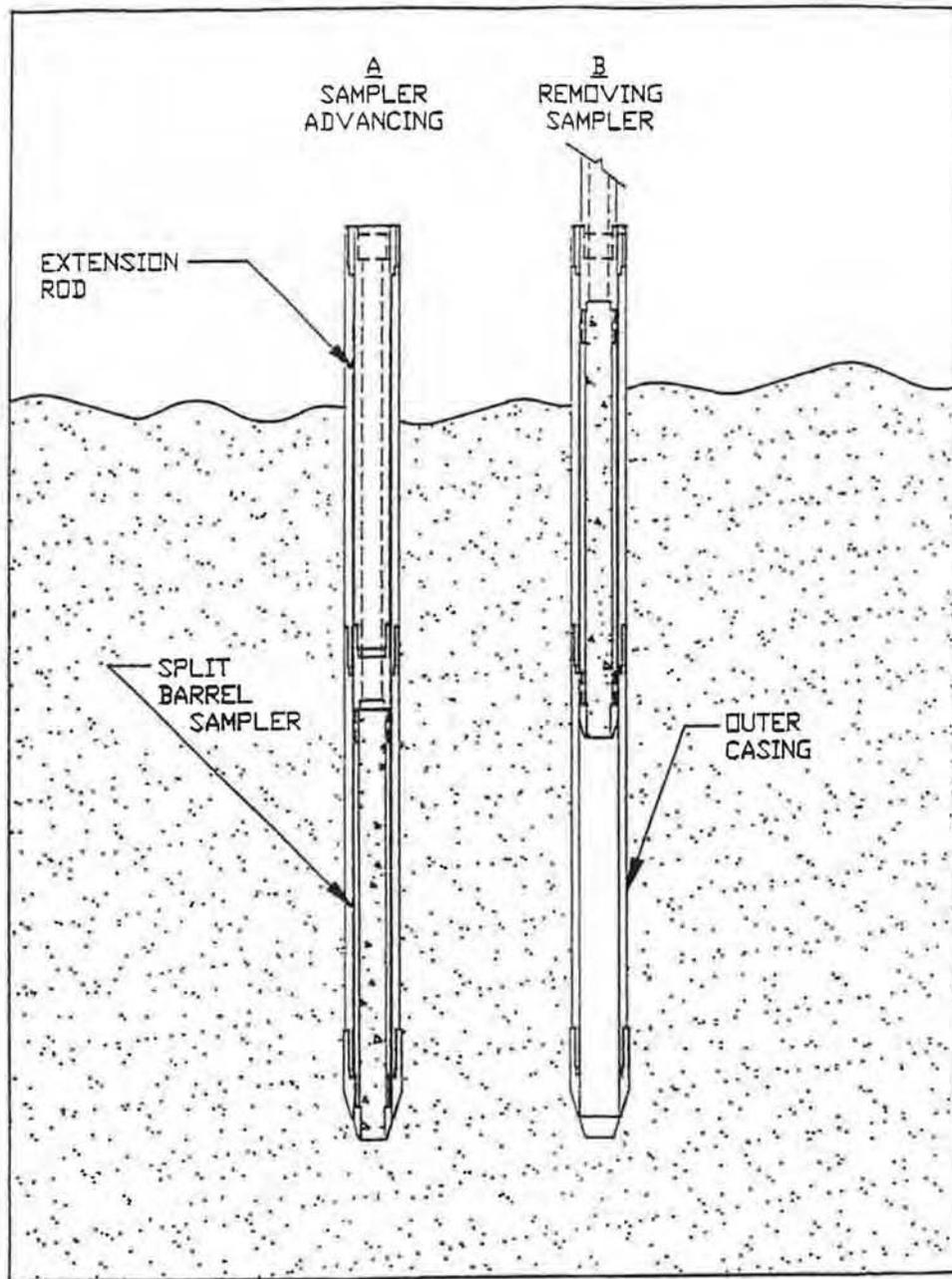


FIG. 1 Split Spoon Sampling, Two Tube System

of the sample from the solid barrel. Without the use of liners, samples are extruded mechanically. Liner lengths should fit sampler barrel lengths. Solid barrel samplers are generally assembled with a removable cutting shoe and a drive head (see Fig. 6). The head provides a backing to hold the liner stationary while the sampler is advanced and serves as a connector to the extension/drive rods. The shoe is manufactured to hold the liner stationary during the soil collection procedure. The liner should be slightly larger than the inner diameter of the cutting shoe. It may be slipped over the cutting shoe (see Fig. 6) or nested inside of the cutting shoe (see Fig. 7). The shoe is

manufactured to cut the sample to a slightly undersized diameter allowing it to pass into the sample liner with a minimum of side friction to reduce sample disturbance. The amount of specimen contact with the inside of the shoe should be held to the minimum distance possible to aid in achieving the maximum amount of recovery.

**7.3.3 Closed Barrel Sampler**—Closed barrel samplers (see Figs. 2 and 3, Figs. 5-8) are devices, which remain sealed shut until an action is taken to open the sample receiving chamber. These samplers are used most often for single events (discrete point sampling) where a sealed sampler is required to avoid

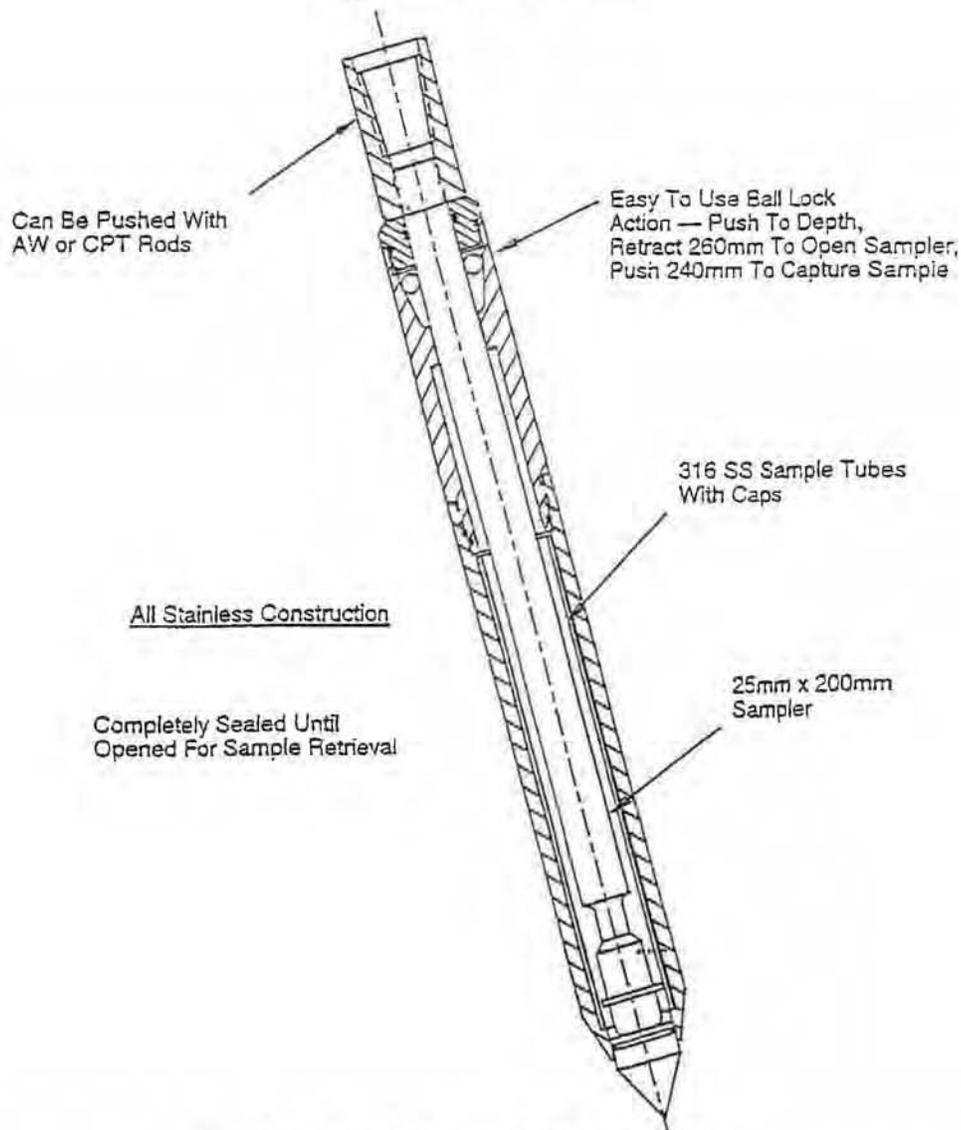


FIG. 2 Sealed Sample Barrel, Single Tube System

cross contamination or in circumstances where borehole wall stability cannot be assured. The shoe sealing device generally is a point designed to allow the continuous flow of soil around and past the sampler until such time as it is removed or released. The piston point can be fitted with seals, such as "O" rings at top and bottom to hold fluid out until sampling the desired interval. The piston rod extends through the sample retaining liner and must be released or removed for the soil to enter (see Fig. 3, Fig. 5, Fig. 7). The piston can be removed manually before sampling or be displaced by the soil entering the sampler chamber. Using the displacement method can result in reduced recovery if sampled soils do not have sufficient strength to displace the piston. Pistons are locked in place by several methods, such as a spring loaded latch. The latch holds several balls (see Fig. 2, Fig. 7, Fig. 8) into a groove in the latch coupling. When the latch is released by lifting up on the latch stem, the balls slip back into the latch chamber

allowing the piston to be removed. Another method uses a locking screw. A reverse thread pin (Fig. 3, Fig. 6) is positioned in the sampler head to prevent the piston from being displaced by the soil when advancing the sampler. At the sampling interval, small diameter extension rods are inserted through the sampler extension/drive rods and rotated clockwise to unscrew the locking pin. A third method uses an inflated packer. An inflated packer (see Fig. 9) is attached to the top of the sampler barrel. The sample barrel is lowered into position in the drive casing and the packer inflated. The packer is deflated to release and the sample barrel is recovered after being advanced the sampling interval.

7.3.4 *Thin Wall Tube*—A 1.0-in. (25.4-mm) diameter thin wall tube (see Fig. 10) is available for use with direct push equipment and is manufactured according to Practice D 1587. Thin wall tubes can be effective when used with dual tube direct push systems as the borehole must be kept clear of

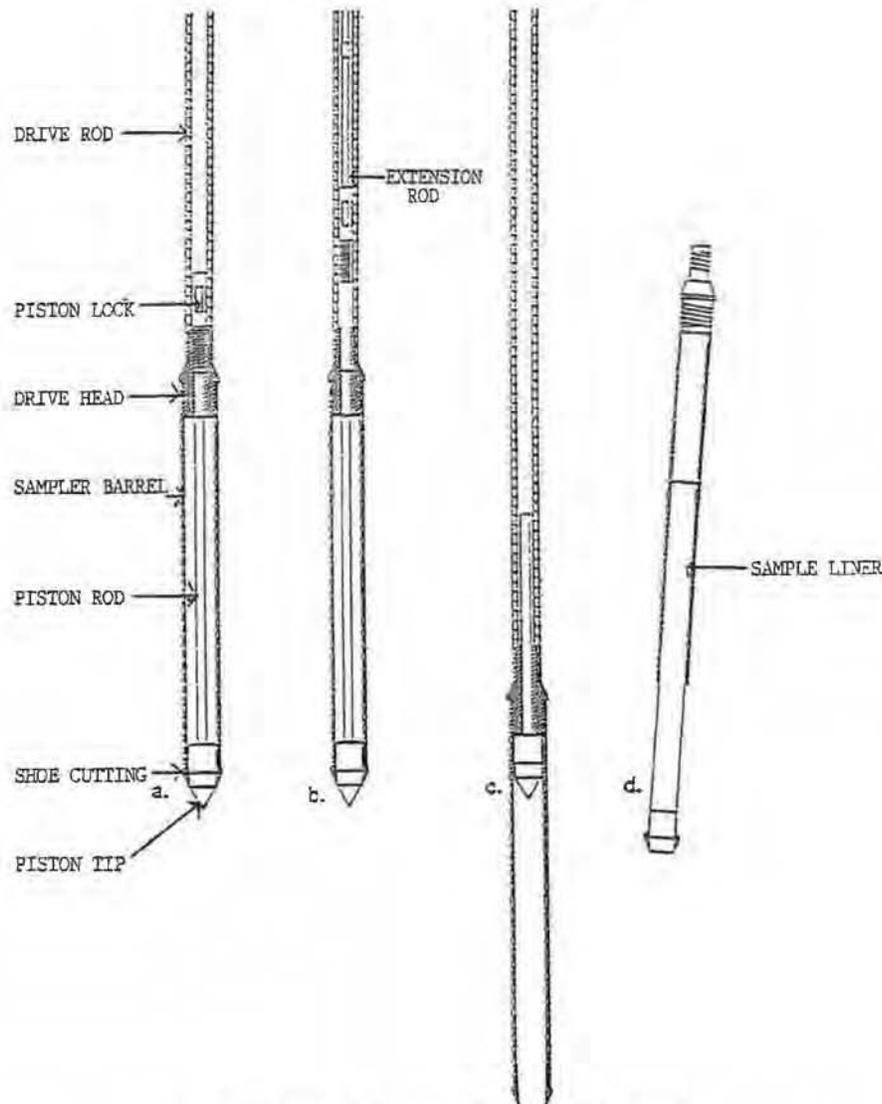


FIG. 3 Driving and Sampling, Single Tube System

- (a) Driving the sealed sampler.
- (b) Removing the stop-pin.
- (c) Collecting a sample
- (d) Recovering sample in liner.

disturbed soil prior to gathering a sample. Thin wall tubes may be effective in cohesive soils with single tube systems when the borehole can be kept clear of disturbed soil. Thin wall tubes must have an outside diameter that will allow passage through the outer casing. The thin wall tube can be operated in accordance with Practice D 1587, or it can be advanced using the percussion hammer or the direct push equipment. The primary use of the thin wall tube is to gather relatively undisturbed samples in cohesive soils. Sealing of thin wall tube ends should be completed in accordance with Practice D 4220. Fixed piston apparatus (see Fig. 10) also is available for use with thin walled tubes. The fixed piston action allows the sampling of very soft formations, which may not be retained in conventional samplers. In certain soil formations, the thin wall tube provides the best method to collect an undisturbed sample.

7.3.5 *Sampler Extension/Drive Rods*—Sampler extension/drive rods are lengths of rod or tube generally constructed of steel to withstand the pushing or percussion forces applied. Extension drive rods are available in various lengths. Rod lengths should be mated with casing and sampling equipment used. Thread types and classes vary between equipment manufacturers. Rod joints can be sealed to prevent fluid intrusion with “O” rings, Teflon<sup>®</sup> washers or Teflon<sup>®</sup> tape. Because of the percussive effort, joint seals should be checked for each sampling effort. Extension/drive rods should have sufficient inside diameter to accommodate the equipment necessary to perform the desired action.

7.3.6 *Sampler Liners*—Sampler liners are used to collect and store samples for shipment to laboratories, for field index testing of samples and for removing samples from solid barrel

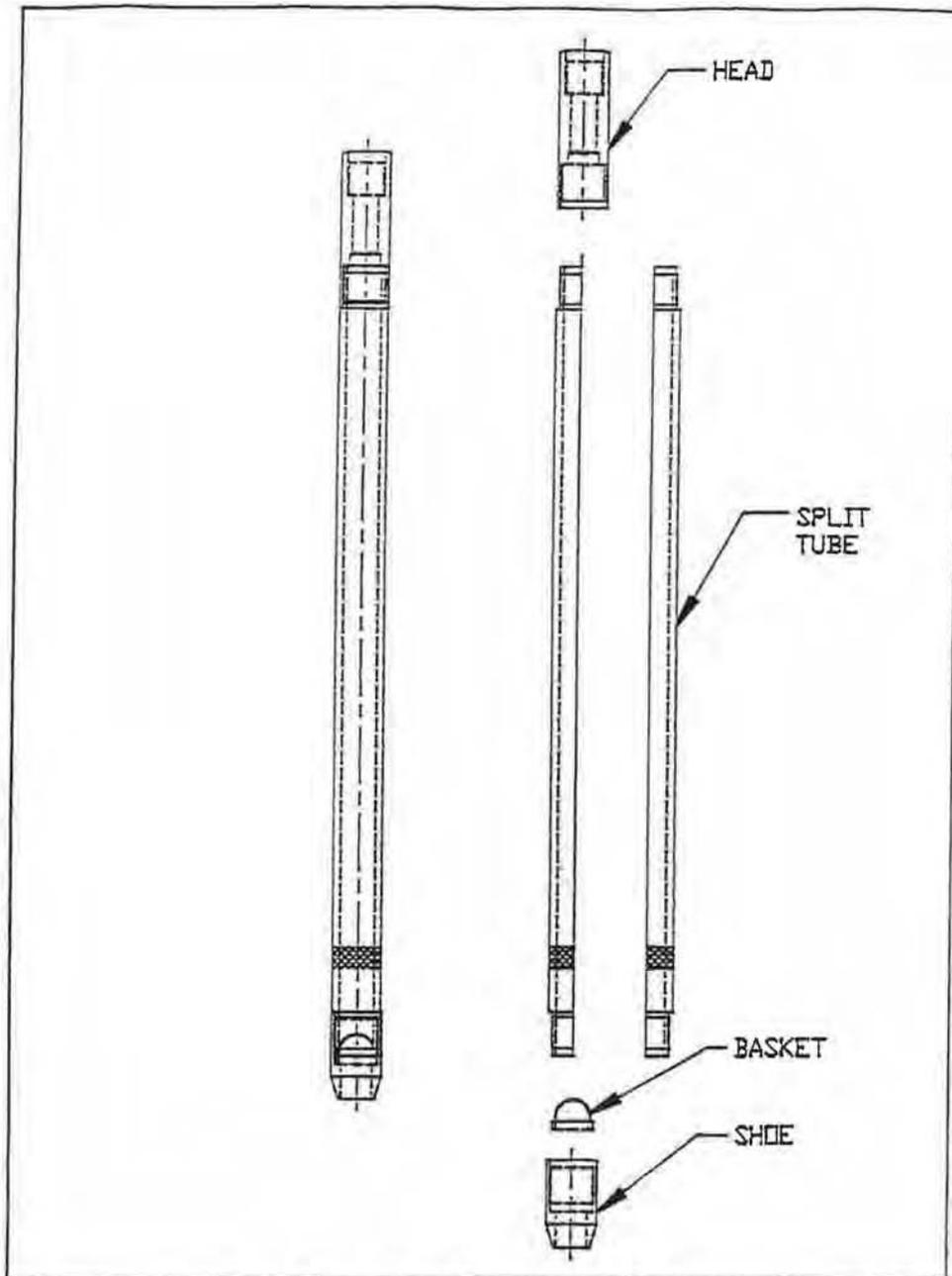


FIG. 4 Split Barrel Sampler, Two Tube System

type samplers. Liners are available in plastics, Teflon<sup>®</sup>, brass, and stainless steel. Other materials can be used as testing needs dictate. Liners are available in lengths from 6 in. (152.4 mm) to 5.0 ft (1.53 m). Liner material selection often is based on the chemical composition of liner/soil to minimize sample reaction with liner. Most liner use is short term as samples are subsampled and preserved immediately on site. A general rule for liner selection is stainless steel for organic compounds and plastic for metals. Teflon<sup>®</sup> may be required for mixed wastes and for long time storage. Liners should be sealed in accordance with Practice D 4220 when samples are collected for

physical testing. Other appropriate procedures must be used when samples are collected for environmental analysis (see Practices D 3694) (1, 2). Liners generally are split in the field for subsampling. Individually split liners are available in some sizes for field use. The liner should have a slightly larger inside diameter than the soil specimen to reduce soil friction and enhance recovery. When a slightly oversized liner is used, the potential for air space exists around the sample. Certain chemical samples may be affected by the enclosed air. Liners having less tolerance may be required and a shortened sampled interval used to reduce friction in the liner. Metal liners can be

A. OPEN

B. CLOSED

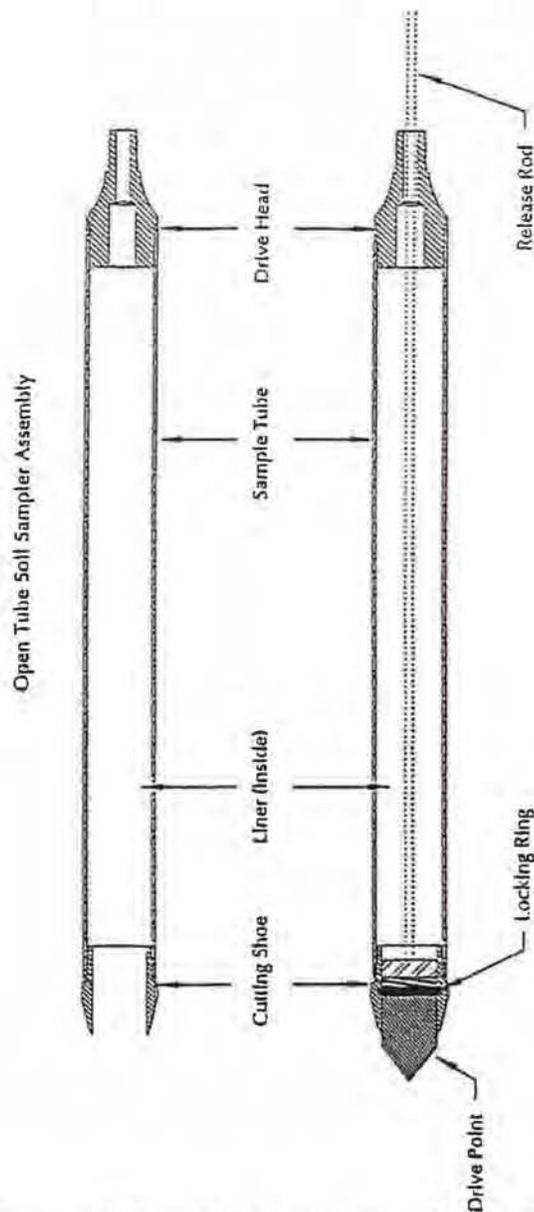


FIG. 5 (A) Open and (B) Closed Piston Sampler Assembly, Single Tube System

reused after proper cleaning and decontamination. Plastic liners should be disposed of properly after use.

7.3.7 *Sample Containers*—Sample containers should be prescribed according to the anticipated use of the sample specimen. Samples taken for chemical testing may require decontaminated containers with specific preservatives. Practice D 3694 provides information on some of the special containers and preservation techniques required (1, 2). These containers generally will be decontaminated to specific criteria. Samples for geotechnical testing require certain minimum volumes and specific handling techniques. Practice D 4220 offers guidance for sample handling of samples submitted for physical testing.

7.4 *Direct Push Power Sources*—Soil probing percussion driving systems, penetrometer drive systems, and rotary drilling equipment may be used to drive casings and direct push soil sampling devices. The equipment should be capable of applying sufficient static force, or dynamic force, or both, to advance the sampler to the required depth to gather the desired sample. The system must have adequate retraction force to remove the sampler and extension/drive rods once the selected strata has been penetrated. Rotation of the drill string can be added during insertion, as well as during retraction if the drive system can impart rotation.

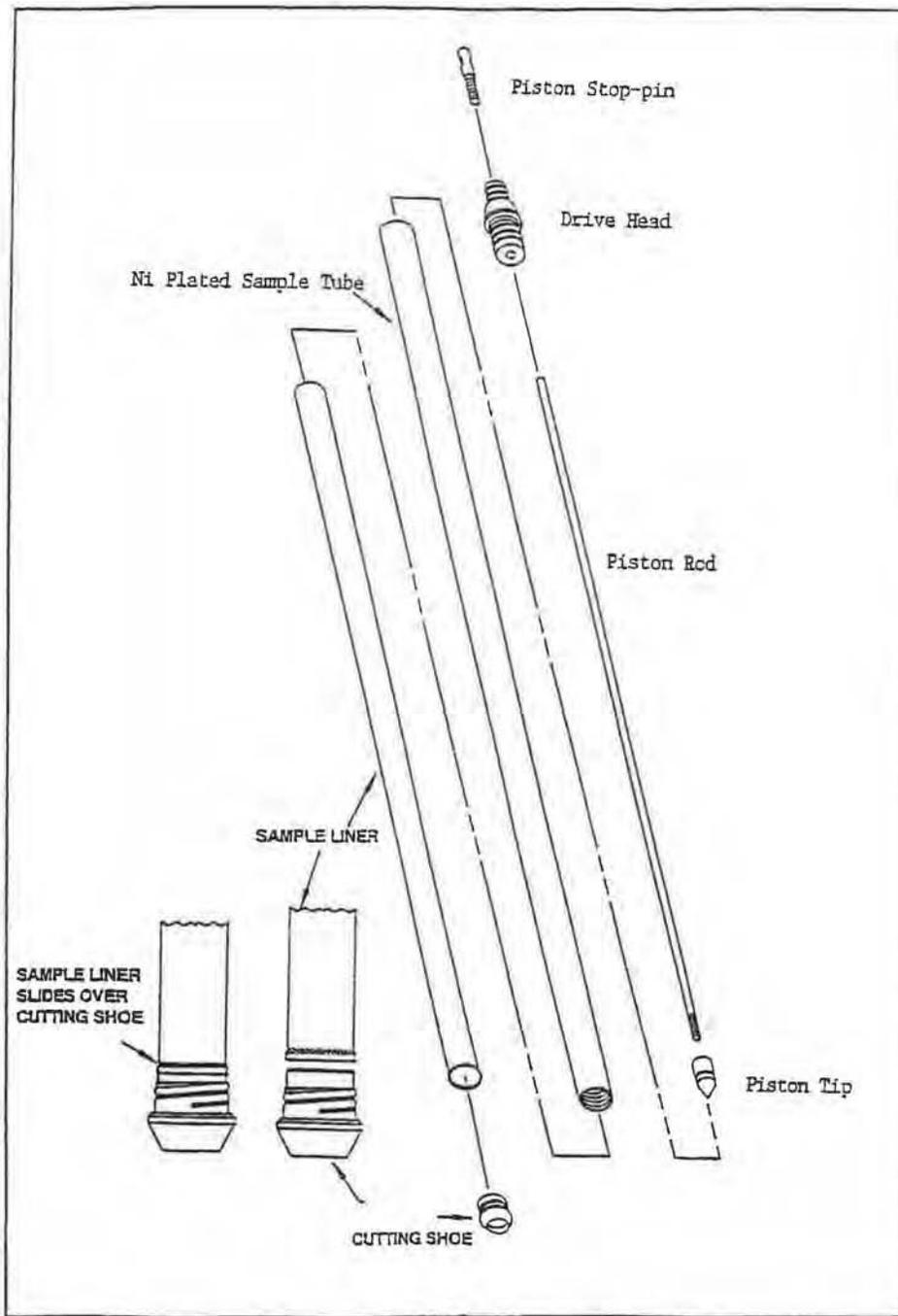


FIG. 6 Sampler Parts, Single Tube System

7.4.1 *Retraction Force*—The retraction force can be applied by direct mechanical pull back using the hydraulic system of the power source; line pull methods using mechanical or hydraulic powered winches, or cathead and rope windlass type devices. Winches used with direct push technology should have a minimum of 2000 lb (907 kg) top layer rating capacity and a line speed of 400 ft (121.96 m)/min to provide effective tool handling. Direct push sampling tools can be retracted by back pounding using weights similar to those of standard

penetration testing practices. Backpounding to recover samples can affect recovery and cause disturbances to the sample. Other forms of extraction, such as jacking, that do not cause undue disturbance to the sample, are preferable.

7.4.2 *Percussion Devices*—Percussion devices for use with direct push methods are hydraulically-operated hammers, air-operated hammers, and mechanically-operated hammers. Hydraulically-operated hammers should have sufficient energy to be effective in moving the samplers through the subsurface

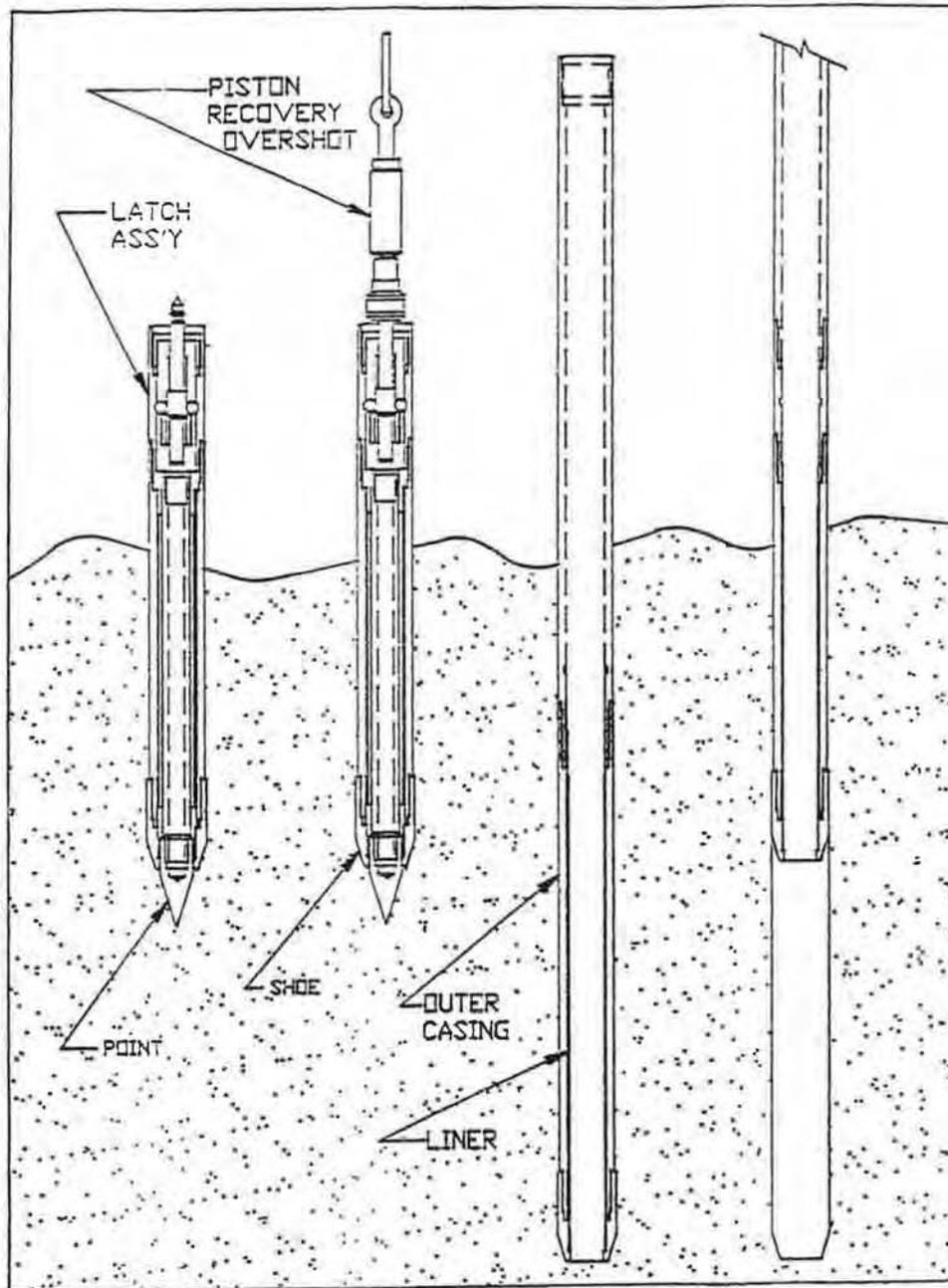


FIG. 7 Closed Solid Barrel Sampler, Single Tube System

strata. The maximum energy application is dependent on the tools used. Hammer energy that exceeds tool tolerance will result in tool damage or loss and will not achieve the goal of collecting high quality samples. Air-operated hammers should be capable of delivering sufficient energy, as well. Hammer systems utilizing hydraulic oil or air should be operated in the range specified by the manufacturer. Manually-operated hammers can be used to advance direct push tools. These hammers can be operated mechanically or manually using cathead and rope. These systems generally involve using 140 lb, standard penetration (see Test Method D 1586) hammers, which can

work well for direct push sampling. In operation, these hammers tend to be slower than hydraulic hammers and can cause tool damage if direct push tools are not designed to take the heavy blows associated with these hammers. The hydraulic- and air-operated hammers strike up to 2000 blows/min. In addition to the energy transferred, the rapid hammer action sets up a vibratory effect, which also aids in penetration. This vibratory effect, along with the percussive effort, may disturb some soil samples.

7.4.3 *Static Push Systems*—Cone penetrometer systems are an example of static push systems. They impart energy to the

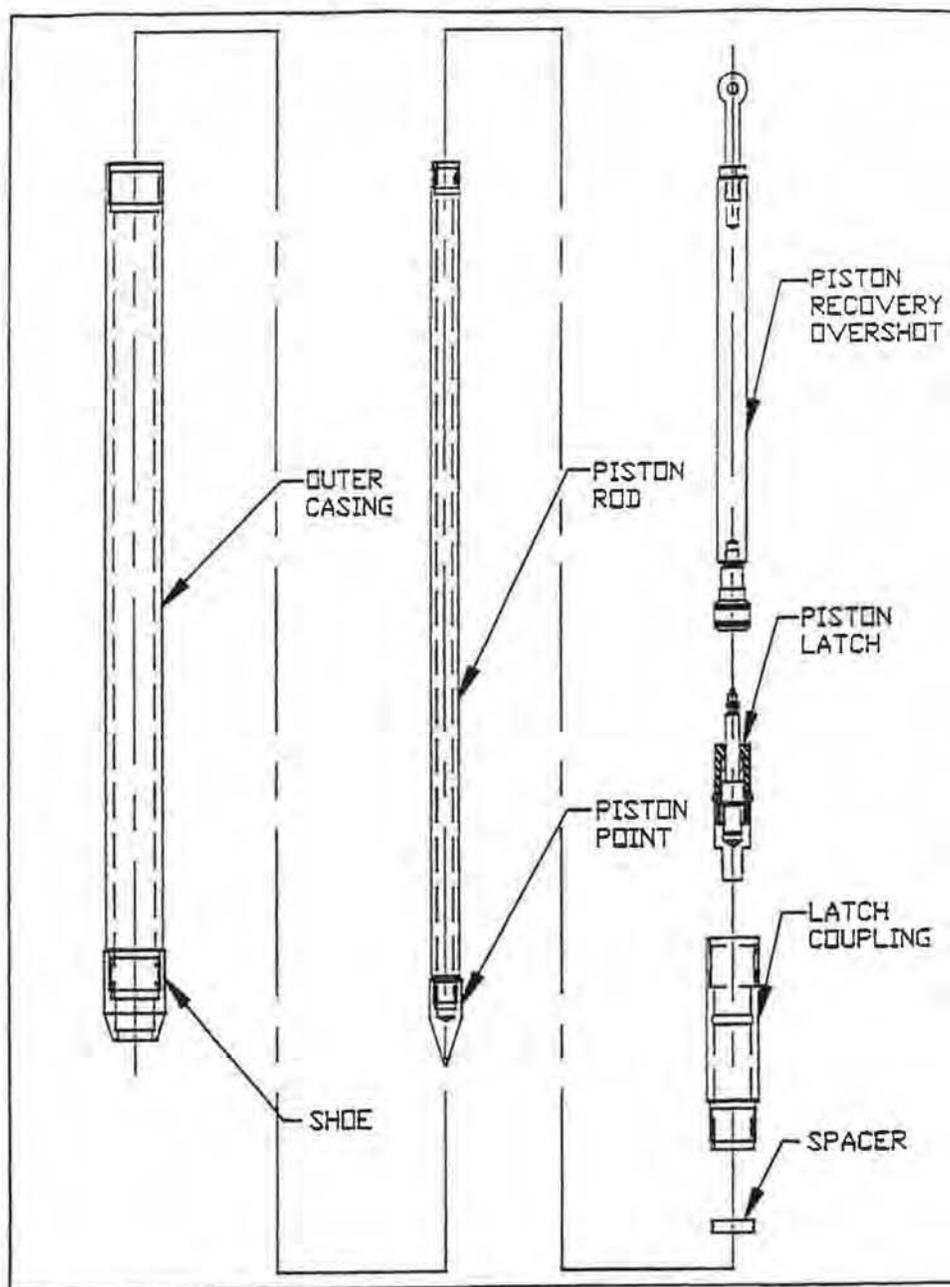


FIG. 8 Closed Solid Barrel Sampler, Single Tube System

sampler and extension rods by using hydraulic rams to apply pressure. The pressure applied is limited to the reactive weight of the drive vehicle. Retraction of the sampler and extension rods is by static pull from the hydraulic rams.

**7.4.4 Vibratory/Sonic Systems**—These systems utilize a vibratory device, which is attached to the top of the sampler extension rods. Reactive pressure and vibratory action are applied to the sampler extensions moving the sampler into the formation. In certain formations, sample recovery and formation penetration is expedited; however, all formations do not react the same to vibratory penetration methods.

**7.4.4.1 Sonic or Resonance Drilling Systems**—These are high powered vibratory systems that can be effective in advancing large diameter single or dual tube systems. They generally have depth capabilities beyond the smaller direct push systems.

**7.4.5 Rotary Drilling Equipment**—Direct push systems are readily adaptable to rotary drill units. The drill units offer a ready hydraulic system to operate percussion hammers, as well as reactive weight for static push. Because most drills are equipped with leveling jacks, better weight application is achieved. Vertical pushing is improved because of the ability to

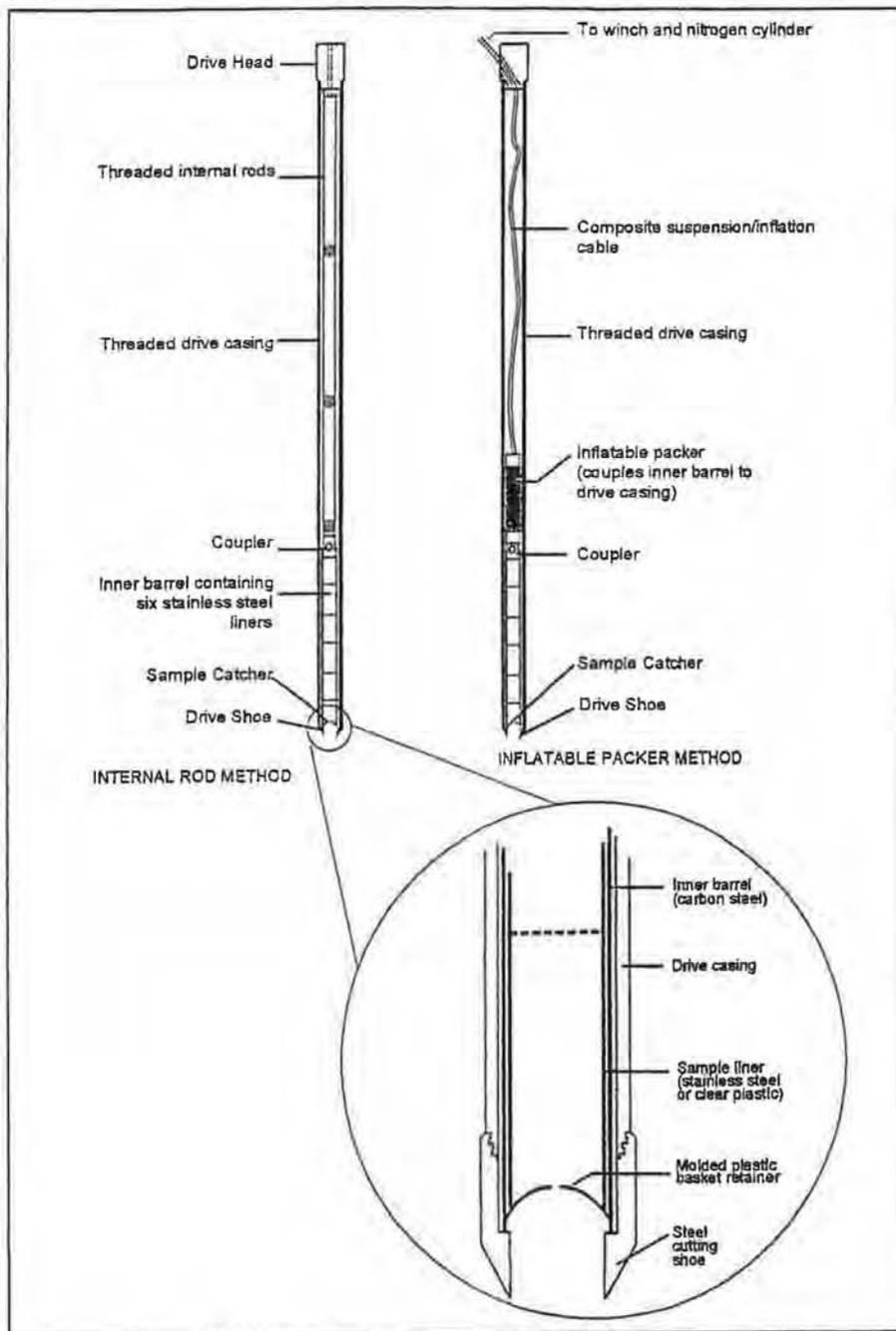


FIG. 9 Sampler Holding Methods, Two Tube System

level the machine. Tool handling is facilitated by high speed winches common to drilling rigs, extended masts for long tool pulls, and longer feed stroke length. Drill units with direct push adaptations also offer drilling techniques should obstacles be encountered while using direct push technology. Large drill units may have reactive weights that can exceed the tool capacity, thereby resulting in damaged tools.

### 8. Conditioning

8.1 *Decontamination*—Sampling equipment that will contact the soil to be sampled should be cleaned and decontaminated before and after the sampling event. Extension rods should be cleaned prior to each boring to avoid the transfer of contaminants and to ease the connecting of joints. Thread

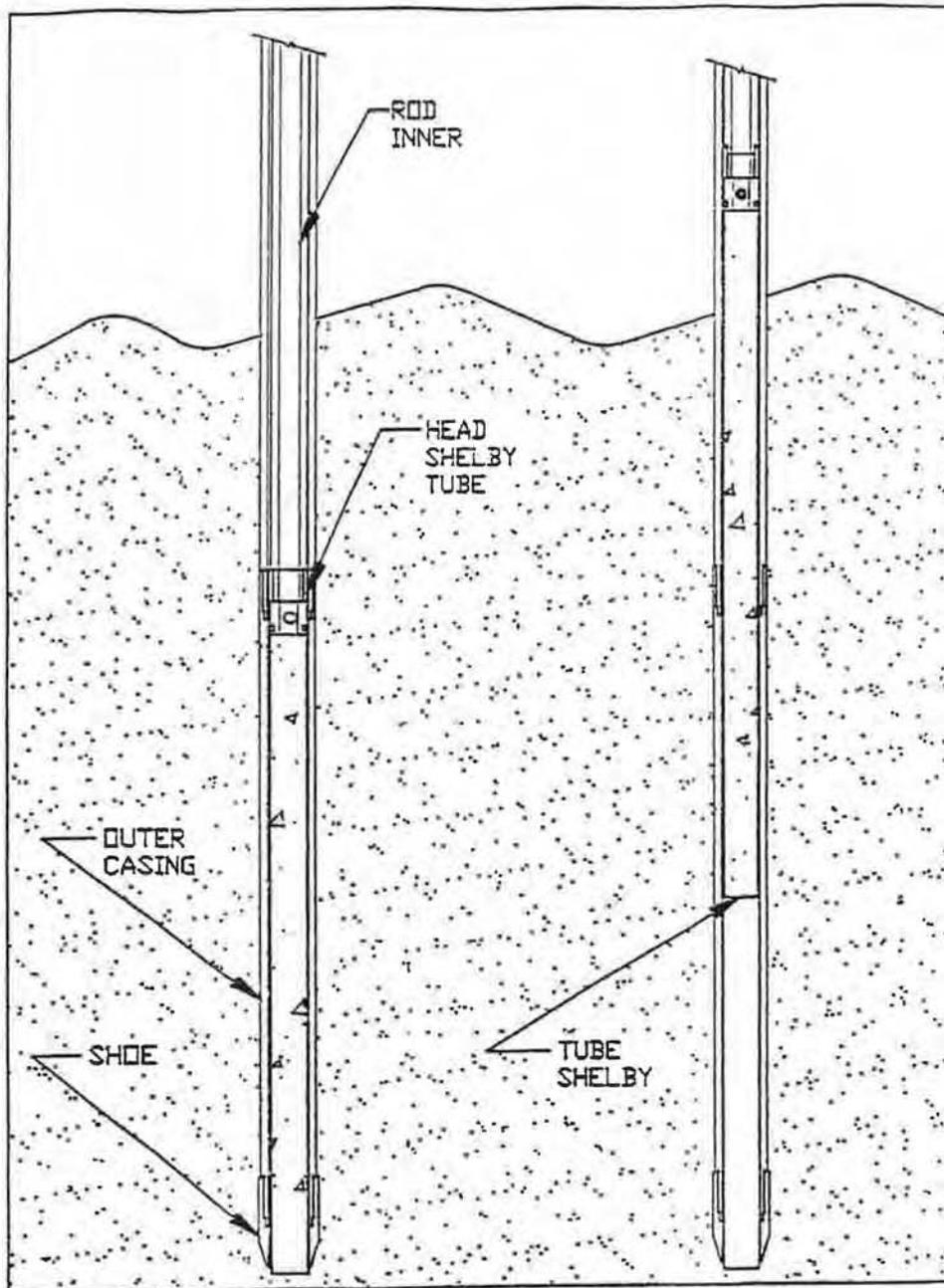


FIG. 10 Thin Wall Tube Sampler, Two Tube System

maintenance is necessary to ensure long service life of the tools. Sample liners should be kept in a sealed or clean environment prior to use. Reusable liners should be decontaminated between each use. All ancillary tools used in the sampling process should be cleaned thoroughly, and if contaminants are encountered, decontaminated before leaving the site. It should not be assumed that new tools are clean. They should be cleaned and decontaminated before use. Decontamination should be performed following procedures outlined in

Practice D 5088 along with any site safety plans, sampling protocols, or regulatory requirements.

8.2 *Tool Selection*—Prior to dispatch to the project site an inventory of the necessary sampling tools should be made. Sample liners, containers, sampling tools, and ancillary equipment should be checked to ensure its proper operation for the work program prescribed. Sampling is expedited by having two or more samplers on site. Since samples can be recovered quite fast, a supply of samplers will allow a boring to be

completed so other functions can be performed while samples are being processed. A backup tool system adaptable to and within the capabilities of the power source should be available should the original planned method prove unworkable. Materials for proper sealing of boreholes should always be available at the site (5-7).

## 9. Procedure

9.1 While procedures for direct push soil sampling with two common direct push methods are outlined here, other systems may be available. As long as the basic principles of practice relating to sampler construction and use are followed, other systems may be acceptable.

9.2 *General Set-Up*—Select the boring location and check for underground and overhead utilities and other site obstructions. Establish a reference point on the site for datum measurements, and set the direct push unit over the boring location. Stabilize and level the unit, raise the drill mast or frame into the drilling position, and attach the hammer assembly to the drill head if not permanently attached. Attach the anvil assembly in the prescribed manner, slide the direct push unit into position over the borehole, save a portion of the sliding distance for alignment during tool advancement, and ready the tools for insertion.

9.2.1 *Tool Preparation*—Inspect the direct push tools before using, and clean and decontaminate as necessary. Inspect drive shoes for damaged cutting edges, dents, or thread failures as these conditions can cause loss of sample recovery and slow the advancement rate. Where permissible, lubricate rod joints with appropriate safe products, and check impact surfaces for cracks or other damage that could result in failure during operations. Assemble samples and install where required, install sample retainers where needed, and install and secure sampler pistons to ensure proper operation where needed.

9.2.2 *Sample Processing*—Sample processing should follow a standard procedure to ensure quality control procedures are completed. View sample in the original sampling device, if possible. Open the sampling device with care to keep disturbance to a minimum. When using liners or thin wall tubes, protect ends to prevent samples from falling out or being disturbed by movement within the liner. Measure recovery accurately, containerize as specified in the work plan or applicable ASTM procedures, and label recovered samples with sufficient information for proper identification. When collecting samples for volatile chemical analysis, sample specimens must be contained and preserved as soon as possible to prevent loss of these components. Follow work plan instructions or other appropriate documents (see Practice D 3694) when processing samples collected for chemical analysis.

### 9.3 Two Tube System:

9.3.1 *Split Barrel Sampling* (see Fig. 1)—Assemble the outer casing with the drive shoe on the bottom, attach the drive head to the top of the outer casing, and attach the sampler to the extension rods. Connect the drive head to the top of the sampler extension rods, and insert the sampler assembly into the outer casing. The sampler cutting shoe should contact the soil ahead of the outer casing to prevent unnecessary sample disturbance. The split spoon cutting shoe should extend a

minimum of 0.25 in. (6.25 mm) ahead of the outer casing. Greater extensions may improve recovery in soft formations. Mark the outer casing to designate the required drive length, position the outer casing and sampler assembly under the drill head, and move the drill head downward to bring pressure on the tool string. If soil conditions allow, advance the sampler/casing assembly into the soil at a steady rate slow enough to allow the soil to be cut by the shoe and move up inside the sample barrel. If advancement is too rapid, it can result in loss of recovery because of soil friction in the shoe. Occasional hammer action during the push may help recovery by agitating the sample surface. If soil conditions prevent smooth static push advancement, activate the hammer to advance the sampler. Apply a continuous pressure while hammering to expedite soil penetration. The pressure required is controlled by subsurface conditions. Applications of excessive down pressure may result in the direct push unit being shifted off the borehole causing misalignment with possible tool damage. Stop the hammer at completion of advancement of the measured sampling barrel length. Release the pressure and move the drill head off the drive head. Attach a pulling device to the extension rods or position the hammer bail and retrieve the sampler from the borehole. At the surface remove the sampler from the extension rods and process. Soil classification is accomplished easily using split barrel samplers as the specimen is available readily for viewing, physical inspection and subsampling when the barrel is opened. Clean, decontaminate, and reassemble the sampler. Reattach the sampler to the extension rod, add the necessary extension rod and outer casing to reach the next sampling interval, and sound the borehole for free water before each sample interval. If water is present, it may be necessary to change sampling tools. Unequal pressure inside the casing may result in blow-in of material disturbing the soil immediately below the casing. Lower the sampler to its proper position, add the drive heads, and repeat the procedure. If it is desired that the pass through certain strata without sampling, install an extension rod point in lieu of the sampler. When the sampling interval is reached, remove the point and install the sampler. Advance the sampler as described. Upon completion of the borehole, remove the outer casing after instrumentation has been set or as the borehole is sealed as described in Section 10 (6).

### 9.3.2 Two Tube System—Other Samplers:

9.3.2.1 *Thin Wall Tubes*—Thin wall tubes (see Fig. 10) can be used with the dual tube system. Attach the tube to the tube head using removable screws. Attach the tube assembly to the extension rods and position at the base of the outer casing shoe protruding a minimum of 0.25 in. (6.25 mm) to contact the soil ahead of the outer casing. Advance the tube, with or without the outer casing, at a steady rate similar to the requirements of Practice D 1587. At completion of the advancement interval, let the tube remain stationary for 1 min. Rotate the tube slowly two revolutions to shear off the sample. Remove the tube from the borehole, measure recovery, and classify soil. The thin wall tube can be field extruded for on-site analysis or sealed in accordance with Practice D 4220 and sent to the laboratory for

processing. Samples for environmental testing generally require the subsampling and preservation of samples in controlled containers. Soil samples generally are removed from the sampling device for storage and shipping. Thin wall tubes should be cleaned and decontaminated before and after use.

9.3.2.2 *Thin Wall Tube Piston Sampler (see Fig. 11)*— Check the fixed piston sampling equipment for proper operation of the cone clamping assembly and the condition of the sealing “O” rings. Slide the thin wall tube over the piston, and attach it to the tube head. Position the piston at the sharpened

end of the thin wall tube just above the sample relief bend. Attach the sampler assembly to the extension rods, and lower the sampler into position through the outer casing. Install the actuator rods through the extension rod, and attach to the actuator rod in the sampler assembly. Attach a holding ring to the top of the actuator rod string, and hook the winch cable or other hook to the holding ring to hold the actuator rods in a fixed position. Attach the pushing fork to the drill head/probe hammer, and slowly apply downward pressure to the extension rods advancing the thin wall tube over the fixed piston into the

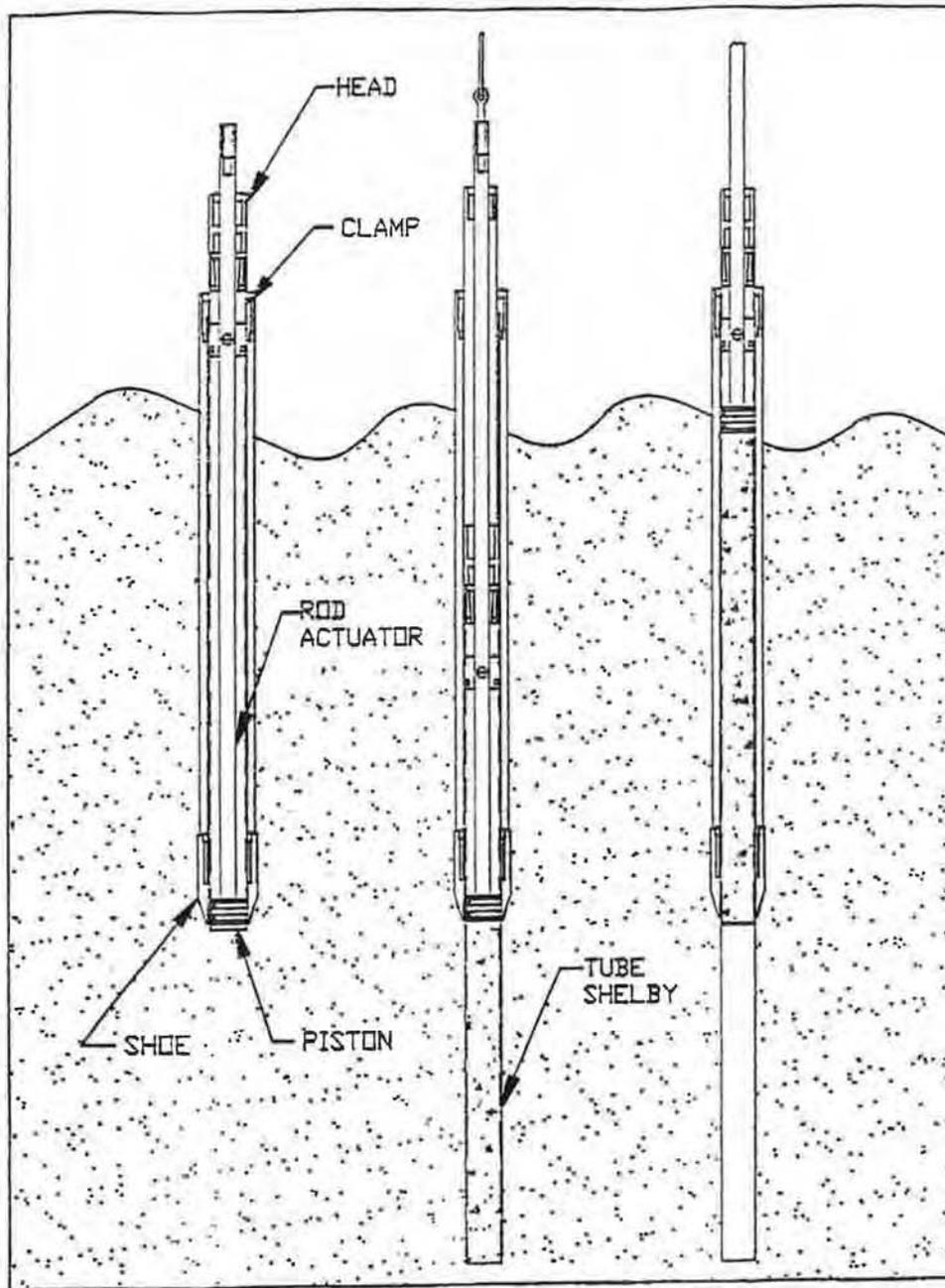


FIG. 11 Fixed Piston Sampler, Two Tube System

soil for the sample increment. Rest sampler 1 min to allow sample to conform to tube. Rotate tube one revolution to shear off sample. Remove sampler assembly from borehole and process sample (6).

**9.3.2.3 Open Solid Barrel Samplers**—Use solid barrel samplers in advance of the outer casing where the soil conditions could cause swelling of split barrel samplers, or where friction against the outer casing precludes its advancement and sampling must still be accomplished. The solid, single, or segmented barrel sampler requires the use of liners for removal of the sample. The sampler must be cleaned and decontaminated before use. Use of the sampler follows the procedure described in 9.3.1.

#### 9.4 Single Tube System:

**9.4.1 Open Solid Barrel Sampler (see Figs. 5 and 6)**—Attached the required liner to the cutting shoe by insertion into the machined receptacle area or by sliding over the machined tube. Insert the liner and shoe into the solid barrel, and attach the shoe (6, 8-11). Attach the sampler head to the sampler barrel providing a backing plate for the liner. Attach the sampler assembly to the drive rod and the drive head to the drive rod. Position the assembly under the hammer anvil and advanced as described in 9.3.1. At completion of the sampling increment, remove the sampler from the borehole. Remove the filled sample liner from the barrel by unscrewing the shoe, cap the liner for laboratory testing or spit open for field processing, and advance the borehole by repeating the procedure. Because the solid barrel cannot be opened for cleaning, it may require more effort for cleaning and decontamination. The open solid barrel sampler is used in soil formations that have sufficient wall strength to maintain a borehole wall without sloughing or cave-in. In soil formations not affording such structure, other sampling methods may be required or the opening sealed. To enhance recovery in some soil strata, it may be necessary to vary the length of the sampling increment. Shorter increments generally improve recovery because of lower sample friction and compression inside the sampler chamber. Sample recovery can be enhanced in some formations by intermittent use of the percussion hammer (6, 8, 10, 11).

**9.4.2 Closed Solid Barrel Sampler (see Figs. 5-7, Fig. 11)**—Insert or attach the sample liner to the shoe, and insert the assembly into the solid barrel sampler. Install sample retaining basket if desired. Attach the latch coupling or sampler head to the sampler barrel, and attach the piston assembly with point and “O” rings if free water is present, to the latching mechanism or holder. Insert the piston or packer into the liner to its proper position so the point leads the sampler shoe. Set latch, charge packer, or install locking pin, and attach assembled sampler to drive rod. Add drive head and position under the hammer anvil. Apply down pressure, hammer if needed, to penetrate soil strata above the sampling zone. When the sampling zone is reached, insert the piston latch release and recovery tool, removing the piston, or insert the locking pin removal/extension rods through the drive rods, turn counter-clockwise, and remove the piston locking pin so the piston can float on top of the sample, or release any other piston holding device. Direct push or activate the hammer to advance the sampler the desired increment. Retrieve the sampler from the

borehole by withdrawing the extension/drive rods. Remove the shoe, and withdraw the sample liner with sample for processing. Clean and decontaminate the sampler, reload as described, and repeat the procedure. Extreme stress is applied to the piston when driving through dense soils. If the piston releases prematurely, the sample will not be recovered from the correct interval, and a resample attempt must be made. The piston sampler can be used as a re-entry grouting tool for sealing boreholes on completion if it is equipped with a removable piston (5, 6, 7, 10, 11).

**9.4.3 Standard Split Barrel Sampler**—Attach the split spoon to an extension rod or drill rod. Using a mechanical or hydraulic hammer drive the sampler into the soil the desired increment, as long as that increment does not exceed the sampler chamber length. Remove the sampler from the borehole, disassemble, and process sample. Standard split barrel samplers can be used, as long as borehole wall integrity can be maintained and the additional friction can be overcome. If caving or sloughing occurs, the sampler tip should be sealed or other sampling tools used (9).

#### 9.5 Quality Control:

**9.5.1 Quality Control**—Quality control measures are necessary to ensure that sample integrity is maintained and that project data quality objectives are accomplished. By following good engineering principles and applying common sense, reliable site characterizations can be accomplished.

**9.5.2 Water Checks**—Water seeping into the direct push casing or connecting rods from contaminated zones may influence testing results. Periodically check for ground water before inserting samplers into borehole or into outer casings in the two tube system. If water is encountered, it may be necessary to switch to the sealed piston type samplers to protect sample integrity. Sealed piston type samples may not always be water tight. Sealing of rod or casing joints can prevent ground water from entering through the joints.

**9.5.3 Datum Points**—Establishment of a good datum reference is essential to providing reliable sample interval depths and elevators. Select datum reference points that are sufficiently protected from the work effort, and that can be located for future reference. Field measurements should be to 0.1 ft (3.05 mm). Measure extension rods as the bore advances to locate sample depth. Mark rods before driving each sample interval to determine accurate measurement of sample recovery and to accurately log borehole depth.

**9.5.4 Sample Recovery**—Sample recovery should be monitored closely and results documented. Poor recovery could indicate a change in sampling method is needed, that improper sampling practices are being conducted, or that sampling tools are incorrect. Sample recovery involves both volume and condition. Poor sample recovery should cause an immediate review of the sampling program.

**9.5.5 Decontamination**—Follow established decontamination procedures. Taking shortcuts may result in erroneous or suspect data.

## 10. Completion and Sealing

**10.1 Completion**—For boreholes receiving permanent monitoring devices, completion should be in accordance with Practice D 5092, site work plan, or regulatory requirements.

10.2 *Borehole Sealing*—Seal direct push boreholes to minimize preferential pathways for containment migration. Additional information and guidance on borehole sealing can be found in Guide D 6001 and in Guide D 5299. State or local regulations may control both the method and the materials for borehole sealing. Regulations generally direct bottom up borehole sealing as it is the surest and most permanent method for complete sealing. High pressure grouting is available for use with direct push technology for bottom up borehole sealing.

10.2.1 *Sealing by Slurry, Two Tube System*—Sound the borehole for free water. If water exists in the casing, place the extension rods, open-ended, to the bottom of the outer casing, as a tremie. Mix the slurry to standard specifications prescribed by regulation or work plan. Pump slurry through the extension/drive rod until it appears at the surface of the outer casing. Remove the extension rods. If no free water exists in the borehole, the slurry can be placed by gravity. Top off the outer casing as it is removed from the borehole.

10.2.1.1 *Slurry Mixes*—Slurry mixes used for slurry grouting of direct push boreholes generally are of lower viscosity because of the small diameter tremie pipes required. Usable mixes are 6 to 8 gal (22.7 to 30.28 L) of water/94-lb (42.64-kg) bag of cement with 5 lb (2.27 kg) of bentonite or 24 to 36 gal (90.84 to 136.28 L) of water to 50 lb (22.68 kg) of bentonite.

10.2.2 *Sealing by Gravity—Two Tube System*—Measure the cased hole to ensure it is open to depth. Slowly add bentonite chips or granular bentonite to fill the casing approximately 2 ft. Withdraw the casing 2 ft and recheck depth. Hydrate the bentonite by adding water. Repeat this procedure as the outer casing is withdrawn. The bentonite must be below the bottom of the casing during hydration. Wetness inside the rods may affect the flow of granular bentonite to the bottom of the casing. Fill the top foot of the borehole with material that is the same as exists in that zone.

10.2.3 *Borehole Sealing Single Tube System:*

10.2.3.1 *Gravity Sealing from Surface*—If the soil strata penetrated has sufficient wall strength to maintain an open hole, then it may be possible to add sealing materials from the surface. Dry bentonite chips or granular bentonite can be placed by gravity. The borehole volume should be determined and the borehole sounded every 10 ft (3 m) to ensure bridging has not occurred. The bentonite should be hydrated by adding approximately 1 pt (0.57 L) of water for each 5 ft of filled borehole. Seal the surface with native material.

10.2.3.2 *Wet Grout Mix Tremie Sealing*—Tremie sealing methods can be used with single tube systems when borehole wall strength is sufficient to maintain an open hole or when extension rods with an expendable point are used to reenter the borehole. The grout pipe should be inserted immediately after the direct push tools are withdrawn or through the annulus of the extension rods that have been reinserted down the borehole for grouting. Care must be taken to not plug the end of the grout pipe. Side discharge grout pipes also can be used to prevent plugging.

10.2.4 *Re-Entry Grouting*—If the borehole walls are not stable, the borehole can be re-entered by static pushing grouting tools, such as an expendable point attached to the extension/drive rods to the bottom of the original borehole. Pump a slurry through the rods as they are withdrawn. High pressure grouting equipment may be beneficial in pumping standard slurry mixes through small diameter gravity pipes. Care must be taken to ensure the original borehole is being sealed.

## 11. Record Keeping

11.1 *Field Report*—The field report may consist of boring log or a report of the sampling event and a description of the sample. Soil samples can be classified in accordance with Practice D 2488 or other methods as required for the investigation (12). Prepare the log in accordance with standards set in Guide D 5434 listing the parameters required for the field investigation program. List all contaminants identified, instrument readings taken, and comments on sampler advancement. Record any special field tests performed and sample processing procedures beyond those normally used in the defined investigation. Record borehole sealing procedures, materials used, and mix formulas on the boring log. Survey or otherwise locate the boring site to provide a permanent record of its replacement.

11.2 *Backfilling Record*—Record the method of sealing, materials used, and volume of materials placed in each borehole. This information can be added to the field boring log or recorded on a separate abandonment form.

## 12. Keywords

12.1 decontamination; direct push; ground water; sealing; soil sampling



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## **Appendix E**

### **Nitrate Extraction and Field Test Procedure**

## 5. Electrical Conductivity Test

Soil samples for the electrical conductivity (EC) test are taken from the 0- to 3-inch depth. Bulked soil samples from across the field can be collected, and two subsamples can be taken for analysis (See Chapter 1, Sampling Guidelines). **Electrical conductivity, pH, and soil nitrate are all measured from the same soil subsample.**

**Materials needed to measure electrical conductivity (EC):**

- 1/8-cup (30 mL) measuring scoop
- 120-mL plastic containers with lid
- EC pocket meter (blue with black cap)
- squirt bottle
- calibration solution (0.01 M KCl)
- distilled water

### Did You Know?

Excess salts in soil can be a detriment to plant health. Salts can also hamper water movement into the soil and increase the occurrence of surface compaction.

### ① Extract Subsample

The soil sample should be thoroughly mixed before taking a subsample. Measure a 1/8-cup level scoop subsample of soil and place it in the plastic container. If soil nitrates will be measured on this subsample (Chapter 7), weigh the subsample for a more accurate estimate of soil nitrates. Enter the subsample weight on the Soil Data worksheet.

### ② Add Water to Subsample and Mix

- Add 1/8-cup (30 mL) of distilled water to the container with the subsample. The resulting soil/water mixture equates to a 1:1 soil to water ratio on a volume basis.
- Put the lid on the container and shake vigorously about 25 times.

**Calibration Tip:** Make sure the EC meter is calibrated before making a measurement. See Appendix C for calibration instructions.



### ③ Measure and Record EC (See Calibration Tip)

- Open the container and insert the EC pocket meter into the soil-water mixture. Take the reading while the soil particles are still suspended in solution. To keep the soil particles from settling, stir gently with the EC pocket meter. Do not immerse the meter above the immersion level (See Appendix C, Figure 1c). Allow the reading to stabilize (stays the same for about 10 seconds).
- Enter the EC reading on the Soil Data worksheet in decisiemens per meter (dS/m). The DiST WP 4 meter gives readings directly in dS/m. For the Microsensor 4 meter, divide the reading by 10, and for the Microsensor 3 meter, divide the reading by 100 to get readings in dS/m.
- Save the soil-water mixture for the pH measurement (Chapter 6).

### ④ Turn the meter off. Thoroughly rinse meter with distilled water and replace cap.

## 6. Soil pH Test

Use the same soil-water mixture prepared in the EC test to conduct the pH Test. **If you are starting with a fresh soil sample, read the introduction and follow Steps 1-3 in the EC Test Chapter on preparing the sample.**

### Materials needed to measure pH:

- 1/8-cup (30 mL) measuring scoop
- plastic specimen bottle
- calibration buffer solutions
- squirt bottle
- pH pocket meter (red with black cap)
- distilled water

### Did You Know?

Soil acidification can also be an indication of excessive N fertilizer applications and N leaching loss.

**Considerations:** If the soil sample is saturated or very wet, a 1:1 ratio, on a volume basis, of soil to water will not be obtained in the soil-water mixture (See Step 2, Chapter 5). Let the soil dry before proceeding with Step 1 in Chapter 5. Also, a small amount of salts diffuse out of the pocket pH meter; therefore, **EC measurements should always be taken first when measuring both EC and pH on the same sample.**

### ① Measure and Record pH

- Make sure to periodically calibrate your pH meter (See Appendix C for instructions). If the meter has not been used in a while, place the meter in tap water for about 5 minutes before calibrating or taking a reading.
- Wait about 10 to 15 minutes after the EC measurement before measuring the pH. This gives the soil particles time to settle. Insert the pH pocket meter into the topmost portion of the solution and turn the meter on. Wait until the reading stabilizes (0-30 seconds), and record the digital reading on the Soil Data worksheet.

### ② Rinse Pocket Meter

- Thoroughly rinse the electrode with distilled water.
- Store the electrode with a few drops of the pH 7 buffer solution and replace the cap. (See Appendix C on storage of pH meter)

**Maintenance Tips:** Check the batteries and calibrate the EC and pH meters periodically. Be sure to clean the meters thoroughly to keep them working properly.



## 7. Soil Nitrate Test ( $\text{NO}_3^-$ )

Use the same sample prepared for the EC and pH tests to measure soil nitrates. **If you are starting with a fresh soil sample, read the introduction and follow Steps 1-3 in the EC Test Chapter on preparing the sample.**

**Materials needed to measure soil nitrate:**

- filter paper
- 120-mL plastic container with lid
- eye dropper
- nitrate/nitrite test strips
- stopwatch or timer
- distilled water

### Did You Know?

Soil nitrates are good measures of plant-available nitrogen, but they can be readily lost from the soil by leaching and volatilization.

### ① Fold Filter

Fold the filter paper in half (into a semicircle). Fold it again, but not quite into a quarter-circle. Leave the edges a little uneven as in **Figure 7.1** (A black line is drawn for demonstration purposes.)



Figure 7.1

### ② Insert Filter Paper into Subsample

Open the filter paper into the shape of a cone and push it (pointed part first) quickly into the jar with the soil/water mixture until it touches the bottom of the jar (**Figure 7.2**). **Wait** until about an eye dropper-full of the solution has seeped through to the inside of the filter paper. (**Note: Inserting the filter paper quickly prevents it from wetting up and tearing as it is inserted.**)



Figure 7.2

### ③ Place Drops on Nitrate Strips

Using the eye dropper and one nitrate/nitrite test strip, place 1 or 2 drops of the filtered solution on each of the strip's two pads. **Note the time.**

**NOTE: One pad measures the amount of nitrite, and the other measures the amount of nitrite and nitrate combined. Nitrite rarely occurs in measurable amounts in soils, so nitrite readings from the test strips are not recorded.**

④ **Measure and Record Nitrate** 

- Align the nitrate/nitrite test strip with the bottom of the bottle with your thumb corresponding to the diagram on the bottle.
- **After 60 seconds**, compare the first pad (furthest from your thumb) along the nitrate scale as shown in **Figure 7.3**. Estimate the nitrate amount according to the degree of color change. Enter the value from the nitrate scale on the Soil Data worksheet in ppm. This value is an estimate of nitrate-N concentration in the extract.



**Figure 7.3**

**NOTE: The nitrate test strips have a shelf-life. Check the expiration date on the bottle.**

**CALCULATIONS:**

**Estimated** (lb NO<sub>3</sub>-N/acre) =  

$$\frac{(\text{ppm extract NO}_3\text{-N}) \times (\text{depth of soil sampled in cm}) \times \text{bulk density} \times 0.89}{10}$$

**Exact** (lb NO<sub>3</sub>-N/acre) =  

$$\frac{(\text{ppm NO}_3\text{-N}) \times (\text{volume water used}) \times (\text{depth of soil sampled, cm}) \times \text{bulk density} \times 0.89}{(\text{dry weight of soil}) \times 10}$$

Volume water used = 30.0 mL + [dry weight of soil x soil water content (g/g)]

**Note:** The maximum nitrate-N reading on the nitrate/nitrite test strip container is 50 ppm. If the sample reading falls into the 50 ppm category, the sample can be diluted to get a better estimate of the actual amount over 50 ppm. To dilute the sample, fill the eye dropper with filtered solution and place five drops in a plastic container. Add five drops of distilled water; mix gently by swirling the container. Take a reading with a new test strip as stated in Step 4. Multiply the estimated nitrate-N in ppm by 2 before using the calculations. If the nitrate reading falls into the category of 50 ppm again, repeat the dilution steps, and multiply the estimated nitrate-N in ppm by 4.

**Did You Know?**

Water samples may be taken from drinking water, well water, tile drainage, drainage ditches, and ponds. Dip a nitrate/nitrite test strip into the water and estimate the nitrate or nitrite concentration from the color chart on the test strip bottle. This test can give you an idea of how much N fertilizer is lost from the soil. (See Chapter 12).