

Direct/Delayed Response Project: Quality Assurance Plan for Soil Sampling, Preparation, and Analysis

by

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A Contribution to the
National Acid Precipitation Assessment Program

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Notice

The information in this document has been funded wholly or in part by the U.S. Environmental Protection Agency under Contract Number 68-03-3249 to Lockheed Engineering and Management Services Company, Inc. and contract number 68-03-3246 to Northrop Services, Inc. It has been subject to the Agency's peer and administrative review, and it has been approved for publication as an Agency document.

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This document is one volume of a set which fully describes the Direct/Delayed Response Project, Northeast and Southeast Soil Surveys. The complete document set includes the major data report, quality assurance plan, analytical methods manual, field operations reports, and quality assurance reports. Similar sets are being produced for each Aquatic Effects Research Program component project. Colored covers, artwork, and the use of the project name in the document title serve to identify each companion document. The proper citation of this document remains:

Bartz, J.K., S.K. Drou  , K.A. Cappel, M.L. Papp, G.A. Raab, L.J. Blume, M.A. Stapanian, F.C. Garner, and D.S. Coffey. 1987. *Direct/Delayed Response Project: Quality Assurance Plan for Soil Sampling, Preparation, and Analysis*. EPA/600/8-87/021. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. 315 pp.

Abstract

The Direct/Delayed Response Project (DDRP) focuses on regions of the United States that have been identified as potentially sensitive to surface water acidification. The Northeastern Soil Survey includes the New England states of Maine, New Hampshire, Vermont, Massachusetts, Connecticut, and Rhode Island, and portions of New York and Pennsylvania. The Southeastern Soil Survey, conducted in the physiographic region known as the Southern Blue Ridge Province, includes the bordering portions of Tennessee, North Carolina, South Carolina, and Georgia.

The specific goals of the DDRP soil surveys are (1) to define soil-physical and soil-chemical characteristics and other watershed characteristics across these regions, (2) to assess the variability of these characteristics, and (3) to determine which of these characteristics are related most strongly to surface-water chemistry.

The purpose of the quality assurance (QA) project plan is to specify the policies, organization, objectives, and QA and quality control (QC) activities needed to achieve the data quality goals of the DDRP. The QA plan is designed to meet the following objectives:

- standardizing sampling, processing, and analytical methods and procedures
- simplifying field operations
- training all personnel
- using QA/QC samples and procedures to verify data
- using field and laboratory audits to ensure that all activities are properly performed and that problems are identified and resolved
- evaluating the reported data and verifying data quality.

This report was submitted in partial fulfillment of Contract Number 68-03-3249 by Lockheed Engineering and Management Services Company, Inc., under the sponsorship of the U.S. Environmental Protection Agency.

Direct/Delayed Response Project:
Quality Assurance Plan for
Soil Sampling, Preparation, and Analysis

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Acknowledgments

Critical reviews by the following individuals were instrumental in the documentation of this project plan and are gratefully acknowledged: E. Knox, M. Meyer, R. W. Arnold, F. T. Miller, E. H. Sautter, K. J. LaFlamme, K. A. Wheeler, D. G. Van Houten, G. H. Lipscomb, T. Gerald, and H. Smith, U.S. Department of Agriculture, Soil Conservation Service; I. Fernandez, University of Maine-Orono; E. Levine, National Aeronautics and Space Administration, Goddard Space Flight Center; D. Coffey, Tetra Tech, Inc., Bellevue, Washington; J. J. Lee and L. H. Liegel, U.S. Environmental Protection Agency; and D. Lammers, U.S. Forest Service.

The support of S. J. Simon, R. E. Cameron, and S. L. Pierett, Lockheed Engineering and Management Services Company, Inc., is gratefully acknowledged.

The following people were instrumental in the completion of this project plan: K. Thornton, FTN and Associates, Little Rock, Arkansas; J. L. Engels, M. L. Faber, and J. M. Nicholson of Lockheed Engineering and Management Services Company, Inc.; Computer Sciences Corporation word processing staff at the U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory, Las Vegas, Nevada; and Donald Clark Associates graphic arts staff at the U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

Finally, recognition belongs to E. P. Meier and P. A. Arberg who have served as technical monitors of this project.

Section 1 **Introduction**

The quality assurance policy of the U.S. Environmental Protection Agency (EPA) requires every monitoring and measurement project to have a written and approved quality assurance (QA) project plan (Costle, 1979a and 1979b). This requirement applies to all environmental monitoring and measurement efforts authorized or supported by EPA through regulations, grants, contracts, or other formal means. The purpose of this QA project plan is to specify the policies, organization, objectives, and quality control (QC) activities needed to achieve the data quality goals of the Direct/Delayed Response Project. All project personnel are expected to be familiar with the policies and objectives outlined in this QA project plan to assure proper interactions

between field and laboratory operations and data management.

EPA guidance (U.S. EPA, 1980) states that the QA project plan must address, in detail or by reference, all 14 items listed in Table 1.1. Method-specific discussions presented in the *Soil Sampling Manual for the Direct/Delayed Response Project Soil Survey* (Blume et al., 1987), *Preparation Laboratory Manual for the Direct/Delayed Response Project Soil Survey* (Bartz et al., 1987), or the *Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey* (Cappo et al., 1987) might not be repeated in this project plan. In these cases, Table 1-1 serves as an index to the appropriate references.

Table 1-1. Sections in this QA Project Plan and in the DDRP Soil Sampling and Analytical Methods manuals where QA subjects are treated

Subject	Section Number		
	QA Project Plan	Soil Sampling Manual	Analytical Methods Manual
1. Project Description	2	1	1
2. Project Organization and Responsibility	3	1, 2, 7	—
3. QA Objectives for Measurement Data	4	—	2
4. Sampling Procedures	6, 7	6	—
5. Sample Custody	6, 7, 8	6, 7	2
6. Calibration Procedures	9	—	2, 3 - 19
7. Analytical Procedures	9	11	3 - 19
8. Data Reduction, Validation, and Reporting	6, 9, 11	5, 6, 11	2, 3 - 19
9. Internal QC Checks	7, 8, 9	11	2

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Table 1-1. Continued

Subject	Section Number		
	QA Project Plan	Soil Sampling Manual	Analytical Methods Manual
10. Performance and System Audits	12	2	—
11. Preventive Maintenance	—	—	3 - 19
12. Procedures for Routine Assessment of Data Precision, Representativeness, Comparability, Accuracy, and Completeness	4, 10	—	2
13. Corrective Actions	9, 10	—	2
14. QA Reports to Management	9, 12	2	—

Section 2

Project Description

The Direct/Delayed Response Project (DDRP) focuses on regions of the United States that have been identified as potentially sensitive to surface water acidification. The Northeastern Soil Survey includes the New England states of Maine, New Hampshire, Vermont, Massachusetts, Connecticut, and Rhode Island, and portions of New York and Pennsylvania. The Southeastern Soil Survey, conducted in the physiographic region known as the Southern Blue Ridge Province, includes the bordering portions of Tennessee, North Carolina, South Carolina, and Georgia. Surface

waters in these two regions were studied during the Eastern Lake Survey (1984) and the National Stream Survey Phase I - Pilot Study (1985), respectively.

The specific goals of the DDRP soil surveys are (1) to define soil-physical and soil-chemical characteristics and other watershed characteristics across these regions, (2) to assess the variability of these characteristics, and (3) to determine which of these characteristics are related most strongly to surface-water chemistry.

Section 3

Project Organization

Figure 3-1 illustrates the operational management structure. The director of the Office of Acid Deposition, Environmental Monitoring, and Quality Assurance (OADEMQA) is the EPA official who has overall responsibility for programs within EPA which address the effects of acidic deposition. The responsibilities of the program director and technical director are as follows:

Program Director

The director of the Aquatic Effects Research Program (program director) is the EPA Headquarters representative for DDRP and is the liaison between the headquarters staff, the laboratory directors, and the National Acid Precipitation Assessment Program (NAPAP). Questions regarding general management and resources should be forwarded to the program director through the technical director.

Technical Director

The technical director performs responsibilities at the discretion of the program director. The technical director's primary role is to maintain the integrity of program objectives, to integrate components of the program, and to see that deadlines are met. The technical director coordinates and integrates the activities of the Environmental Research Laboratory at Corvallis, Oregon (ERL-C), the Environmental Monitoring Systems Laboratory at Las Vegas, Nevada (EMSL-LV), and Oak Ridge National Laboratory (ORNL) at Oak Ridge, Tennessee. The technical director also coordinates peer review, resolves issues of responsibility, and disseminates information to the public. The technical director represents the program director as necessary and informs the program director of EPA laboratory activities, progress, and performance.

The roles of the laboratories are as follows:

ERL-C: ERL-C is a focal point for the soil surveys. Responsibilities of ERL-C staff for all phases of the program include:

- Developing experimental design for soil sampling.
- Developing protocol for selection of sampling sites.
- Preparing sampling protocols (jointly with EMSL-LV).
- Collecting supplemental historical and other available data on each sampling site.
- Analyzing data (jointly with EMSL-LV).
- Interpreting data.
- Preparing reports (final and progress reports with contributions from the other laboratories relative to their responsibilities).
- Assessing and resolving all science-related issues other than quality assurance/quality control (QA/QC) data management (jointly with other laboratories as necessary).
- Coordinating survey activities with NAPAP management staff.

EMSL-LV: The Las Vegas laboratory has expertise in matters relating to QA/QC, logistics, analytical services, and sampling protocols. The responsibilities of personnel at EMSL-LV include:

- Developing QA/QC procedures for all components of the survey except data management (a joint responsibility of ORNL and ERL-C).

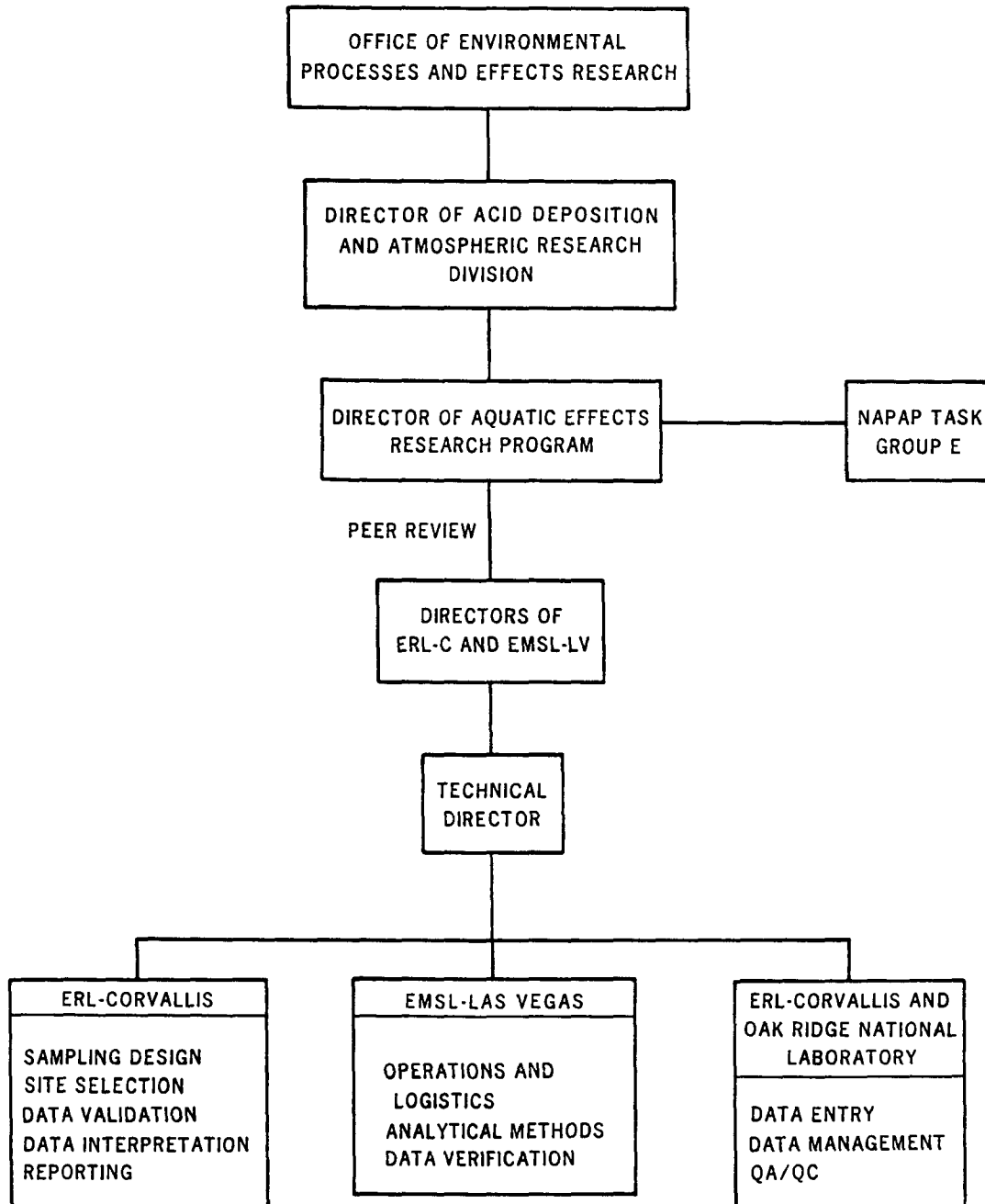


Figure 3-1. Operational management structure for the soil surveys of the Direct/Delayed Response Project, a project of the Aquatic Effects Research Program.

- Preparing all sampling protocols (jointly with ERL-C).
- Preparing a soil sampling and preparation manual.
- Preparing an analytical methods manual.
- Coordinating logistical support and equipment needs for all field operations.
- Training field personnel in DDRP soil survey protocols.
- Distributing all samples to analytical laboratories.
- Developing and implementing QA/QC procedures for verification of field data and analytical laboratory data.
- Preparing and implementing the QA project plan.

- Independently assessing field measurements and laboratory data quality, i.e., bias and variability.
- Assessing and resolving problems pertaining to QA/QC, logistics, and analytical services.

ORNL: ORNL has expertise in managing, manipulating, and restructuring large data bases to satisfy data analysis needs. ERL-C oversees the activities of ORNL, which has responsibilities for:

- Developing and maintaining a data management system.
- Entering all field, laboratory, and support data into the data base and simultaneously assuring entry quality.
- Preparing computer-generated summary tables, statistics, and graphics for reports.

Section 4

Quality Assurance Objectives

Quality assurance (QA) objectives are required for three phases of data collection: (1) soil description and sample collection, (2) sample preparation, and (3) laboratory analysis. The approach selected for data collection provides a balance between constraints of time and cost and the quality of data necessary to complete the research objectives of the project. The QA plan is designed to meet the following objectives:

- Standardizing sampling, processing, and analytical methods and procedures.
- Simplifying field operations.
- Training all personnel.
- Using QA/QC samples and procedures to verify data.
- Using field and laboratory audits to ensure that all activities are properly performed and that problems are identified and resolved.
- Evaluating the reported data and verifying data quality.

Each phase of data collection is addressed in the following sections.

4.1 Soil Sampling

4.1.1 Precision and Accuracy

A representative of the Soil Conservation Service (SCS) state soils staff independently describes a minimum of one site per field crew. These independent pedon descriptions are used to assess the variability in site descriptions among soil scientists. The SCS representative monitors adherence to protocol for site selection, labeling, and sampling. The soil profile is described on the same face of the pit as described by the field crew. The

representative makes the assessment while the crew is describing and sampling the pedons. Written reviews are submitted to the sampling task leader at ERL-C within two weeks. Major problems are reported verbally within two working days.

The Regional Coordinator/Correlator (RCC) must be a qualified soil scientist with several years experience in soil profile description and soil mapping. The RCC monitors one site per field crew for adherence to SCS standards, procedures, and sampling protocol modifications as presented in this document, and performs an independent duplicate profile description. At least one site in each state is monitored with the SCS state soils staff representative while the remaining sites may be monitored independently. The RCC also ensures that state soils staff performs duplicate profile descriptions. During this process, the RCC identifies, discusses, and resolves any significant problems. Written reports are submitted to the sampling task leader at ERL-C within two weeks. The resolution of major problems is reported verbally within two working days.

The quality assurance/quality control (QA/QC) representative audits each field sampling crew at least once to ensure adherence to sampling protocol. Written reports are submitted to the QA manager at EMSL-LV within two weeks. Major problems are reported verbally within two working days. The QA manager is responsible for conveying any major problems to the technical monitor or technical director.

A small percent of the sampling units is selected randomly by EPA for sampling to determine the within-delineation variability. These replicate pedons, called paired pedons, are selected before sampling begins. The paired pedon and the routine pedon from a representative site for each selected unit are sampled on the same day by the same field crew.

Sample pits are located accurately on the soil survey maps, and the pit dimensions and the long azimuth are recorded. The pit face from which samples are removed is recorded, and the location of the pit in the field is flagged or identified so that the site can be revisited. The soil profile is described according to SCS protocols.

One horizon per day is sampled in duplicate by each field crew (see Section 7.0). One field duplicate is included in each set of samples sent to a preparation laboratory.

4.1.2 Representativeness

The primary concerns in the selection of sampling sites are (1) to assess soil characteristics, (2) to integrate information on parent material, internal drainage, soil depth, slope, and vegetative cover, and (3) to determine representative sampling classes. Soils which have been identified in the study regions have been combined into groups, or sampling classes, which are either known to have or are expected to have similar chemical and physical characteristics. Each of the sampling classes can be sampled across a number of watersheds in which they occur. In this approach, a given soil sample does not represent the specific watershed from which it came. Instead it contributes to a set of samples which collectively represent a specific sampling class on all DDRP watersheds within the sampling region. The lead soil scientist of the sampling party selects a sampling site representing the designated sampling class and vegetation class within the designated watershed according to the protocols documented in Blume et al. (1987).

4.1.3 Completeness

Soil sampling protocols require the sampling of 100 percent of the designated pedons and of the prerequisite number of horizons. If samples are lost, spilled, or mislabeled, it is possible to return to the field and resample the same site. If a sampling site is inaccessible, the reason for excluding the site must be formally documented by the field crew (refer to Section 5.4.1).

4.1.4 Comparability

The use of standard SCS methods, protocols, and forms for the sampling phase provide field and analytical data that are comparable to data generated from SCS investigations and other studies which have utilized these standardized methods.

4.2 Sample Preparation

4.2.1 Precision and Accuracy

The preparation laboratory combines sets of field samples into one batch containing a maximum of 39 routine and duplicate samples. After processing, i.e., air-drying, crushing, sieving, and homogenization, one bulk sample is split into two subsamples which are termed preparation duplicates. Comparison of physical and chemical data for these duplicates allows evaluation of the subsampling procedure.

4.2.2 Representativeness

Each bulk soil sample is processed by a preparation laboratory to obtain a homogeneous sample. Homogenization is accomplished by passing the sample through a Jones-type riffle splitter at least seven times. The riffle splitter also is used for subsampling. All samples not being processed are stored at 4°C by the preparation laboratory.

4.2.3 Completeness

Each batch of samples sent to a contractor analytical laboratory includes the preparation duplicates.

4.2.4 Comparability

All preparation laboratories process bulk samples according to protocols documented in Bartz et al. (1987). Strict adherence to protocols should result in comparability among preparation laboratories.

4.3 Laboratory Analysis

4.3.1 Precision and Accuracy

The data quality objectives (DQOs) for precision and accuracy of the physical and chemical analyses of routine soil samples are presented in Table 4-1 (U.S. EPA, 1985). The structure of Table 4-1 is as follows:

Reporting Units - specifies the units in which the laboratory data should be reported.

Reporting Format - specifies the significant figures to which the data should be reported.

Expected Range - specifies the range of values expected to occur naturally in the soil sampled, independent of measurement error.

Lower Reporting Limit - this value has been extrapolated to that of the reporting unit; if the sample values are lower than stated, the "limit of reproducibility" is approached.

Precision at the Lower Limit - serves as a guideline to define the acceptable absolute percent standard deviation beyond which the analytical reproducibility for low concentration samples is questionable and often not attainable.

Precision at the Upper Limit - serves as a guideline to define the acceptable percent relative standard deviation beyond which the analytical reproducibility for high concentration samples is questionable.

The values given for precision at the lower limit are absolute; the upper limit values are relative. This eliminates unrealistic, restrictive precision requirements for low concentration samples.

Initial DQOs were established on the basis of the requirements of EPA data users and the selection of appropriate methods to obtain the data. The initial DQO values were reviewed by persons familiar with analytical methods and techniques for soil characterization including soil chemists, laboratory directors, and laboratory personnel. Modifications were implemented based on reviewers' comments and the limitations of the particular analytical procedure or instrument. Because of the greater heterogeneity of the material in the organic horizons, attaining specific precision limits for organic horizons may be difficult for many of the analyses. Precision objectives for organic horizons should be reevaluated as data become available and should be changed if necessary. If the data quality goals cannot be met during the course of the project, the actual quality of the data will be used to reassess the intended use of the data and to document the implications derived from the survey. Therefore, the actual data quality achieved may require different conclusions or modifications in the level of confidence of conclusions and decisions.

4.3.2 Representativeness

A representative subsample is shipped from the preparation laboratory to the contractor analytical laboratory. For each analysis, the analytical laboratory must remove an aliquot from the subsample. Personnel at the analytical laboratory mix the soil material thoroughly to ensure the representativeness of the aliquot. All samples not in use are stored at 4°C by the contractor analytical laboratory.

4.3.3 Completeness

The objective for the complete analysis of all samples collected is 90 percent or better for all parameters. One hundred percent completeness is possible if sufficient sample is available to complete all analyses, reanalyses, and duplicate analyses.

Table 4-1. Data Quality Objectives (U.S. EPA, 1985)

Parameter	Reporting Unit	Reporting Format	Expected Range	Lower Reporting Limit	Precision at Lower Limit ^a	Precision at Upper Limit ^b
1. Sand ^c	dry wt	±0.1%	0-98% (of <2-mm	1.0%	±1.0% of	1% of absolute value
2. Silt ^c	"	"	10-80%	"	"	±1.0%
3. Clay ^c	"	"	0-70%	"	"	"
4. Rock Fragments (2-20mm) ^c	"	±5% of total sample weight	0-100%	0%	±20% of total sample weight	±20% of total sample weight
5. Bulk Density	g/cm ³	±0.01%	0.2-2.0	0.20 g/cm ³	±0.1 g/cm ³	±0.1 g/cm ³
6. pH in Water	pH units	±0.01 units	2.5-7.0	—	±0.15 units	±0.15 units
7. pH in 0.01 M CaCl ₂	"	"	2.0-7.0	—	"	"
8. pH in 0.002 M CaCl ₂	"	"	2.0-7.0	—	"	"
9. Organic C	% dry wt	±0.01%	0-50%	0.05%	±0.05 wt %	±15% of reported value
10. Inorganic C	"	"	0-20%	±0.1%	±0.1%	±10% of reported value
11. Total N	"	0.01%	0-2.0%	0.01%	±0.01 wt %	"
12. Total S	"	±0.001%	0-0.250%	0.001%	"	"
13. CEC (NH ₄ OAc)	meq/100 g	±0.01 meq/100 g	1.0-200	0.1 meq/100 g		±0.25 meq/100 g ^c
14. CEC (NH ₄ Cl)	"	"	0.2-100	"	"	"
15. Exchangeable Ca (in NH ₄ OAc)	"	"	0-10.0 (≤100 in 0 horizon) ^c	0.03 meq/100g	±0.03 eq/100 g	±15% of reported value
16. " Mg	"	"	0-1.5 (10.0 ") ^c	"	"	"

^a Because of the greater inherent heterogeneity of the material in organic horizons, attaining these precision limits for organic horizons may be difficult for many of the analyses. Precision objectives for organic horizons will be reevaluated as data become available and will be changed if necessary.

^b Parameter determined on mineral horizons only.

^c The initial range listed is for mineral soil horizons; the second range in parentheses is for organic horizons.

(continued)

Table 4-1. (Continued)

Parameter	Reporting Unit	Reporting Format	Expected Range	Lower Reporting Limit	Precision at Lower Limit ^a	Precision at Upper Limit ^a
17. * Na	"	"	0-0.5 (2.0 *) ^c	"	"	"
18. * K	"	"	0-1.0 (5.0 *) ^c	"	"	"
19. Exchangeable Ca (in NH ₄ Cl)	"	"	0-10.0 (100.0 *) ^c	"	"	"
20. * Mg	"	"	0-2.5 (10.0 *) ^c	"	"	"
21. * Na	"	"	0-0.25 (1.0 *) ^c	"	"	"
22. * K	"	"	0-1.0 (5.0 *) ^c	"	"	"
23. Ca Exchangeable in	mg/L	±0.1%	0-100	10 mg/L	±5.0% of reported value	±5.0% of reported value
24. Mg 0.002 M						
25. Na CaCl ₂ 26. K 27. Fe 28. Al	meq/100 g	Data are expected to be comparable to other exchangeable cation data; however, no reliable data are available to support specific data quality objectives.				
29. Fe (Pyrophosphate % dry wt		±0.01%	0-7.5	0.05%	±0.05% wt %	±15% of reported value
30. Al Extractable)	"	"	0-6.0	"	"	"
31. Fe (Acid-Oxalate	"	"	0-7.5	"	"	"
32. Al Extractable)	"	"	0-6.0	"	"	"
33. Fe (Citrate-Dithionite	"	"	0-7.5	"	"	"
34. Al Extractable)	"	"	0-6.0	"	"	"
35. SO ₄ - Water Extractable	mg S/kg dry wt	±0.1	0-100	1.0 mg/kg	±1.0 mg/kg	±10% or reported value

^a Because of the greater inherent heterogeneity of the material in organic horizons, attaining these precision limits for organic horizons may be difficult for many of the analyses. Precision objectives for organic horizons will be reevaluated as data become available and will be changed if necessary.

^b Parameter determined on mineral horizons only.

^c The initial range listed is for mineral soil horizons; the second range in parentheses is for organic horizons.

(continued)

Table 4-1. (Continued)

Parameter	Reporting Unit	Reporting Format	Expected Range	Lower Reporting Limit	Precision at Lower Limit ^a	Precision at Upper Limit ^a
36. SO ₄ - PO ₄ Extractable	"	"	0-200	±1.0 mg/kg	±0.05 mg/L	±5% of reported value
37. Six points on thru sulfate adsorption 42. isotherm	mg S/L (in equilibrated solution)	0.01	0-35	±0.05 mg/L	±0.05 mg/L	±5% of reported value
43. BaCl ₂ -TEA Exchangeable Acidity	meq/100 g	±0.01	0-100 (<250 in 0 horizon) ^c	0.5meq/100g	±0.5meq/100g	±20% of reported value
44. KCl Exchangeable Acidity	"	"	0-20	"	"	"
45. KCl Exchangeable	"	"	"	"	"	"

^a Because of the greater inherent heterogeneity of the material in organic horizons, attaining these prec. difficult for many of the analyses. Precision objectives for organic horizons will be reevaluated as data become available and will be changed if necessary.

^c The initial range listed is for mineral soil horizons; the second range in parentheses is for organic horizons

4.3.4 Comparability

Comparability is assured by the uniform use of procedures documented in Capps et al. (1987) and by the use of uniform units for reporting data as specified on the data summary sheets. The QA procedures required for contractor analytical laboratories (see sections

9 and 10) allow for determination of interlaboratory and intralaboratory bias so that results can be compared. In addition, the analytical techniques and methods used to determine the soil parameters allow the data to be compared to other data bases compiled from results that were obtained by using the same or comparable techniques and methods.

Section 5

Sampling Strategy

5.1 Northeastern Soil Survey

5.1.1 Watershed Selection

The objectives of the DDRP focus on making regional inferences. For this reason, the 150 watersheds selected for mapping of soils and watershed characteristics must constitute a representative sample of the region. The 773 watersheds included in Region I of the National Surface Water Survey (NSWS) provided an excellent starting point from which to draw a subsample of 150 for the Northeastern soil survey of the DDRP for two reasons: (1) the Region I NSWS lakes were selected according to a rigorous probability sampling method, i.e., stratified by five subregions and three alkalinity classes within each subregion, and (2) water chemistry information was available from NSWS for these lakes.

The 150 watersheds studied in the DDRP also are part of the Phase II Lake Monitoring Program of the NSWS. This provides a data set that contains both water-chemistry and watershed information; therefore, the procedure used to select these watersheds incorporated criteria relevant to both the DDRP and the NSWS. The preliminary selection procedure for the NSWS consisted of five steps which are summarized as follows:

1. Lakes of low interest, e.g., too shallow, highly enriched, capacity-protected, polluted by local activities, or physically disturbed, were excluded.
2. Lakes too large to be sampled, i.e., greater than 2,000 ha, were excluded.
3. A cluster analysis was performed on a set of chemical and physical variables to group the remaining 510 lakes into three clusters of lakes with similar characteristics.

4. A subsample of 60 lakes was selected from each cluster, then the three subsamples were weighted to represent the overall population of lakes in the Northeast.

5. Lakes with watersheds too large to be mapped at the required level of detail, i.e., watersheds greater than 3,000 ha, were excluded from the subsamples.

This procedure identified 148 lakes and watersheds spread across the three clusters. The three groups differ primarily in their alkalinities, pH levels, and calcium concentrations. To maintain the ability to regionalize conclusions drawn from the sample of 148 watersheds, the precision of information characterizing each of these watersheds should be comparable, and each cluster should be described at the same level of detail as the others.

5.1.2 Watershed Mapping

During the spring and summer of 1985, 145 of the 148 watersheds were mapped. Approximately 440 mapping units were identified in the 148 watersheds. Sampling each of the 440 mapping units is not necessarily the best way to describe the chemistry of the soils in a region. A better procedure is to combine the mapping units into groups, or sampling classes, which are either known or expected to have similar chemical characteristics. Each of these sampling classes can be sampled from a number of watersheds, and the mean characteristics of each sampling class can be computed. The mean values and the variance about the mean can be used to construct area- or volume-weighted estimates of the characteristics for each watershed. For this procedure to work, at least five samples must be taken to characterize the variability of each sampling class. The goal of this sampling plan is to develop a method of grouping the

large number of soils into a reasonable number of sampling classes.

5.1.3 Sampling Classes

5.1.3.1 Soil Mapping Data Base--

The data base contains about 2,200 observations initially recorded on field forms during the soil mapping of 145 watersheds selected as part of the DDRP and the Phase II lakes survey. This information, which was considered in aggregating similar soils into sampling classes, includes:

- soil taxonomic class (series, subgroup, great group)
- family texture
- parent material
 - origin
 - mode of deposition
- drainage class
- slope class
- slope configuration
- geomorphic position
- dominant landform
- surface stoniness
- percent inclusions
- percent of soils occurring in complexes
- estimated depth to bedrock
- estimated depth to permeable material.

The data base also includes the area of each mapping unit, the number of occurrences, and the percent of the watershed area. Separate data files exist for vegetation type, vegetation class, and geology. A comparison of vegetation types to Society of American Foresters (SAF) cover types is given in Table 5-1.

5.1.3.2 Evaluation of Sampling Classes--

Initially, a taxonomic approach was used to identify 38 sampling classes as a foundation for aggregating similar soils. Taxonomic classification is based on similarities among soil properties. This taxonomic scheme was

modified to reflect the major factors which are thought to influence soil chemistry, e.g., drainage class and parent material.

5.1.4 Watershed and Sampling Class Selection

5.1.4.1 Sampling Class Objectives--

The goal of this part of the sample selection procedure is to determine which sampling classes are sampled in which watersheds. The sites are selected to meet the following objectives:

1. To characterize all the sampling classes with similar levels of precision.
2. To describe the variation in watershed characteristics.
3. To describe the variation in the acid-neutralizing capacity (ANC) clusters developed from the lake survey.

5.1.4.2 Sampling Class Constraints--

To meet these three objectives, a series of constraints based on the allocation of samples to sampling classes and watersheds must be met. These constraints are:

1. Approximately equal numbers of samples must be taken from each sampling class.
2. Approximately two samples must be taken from each watershed.
3. Not more than one sample may be taken from each sampling class in each watershed.
4. Samples must be selected over the range of ANC clusters within each sampling class.

The method uses a simple selection algorithm to randomly select watersheds and sampling classes within these constraints.

Table 5-1. Comparison of Coniferous, Deciduous, and Mixed Vegetation Types to Society of American Foresters (SAF) Forest Cover Types

SAF Cover Type Name	Cover Type Number
Coniferous Vegetation Types	
Jack Pine	1
Balsam Fir	5
Black Spruce	12
Black Spruce - Tamarack	13
White Spruce	107
Tamarack	38
Red Spruce	32
Red Spruce - Balsam Fir	33
Red Spruce - Fraser Fir	34
Northern White Cedar	37
Red Pine	15
Eastern White Pine	21
White Pine - Hemlock	22
Eastern Hemlock	23
Deciduous Vegetation Types	
Aspen	16
Pin Cherry	17
Paper Birch	18
Sugar Maple	27
Sugar Maple - Beech - Yellow Birch	25
Sugar Maple - Basswood	26
Black Cherry - Maple	28
Hawthorn	109
Gray Birch - Red Maple	19
Beech - Sugar Maple	60
Red Maple	108
Northern Pin Oak	14
Black Ash - American Elm - Red Maple	39
Mixed Vegetation Types	
Hemlock - Yellow Birch	24
Red Spruce - Yellow Birch	30
Paper Birch - Red Spruce - Balsam Fir	35
White Pine - Chestnut Oak	51
White Pine - Northern Red Oak - Red Maple	20

5.1.4.3 Selection Algorithm--

The selection method proceeds through a series of stages. Whenever possible, the rationale for the particular approach taken is described and cross-referenced with the objectives and constraints.

The selection method is based on the use of a systematic, weighted, random sample of the watersheds that contain any given sampling class. First, the number of samples to be taken in each sampling class is determined (Constraint 1).

5.1.4.3.1 The first step in the selection process involves constructing a matrix of the occurrences of each sampling class in each watershed. This matrix is used to: (1) prepare a list of the watersheds that contain each sampling class, and (2) determine the number of different sampling classes in each watershed.

After the number of watersheds represented in each sampling class is determined, it is possible to allocate the samples to be taken from each watershed into sampling classes (given Constraint 3).

Using eight samples per sampling class as a goal for selection, the following sample allocation occurs: eight samples are allocated to each sampling class when there are more than eight watersheds; when there are eight or fewer watersheds, one sample is allocated to each watershed.

5.1.4.3.2 Next, watersheds are selected within each sampling class. Constraints 2 and 4 are important in this process.

If watersheds are selected randomly within each sampling class, the watersheds that contain a large number of sampling classes have more samples allocated to them than the watersheds that have few sampling classes. To counteract this effect and to approach an approximately equal number of samples per watershed, the watersheds are weighted (during the random selection procedure) by the inverse of the number of sampling classes that they contain.

For example, if one watershed contains four different sampling classes, it is exposed to the sample selection procedure four times. In other words, it is given one quarter of the weight of a watershed that contains only one sampling class. When this technique is used, both watersheds have an approximately equal probability of being selected. This scheme works properly if there are equal numbers of watersheds considered in each sampling class; the presence of unequal numbers causes some deviation from the most desirable distribution of samples.

To avoid overemphasizing the very common soils, only one sample is taken from each watershed that contains only one sampling class. All named soils in a soil complex are counted as occurrences in their respective sampling classes. For example, a Tunbridge-Lyman soil complex in a watershed mapping unit is considered one occurrence of sampling class S12, which contains the Tunbridge series, and one occurrence of sampling class S13, which contains the Lyman series.

Watersheds within sampling classes are sorted by ANC cluster. When the weights described above are used, a systematic, weighted, random sample is taken. A random starting point is selected from the list of watersheds; then watersheds are selected at regular intervals from the (weighted) list. This method ensures a selection across the range of ANC clusters.

To ensure that a watershed is not sampled more than once for a given sampling class, the weight assigned should not be larger than the interval used in the systematic sampling. Weights should be scaled down if they exceed the systematic sampling interval.

5.1.4.3.3 After this procedure has been followed for each sampling class, the initial selection of watersheds and sampling classes can be summarized. Three options are possible at this point:

1. The weighting factors can be adjusted iteratively until the allocation is acceptable.

2. Samples can be moved arbitrarily among watersheds to reach the desired allocation.
3. The selection can be accepted as adequate.

If the selection is not considered adequate, the most acceptable solution is to repeat the procedure with adjusted weights. This process could be automated, if necessary, with the weight of a watershed being increased until the watershed receives sufficient samples.

The method of sampling class and watershed selection outlined here is designed to satisfy the objectives and constraints listed in sections 5.1.4.1 and 5.1.4.2. Given the nature of the constraints, it is likely that there is no single, perfect solution; however, this method allows the production of an acceptable selection that is a compromise between the demands of the different objectives.

5.2 Southeastern Soil Survey

The sampling strategy for the Southeastern soil survey is similar to that for the Northeastern soil survey.

5.3 Final Sampling Locations

Generally, soil surveys identify and describe soils at the level of series and phases. The DDRP is interested in obtaining soil samples that are integrative or representative of the sampling classes in the region. A sampling class may contain six or seven similar soils. The sampling purpose is to describe the characteristics of the sampling class rather than to describe the characteristics of a specific soil phase. All soils within a sampling class are considered similar in soil chemistry; therefore, the specific sampling location within a sampling class can be selected at random. The procedures described in this section are intended (1) to describe the range of variability of soil characteristics within each sampling class, and (2) to ensure that each sampling class is characterized at the same level of precision.

Determining the potential sampling locations within the watershed is a two-step process.

5.3.1 Sampling Site Selection

There are five steps in selecting representative sampling sites within a sampling class:

NOTE: Steps 1 through 5 are completed by ERL-C. Maps that show the five random points, as discussed in Step 3, are given to each SCS sampling crew.

1. Prepare a list of all mapping units and the sampling class or classes in which they occur. Most mapping units occur only in one sampling class; complexes may occur in two or more sampling classes. For each complex, record the proportion of area occupied by each soil series in the complex (from the mapping unit description). This proportion should be the average proportion, excluding the area occupied by inclusions.
2. For each watershed, obtain the watershed maps, and identify the sampling classes selected for that watershed. Mapping-unit delineations for each soil series must be aggregated and identified for each sampling class.
3. Transfer a grid that has a cell size of about 2 acres to a Mylar sheet. Overlay the grid on the watershed map. Select a set of random coordinates (using a computer program), and determine if the point they represent intersects one of the sampling classes selected on that watershed. If the point does not fall within the selected sampling class, draw another pair of random coordinates. Continue this process until five random points have been identified in each sampling class. Record their order of selection from 1 through 5. Some sampling locations may not be accessible; therefore, alternate locations must be provided.

4. If the point falls on a mapping unit that is a complex, draw a random number, Y, between zero and the total percentage of the soils in the complex, e.g., a 50 to 30 percent complex of Tunbridge-Lyman would sum to 80, so the maximum random number is 80. Determine the percentage of the area in the desired sampling class, e.g., Tunbridge is 50 percent. Call this number X. If X is less than Y, draw another set of coordinates. This procedure minimizes the probability that complexes are overselected for sampling.
5. For each location selected, overlay appropriate maps and note the vegetation class associated with each point as (1) coniferous, (2) deciduous, (3) mixed, (4) open dryland, or (5) open wetland.

Within the sampling class, sample the pedon that has one or more of the soils in the sampling class and that has one or more of the vegetation classes noted above.

5.3.2 Sampling Site Locations

The general vicinity of the site is located on the watershed soil map. Soil maps marked with the random points are distributed before the sampling crew leaves for the field. Each point, i.e., starting point, marked on the map represents the origin of a circle with a 150-m radius, i.e., a sampling site. Within the area of the sampling site, there may be inclusions, rock outcrops, a soil complex, or other factors that make finding a soil of the specific sampling class difficult. The following procedure is used by the sampling crew to select the specific sampling site in the watershed:

1. Refer to the assigned sampling class and vegetation class for a specific watershed. For each sampling class to be sampled on the watershed, refer to a list of the soil series that are part of the sampling class. Also refer to a map that clearly shows the five predetermined random points prioritized from first to fifth for selection.

2. Go to the location of the starting point of the first potential sampling site indicated on the map. If that location is inaccessible but some part of the sampling site is accessible, go to Step 4. If the entire sampling site is inaccessible, note the reasons in the field logbook and on the SCS-232 field data form (refer to Appendix A), and go to the next potential sampling site.
3. If the location is accessible and the soil at the site is in the selected sampling class and the vegetation class is appropriate, sample the pedon.
4. If the starting point is inaccessible as described in Step 2 or if the starting point is accessible but does not contain the specified sampling class or vegetation class, then the following procedures are required:
 - From a random-number table, select a random number between 1 and 8 where 1 represents the direction northeast, 2 represents east, 3 represents southeast, 8 represents north.
 - Transect potential sampling points in 10-m intervals along a 150-m straight line in the chosen direction until the first occurrence of the proper combination of sampling class and vegetation class is found. If a proper combination of sampling class and vegetation class is not obtained after five transects, go to the next potential sampling site on the list.
 - Record the direction of each transect, e.g., southwest (SW) or north (N), and the number of the sampling point, i.e., 1 through 15, on the SCS-232 field data form.
 - If none of the five potential sampling sites yields an accessible pedon with the specified sampling class and vegetation class, call the sampling task leader as soon as possible.

5.4 Special Conditions

5.4.1 Inaccessible Watersheds

An attempt should be made to sample every watershed. Some watersheds may be inaccessible or may have inaccessible areas. In addition, access to a sampling site may be denied by the landowner. Alternative sampling classes are selected during the random selection process for backup sampling locations to ensure an equitable distribution of samples among watersheds. Each field crew must formally document the reasons for excluding a watershed or sampling site.

5.4.2 Inclusions

For this study, an inclusion is a soil associated with a sampling class other than the one being sampled; therefore, its chemical properties are described when the other sampling class is sampled. Because it is not representative of the soils in the sampling class, an inclusion located on a randomly selected site should not be sampled. The procedure described earlier accommodates this contingency.

5.4.3 Agricultural Sites

The open-dryland class contains some cultivated land. If a cultivated site has been selected randomly as a sampling location and if access permission has been obtained, the site is sampled. Agricultural practices may alter the chemical characteristics of the soils; therefore, if a cultivated site is sampled, that land use must be noted on the field form. During statistical analyses and subsequent

modeling, these samples may or may not be incorporated as representative of watershed soil chemistry.

5.4.4 Unsuitable Sampling Sites

Some land use classes generally are unsuitable for sampling, e.g., urban land, barren land, and waste disposal land. The crew leader decides if a sampling site is unsuitable. Documentation of the land use and reasons for the decision whether sampled or not sampled are entered into the log book.

5.5 Paired Pedons

Paired pedon sites for sampling are selected and assigned in advance by ERL-C. These sites are sampled in conjunction with the corresponding routine pedon. The paired pedon should be treated as a routine pedon when assigning the sample code.

The crew leader determines the location of the paired pedon by:

- Establishing sufficient distance between the two sampling locations to avoid disturbance of the paired pedon from sampling of the routine pedon.
- Using the same sampling unit and vegetation class as the routine pedon.
- Using the same slope position as the routine pedon.
- Using the same profile description and sampling protocol as the routine pedon.

Section 6 Operations

6.1 Profile Description

After the sampling site is located as described in Section 5.0, a pit large enough for sampling all major horizons is excavated to a depth of 1.5 m in the Northeast, 2.0 m in the Southeast, or to bedrock. The soil profile is described according to SCS protocols, and the data is recorded on the SCS-232 field data form (see Appendix A). Other descriptive information such as pesticide and herbicide contamination also is recorded on the field data form.

The sampling site is identified by a unique descriptor composed of the following numbers separated by hyphens: (1) the six-digit site identification code (ID) which incorporates the region, subregion, alkalinity class, and ID numbers, (2) the random site ID, i.e., a number from one to five, (3) the three-digit sampling class ID, and (4) the three-digit azimuth, measured in degrees and perpendicular to the described pit face.

6.2 Sampling

Precautions should be taken to avoid contamination when sampling the pedon. A wet pedon of mineral soil should be sampled from the base of the profile toward the top in order to avoid the sloughing of upper horizons onto the lower horizons. Other precautions include the draining of saturated soils before sampling; however, soil water should not be drained from sampled material. Also, handling the sample should be minimized.

Samples of approximately 5.5 kg of less than 20-mm material are taken so that at least 2 kg of less than 2-mm material are available after processing. Sample bags are labeled with Label A which identifies the date the sample was taken, the crew that took the sample, the

site, the sample code, the horizon depth, and the assigned set ID. The twelve-digit sample code is an alpha-numeric coding of the sample type, i.e., routine or field duplicate; number of bags filled per sample; the two-digit SCS state code; the three-digit SCS county code; the three-digit county pedon number; and the two-digit horizon number. The identification and sample numbering scheme yields unique alpha-numeric labels for each pedon and for each sample taken within the pedon.

Samples are kept as cool as possible in the field and in transport to the preparation laboratory. To maintain an ambient air temperature of 4°C, samples are stored in coolers with frozen gel packs. When sampling sites are remote, samples are stored in rented cold lockers prior to delivery to the preparation laboratory.

For the determination of bulk density, natural soil clods are sampled in triplicate from each mineral soil horizon. The clods are placed in hairnets, are moistened with a water mist, and are dipped in a saran solution to preserve their structural integrity for transport to the preparation laboratory.

For further information regarding sampling protocols, refer to Blume et al. (1987).

6.3 Sample Custody

Legal chain-of-custody procedures are unnecessary for this study; however, sample handling and storage procedures must be documented. Prior to delivery of the samples to the preparation laboratory by SCS personnel, the amount of time that samples are unrefrigerated must be minimized. An overnight air courier is used for shipment of all samples from the preparation laboratory to the analytical laboratory.

Section 7

Soil Sampling Internal Quality Control

Each field crew samples one horizon in duplicate on each day of sampling activity. The horizon for replicate sampling is chosen at the discretion of the field crew; however, the type of horizon is alternated so that field duplicates for each field crew are sampled across the complete range of possible horizons.

The sampling procedure specifies that the field duplicate and paired routine sample are sampled simultaneously. Trowelsful of soil are removed from the pit face and are placed alternately into sample bag 1 and then into sample bag 2, until two samples of fine earth material equal to approximately 5.5 kg each are collected. If sieving is necessary to remove rock fragments greater than 20 mm, two

options exist: (1) each sample may be collected on a plastic sheet then sieved into a sample bag, or (2) if two 20-mm sieves are available, each sample may be sieved directly into a sample bag.

The field duplicates are processed by a preparation laboratory and are analyzed by a contractor analytical laboratory. The analytical results are used to assess the variability attributed to sampling, preparation, and analysis.

For the determination of bulk density, natural soil clods are sampled in triplicate from each mineral horizon; however, a duplicate set of three clods is not taken.

Section 8

Preparation Laboratory Internal Quality Control

8.1 Sample Receipt

All field samples received by the preparation laboratory are checked in by preparation laboratory personnel. The following information is recorded in a logbook: (1) date received, (2) time received, (3) who delivered samples, (4) who received samples, (5) condition of samples, including notation by sample code of any problems, (6) set ID numbers, and (7) total number of samples. This logbook must be submitted to EMSL-LV at the end of the project.

8.2 Sample Processing

Each preparation laboratory splits one routine sample per batch into two samples. The preparation duplicates are analyzed by a contractor analytical laboratory. The results provide a measure of the variability attributed to subsampling and analysis.

8.3 Inorganic Carbon

For the visual determination of inorganic or carbonate carbon, a quality control (QC)

detection limit sample is used to test the ability of the analyst to see effervescence. The QC detection limit sample is prepared by spiking noncalcareous, less than 2-mm soil material with 1 percent (wt/wt) reagent-grade CaCO_3 powder or natural dolomite, $\text{CaMg}(\text{CO}_3)_2$, ground to pass a 60-mesh sieve. A QC calibration sample, prepared by spiking noncalcareous, less than 2-mm soil material with 5 percent (wt/wt) reagent grade CaCO_3 or natural dolomite, also is used by the analyst.

8.4 Bulk Density

Two or three soil clods are collected for each horizon sampled; therefore, duplicate or triplicate analyses are possible.

8.5 Raw Data

All raw data recorded in logbooks or on data sheets must be submitted to EMSL-LV at the end of the project (see Section 13.2).

Section 9

Analytical Laboratory Procedures and Internal Quality Control

9.1 Sample Receipt

All samples received by the contractor analytical laboratory are checked in by a receiving clerk who (1) records on the shipping form the date samples are received, (2) checks the samples to identify discrepancies with the shipping form, and (3) mails copies of the completed shipping forms to the Sample Management Office (SMO) and the project officer or designee. If there are any discrepancies or problems such as leakage in shipping or insufficient sample, the QA manager designee must be notified immediately. The receiving clerk retains a copy of the completed shipping form for the laboratory records. The samples are refrigerated at 4°C as soon as possible and must be refrigerated when not in use.

The samples received by the contract or analytical laboratory have been prepared, i.e., air-dried and crushed to pass a 2-mm sieve. During shipping, the sample material within each container segregates both by particle size and by density; therefore, each sample must be homogenized by thorough mixing prior to the removal of aliquots for analysis. One method of mixing is to place sample material on a large square of heavy paper. Each corner of the paper is lifted alternately and the soil is rolled toward the opposite corner. This process is continued until the soil is mixed thoroughly, at least 20 passes from each corner is

recommended. Alternative methods of homogenizing the sample may be used. Prior to the removal of an aliquot for analysis, the sample is mixed thoroughly by rolling the sample container. After an aliquot is removed for analysis the sample should be returned to the refrigerator as soon as possible. After all analyses have been completed and the results have been checked, samples should remain in refrigerated storage at 4°C in case reanalyses are necessary.

9.2 Sample Analysis

Procedures specified in the analytical methods manual (Cappo et al., 1987) are to be followed exactly for each parameter. Table 9-1 summarizes the parameters to be measured and the corresponding analytical techniques. Table 4-1 lists the required precision and expected range for parameters specified by ERL-C. Required detection limits for each parameter are given in Table 9-2.

9.3 Analytical Laboratory Documentation for Quality Control

The following documents must be updated constantly at the analytical laboratory and must be available to the analysts and the supervisor involved in the project:

Table 9-1. List of Parameters and Corresponding Analytical Techniques

<u>Parameter</u>	<u>Method</u>
Moisture	Gravimetric
Sand	Sieve/gravimetric
Silt	Pipet/gravimetric
Clay	Pipet/gravimetric
pH in deionized water pH in 0.01 M CaCl ₂ pH in 0.002 M CaCl ₂	Combination electrode/millivoltmeter
Total C	Elemental analyzer
Total N	Elemental analyzer
Total S	Elemental analyzer
Inorganic C	Coulometric
CEC (NH ₄ OAc saturating solution) CEC (NH ₄ Cl saturating solution)	Autotitration/flow injection analyzer
Ca Mg Exchangeable in NH ₄ OAc, NH ₄ Cl, and CaCl ₂ Na	Flame atomic absorption spectroscopy, inductively coupled plasma atomic emission spectroscopy (or flame atomic emission spectroscopy for Na only)
K Exchangeable in NH ₄ OAc, NH ₄ Cl, and CaCl ₂	Flame atomic absorption spectroscopy or flame atomic emission spectroscopy
Fe Exchangeable in CaCl ₂ : extractable in pyrophosphate, acid-oxalate, and citrate-dithionite	Flame atomic absorption spectroscopy or inductively coupled plasma atomic emission spectroscopy
Al Extractable in pyrophosphate, acidoxalate, and citrate-dithionite	
Al Exchangeable in CaCl ₂ and KCl	Inductively coupled plasma atomic emission spectroscopy
Nitrate (NO ₃ ⁻) water extractable	Ion chromatography
Sulfate (SO ₄ ²⁻) water extractable, phosphate extractable, and sulfate adsorption 6-point isotherm	Ion chromatography
Exchangeable acidity in BaCl ₂ -Triethanolamine and KCl saturating solutions	Titrimetric
Specific surface	Gravimetric

Table 9-2. Required Detection Limits, Expected Ranges, and Intralaboratory Relative Precision Goal

Parameter	Matrix	Calculated Reporting Units	Contract- Required Calculated Detection Limit	Contract- Required Instrumental Detection Limit	Expected Range	Intralaboratory Relative Precision Goal (%)*
Particle size	—	wt % †	—	—	0-100% ^a	5%
pH	—	pH	—	—	2.5-7.0	0.05
Total C	—	wt %	0.010%	—	0-50%	10%
Inorganic C	—	wt %	0.010%	—	0	15%
Total N	—	wt %	0.010%	—	0-20%	10%
Total S	—	wt %	0.010%	—	0-0.25%	10%
CEC (FIA)	—	meq/100g	—	0.010 meq	0.2-200	10%
CEC (titration)	—	meq/100g	0.01meq	—	0.2-200	10%
Na ⁺	all	meq/100g	—	0.50 mg/L	0.00-0.50	10%
K ⁺	all	meq/100g	—	0.050 mg/L	0.00-1.00	10%
Mg ²⁺	all	meq/100g	—	0.050 mg/L	0.00-1.50	10%
Ca ²⁺	CaCl ₂	meq/100g	—	2.00 mg/L	—	10%
Ca ²⁺	other	meq/100g	—	0.050 mg/L	0.00-8.00	10%
Al ³⁺	CaCl ₂	meq/100g	—	0.050 mg/L	—	10%
Al ³⁺	KCl	meq/100g	—	0.10 mg/L	—	10%
Al ³⁺	other	wt %	—	0.50 mg/L	—	10%
Fe ³⁺	CaCl ₂	meq/100g	—	0.050 mg/L	—	10%
Fe ³⁺	other	wt %	—	0.50 mg/L	—	10%
SO ₄ ²⁻	all	mg S/kg soil	0.32 mg/kg	0.10 mg S/L	0-200	5%
NO ₃ ⁻	water	mgN/kg soil	—	0.10 mg N/L	—	5%
SO ₄ ²⁻ , adsorption		mg S/L	0.32 mg/L	0.10 mg S/L	0.35	5%
Exchangeable acidity	KCl	meq/100g	0.40 meq	—	0-100	10%
Exchangeable acidity	BaCl ₂ -TEA	meq/100g	0.25 meq	—		
Specific surface		m ² /g	—	—	1.0-800	10%

* Unless otherwise noted, this is the relative precision at concentrations above 10 times instrumental detection limits.

† All values are determined on an oven-dry weight basis.

- Laboratory standard operating procedures (SOPs) - detailed instructions about the laboratory and instrument operations.
- Laboratory quality assurance plan - clearly defined laboratory protocol, including personnel responsibilities and use of QC samples.
- List of in-house samples - includes dates for completion of analyses, allowing the analysts to schedule further analyses.
- Instrument performance study information - information about baseline noise, calibration standard response, precision as a function of concentration, and detection limits; used by analysts and supervisor to evaluate daily instrument performance.
- QC charts - with 99 percent and 95 percent control limits for all quality control calibration samples (QCCS) and detection limit QC samples; generated and updated for each batch. The same QCCS must be used throughout each control chart in order to ensure the continuity of the control chart. (Note: The purpose of preparing QCCS control charts is to ensure that the actual control limits do not exceed the limits given in Table 9-3.)
- Data QC report - report by laboratory manager reviewing QC results for each parameter; specifies flags (see

Table 9-4) that are used (1) to document all results that are outside statistically established QC limits and (2) to identify samples that will require reanalysis before data are submitted.

9.4 Internal Quality Control Within Each Method

Internal quality control is an integral part of any measurement procedure and ensures that results are reliable. A summary of internal QC procedures for each method is given in Table 9-5. QC procedures are detailed in the appropriate method description in the analytical methods manual (Cappo et al., 1987). Details on internal QC procedures are described below.

9.4.1 Initial Calibration

All calibration standards are prepared in concentration units of mg/L or as specified in the procedure. A calibration curve for each analytical method is established by using a minimum of three points within the linear range. The use of at least a three-point calibration curve is required in place of the manufacturer's recommendations for the instrumentation, unless the manufacturer's recommendations for the instrumentation require more than three points within the linear range. The concentration of standards must bracket the expected sample concentration without exceeding the linear range of the instrument. Occasionally the standards suggested by a method must be adjusted to meet this requirement. The lowest standard should not be greater than 10 times the detection limit.

Table 9-3. Maximum Control Limits for QC Samples

Parameter	Maximum Control Limit for QC Sample (% Deviation from Theoretical Concentration of QC Sample)
Particle	*
pH	± 0.1 unit
Total C	±10%
Inorganic C	±15%
Total N	±10%
Total S	±10%
CEC	±10%
Na ⁺	±10%
K ⁺	±10%
Mg ⁺⁺	±10%
Ca ⁺⁺	±10%
Al ⁺⁺	±10%
Fe ⁺⁺	±10%
NO ₃ ⁻	± 5%
SO ₄ ⁻	± 5%
SO ₄ ⁻ , Adsorption	± 5%
Specific Surface	±10%

*Refer to Section 4.12, Particle-Size Analysis
in Capps et al., 1987.

Table 9-4. Laboratory/Field Data Qualifiers

Data Qualifier	Indicates
A	Instrument unstable.
B	Redone, first reading not acceptable.
F	Result outside criteria with consent of QA Manager.
G	Result obtained from method of standard additions.
J	Result not available; insufficient sample volume shipped to laboratory.
L	Result not available because of interference.
M	Result not available; sample lost or destroyed by laboratory.
N	Result outside QA criteria.
P	Result outside criteria, but insufficient volume for reanalysis.
R	Result from reanalysis.
S	Contamination suspected.
T	Container broken.
U	Result not required by procedure; unnecessary.
X	No sample.
Y	Available for miscellaneous comments.
Z	Result from approved alternative method.

Table 9-5. Summary of Internal Quality Control

Parameter	Procedure	Control Limits	Corrective Action
Moisture	<u>Laboratory Triplicate Analysis</u>		
	Analyze two additional portions of one sample in each batch.	Precision should be within 10% relative standard deviation (RSD).	Analyze a second sample in triplicate. If not within control limits, check temperature stability of the oven and repeat triplicate analyses.
Particle Size Analysis	<u>QC Calibration Sample Analysis</u>		
	Analyze a QCCS after every 10 or fewer samples.	Precision should be $\pm 5\%$ for sand, silt, and clay fractions $\geq 5\%$ (wt/wt).	Recalibrate balance, volumetric pipet, and thermometer. Check water bath or room temperature. Then re-analyze QCCS and samples bracketed by the affected QCCS.
	<u>Laboratory Duplicate Analysis</u>		
	Analyze a second portion of one sample in every batch.	Precision should be $\pm 5\%$ for sand, silt, and clay fractions $\geq 5\%$ (wt/wt).	Analyze a second sample in duplicate. Determine the source of imprecision; homogenization of sample may have been inadequate. Recalibrate balance. Check sieves for broken wires. Reanalyze the batch.
Specific Surface	<u>QC Calibration Sample Analysis</u>		
	Analyze 1 QCCS per batch of 21 or fewer samples, and 2 QCCSs per batch of 22 or more samples. Note: N ₂ adsorption standards may be purchased from Duke Scientific Corp. Palo Alto, California.	Precision should be within 10% RSD.	Continue desorption of ethylene glycol monoethyl ether (EGME) with continuous vacuum. Check CaCl ₂ in desiccator; if hydrated, replace. Recalibrate balance. Reanalyze QCCS and all affected samples.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Specific Surface (continued)	<u>Laboratory Triplicate Analysis</u>		
	Analyze two additional portions of one sample in every batch.	Precision should be within 10% RSD.	Analyze a second sample in triplicate. Check for vacuum in desiccator. Recalibrate balance. Reanalyze the batch.
	<u>Reagent Blank Analysis</u>		
	Analyze three reagent blanks per batch containing an amount of EGME equal to the greatest quantity required to saturate the soil samples.	Blanks show no EGME residual at end of equilibrium period.	No correction.
		Blanks show residual EGME at end of equilibrium period.	Determine if EGME reagent is old or otherwise contaminated. Purchase new reagent and reanalyze the batch.
pH	<u>Calibration and Standardization</u>		
	<u>Sample Analysis</u>		
	Calibrate pH meter for the range of pH expected in the soil (usually pH = 4 and pH = 7 standards). Analyze a QCCS immediately after calibration and after analyzing every 10 or fewer samples.	The value of the QCCS must be 4.00 ± 0.05 .	Recalibrate pH meter and reanalyze fresh QCCS. Check wiring, static electricity, and solution level in electrode, then reanalyze fresh QCCS. Replace electrode of pH meter, then reanalyze fresh QCCS.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
pH (continued)	<u>Reagent Blank Analysis</u>		
	Analyze one blank of each suspension solution.	The value should be between pH = 4.5 and 7.5.	Determine source of contamination. Prepare new solutions for reanalysis for batch.
	<u>Laboratory Triplicate Analysis</u>		
	Analyze two additional portions of one sample in every batch.	Precision should be ± 0.10 units.	Analyze a second sample in triplicate. Check for contamination in the suspension solution. Prepare new solutions for reanalysis of batch.
Cation Exchange Capacity (titration)	<u>Calibration and Standardization For Distillation/Titration Method</u>		
	Acid for titration must be re-standardized weekly.	Normality of acid changes more than 5 percent.	Prepare new solution.
	Calibrate pH meter (titrator) for range of pH expected in the titration (end point pH = 4.60). Analyze QCCS immediately after calibration and after every 10 or fewer samples.	The value of the pH QCCS must be 4.00 ± 0.05 .	Recalibrate pH meter and reanalyze fresh QCCS. Check wiring, static electricity, and solution level in electrode, then reanalyze fresh QCCS. Replace electrode or pH meter, then reanalyze fresh QCCS.
	Calculate instrumental detection limit based upon a minum titration, i.e., smallest possible volume, and normality of acid.	Instrumental detection limit must not exceed the contract-required detection limit (CRDL).	Use a more dilute titrant.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Cation Exchange Capacity (FIA)	<u>Calibration and Standardization for Flow Injection Analysis</u>		
	Determine instrumental detection limit.	Instrumental detection limit must not exceed the CRDL.	Check for possible contamination. Optimize instrumentation, e.g., wavelength.
	Analyze a detection limit QC sample.	Value must be within 20% of the theoretical concentration.	Identify and correct problem. Acceptable result must be obtained prior to sample analysis.
	One calibration blank ("0" mg/L standard) and three reagent blanks (reagents carried through the analytical procedure) per analytical batch.	Blank is less than the CRDL. Blank exceeds the CRDL.	No correction. Investigate the element source of contamination, then reanalyze all samples associated with the high blanks.
	QCCS must be run every 10 or fewer samples if flow injection analysis is used.	Measure each cation exchange capacity (CEC) and plot the results on a control chart. Develop 99% and 95% confidence limits. Required precision is within 10%	Recalibrate. Analyze a second QCCS and all samples bracketed by the affected QCCS.
Cation Exchange Capacity (both)	<u>Laboratory Duplicate Analysis</u>		
	Analyze a second portion of one sample in each batch for each saturating solution.	Precision should be within 10% RSD.	Analyze a second sample in duplicate. Check for contamination, e.g., atmospheric NH_4^+ or CO_3 . Recalibrate the balance, sample diluter how injection analyzer (FIA), or titrator. Reanalyze the batch.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Cation Exchange Capacity (both) (continued)	<p><u>Matrix Spike Sample Analysis</u></p> <p>One spike is required for each analytical batch. Add standard solution of NH_4Cl or $(\text{NH}_4)_2\text{SO}_4$ at a level approximately equal to the endogenous level or 10 times the instrumental detection limit, whichever is greater. Samples for flow injection analysis may be split, and the spike is added to one split. The distillation/titration method requires that a duplicate sample be extracted, then spiked for analysis.</p>	Calculate the percent recovery. Acceptable range is $100 \pm 15\%$.	Repeat on two additional samples. If either or both are outside the control limits, analyze the batch by the method of standard additions.
Metals - Na, K, Ca, Mg, Fe, and Al by AAS and ICPES	<p><u>Calibration and Standardization Sample analysis</u></p> <p>Calibrate the spectrometer as required in the analytical method. Analyze a QCCS immediately after calibration and after analysis of every 10 or fewer samples.</p>	Calculate the QCCS value from the calibration curve, and plot the result on a control chart. Develop the 99% and 95% confidence limits (warning and control). Acceptable range is $\pm 10\%$.	Recalibrate instrument, prepare new stock and calibration standards if necessary. Analyze a second QCCS and all samples bracketed by the affected QCCS.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Metals - Ca, Mg, K, Na, Fe, and Al by AAS and ICPEs (continued)	Verify calibration linearity. Determine linear dynamic range.	Linearity as determined by a least squares fit should not be less than 0.99.	Check calibration standards to see if properly prepared. Prepare new stock and calibration standards, if necessary, and recalibrate. Follow instrumental manufacturer's troubleshooting procedures.
	Determine the instrumental detection limits.	Instrumental detection limits must not exceed the CRDL for each element.	Check for possible contamination. Optimize instrumentation, e.g., wavelength, burner or torch position, oxidant and fuel pressures, nebulizer flow rate, integrity of impact bead or spoiler, optical alignment.
	Analyze a detection limit QC sample.	Value must be within 20% of the theoretical concentration.	Identify and correct problem. Acceptable result must be obtained prior to sample analysis.
	One calibration blank ("0" mg/L standard) and one reagent blank (any necessary reagents carried through the analytical procedure) per analytical batch.	Blank is less than the CRDL. Blank exceeds the CRDL.	No correction. Investigate and eliminate source of contamination, then reanalyze all samples associated with the high blank.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Metals - Ca, Mg, K, Na, Fe, and Al by AAS and ICPES. (continued)	<u>Matrix Spike Sample Analysis</u> To one solution in each batch add standard solution of analyte at a level approximately equal to the endogenous level or 10 times the instrumental detection limits, whichever is greater. Check recovery in each matrix.	Calculate the percent recovery. Acceptable recovery is $100 \pm 15\%$.	Repeat on two additional samples. If either or both are outside the control limits, analyze batch by the method of standard additions.
	<u>Laboratory Duplicate Analysis</u> Analyze a second portion of one sample in each batch for each analyte.	Precision should be within 10% RSD.	Analyze a second sample in duplicate. Recalibrate balance, repipet, and sample diluter. Check for source of contamination. Reanalyze the batch.
Exchangeable Acidity - BaCl ₂ -TEA, KCl	<u>Standardization</u> The solutions used for titration must be restandardized weekly.	Normality of solution changes more than 5%.	Prepare new solution.
	Calculate instrumental detection limit, based upon a minimum titration, i.e., smallest possible volume, and normality of titrants.	Contract-required instrumental detection limits must not be exceeded.	Use more dilute titrants.
	<u>Laboratory Duplicate Analysis</u> Analyze a second portion of one sample in each batch for each method.	Precision should be within 10% RSD.	Analyze another sample in duplicate. Determine source of difficulty, e.g., reduce normality of titrant, replace electrode, or recalibrate titrator. Reanalyze the batch.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Exchangeable Acidity (continued)	<u>Reagent Blank Analysis</u>		
	Three reagent blanks per batch are required for each exchangeable acidity method.	Blanks for KCl method are equal to or less than twice the CRDL. Blanks for BAcL ₂ -TEA method should have a %RSD ≤5%.	Determine source of contamination. Eliminate the problem, then reanalyze samples associated with the high blank(s). Determine and eliminate source of variation, then reanalyze the batch.
Sulfate and Nitrate	<u>Calibration and QA Calibration Sample Analysis</u>		
	Calibrate as required in the analytical methods. Analyze a QCCS immediately after calibration and after analysis of every 10 or fewer samples.	Calculate the QCCS value from the calibration curve, and plot the result on a control chart. Develop the 99% and 95% confidence limits (warning and control). Acceptable range is ±5%.	Recalibrate instrument. Prepare new stock and calibration standards, if necessary. Analyze a second QCCS and all samples bracketed by the affected QCCS.
	Verify calibration linearity. Determine linear dynamic range.	Linearity as determined by a least squares fit should not be less than 0.99.	Check calibration standards to see if properly prepared. Prepare new stock and calibration standards, if necessary, and recalibrate. Follow instrumental manufacturers trouble-shooting procedures.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Sulfate and Nitrate (continued)	Determine instrumental detection limits.	Instrumental detection limits must not exceed the CRDL.	Check for possible contamination. Optimize instrumentation.
	<u>Resolution Check</u>		
	Once per analytical run (day), check resolution of the anion separator column by analyzing a standard containing SO_4^{2-} , NO_3^- , and NO_2^- in equal 1-mg/L concentrations. Set instrument for a nearly full-scale response on the most sensitive range used.	Resolution must exceed 60%.	Clean or replace anion separator column, then repeat calibration and resolution check.
	<u>Calibration and Reagent Blank Analysis</u>		
	One calibration blank ("0" mg/L standard) and one reagent blank (necessary reagents carried through the analytical procedure) per analytical batch.	Blank is equal to or less than the CRDL. Blank exceeds the CRDL.	No correction. Investigate and eliminate source of contamination, then reanalyze all samples associated with the high blank.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Sulfate and Nitrate (continued)	<u>Matrix Spike Sample Analysis</u> To one sample in each batch, add standard solution of analyte at a level approximately equal to the endogenous level or 10 times the instrumental detection limit, whichever is greater.	Calculate the percent recovery. Acceptable range is $100 \pm 15\%$.	Repeat on two additional samples. If possible, determine and eliminate the source of the interference, then repeat analyses. If either or both are outside the control limits, analyze the batch by the method of standard additions.
	<u>Laboratory Duplicate Analysis</u> Analyze a second portion of one sample in each batch for each extraction procedure.	Precision should be within 5% RSD.	Analyze a second sample in duplicate. Recalibrate balance, repipet, and sample diluter. Check for source of contamination. Reanalyze the batch.
Total S, C, N	<u>Calibration and QC Calibration Sample Analysis</u> Calibrate and standardize induction furnace and titrator as described in method and instrument manual. Analyze a QCCS immediately after calibration and after analysis of every 10 or fewer samples.	Measure analyte and plot result on a control chart. Develop the 99% and 95% confidence limits (control and warning). Precision required is 10%.	Recalibrate and then analyze a second QCCS and all samples bracketed by the affected QCCS.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Total S, C, N (continued)	Verify calibration linearity Determine linear dynamic range.	Linearity as determined by a least squares fit should not be less than 0.99.	Check calibration standards to see if properly prepared. Prepare new stock and calibration standards; if necessary, recalibrate. Follow instrumental manufacturer's troubleshooting procedures.
	Determine instrumental detection limits.	Instrumental detection limits must not exceed the CRDL.	Check for possible contamination, e.g., purity of gas. Optimize instrumentation.
	<u>Calibration Blank Analysis</u>		
	Analyze one calibration blank per batch.	Blank is less than the CRDL.	No correction.
		Blank exceeds the CRDL.	Eliminate source of contamination then reanalyze all samples associated with high blank.
	<u>Matrix Spike Sample Analysis</u>		
	To one sample per batch add a standard amount of analyte at the endogenous level or 10 times instrumental limit, whichever is greater.	Calculate the percent recovery. Acceptable range is $100 \pm 15\%$.	Repeat on two additional samples. If possible, determine and eliminate the source of the interference, then repeat analyses. If either or both are outside the control limits, analyze the batch by the method of standard additions.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Total S, C, N (continued)	<u>Laboratory Duplicate Analysis</u> Analyze a second portion of one sample in every batch for each procedure.	Precision should be within 10% RSD.	Analyze a second sample in duplicate. Increase sample size, e.g., use two combustion boats. Decrease particle size to pass a finer mesh. Sample may be inhomogenous. Check for source of contamination. Recalibrate the instrument, then reanalyze the batch.
Inorganic Carbon	<u>Calibration and QA Calibration Sample Analysis</u> Calibrate as required in analytical methods. Analyze a QCCS immediately after calibration and after analysis of ever 10 or fewer samples. Verify calibration linearity. Determine linear dynamic range. Determine instrumental detection limit.	Calculate the QCCS value from the calibration curve, and plot the result on a control chart. Develop the 99% and 95% confidence limits (control and warning). Acceptable range is 15% RSD. Linearity as determined by a least squares fit should not be less than 0.99. Instrumental detection limit must not exceed CRDL.	Recalibrate instrument. Prepare new stock and calibration standards, if necessary. Analyze a second QCCS. Check working standards to see if properly prepared. Prepare new stock and calibration standards, if necessary, and recalibrate. Check for possible contamination. Optimize instrumentation.

(continued)

Table 9-5. Continued

Parameter	Procedure	Control Limits	Corrective Action
Inorganic Carbon (continued)	<u>Calibration Blank Analysis</u>		
	Analyze one calibration blank per batch.	Blank is equal to or less than the CRDL.	No correction.
		Blank exceeds the CRDL.	Investigate and eliminate source of contamination, then reanalyze all samples associated with the high blank.
	<u>Laboratory Duplicate Analysis</u>		
	Analyze a second portion of one sample per batch.	Precision should be within 15% RSD.	Analyze a second sample in duplicate. Recalibrate balance. Sample may be inhomogenous. Check for source of contamination. Reanalyze the batch.
	<u>Matrix Spike Sample Analysis</u>		
	To one sample in each batch, add analyte at a level approximately equal to the endogenous level or 10 times the instrumental detection limit, whichever is greater.	Calculate the percent recovery. Acceptable range is $100 \pm 15\%$.	Repeat on two additional samples. If possible, determine and eliminate the source of the interference, then repeat analyses. If either or both are outside the control limits, analyze the batch by the method of standard additions.

Next, the linear dynamic range (LDR) for the initial calibration is determined. If during the analysis the concentration of a sample falls above the LDR, two options are available. The first option is to dilute and reanalyze the sample. In this case, the diluent should have the same matrix as the sample matrix. The second option is to calibrate two concentration ranges. Samples are first analyzed on the lower concentration range. Any samples whose concentrations exceed the upper end of the LDR are then reanalyzed on the higher concentration range. If this option is performed, separate QC calibration samples (QCCSs) must be analyzed and reported for each range.

Spectroscopic-grade or high purity chemicals are required for primary standards when analysis is done by atomic absorption or emission methods. Also, calibration standards must have the same matrix as the solutions being analyzed. In order to meet the detection limits, some procedures require that the matrix, i.e., extracting or saturating solutions, be prepared from high purity chemicals.

9.4.2 Calibration Blank

One calibration blank per batch is analyzed immediately after the initial calibration to check for baseline drift. The calibration blank is defined as a "0" mg/L standard and contains only the matrix of the calibration standards. The observed concentration of the calibration blank must be less than or equal to the detection limit. If it is not, rezero the instrument and recheck the calibration.

9.4.3 Quality Control Calibration Samples (QCCS)

Immediately after standardization of an instrument, a QCCS containing the analyte of interest at a concentration in the midcalibration range is analyzed. The QCCS may be obtained commercially or may be prepared by the analyst from a source which is independent of the calibration standards. The QCCS

is analyzed to verify the calibration curve prior to any sample analysis, after every 10 samples, and after the last sample in each batch.

The observed value for the QCCS should be corrected for the calibration blank. The observed concentration for the QCCS is plotted on a control chart, and the 99 percent and 95 percent confidence intervals are developed. The 99 percent confidence interval must not differ from the theoretical value by more than the limits given in Table 9-3. A value outside the 99 percent confidence interval is unacceptable. When an unacceptable value for the QCCS is obtained, the instrument is recalibrated, and all samples up to the last acceptable QCCS are reanalyzed.

After each day of analysis, the control charts are updated. Cumulative means and new warning and control limits, i.e., 95 percent and 99 percent confidence intervals, are calculated. Bias for a given analysis is indicated by at least seven successive points on one side of the cumulative mean. If bias is indicated, analysis must be stopped until an explanation is found.

The same QCCS must be used to establish all values on a given control chart to ensure continuity.

9.4.4 Detection Limit Quality Control Samples

One detection limit QC sample is analyzed per batch. This is a low-level QC sample that contains the analyte of interest at a concentration two to three times above the required detection limit. The purpose of the detection limit QC sample is to eliminate the necessity of formally determining the detection limit on a daily basis. The measured value must be within 20 percent of the theoretical concentration. If it is not, the problem must be identified and corrected, and an acceptable result must be obtained prior to sample analysis.

9.4.5 Reagent Blank

For methods that require sample preparation, a reagent blank for each group of samples processed is prepared and analyzed. A reagent blank is defined as a sample composed of all the reagents, in the same quantities, used in preparing an actual sample for analysis. The reagent blank undergoes the same digestion and extraction procedures as an actual sample. The concentration of the reagent blank must be less than or equal to the detection limit. If the concentration exceeds this limit, the source of contamination must be investigated and eliminated. A new reagent blank is then prepared and analyzed, and the same criteria are applied. All samples associated with the "high" blank must be reprocessed and reanalyzed after the contamination has been eliminated.

9.4.6 Preliminary Sample Analysis

Approximately seven samples and a reagent blank are analyzed prior to matrix spike and duplicate analyses so that approximate endogenous sample concentrations may be determined.

9.4.7 Matrix Spike Analysis

One matrix spike sample is prepared for each procedure, as specified.

9.4.7.1 Liquid Samples--

For liquid samples, a matrix spike sample is prepared by spiking an aliquot of a solution with a known quantity of analyte prior to analysis. The spike concentration must be approximately equal to the endogenous level or 10 times the detection limit, whichever is larger. Also, the volume of the added spike must be negligible, i.e., less than or equal to 0.01 of the sample aliquot volume. The spike recovery must be within 100 ± 15 percent to be acceptable.

If the recovery is not acceptable, two additional, different samples must be spiked

with the analyte in question and must be analyzed. If the recovery for one or both samples is not within 100 ± 15 percent, the entire batch must be analyzed for the analyte in question by the method of standard additions. The method of standard additions is performed by analyzing the sample, analyzing the sample plus a spike at about the endogenous level, and analyzing the sample plus a spike at about twice the endogenous level. The concentration of the matrix spike sample must not exceed the linear range of the instrument. If it does, the spiked sample must be diluted before analysis. The percent spike recovery is calculated as follows:

$$\frac{\text{value of sample - sample value} \\ \text{plus spike} \quad \quad \text{of unspiked}}{\text{value of spike added}} \times (100)$$

9.4.7.2 Solid Samples--

Matrix spikes for solid samples, e.g., for analysis of total carbon and total nitrogen, are prepared by adding a known weight of material containing the analyte of interest to a sample of known weight. The spike concentration should be twice the endogenous level or 10 times the detection limit, whichever is larger. The concentration of the matrix spike must not exceed the linear range of the instrument. Although it will not be negligible, the weight of the spike material should be considered negligible for the purposes of calculation.

The spike recovery must be within 100 ± 15 percent to be acceptable. If the recovery is not acceptable, two additional, different samples must be spiked with the analyte in question and must be analyzed. If the recovery for one or both samples is not within 100 ± 15 percent, the entire batch must be analyzed for that analyte by the method of standard additions.

9.4.8 Duplicate Sample Analysis

One sample per batch is prepared and analyzed in duplicate for each parameter. Some procedures require triplicate analysis.

Refer to the specific method in Cappo et al. (1987).

Calculate the percent relative standard deviation (%RSD) as follows:

$$\%RSD = \frac{s}{\bar{X}} \times 100$$

$$\text{where } s = \left(\frac{\sum (X - \bar{X})^2}{n - 1} \right)^{1/2}$$

The relative standard deviation is plotted on a control chart, and 99 percent and 95 percent confidence intervals are established. These confidence intervals represent control and warning limits, respectively. Initial control limits are set at the precision levels given in Table 9-3. If duplicate values fall outside the control limits, an explanation must be sought, e.g., instrument malfunction or calibration drift. A second, different sample must then be analyzed in duplicate. No further samples should be analyzed until duplicate sample results are within the control limits.

Because %RSD is affected by concentration, this criterion is applied only when the mean of duplicate analyses exceeds the detection limit by a factor of 10.

9.4.9 Ion Chromatography Resolution Test

An ion chromatography resolution test is performed once per analytical run by analyzing a standard that contains concentrations of approximately 1 mg/L for each of SO_4^{2-} , PO_4^{3-} , and NO_3^- . If the resolution does not exceed 60 percent, the column should be replaced, and the resolution test should be repeated.

9.4.10 Continuing Sample Analysis

The remaining samples are analyzed if the detection limit QC sample, QCCS, reagent blank, matrix spike, and duplicate samples are within the required limits. After every 10 or fewer samples and after the last sample, a QCCS is analyzed to periodically verify the calibration curve. If the measured value of the QCCS differs from the theoretical value by more than the limits given in Table 9-3, the instrument must be restandardized, and the previous 10 samples must be reanalyzed.

9.5 Instrumental Detection Limits

Instrumental detection limits (IDLs) are determined and recorded monthly for each parameter except pH. For this study, the detection limit is defined as three times the standard deviation of 10 nonconsecutive replicate calibration blank analyses run on separate days. In some analyses, such as ion chromatography, a signal may or may not be obtained for a blank analysis. If a signal is not obtained for a blank analysis, the instrumental detection limit is defined as three times the standard deviation of 10 nonconsecutive replicate analyses of a standard whose concentration is four times the lesser of the actual detection limit or the required detection limit.

9.6 Reagent Blank Correction for Spectrometric and Ion Chromatographic Procedures

For all spectrometric and ion chromatographic procedures presented in Cappo et al.

(1987), the equations presented in the calculations subsections assume that the concentration of the analyte in solution has been corrected for the reagent blank. The reagent blank, composed of all the reagents in the same quantities used for actual samples, undergoes the same manipulations as actual samples and therefore should reflect any analyte contamination from the sample matrix or analytical procedure. Specifically, the actual (corrected) solution concentration is equal to the analyte concentration in the sample solution minus the analyte concentration in the reagent blank.

9.7 Data Reporting

The data forms used by the analytical laboratory are provided in Appendix B. The raw data are recorded on forms 115, 116, 303b, 306, and 308. The pH, moisture, and particle size analysis results are summarized on forms 103a and 103b. Data that are corrected both for blanks and dilutions are summarized on the 200-series forms. Data are annotated by using the data qualifiers listed in Table 9-4, if applicable. Results should be reported to the same number of decimal places as listed in Table 9-6; however, no more than four significant figures should be reported. Forms 109 through 114 contain quality control data. After

a form is completed, the laboratory manager must sign it to indicate that he or she has reviewed the data and that the samples were analyzed exactly as described in the procedure.

All deviations from the analytical protocol must be documented. All original raw data such as data system printouts, chromatograms, notebook, individual data sheets, QC charts, and standard preparation data should be retained.

9.8 Evaluation of Quality Control Data

Each laboratory will make a report by telephone to the QA manager or other authorized representatives, as directed. The objective of these reports is to keep the QA manager informed of the status of the internal QC and external QA checks in the laboratory in order to identify and solve problems that may arise. The reports also allow the QA manager to obtain preliminary results for the blanks, duplicates, and audit samples. Otherwise, these data would not be available for QA/QC checks until the data packages are received from the laboratories. During the telephone contact, the QA manager or designer records all interaction in a bound logbook.

Table 9-6. List of Decimal-Place Reporting Requirements

Parameter	Number of Decimal Places in Reported Results*
Moisture content	3
Particle size	1
pH	2
Total C	3
Inorganic C	3
Total N	3
Total S	2
CEC	3
Na ⁺	3
K ⁺	3
Mg ⁺⁺	3
Ca ⁺⁺	3
Al ⁺⁺	3
Fe ⁺⁺	3
NO ₃ ⁻	2
SO ₄ ⁻	2
SO ₄ ⁻ adsorption	3
Exchangeable acidity	2
Specific surface	4

*Report to a maximum of four decimal places.

After each day of analysis at the contractor laboratory, control charts are updated and new control and warning limits are calculated. The contractor QA chemist then performs a QC audit in which all the pertinent data are reviewed. Any values that lie outside the control or warning limits are checked to verify that they are not the result of a transcription error. If bias is indicated by seven successive points on one side of the cumulative mean, analysis is stopped and an explanation is sought. Copies of the plots are given to the contractor analytical laboratory supervisor and to each analyst.

Section 10

Performance and System Audits

10.1 Soil Samples to Estimate Precision

Three kinds of paired quality assurance samples are included in each batch of soil samples submitted to an analytical laboratory: (1) field duplicates, (2) preparation duplicates, and (3) audit samples.

One horizon per crew, per day is sampled in duplicate as specified in Blume et al. (1987). The field duplicate undergoes all preparation steps in order to estimate variation in sampling a horizon.

One sample per batch is chosen by the preparation laboratory to be split into two subsamples. The preparation duplicates are included to estimate the range in physical and chemical characteristics for splits of the sample material.

Two audit samples that are replicates from a homogenized bulk sample are sent to the analytical laboratory via the preparation laboratory. The audit samples do not undergo further processing at the preparation laboratory. These samples are double-blind QA samples, i.e., the analytical laboratory does not recognize an audit sample as a QA sample and does not know its predetermined composition. The audit samples are used to assess analytical within-batch precision and to estimate interlaboratory bias. Appendix C presents the plan for laboratory audit samples.

10.2 Field Sampling On-Site Evaluation

Each field sampling crew can expect at least one on-site evaluation during the course of the sampling effort. This is an on-site inspection to review site selection, profile description, sampling procedures, and QA efforts. The questionnaire given in Appendix D is used to assist in the evaluation.

The QA auditor conducts an in-depth review of all field operations for compliance with the sampling protocols. This includes, but is not limited to: (1) interviewing the sampling crew, (2) accompanying the sampling crew during a sampling excursion, and (3) writing a summary report with results, observations, and recommendations. If there are any problems, the evaluator must attempt to correct them by reference to or interpretation of the sampling protocols after the daily sampling has been completed. All problems are brought to the attention of the QA manager at EMSL-LV within two working days. The QA manager is responsible for conveying any major problems to the technical monitor or technical director.

10.3 Preparation Laboratory On-Site Evaluation

Each preparation laboratory can expect a minimum of two on-site evaluations. The first on-site evaluation is performed *before* samples are received. The purpose of this evaluation is to assess the facilities, including refrigerated storage and areas for soil drying and for sample processing, i.e., crushing, sieving, and splitting. The questionnaire in Appendix E is used to assist in the evaluation. The auditor brings any problems to the attention of the laboratory manager. All observations are summarized in an evaluation report that is submitted to the QA manager at EMSL-LV.

The second on-site evaluation is conducted about a third of the way through sample processing. After reviewing the previous evaluation report, any changes since the first on-site evaluation are noted on the questionnaire. Also, any problems identified must be corrected and brought to the attention of the QA manager. A summary report is written for this and any additional on-site evaluations and is submitted to the QA manager at EMSL-LV.

10.4 Analytical Laboratory On-Site Evaluation

Each analytical laboratory can expect a minimum of two on-site evaluations. The first on-site evaluation is performed *after* the laboratory has analyzed successfully a set of pre-award performance evaluation (PE) samples for the contract-required parameters, or *during* the PE sample analyses (see Appendix F). The PE samples contain up to the maximum number of required analytes in the expected analytical ranges. The pre-award scoring sheet given in Appendix G is used to score the PE sample results. Grading emphasizes analytical accuracy, but a substantial portion of the grade depends on meeting the QA, reporting, and deliverable requirements. The EPA QA manager or an authorized representative

conducts an in-depth review of all laboratory functions that are pertinent to the analyses. The questionnaire in Appendix H is used to assist in the on-site laboratory evaluation. The auditor brings any problems to the attention of the laboratory manager for corrective action. All observations are summarized in an evaluation report that is submitted to the QA manager at EMSL-LV.

The second on-site evaluation is conducted approximately a third of the way through sample analyses. The evaluation questionnaire is completed with emphasis on all changes since the first on-site evaluation. During the second on-site evaluation, audit sample data and QC data received to date are reviewed. An evaluation report is written for this and any additional on-site evaluations and is submitted to the QA manager at EMSL-LV.

Section 11 Acceptance Criteria

11.1 Audit Sample Results

Acceptance windows for single values from audit samples are based on previous interlaboratory analyses of the same sample material by the same protocols. The objective of creating windows is to predict intervals for acceptable single future values based on a sample mean (\bar{X}) and sample standard deviation (s) computed from n previously observed values. The limits of the windows are determined by using a t-statistic (t).

$$t = \frac{Z}{\sqrt{\frac{\mu}{r}}}$$

is a Student's t

where:

Z is the standard normal variate, having a normal distribution with a mean of 0 and a variance of 1;

μ is a variable with a chi-square distribution with r degrees of freedom, and Z and μ are independent.

The observed values $X_1, X_2, X_3, \dots, X_n$ are independent and have a normal distribution with a population mean (μ) and variance (σ^2). A $(1 - \alpha)$ prediction interval for a single future value y is needed. Let \bar{X} equal sample mean and s equal sample standard deviation. It is known that:

$$y \sim N(\mu, \sigma^2) \text{ and } \bar{X} \sim N(\mu, \left(\frac{\sigma^2}{n}\right))$$

$$\text{Therefore, } y - \bar{X} \sim N\left(0, \sigma^2 \left(1 + \left(\frac{1}{n}\right)\right)\right)$$

$$Z = \frac{y - \bar{X}}{\sigma \sqrt{1 + \frac{1}{n}}} \sim N(0,1)$$

$$\mu = \frac{n-1}{r} \left(\frac{s^2}{\sigma^2}\right) \sim \chi^2(n-1) \text{ and } r = n-1.$$

Substituting,

$$t = \frac{\frac{y - \bar{X}}{\sigma \sqrt{1 + \frac{1}{n}}}}{\sqrt{\frac{(n-1)s^2}{(n-1)\sigma^2}}} = \frac{y - \bar{X}}{s \sqrt{1 + \frac{1}{n}}}$$

The upper and lower limits of the window can be formalized as follows:

$$\bar{X} + (t)(s) \sqrt{1 + \frac{1}{n}} = \text{upper limit of the window}$$

$$\bar{X} - (t)(s) \sqrt{1 + \frac{1}{n}} = \text{lower limit of the window}$$

The Student's t-value has $n-1$ degrees of freedom. The t-value is for a two-tailed test with a cumulative probability of 0.95, i.e., 2.5 percent probability on either side.

For predicting future values, wider windows than the standard 95 percent confidence interval about the mean are desirable. As the number of observed values increases, more variance occurs because of chance alone.

Initially, there may not be sufficient data ($n < 10$) available to provide good interval estimates. Arbitrary criteria may be used until 10 or more values are available. The windows should be updated periodically as more data are accumulated.

To detect outliers, a statistical test, e.g., Grubbs' test (Grubbs, 1969), is applied to the data before interval estimation. The outliers are excluded from the computation of the windows.

Windows for matrix spike analysis results are computationally identical to those for audit sample results.

11.2 Replicate Analysis Results

Acceptance criteria for the relative standard deviation (RSD) are based on the upper 95th percentile of observed values of RSD. Because RSD is affected by concentration, these criteria are applied only when the mean of the duplicate or triplicate analyses exceeds the contract-required detection limit (CRDL) by a factor of 10.

Arbitrary acceptance criteria may be used until sufficient (at least 10) RSD values have been observed.

The distribution of RSD values cannot be estimated accurately until sufficient RSD

values have been observed. It is recommended that no outlier test be applied until the distribution has been estimated.

11.3 Corrective Action

Laboratories which fail to meet the acceptance criteria for analysis of audit samples, matrix spikes, or replicates are required to repeat the analysis that produced the questionable results. If results from the second analysis are still unacceptable, further corrective action must be initiated.

Section 12

Data Management System

The purpose of the data base management system is to assemble and store data generated as part of the DDRP, to provide basic reports of the survey results, to perform simple statistical analyses, and to provide data security. The relationship of data base management to the overall soil survey is shown in Figure 12-1.

All data sets are protected from unauthorized or accidental access by individual, system, and file password protection.

The data are stored in three major data sets: (1) a raw data set, (2) a verified data set, and (3) a validated data set.

12.1 Raw Data Base

At ORNL, the Statistical Analysis System (SAS) is used to enter the field data, preparation laboratory data, and analytical laboratory data (analytical results and data qualifiers, see Table 9-5) into the raw data base. These data are also sent to the EMSL-LV QA staff for concurrent data analysis. The SAS full-screen editor procedure is used to provide gross error checking as data are entered. All data are entered into two separate data sets by two different operators. For the DDRP data base, a comparison program is used to compare the two data sets and to identify any inconsistencies. This double entry and comparison process allows typographical errors to be identified and removed from the data base.

12.2 Verified Data Base

As the field and analytical laboratory data are received by EMSL-LV QA group, all

data are reviewed. The analytical data are processed by an on-line quality assurance system being developed by EMSL-LV QA staff. Problems with the data are flagged as deemed necessary by the QA staff. Data are examined for reporting errors and may be modified in the data base. Also, reanalysis may be requested. Old data values are maintained in the raw data base as a historical file.

In addition to the standard QA analysis, various printouts are supplied to the QA manager to point out intralaboratory or interlaboratory bias as well as discrepancies in blanks, audits, or other QA/QC samples. The overall outcome is a verified data base in which all values are either qualified or replaced with missing value codes. EMSL-LV coordinates with sampling crews, preparation laboratories, and the contractor laboratories to make all appropriate corrections in the data.

12.3 Validated Data Base

A computer printout of the verified data base is sent to ERL-C for data validation. The validation procedure consists of a final review of all data for internal and regional consistency and uses all the QA/QC information available.

The validation process compares data for a set of variables against a much narrower range established from internal chemical relationships and data from each sampling class.

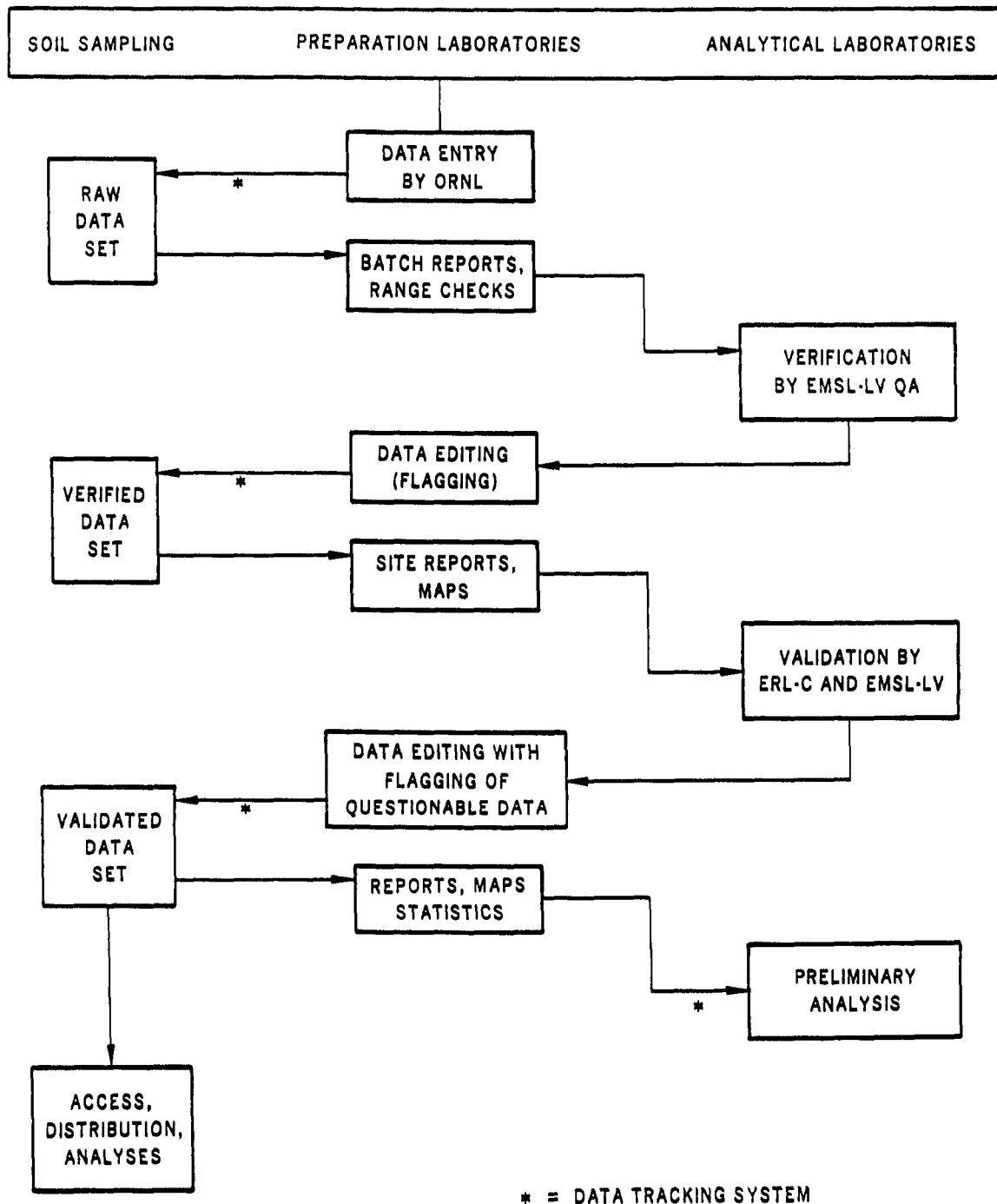


Figure 12-1. Data management for the DDRP Soil Survey.

The validation step incorporates soil chemistry to identify intrasite sample inconsistencies. Sample data are checked by examining relationships between paired data, such as pH H₂O versus pH CaCl₂ and cation exchange capacity versus specific surface. Samples flagged as questionable are subjected to further review. Intersite validation consists of comparing profile data for a single pedon with profile data for all pedons in the sampling class. Data that contrast with nearby sites

can be flagged for more detailed review. Data from analytical replicates, audits, and other paired QA samples are also reviewed. The validation process increases the integrity of the data base by using a systems approach to determine that data are reasonable. After the validated data are transferred to the validated data base, the data base will be released by EPA and will be made available to all data users.

Section 13 ***Review of Data***

As the field, preparation laboratory, and analytical laboratory data are received by EMSL-LV QA staff, all data are reviewed as described in the following subsections.

13.1 Field Data Review

Field data forms are reviewed by:

- Checking the accuracy of the ID numbers.
- Reviewing all profile descriptions and associated data.
- Contacting SCS or referring to field notes to correct any errors.
- Notifying ORNL if the data base is affected by any changes.
- Recording all interactions with ORNL, EPA, and SCS in a bound logbook.

13.2 Preparation Laboratory Batch Assignment and Data Review

Form 101 is reviewed by:

- Checking all sample codes against ID numbers on the field data forms.
- Checking for inclusion of duplicates and audit samples.
- Recording identity of audit samples.
- Checking analytical data.
- Contacting preparation laboratory or referring to the preparation laboratory logbook for Label A to correct any errors.

- Notifying ORNL if the data base is affected by any changes.
- Notifying contractor analytical laboratory and Sample Management Office (SMO) if any changes affect sample analysis or data reporting.
- Recording all interactions with preparation laboratories, ORNL, SMO, and contractor analytical laboratories in a bound logbook.

Form 102 (shipping form) is reviewed by:

- Recording date that form is received from contractor analytical laboratory.
- Checking Form 102 against Form 101 to verify analytical laboratory name and number of samples.
- Verifying that prepared rock fragments were shipped if organic carbon is to be determined.
- Calling contractor analytical laboratory to discuss condition of samples upon receipt, and date and time of receipt.
- Calling other involved parties to correct any problems.
- Recording all interactions in a bound logbook.

13.3 Analytical Laboratory Data Review

13.3.1 Communications

Frequent communications, i.e., two or three contacts each week, are maintained with each contractor analytical laboratory to obtain current sample data and to discuss any problems that may occur during analyses. Data

may be available via electronic transfer. Data that are received verbally are recorded in a bound logbook. These preliminary data are reviewed for anomalies. If a problem is identified, the laboratory is notified. Corrective action or reanalysis may be suggested. All interactions with each laboratory are recorded in a bound logbook.

Contractual issues are referred to the QA manager and to the contract officer. Major technical issues are referred to the QA manager.

13.3.2 Preliminary Data Package Review

Each data package is reviewed by:

- Reviewing cover letter.
- Completing Data Package Completeness Checklist (given in Appendix I) to review internal QC data, data completeness, and data qualifiers used.
- Notifying the contractor laboratory of any major discrepancies and recording corrective action.

13.3.3 Computer Review of Analytical Data

The National Computer Center (NCC), Research Triangle Park, North Carolina, receives a magnetic tape from ORNL. The magnetic tape, containing all analytical data, is accessed as follows:

- (1) Each magnetic tape received by the NCC tape library is given a volume serial number and a BIN number. A BIN number indicates the physical location of the tape.
- (2) EMSL-LV QA computer support contacts the NCC tape library to obtain the volume serial number and the BIN number. Upon request from EMSL-LV QA staff, the tape is loaded.

The QA staff runs the data through programs that check laboratory QC, paired QA data, and the internal consistency of data. These programs generate lists of data that are exceptions to predetermined criteria. These exceptions are subject to the scrutiny of the QA staff. Corrective action for exceptions includes requests that the contractor analytical laboratory confirm the data or reanalyze the samples for which the data are anomalous.

Section 14

Data Verification

14.1 Verification of Field Data

14.1.1 Verification of Sampling Class and Vegetation Class

This verification involves using the list of sampling classes and corresponding vegetation classes as supplied by ERL-C to identify the appropriate sampling class and vegetation class for the specific pedon. Each field data form lists the watershed ID, random point, sampling class, and aspect on the first line of the location description and free form site notes. This information is checked against the information from ERL-C.

14.1.2 Review of the Field Data Forms for Completeness and Misnomers

Each field data form is reviewed for:

- Left and right justification of letters and numbers.
- Correctness of code values and coding, e.g., coding 0 as Ø and not as 0 or misplacing decimals.
- Completeness: many forms lack complete information for certain parameters; parameters not listed in Blume et al. (1987) and missing data are considered incomplete.

Reference information used in the review includes (1) instructions for using the SCS-232 field data form, (2) coding values found on the SCS-232 form (see Appendix A), (3) Soil Survey Manual, (4) National Handbook of Plant Names, and (5) Land Resource Regions and Major Land Resource Areas of the Northeast United States U.S. Department of Agriculture Soil Conservation Service ([USDA/SCS], 1985).

After problems have been identified, a discrepancy form describing these problems will be sent to the SCS field crew. The form consists of:

- Tracking number to identify the specific SCS-232 field data form, watershed ID number.
- Soil series name.
- Pedon sample number.
- Description of problem, i.e., discrepancy or missing data.
- Old value, i.e., value thought to be incorrect or question mark if value is missing.
- New value, i.e., value supplied if possible or to be filled in by SCS field crew.
- Signature of SCS personnel to acknowledge the discrepancy and change.

The SCS field crew checks the discrepancy form against the SCS-232 forms, fills in the appropriate areas, and returns the discrepancy form. The form is rechecked by EMSL-LV QA staff and is used to edit the local working copy of the raw data base (see Section 14.1.4).

Discrepancy forms are sent to field crews after review of approximately 40 data forms, i.e., weekly. Copies of the discrepancy forms are filed at EMSL-LV.

14.1.3 Verification of Soil Descriptive Parameters

This step in verification of each soil parameter on the field data form depends on the type of information needed for verification. Some parameters must be checked against logbooks or analytical laboratory data; other

parameters require comparison against soil taxonomic criteria; field-observed parameters may not be possible to verify.

Verification of the field data is accomplished with a computer program designed to check every parameter on the 232 form. The checks include:

- Appropriate coding.
- Missing information.
- Field parameter versus field parameter, e.g., texture modifier versus percent rock fragments.
- Field parameter versus analytical parameter, e.g., field pH versus laboratory pH.

The last two checks are exception programs that examine internal data consistency.

Page 1 of 4 of SCS-232 Field Form

NOTE: The following parameters are found in sequence on the field data form.

- A. Soil Series Name-Verification described in Section 14.1.1.
- B. Sample Number-Verification against logbook.
- C. Major Land Resource Areas (MLRA)-Verification against MLRA map (USDA/SCS, 1985).
- D. Latitude and Longitude-Verification against watershed latitude and longitude information supplied by ERL-C.
- E. Date-Verification against logbook; set ID from preparation laboratory logbook.
- F. Slope
 - 1. % - field-observed: coding and completeness check.

- 2. Shape (SHP) - field-observed: coding and completeness check.
- 3. Local Physiographic Component (GM) - field-observed: coding and completeness check.
- 4. Aspect (ASP) - field-observed: coding and completeness check.
- 5. Microrelief - field-observed: coding and completeness check
 - a. Kind (K)
 - b. Variation (A)
 - c. Pattern (P)
 - d. Position (POS)

G. Physiography

- 1. Regional (RG) - coding and completeness check.
- 2. Local (LOC) - coding and completeness check.

H. Pedon Classification - (all parameters in this category). Verification based on taxonomic description of soil series.

I. Precipitation - field crews not required to describe parameter.

J. Water Table

- 1. Depth - field-observed: coding and completeness check.
- 2. Month - verification against date.
- 3. Kind (KD) - field-observed: coding and completeness check K. Land Use (LU) Verification against MLRA, vegetation class, and vegetation species.

L. Stoniness Class-field-observed: coding and completeness check.

M. Estimated Permeability (PM)-verification against texture for each horizon.

N. Soil Drainage Class (DR)-verification against sampling class description.

O. Elevation Meters-verification against U.S. Geological Survey soil topographical map, if necessary. Otherwise coding and completeness check.

P. Parent Material

1. Degree of weathering or bedding inclination (w) field-observed: coding and completeness check.

2. Mode of accumulation or deposition (M) coding and completeness check.

3. Origin or source of parent materials (orig) verification against sampling class description.

Q. Temperature-parameter not required to be described by field crew.

R. Moisture Regime (MST RGE)-coding and completeness check.

S. Weather Station Number-parameter not required to be described by field crew.

T. Control Section-coding and completeness check.

U. Erosion (ERWA)-parameter not required to be described by field crew.

V. Runoff (RNOF)-coding and completeness check.

W. Diagnostic Features

1. Depth-
2. Kind (KND) } should correspond to
 } horizon description and
 } relative taxonomy

X. Flooding

1. Frequency - } field-observed (usually
2. Duration } not filled in)

Y. Vegetation-verification against National Handbook of Plant Names (see Section 14.1.2, Item 4) and vegetation class specified by ERL-C (see Section 14.1.1)3.

Z. Location Description and Freeform Site Notes-verification for watershed ID, random site, sampling class, and aspect against information from ERL-C; must be coded in first 17 spaces.

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A. Depth Upper/Lower-coding and completeness check.

B. Horizon Designation-coding and completeness check.

C. Thickness-Average thickness should correspond approximately with difference of upper and lower depth parameters.

D. Moist Color

1. Location - field-observed: coding and completeness check.

2. Percentage (%) - field-observed: coding and completeness check.

3. Color - field-observed: coding and completeness check.

4. Texture - verification against analytical data.

5. Texture modifier - coding and completeness check.

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A. Structure

1. Grade (GRD)

2. Size (SZ)-field observed: coding and completeness check

3. Shape (SHP)

B. Consistence-Field-observed: coding and completeness check.

C. Mottles-Field-observed: coding and completeness check.

D. Boundary-Field-observed: coding and completeness check.

E. Field Measured Properties

1. Kind - verification against horizon designation and texture.
2. Amount - pH value may be correlated to analytical data.
3. Soil Water - field-observed: coding and completeness check.

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A. Roots

- | | | |
|---|---|--|
| <ol style="list-style-type: none">1. Quantity (QT)2. Size (SZ) -3. Location (LOC) | } | field-observed:
coding and
completeness
check |
|---|---|--|

B. Pores-Parameter not described by field crew.

C. Concentrations-Parameter not described by field crew.

D. Rock Fragments

1. Kind - verification against parent material origin.
2. Percentage (%) - verification against texture class and texture modifier.
3. Size (SZ) - verification against texture modifier.

14.1.4 Methods Used to Treat Outliers

In this section, the term outlier refers to:

- Information identified through discrepancy forms.
- Codes input incorrectly.
- Exception program outliers.
- Computer program outliers.

Discrepancy form outliers and input errors are corrected and other outliers are flagged (see Table 14-1) through an editing program. Editing is done on a working copy of the official raw data base supplied from ORNL via NCC (see Section 13.3.3). All editing changes are made to this data base, thereby

Table 14-1. Flags for the Verification of field data

AO - missing value	EO - correlation outlier
BO - invalid code	FO - value inappropriate for state
CO - alpha character in numeric field	GO - missing value; with explanation
DO - numeric character in alpha field	HO - miscellaneous flag, for unique problems

protecting the official raw data base. Upon entering the editing program, a subset of the field data is keyed in by the sample number, state, and county. This subset is copied into a temporary working file for manual editing. When editing of the work file is finished, the manual editing system is exited. The edited information and the original field data are sent automatically to a transaction file. The transaction file is printed and reviewed at the end of an editing session.

After the edits have been checked, the local master data base is updated. All edited information in the transaction file is applied to the local master data base, replacing the original data. This information also enters the history file, i.e., the record of all transactions made to the local master data base. After the process of correcting the local master data base is completed, the data base becomes the verified master data base.

Copies of the verified data base and a hard copy of the history file are sent to ORNL. ORNL compares the official raw data base with the verified master data base. Any anomalies between the data bases should correspond to the history file. After both data bases are proofed by ORNL, the official raw data tape is stored, and the official verified data tape becomes available for the next user.

14.2 Verification of Physical and Chemical Data

14.2.1 Exceptions Programs for Internal Consistency of Data

Simple mathematical relationships are used to examine the internal consistency of

data for each sample. For each relationship it is expected that approximately 10 percent of the data will not comply with these relationships. These anomalous data are examined by a soil chemist who qualifies them or assigns appropriate flags (see Appendix M). The following relationships are examined in qualifying the data:

- (1) Laboratory-determined pH values should relate as follows:

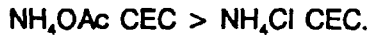
$$\text{pH H}_2\text{O} > \text{pH 0.002 M CaCl}_2 > \text{pH 0.01 M CaCl}_2$$

CaCl₂ solution masks the effect of soluble salts in soils. Ca²⁺ ions displace H⁺ ions from exchange sites; the H⁺ ion concentration in solution increases, and the result is the measurement of a lower pH.

- (2) Field pH should be greater than laboratory-determined pH. Field pH is not available for all samples; however, when field pH is available, this comparison is made. This relationship occurs because laboratory samples are dried during sample processing, whereas field pH is determined on a field-moist sample.
- (3) Phosphate-extractable sulfate should be greater than water extractable sulfate.

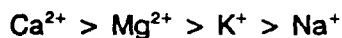
Phosphate-extractable sulfate approximates the total adsorbed sulfate; water-extractable sulfate approximates that which readily enters soil solution.

- (4) Cation exchange capacity (CEC) should relate as follows:



A higher CEC is measured by using a buffered (pH 7) NH_4OAc saturating solution to determine CEC in an acid soil. With an increase in pH, H^+ ions are displaced. This creates more exchange sites for retention of NH_4^+ . The NH_4Cl saturating solution is unbuffered; therefore, cation exchange takes place at the soil pH, resulting in the measurement of a lower CEC.

- (5) Exchangeable cations should relate as follows:



except in the presence of illitic clays where:



The first relationship occurs because of the natural abundance of the cations and because of their hydrated radii. Illitic clays provide an exception because they are potassium rich.

- (6) Exchangeable acidity should relate as follows:



The BaCl_2 -TEA solution is buffered to a pH of 8, and this results in measurement of *total* potential acidity. KCl is a neutral salt; therefore, values obtained are more representative of natural exchangeable acidity in field soils.

- (7) The summation of sand, silt, and clay should equal 100 percent. Also, the sand and silt fractions should sum to equal total sand and silt.
- (8) The field-determined particle-size estimates should be approximately

equivalent to particle-size data measured in the laboratory.

- (9) Soil permeability estimates should compare to particle-size data.
- (10) Each SO_4 adsorption isotherm, in adherence to the Langmuir Equation, should be linear up to the point of surface saturation.
- (11) Total carbon (C) should be greater than total nitrogen (N). The ratio of C to N should fall within a known range.
- (12) A plot of CEC versus percent clay should display a proportional relationship reflecting the relationship of CEC to the amount of clay.
- (13) Specific surface versus SO_4 adsorption, CEC, and exchangeable cations are proportional relationships. An increase in specific surface should show a corresponding increase in the other parameters.
- (14) The summation of exchangeable acidity and exchangeable basic cations should be approximately equal to CEC. In soils, Ca^{2+} , Mg^{2+} , Na^+ , K^+ , Al^{3+} , and H^+ are the predominant cations; therefore, the summation should be approximately equal to the CEC. Some variation occurs because of organic chelation and the presence of organic cations.

14.2.2 Other Exceptions Programs

Exceptions programs also check laboratory QC and paired QA data against predetermined criteria. These programs generate lists of data that are examined by the QA staff. Corrective action includes requests for confirmation of data or reanalyses of batches for which data are outside the criteria (see Appendix M).

14.2.3 Methods Used to Treat Outliers

Misreported data and data from reanalyses are edited as described in Section 14.1.4 for field data. Outliers generated by exceptions programs are flagged according to category (see Table 14-2). The edited files are applied to the local master data base to

produce the verified master data base, as described in Section 14.1.4.

14.3 Reporting Scheme

Lists of flagged data are hard-copied and filed. Progress and major problems are reported to the EPA technical monitor at EMSL-LV.

Table 14-2. Flags for the Verification of Analytical Data

Miscellaneous

AO* Value missing

Generated by Appropriate Blank Exception Program

B3* Internal (laboratory) calibration or reagent blanks are $>2 \times$ CRDL and contribute $>50\%$ to the sample concentrations in the batch.

B4** Potential negative sample bias based on internal (laboratory) blank data.

B5** Calibration blank $>1.05 \times$ reagent blank.

Generated by Duplicate Precision Exception Program

D1** Field duplicate precision exceeded the maximum expected percent relative standard deviation (%RSD), and either the routine or the duplicate value was $\geq 10 \times$ CRDL.

D2** Field duplicate precision exceeded the maximum expected %RSD, and both the routine and the duplicate sample concentrations were $\geq 10 \times$ CRDL.

D3** Internal (laboratory) replicate precision exceeded the maximum contract required %RSD, and either the routine or the duplicate sample concentration was $\geq 10 \times$ CRDL.

D4** Internal (laboratory) replicate precision exceeded the maximum contract required %RSD, and both the routine and duplicate sample concentrations were $\geq 10 \times$ CRDL.

D5** Preparation duplicate precision exceeded the maximum expected %RSD, and either the routine or the duplicate value was $\geq 10 \times$ CRDL.

D6** Preparation duplicate precision exceeded the maximum expected %RSD, and both the routine and the duplicate sample concentrations were $\geq 10 \times$ CRDL.

D7** Audit duplicate precision exceeded the maximum expected %RSD, and either of the audit sample concentrations was $\geq 10 \times$ CRDL.

D8** Audit duplicate precision exceeded the maximum expected %RSD, and both audit pair concentrations were $\geq 10 \times$ CRDL.

Generated for Known Relationships of Sulfur Isotherms

KO** Elemental parameter out of range; used for total C, N, and S only.

K1** Organic soil (total C 20-60%) and $\text{SO}_4 - \text{H}_2\text{O} > 1.05 \times \text{SO}_4 - \text{PO}_4$.

K2** Mineral soil (total C 0-20%) and $\text{SO}_4 - \text{H}_2\text{O} > 1.05 \times \text{SO}_4 - \text{PO}_4$.

(continued)

Table 14-2. (Continued)

K3** Organic soil: $1,000 \times \text{Total S} < \text{SO}_4 - \text{PO}_4$ or $\text{SO}_4 - \text{H}_2\text{O}$.

K4** Mineral soil: $3,000 \times \text{Total S} < \text{SO}_4 - \text{PO}_4$ or $\text{SO}_4 - \text{H}_2\text{O}$.

Generated by Detection Limit Exception Program

L1* Instrumental detection limit (IDL) exceeded contract required detection limit (CRDL) and sample concentration was $< 10 \times \text{CRDL}$.

Miscellaneous

MO* Value was obtained by using a method that is unacceptable according to the contract.

D8** Audit duplicate precision exceeded the maximum expected %RSD, and both audit pair concentrations were $\geq 10 \times \text{CRDL}$.

Generated for Known Relationships of Sulfur Isotherms

KO** Elemental parameter out of range; used for total C, N, and S only.

K1** Organic soil (total C 20-60%) and $\text{SO}_4 - \text{H}_2\text{O} > 1.05 \times \text{SO}_4 - \text{PO}_4$.

K2** Mineral soil (total C 0-20%) and $\text{SO}_4 - \text{H}_2\text{O} > 1.05 \times \text{SO}_4 - \text{PO}_4$.

K3** Organic soil: $1,000 \times \text{Total S} < \text{SO}_4 - \text{PO}_4$ or $\text{SO}_4 - \text{H}_2\text{O}$.

K4** Mineral soil: $3,000 \times \text{Total S} < \text{SO}_4 - \text{PO}_4$ or $\text{SO}_4 - \text{H}_2\text{O}$.

Generated by Detection Limit Exception Program

L1* Instrumental detection limit (IDL) exceeded contract required detection limit (CRDL) and sample concentration was $< 10 \times \text{CRDL}$.

Miscellaneous

MO* Value was obtained by using a method that is unacceptable according to the contract.

Generated by Audit Check Program

NO** Audit sample value exceeded upper control limit.

N1** Audit sample value was below lower control limit.

N2** Audit sample value exceeded control limits; audit sample preparation procedure is suspect.

Generated by QCCS Exception Program(s)

Q1** Quality control calibration sample (QCCS) was above contractual criteria.

Q2** QCCS was below contractual criteria.

Q3** Insufficient number of QCCSs were measured.

Q4** Detection limit QCCS was not $3 \times \text{CRDL}$ and measured detection limit (DL) QCCS value was not within 20% of the theoretical concentration.

Generated by Matrix Spike Program

S1** Percent recovery of matrix spike was above contractual criteria ($100 \pm 15\%$).

S2** Percent recovery of matrix spike was below contractual criteria ($100 \pm 15\%$).

(continued)

Table 14-2. (Continued)

Miscellaneous

WO* Air dry sample weight was not within contractual requirement.

Miscellaneous (*flagged data not to be included in any statistical analyses*)

XO* *Invalid* but confirmed data based on QA/QC data review.

X1* *Invalid* but confirmed data - potential gross contamination of sample or parameter.

X2* *Invalid* but confirmed data - potential sample switch.

* Sample Flag: Flag the affected parameter for the *affected* samples only.

** Parameter Flag: Flag the affected parameter for *ALL* samples in the batch (the assumption is that QA/QC represents all samples in the batch).

Section 15

Quality Assurance Plan for Mineralogy

15.1 Introduction

Mineralogical analyses are performed on a subset of soil horizons studied during the DDRP soil survey. The methods used for mineralogical analyses include X-ray diffraction spectrometry, wavelength-dispersive X-ray spectrometry, and scanning electron microscopy/energy-dispersive X-ray spectrometry (Cappo et al., 1987). To meet requirements for data precision, accuracy, representativeness, and completeness, specialized QA/QC procedures are presented for use with these analytical methods.

15.2 Project Description

ERL-C designated a subset of the soil samples for mineralogical analysis. The specific goals of the mineralogical study include:

- Identifying and quantifying the clay minerals present in the soils.
- Identifying and quantifying the other minerals present in the soils.
- Characterizing the chemistry of the whole sample and of the clay fraction.
- Assessing the variability of the mineralogical and chemical characteristics.
- Establishing the chemical contribution that mineral weathering makes to the soil.
- Assessing the effect that clay content and heavy-mineral content have on the acid-neutralizing capacity of the soil.

15.3 Project Organization

Section 3.0 addresses project organization.

15.4 Quality Assurance Objectives

15.4.1 Soil Sampling

Section 4.1 addresses soil sampling.

15.4.2 Sample Preparation

15.4.2.1 Precision and Accuracy--

After processing, i.e., air-drying, disaggregating, sieving, and homogenization, the preparation laboratory uses a Jones-type riffle splitter to prepare 500-g subsamples from the routine soil samples and special interest watershed (SIW) samples designated by ERL-C. Comparison of physical and chemical data for these duplicates allows evaluation of the subsampling procedure.

15.4.2.2 Representativeness--

After homogenization as described in Section 4.2.2, each subsample is reduced to a 500-g aliquot by successive passes through a Jones-type riffle splitter. This procedure maintains the representativeness of the sample.

15.4.2.3 Completeness--

Samples from mineral soil horizons designated by ERL-C are analyzed for mineralogy. Sample batches sent to each mineralogical laboratory include 23 percent QA/QC samples for the routine air-dry soil samples and for the SIW samples. Each sample batch consists of 20 routine or SIW samples, 3 duplicates, and 3 audit samples. One audit sample in each batch is a synthetic sample.

15.4.2.4 Comparability--

All preparation laboratories process bulk samples according to protocols documented in Bartz et al. (1987). Strict adherence to protocols is required to ensure comparability among preparation laboratories.

15.4.3 Laboratory Analysis

15.4.3.1 Precision and Accuracy--

The data quality objectives (DQOs) for precision and accuracy of the analyses are presented in Table 15-1. The structure of Table 15-1 is as follows:

Reporting Units - specifies the units in which the laboratory data should be reported.

Reporting format - specified the significant figures to which the data should be reported.

Expected Range - specifies the range of values expected to occur naturally in the soil sampled, independent of measurement error.

Lower Reporting Limit - this value has been extrapolated to that of the reporting unit; if the sample values are lower than stated, the "limit of reproducibility" is approached.

Table 15-1. Mineralogical Data Quality Objectives

Parameter	Reported Unit	Reported Format	Expected Range	Lower Reporting Limit	Precision at Lower Limit	Precision at Upper Limit
1. Minerals in <2-mm fraction	%	±1%	0-100%	2%	±0.1%	±0.1%
2. Minerals <0.002-mm fraction	%	±1%	0-100%	2%	±0.1%	±0.1%
3. Reference Intensity Ratios	D	±0.01 units	10,000 cps	0.0%	0.1%	0.1%
4. Light Minerals	wt %	±0.1%	0-80%	NA	NA	NA
5. Heavy Minerals	wt %	±0.1%	0-20%	NA	NA	NA
6. Clay Minerals	wt %	±0.1%	0-100%	NA	NA	NA
7. Morphological features	NA	Written description with photograph	NA	NA	NA	NA

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8. Na	%Na ₂ O	±0.1%	0-10%	0.6%	1.0%	1.0%
9. Mg	%MgO	±0.1%	0-5%	1.0%	1.0%	1.0%
10. Ca	%CaO	±0.1%	0-5%	1.0%	1.0%	1.0%

D = Dimensionless number
NA = Not applicable

(continued)

Table 15-1 (Continued)

Parameter	Reported Unit	Reported Format	Expected Range	Lower Reporting Limit	Precision at Lower Limit	Precision at Upper Limit
11. Al	%Al ₂ O ₃	±0.1%	0-5%	1.0%	1.0%	1.0%
12. Si	%SiO ₂	±0.1%	0-20%	4.0%	1.0%	1.0%
13. P	ppm P ₂ O ₅	±1 ppm	<1%	100 ppm	1.0%	1.0%
14. Cl	ppm	±1 ppm	<1%	120 ppm	1.0%	1.0%
15. K	%K ₂ O	±0.1%	0-10%	0.8%	1.0%	1.0%
16. Ti	%TiO ₂	±0.1%	<1%	0.1%	1.0%	1.0%
17. Cr	ppm	±1 ppm	<1%	400 ppm	1.0%	1.0%
18. Mn	ppm MnO ₂	±1 ppm	<1%	300 ppm	1.0%	1.0%
19. Fe*	%Fe ₂ O ₃	±0.1%	0-20%	0.8%	1.0%	1.0%
20. Co	ppm	±1 ppm	<1%	50 ppm	1.0%	1.0%
21. Ni	ppm	±1 ppm	<1%	50 ppm	1.0%	1.0%
22. Cu	ppm	±1 ppm	<1%	50 ppm	1.0%	1.0%
23. Zn	ppm	±1 ppm	<1%	50 ppm	1.0%	1.0%
24. Rb	ppm	±1 ppm	<1%	30 ppm	1.0%	1.0%
25. Sr	ppm SrO	±1 ppm	<1%	50 ppm	1.0%	1.0%
26. Ba	ppm	±1 ppm	<1%	50 ppm	1.0%	1.0%
27. La	ppm	±1 ppm	<1%	200 ppm	1.0%	1.0%
28. Ce	ppm	±1 ppm	<1%	200 ppm	1.0%	1.0%
29. Pb	ppm	±1 ppm	<1%	40 ppm	1.0%	1.0%
30. Th	ppm	±1 ppm	<1%	40 ppm	1.0%	1.0%
31. U	ppm	±1 ppm	<1%	40 ppm	1.0%	1.0%
32. Zr	ppm	±1 ppm	<1%	200 ppm	1.0%	1.0%
33. S	ppm	±1 ppm	<1%	320 ppm	1.0%	1.0%

*Fe represents both +2 and +3 oxidation states of Iron and is reported as %Fe₂O₃.

Precision at the Lower Limit - serves as a guideline to define the acceptable absolute percent standard deviation beyond which the analytical reproducibility for low concentration samples is questionable and often not attainable.

Precision at the Upper Limit - serves as a guideline to define the acceptable percent relative standard deviation beyond which the analytical reproducibility for high concentration samples is questionable.

15.4.3.2 Representativeness--

Section 4.3.2 addresses representativeness.

15.4.3.3 Completeness

Section 4.3.3 addresses completeness.

15.4.3.4 Comparability

Section 4.3.4 addresses comparability.

15.5 Strategy of Sample Selection for Mineralogical Analysis

The strategy used to select specific watersheds and sampling sites is described in Section 5.0. This section details the selection of a subsample of soil horizons for mineralogical analysis from all horizons sampled.

15.5.1 Constraints

A dBase III file is used to index the pedons sampled into sampling classes, e.g., E2, and within each sampling class by lake ID, e.g., 1A1-012. Histic soils, i.e., sampling classes H1, H2, and H3, are not candidates for

mineralogical analyses because they do not have a significant mineral component; therefore, samples for mineralogical analyses are selected randomly from the remaining pedons within the sampling classes.

For the routine and special interest watersheds, samples were selected in pairs from each mineral sampling class; one sample is selected from the most weathered mineral horizon, and one is selected from the least weathered mineral horizon within a pedon. Additional pairs of samples are selected from the E2, I2, I33, S12, and S16 sampling classes. The I33 sampling class (Inceptisol order) represents the largest land surface area studied, and the S12 sampling class (Spodosol order) represents the next largest land area studied. Additional mineralogical samples are chosen from the E2, I2, and S16 sampling classes because these classes are thought to be regionally representative of the mineral soil orders mapped.

For quality control, 15 percent of the samples are collected in duplicate. These duplicate samples are selected randomly.

15.5.2 Limitations to Selection Criteria

The following situations disqualify a specific horizon as a choice for mineralogical analyses:

- **Lithological discontinuity** - A lithological discontinuity within a pedon indicates that the upper mineral horizons were developed from a parent material other than the one present in the C horizon. In this situation, the mineralogical relationship between the upper horizons and the C horizon is ambiguous. If a pedon is disqualified for this reason, the selection procedure is repeated until a suitable pedon is selected.
- **Horizon not sampled** - Within a pedon, a horizon described on the field data form and assigned a sample code may not be sampled if the quantity of soil is insufficient. When a pedon containing an

unsampled horizon is chosen randomly for mineralogical analyses, the selection procedure is repeated until a qualifying pedon is selected.

If a sampling class contains pedons that have only one mineral horizon, it is difficult to study the extent of mineral weathering. Therefore, rather than selecting paired samples from the same pedon, two different pedons are selected randomly from the sampling class.

15.5.3 Selection Procedure

A random number, X , was generated on a Hewlett Packard-15C calculator, where $0 < X < 1$. Next, X was multiplied by the number of pedons, N , within the specific sampling class. The decimal portion of the resulting number was truncated to give an integer. To this integer, one (1) was added to result in a random number, i , which ranged from one (1) to N . Counting from the first pedon in each indexed sampling class, the i th pedon was selected for mineralogical analyses.

The procedure was repeated until one unique pedon was selected from each of the eligible sampling classes. For sampling classes E2, I2, I33, S12, and S16, the procedure was repeated to select a total of four unique pedons from each sampling class.

Fifteen duplicate samples were chosen from the first fifteen paired samples by the toss of a coin. From each pair, either the most weathered or the least weathered mineral horizon was selected as a duplicate sample.

This selection procedure was repeated to select mineralogical samples from the special interest watersheds.

15.6 Sampling Internal Quality Control

Sampling internal quality control is detailed in Section 7.0.

15.7 Preparation Laboratory Internal Quality Control

Preparation laboratory internal quality control is discussed in detail in Section 8.0.

One audit sample per batch is synthetic. It is comprised of separate, naturally occurring minerals which have been combined in known weights. The other two audit samples are taken from the audit samples prepared from the C, B_s, and B_w horizons.

The preparation laboratory splits a 500-g aliquot from each bulk soil sample. This is stored in a 500-mL high-density polyethylene bottle for shipment to EMSL-LV. EMSL-LV prepares the sample batches and ships the batches to the mineralogical laboratory. A mineralogical batch of 26 samples consists of 20 routine or SIW samples, 3 duplicates, and 3 audit samples.

15.8 Laboratory Procedures

Analytical and mineralogical procedures are detailed in Cappo et al. (1987). Table 15-2 summarizes the parameters determined and the corresponding analytical techniques.

15.9 Mineralogical Laboratory Internal Quality Control

15.9.1 Sample Receipt

Section 9.0 addresses sample receipt and laboratory documentation for quality control.

15.9.2 X-ray Diffraction Spectrometry

15.9.2.1 Sample Preparation and Analysis--

Each sample must have a uniform particle size (less than or equal to 0.002 mm) prior to analysis to reduce the matrix adsorption effect. A titanium carbide ring-and-puck pulverizer is recommended for initial particle-size reduction to about 0.040 mm. For the final particle-size reduction to ≤ 0.002 mm, an automated mortar and pestle with the addition of acetone is required. For the first five samples of the first batch, a check on the particle-size distribution of the prepared sample is required. Each batch of samples including a duplicate sample is prepared by the same technician.

15.9.2.2 Initial Alignment and Continuing Calibration--

An initial alignment is performed with National Bureau of Standards (NBS) standard reference material (SRM) number 640A silicon powder as required in the analytical method. As a part of an on-going check on the alignment and intensity of the X-ray tube, the silicon powder calibration standard is X-rayed after half the samples are X-rayed and after the last sample has been X-rayed. All three patterns are included in the data package.

Table 15-2. Mineralogical Parameters and Corresponding Analytical Techniques

Parameter	Method
Mineralogy of <2-mm and <0.002 mm fractions	X-ray diffraction spectrometry
Elemental analysis of bulk sample and of clay fraction	Wavelength-dispersive X-ray spectrometry
Mineralogy of heavy mineral fraction	Scanning electron microscopy/energy dispersive X-ray spectrometry
Morphological features of samples	Scanning electron microscopy/energy dispersive X-ray spectrometry

15.9.2.3 Instrumental Requirements--

Copper K_α radiation is required for these analyses. The goniometer speed is determined by the intensity of the X-rays generated and is dependent upon the brand and age of the diffractometer. Refer to Cappelletti et al. (1987) for the requirements. The patterns are stored digitally in the computer until they are printed for the data package.

15.9.2.4 Determination of the Reference Intensity Ratios--

Each diffractometer yields slightly different patterns and reflection intensities. To establish the reference intensity ratios for the external standard, pure corundum is mixed with quartz, albite, orthoclase, hornblende, montmorillonite, illite, and kaolinite in equal amounts. This corundum reference standard is X-rayed, the area under the strongest peak of each mineral is integrated, and the reference intensity ratios are computed. As part of the internal QC, the corundum-reference standard must be X-rayed after every 60 samples for the <2-mm and <0.002-mm randomly oriented powder mounts only. The calculation of the reference intensity ratios is based on the most recent analysis of the standard.

15.9.2.5 Data Reporting--

All required X-ray diffraction (XRD) patterns are included with the data package. Each pattern is indexed. Indexing includes marking the 2θ in 1° increments, marking the 2θ of the starting and ending points of the pattern, and labeling each peak with a 2θ number, the equivalent angstrom units, the mineral name, and the number of the Joint Committee on Powder Diffraction Standards (JCPDS) card used to identify each mineral. On each pattern, the sample number, size fraction, type of mount (i.e., oriented or randomly oriented), treatments, date of analysis, goniometer speed, scale, and the millivolt (mV), milliamperes (mA), and time constant settings are recorded.

15.9.3 Wavelength-dispersive X-ray Spectrometry

15.9.3.1 Sample Preparation and Analysis--

The <2-mm fraction and the <0.002-mm fraction are pelletized and analyzed as separate samples. Half-batch lots are analyzed if the X-ray fluorescence (XRF) carousel does not accommodate a full batch of samples.

15.9.3.2 Background Signal Corrections--

Background signals are determined and subtracted by software developed at Oregon State University. For each fixed channel, the dependence of the background signal on the average atomic number, Z , of the sample matrix is established from measurements of 30 samples. These consist of pure-element oxides, salts, and mixtures of salts and oxides, which represent a Z range from 10 to 25. This range accurately represents the Z range expected in naturally occurring samples such as soils, rocks, and ocean sediments. The measured background signals (I_{peak}) for the fixed channel are related to measurements of the scattered continuum (I_{cont}) obtained at one of several 20° angles.

$$\begin{aligned} I_{\text{peak}} &= k I_{\text{cont}} \\ k &= I_{\text{peak}}/I_{\text{cont}} \end{aligned}$$

Plots of k versus I_{cont} permit calculation of k if I_{cont} is known. For routine sample measurements, I_{cont} is measured, then the software calculates k for each fixed channel. The background is subtracted automatically because $I_{\text{peak}} = k I_{\text{cont}}$.

15.9.3.3 Spectral Interferences--

Spectral interferences are greatly minimized through the inherently high resolution of the wavelength-dispersive XRF. Some peak overlaps do occur, however. For each overlap situation, standards containing a fixed concentration of analyte and a varying concentration of the interfering element are prepared and

analyzed. The appropriate functional relationships are developed to permit software to predict and subtract the contribution of an interfering element to the measured analyte signal.

15.9.3.4 Corrections for Interelement Effects--

Interelement effects are dealt with through software similar to that developed by Criss' Software, Inc. The software is used to convert from measured X-ray fluorescence line intensities to chemical composition. The software uses measured net intensities from standards to establish a set of theoretical and empirical coefficients that fit the concentration versus intensity relationships over the range of compositions represented by the standards.

15.9.3.5 Initial Calibration--

A suite of at least 25 certified standard rock and sediment samples is analyzed by XRF, according to the same measurement parameters that are used for routine samples. Background signals are subtracted, and possible spectral interferences are corrected for as described in Section 15.10.3.2 and Section 15.10.3.3. Software described in Section 15.10.3.4 is used to establish data files which contain the calibration information required to convert the measured net intensities from routine samples to elemental concentrations. The calibration of the XRF using the software requires entering the known elemental concentrations for the standards and their measured net intensities. The measured intensities for the standards may be scaled in any appropriate manner as long as the scaling is applied consistently. To ensure that the calibration is not affected by differences in instrument response due to such factors as replacement of a detector, changing of a tank of detector gas, or long-term drift, all sample and standard net element signals are divided by the corresponding monitor net element signal. A monitor sample is measured several times during the analysis of each suite of samples, and the same monitor sample is used for all sample and standard runs. The instrument is calibrated in terms of signal ratios.

15.9.3.6 Quality Control Calibration Standards--

QCCSs are rock standards certified by the United States Geological Survey (USGS). The USGS standards are obtained already ground, and the analyst at the contractor laboratory pelletizes and analyzes the standards. Spectra are stored digitally for cross-referencing by the software.

15.9.3.7 Dispersion Crystals--

The manufacturer sets the dispersion crystals in the spectrometer for the simultaneous analysis of 25 elements. There are three adjustable spectrometers available for sequential analysis of elements that are not among the 25 analyzed in the simultaneous mode.

15.9.3.8 X-ray Target--

The manufacturer provides a rhodium target in the X-ray tube.

15.9.3.9 Acquisition--

Spectral acquisition is 300 seconds.

15.9.3.10 Duplicate Sample Analysis--

Duplicate analysis is performed on a separate portion of each thirteenth routine sample.

15.9.3.11 Continuing Sample Analysis--

A monitor standard is included in the sample set. The monitor standard measures instrument performance and must be analyzed three times or more during the analysis of each set of samples; the check standard is treated as a normal sample so that its measured concentrations may be checked after each set of samples has been analyzed. The data is stored digitally for later printing.

15.9.3.12 Instrumental Detection Limit--

The instrumental detection limit is established for each element. The concentration at the detection limit, C_L , is defined as that amount of analyte which gives a net line intensity equal to three times the square root of the background intensity for a specified counting time.

15.9.3.13 Data Reporting--

Results obtained from each kind of analysis are recorded on the data forms in Appendix J. After a sample is analyzed completely, the results are summarized on the summary data forms and are annotated by the data qualifiers listed in Table 9.5, if applicable. Results should be reported to the number of decimal places in the current instrumental detection limit to a maximum of three significant figures. The laboratory manager must sign each completed form to indicate that he or she has reviewed the data and that the samples were analyzed exactly as described in the protocol. Any deviations from protocol require authorization of the QA manager prior to sample analysis.

15.9.4 Scanning Electron Microscopy/Energy-Dispersive X-ray Spectrometry (SEM/EDXRF)

15.9.4.1 Sample Preparation and Analysis--

For this method, the light and heavy minerals of the very fine sand fraction (0.105-0.053 mm) and the clay fraction (less than or equal to 0.002 mm) are studied. These procedures are described in Cappelletti et al. (1987).

15.9.4.2 Quality Assurance Objectives for Energy-Dispersive X-ray Analysis--

15.9.4.2.1 Precision -- Precision must be within 2 percent relative standard deviation when not limited by counting statistics. Precision is

assessed by computing the standard deviation of measurements from the QC calibration standard. Individual standard deviations are computed for each element in the standard. An overall value is computed as the mean and individual standard deviation.

15.9.4.2.2 Accuracy -- Accuracy must be within 5 percent of true concentration when not limited by counting statistics. As elemental concentrations approach the detection limits, precision and accuracy become poorer because of the effect of counting statistics.

15.9.4.2.3 Completeness -- All samples submitted are analyzed. Data completeness is computed by the following equation:

$$\text{completeness, \%} = (100) \left(\frac{\text{Number samples analyzed}}{\text{Number samples received}} \right)$$

15.9.4.2.4 Representativeness -- The analysis area is an elliptical spot about 10 by 12 mm near the center of the pellet.

15.9.4.3 Calibration Procedures and Frequency--

The instrument is calibrated with NBS-certified or USGS-certified standard reference materials. For each XRF analytical batch, a multielement QC calibration standard is analyzed. Measured concentrations of the QC calibration standard are compared with actual concentrations. If the results show a trend or drift, recalibration is required. The instrument generally maintains calibration stability for 3 to 4 months.

15.9.4.4 Energy Dispersive X-ray Fluorescence (XRF) Analyzer-

Atoms in the sample are excited from their ground state to higher energy levels by radiation from an X-ray tube. These excited atoms emit X-rays of discrete energies as they return to their normal ground-state energy level. The energy of these X-rays is characteristic of the emitting element and is used to identify the element qualitatively. The number of observed X-rays, which is proportional to the number of atoms, is used to determine

quantitatively the concentration of a specific element through a direct comparison (by the software) with certified reference standards.

There are potential spectral interferences with the energy dispersive (ED) XRF method because of its low resolution relative to the number and spacing of possible X-ray lines. Correction factors are determined by analyzing single-element standards and by quantifying their interference with other elements. Samples exhibiting chemical composition uncharacteristic of normal samples may require additional corrections. The software automatically makes all the calculations and corrections.

15.9.4.5 Data Reduction, Validation, and Reporting--

For each element measured, data in units of ppm or percent are processed at the time of analysis. These calculations are an integral part of the analytical software. Results are recorded in both floppy disk and hardcopy formats. In addition, the raw spectra are saved on floppy disk.

15.9.4.6 Internal Quality Control

A multielement standard is analyzed after every batch of 15 samples. Results of that analysis are compared with true concentrations. If the deviation is greater than 2 percent, all samples of that batch must be reanalyzed.

15.9.4.7 Preventive Maintenance--

The Si(Li) detector is cooled with liquid nitrogen by filling the Dewar flask every week. Routine cleaning and maintenance is performed semiannually.

15.10 Acceptance Criteria

The acceptance criterion for the relative standard deviation (RSD) of duplicate sample results is based on the upper 95th percentile of observed values of RSD. Because the RSD is affected by concentration, this criterion is applied only when the mean of the duplicate

analyses exceeds the contract-required detection limit by a factor of 10. Arbitrary acceptance criteria are used until sufficient (at least 20) RSD values have been observed. No outlier test is applied to the RSD values prior to estimating the upper 95th percentile.

15.11 Data Management System

Section 12.0 describes the data management system.

15.12 Performance and System Audits

15.12.1 QA/QC Samples

Reference standards are USGS-certified rock samples for the XRF methods. Microprobe standards and the corundum used in the semiquantitative X-ray diffraction (SQXRD) method are certified by the manufacturers.

15.12.2 Laboratory On-Site Evaluations

Each mineralogical laboratory can expect two on-site evaluations. A QA representative makes the first on-site evaluation before analysis begins and makes another during analysis. The questionnaire in Appendix K is completed during this evaluation.

15.13 Review of Mineralogical Data

15.13.1 Communications

Section 13.3.1 addresses communications.

15.13.2 Preliminary Data Package Review

Each sample data package is reviewed as described below:

- Log sample data package into master tracking notebook and indicate in notebook if data package arrived late.
- Review cover letter.
- Complete Data Package Completeness Checklist (given in Appendix L) to review internal QC data, data completeness, and data qualifiers used.
- Notify the contractor laboratory of any major discrepancies, and record corrective action.

15.13.3 Quality Assurance Reports to Management

Results of precision, accuracy, and completeness are included in the final summary report. Also included is a discussion of data quality and of all specific deviations from protocol and from the QA plan.

15.14 Data Verification

The data package is reviewed for completeness of the required patterns. The XRD patterns from the routine samples are reviewed for completeness of the required indexing

information. Each mineral in the pattern is compared to the duplicates, audit samples, and JCPDS card file that is the accepted reference standard used to identify the mineral for the qualitative portion of the SQXRD data verification. Reference intensity ratios (RIR) are checked against the corundum standard for the quantitative portion of the SQXRD data verification. The percent clay data from the SQXRD analysis along with the chemical composition (XRF), cation exchange capacity, and specific surface data are used in a simultaneous linear equation clay analysis (SLECA) computer program which refines the clay data.

The elemental analysis data for the soils are reviewed for completeness as described in Cappelletti et al. (1987). The elemental data are compared against the duplicates, audit sample, and reference standard data.

The SEM/EDXRF pictures and elemental data are reviewed for completeness as described in Cappelletti et al. (1987). The pictures are reviewed and are compared with the minerals identified by SQXRD and with the elemental compositions identified by the EDXRF analyses. The EDXRF elemental data are compared with the data for the reference standards.

Section 16 ***References***

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Appendix A

Forms and Legends for Reporting Field Data

Field data describing each sampled pedon are recorded on the SCS-232 form. This appendix also includes specific information on the abbreviations used on this form, as well as the soil description codes that are used in completing it.

FORM SCS-SOI-232

U.S. DEPARTMENT OF AGRICULTURE
SOIL CONSERVATION SERVICE

SOIL DESCRIPTION

SOIL SERIES NAME										SAMPLE NUMBER										LATITUDE										LONGITUDE										DATE																			
ST COUNTY UNIT										DEG MIN SEC										DEG MIN SEC										MO DAY YEAR																													
SLOPE SHAPE CODES (1-5)										PEDON CLASSIFICATION										PRECIP										WATER TABLE										TEMP																			
ELEVATION METERS										PARENT MATERIAL										AVERAGE AIR TEMPERATURES °C										AVERAGE SOIL TEMPERATURES °C										WEATHER STATION NUMBER																			
CONTROL SECTION										DEPTH										DEPTH										DEPTH										DEPTH										FLOODING									
VEGETATION SPECIES										DESCRIPTORS NAMES										CREW ID																																							

SOIL DESCRIPTION CODES FOR FORM SCS-SOI-232

SLOPE SHAPE CODES (1-5) 1 Convex 2 Plane 3 Concave 4 Inclining 5 Complex										GEOMORPHIC POSITION CODES (GM) 01 Summit crested hills 02 Shoulder crested hills 03 Backslope crested hills 04 Backslope sideslope 05 Footslope sideslope 06 Footslope headslope 07 Summit interfluve 08 Shoulder interfluve 09 Backslope headslope 10 Backslope interfluve 11 Footslope headslope 12 Footslope interfluve 13 Torslope crested hills 14 Torslope sideslope 15 Not applicable										SLOPE ASPECT CODES (ASP) 1 Northeast 2 East 3 Southeast 4 South 5 Southwest 6 West 7 Northwest 8 North										PEDON POSITION CODES (POS) 1 On the crest 2 On slope and crest 3 On upper third 4 On middle third 5 On lower third 6 On a slope 7 On a slope and depression 8 In a depression 9 In a depression										PHYSIOGRAPHY (PHYS) A Coastal Plains B Intermountain basin C Lake plains D River valley E Glaciated uplands F Glaciated lowland G Basin H Level or undulating uplands I Mountains or deeply dissected plateaus J High hills K Piedmonts L Plateaus or tablelands M Mountain valleys or canyons										LOCAL LANDFORM CODES (LC) A Fall B Bog C Curbs or hogback D Dome or volcanic cone E Escarpment F Broad plain G Gully H Abandoned channel I Inside or mountainside J Moirine K Kamefield L Drumlin M Mound or butte N Low sand ridge O Flood plain P Playa or alluvial flat Q Upland slope R Sand dune or hill S Terrace stream or lake T Terrace outwash or marine V Pediment W Swamp or marsh X Salt marsh Y Barrier bar Z Back barrier flat										PERMEABILITY CODES (PM) 1 Very slow <0.06 in/hr 2 Slow 0.06 - 0.2 in/hr 3 Moderately slow 0.2 - 0.6 in/hr 4 Moderate 0.6 - 2.0 in/hr 5 Moderately rapid 2.0 - 6.0 in/hr 6 Rapid 6.0 - 20.0 in/hr 7 Very rapid >20.0 in/hr										DIAGNOSTIC FEATURE CODES (DF) 1 Very poorly drained 2 Poorly drained 3 Somewhat poorly drained 4 Moderately well drained 5 Well drained 6 Somewhat excessively drained 7 Excessively drained										MOISTURE REGIME CODES (MR) AR Aridic moisture regime US Ustic moisture regime AQ Aquic moisture regime UE Ustic moisture regime UD Ustic moisture regime UO Ustic moisture regime PU Perudic moisture regime										EROSION CODES (ER) 0 None 1 Slight 2 Moderate 3 Severe										STONINESS CLASS (ST) 1 Class 1 2 Class 2 3 Class 3 4 Class 4 5 Class 5									
--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	---	--	--	--	--	--	--	--	--	--	---	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	---	--	--	--	--	--	--	--	--	--	---	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	---	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

FORM SCS-SOI-232 (Continued)

DEPTH	HORIZONTAL DESIGNATION		THICKNESS AVG MAX MIN	DRY COLOR			MOIST COLOR			TEXTURE				
	UPPER LOWER	D S C		MASTER LETTER	SUFFIX	HUE	V A L	C H R	L O C	HUE	V A L	C H R	CLASS	MOD
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														

COLOR LOCATION AND TREATMENT
0 - upper bed; 2 - ped exterior;
1 - ped interior; 3 - rubbed or crushed

TEXTURE CLASSES

C. Clay	CND. Cinder
CL. Clay loam	COS. Coarse sand
COSL. Coarse sandy loam	CSCL. Coarse sandy clay loam
FM. Fragmental material	FS. Fine sand
FSL. Fine sandy loam	G. Gravel
L. Loam	LCOS. Loamy coarse sand
LFS. Loamy fine sand	LS. Loamy sand
LVS. Loamy very fine sand	S. Sand
SC. Sandy clay	SCL. Sandy clay loam
SG. Sand and gravel	SL. Silt
SIC. Silty clay	SICL. Silty clay loam
SIL. Silty loam	SL. Sandy loam
VLS. Very fine sand	VSL. Very fine sandy loam
ICE. Ice in frozen soil	GYP. Gypsiferous earth
VAR. Variable	GE. Gypsiferous earth
DE. Dermalaceous earth	FB. Fibric material
MARL. Marl	MPT. Mucky peat
MUCK. Muck	PDOM. Partially decomposed organic
UDOM. Undecomposed organic	PEAT. Peat
SP. Sapric material	HM. Humic material
DPWD. Oxide protected weathered bedrock	CEM. Cemented
U. Unknown texture	UWB. Unweathered bedrock
WB. Weathered bedrock	IND. Indurated

TEXTURE MODIFIERS

RY. Brittle	GRG. Coarse gravelly
ST. Sticky	SH. Shaly
CR. Cobbly	SY. Silty
CB. Extremely cobbly	CY. Cindery

CNV. Extremely channery	SM. Mucky
CRV. Very cherty	GVV. Very gritty
FLV. Very flaggy	BVZ. Extremely bouldery
GRF. Fine gravelly	STZ. Extremely stony
GRE. Extremely gravelly	CBV. Very cobbly
SHZ. Extremely shaly	CMV. Very channery
SVZ. Extremely silty	CRZ. Coarse cherty
SR. Stratified	FL. Flaggy
GV. Gravelly	GR. Gravelly
BVV. Very bouldery	GRV. Very gravelly
STV. Very stony	SHV. Very shaly
CRZ. Angular cobbly	STV. Very silty
CH. Channery	AV. Ashy
CR. Cherty	PT. Peaty
CRX. Extremely cherty	GVZ. Extremely gritty
FLX. Extremely flaggy	RB. Rubbly

GRADE OF STRUCTURE

0. Not used
1. Weak
2. Moderate
3. Strong
4. Very strong
5. Weak and moderate
6. Moderate and strong

STRUCTURE SHAPE

PL. Platy
COL. Columnar
SBK. Subangular blocky
CR. Crumb
WED. Wedge
LF. Lenticular

FM. Fine and medium
M. Medium
MC. Medium and coarse
CO. Coarse
VC. Very coarse
CV. Coarse and very coarse

SIZE OF STRUCTURE

EF. Extremely fine
VF. Very fine
F. Fine
FM. Fine and medium
M. Medium
MC. Medium and coarse
CO. Coarse
VC. Very coarse
CV. Coarse and very coarse

DRY CONSISTENCE

L. Loose
S. Soft
SH. Slightly hard
H. Hard
VM. Very hard
EH. Extremely hard

MOIST CONSISTENCE

L. Loose
VR. Very friable
FR. Friable
FI. Firm
VFI. Very firm
EFI. Extremely firm

OTHER CONSISTENCE

WSM. Weakly smeary
SM. Strongly smeary
MS. Moderately smeary
B. Brittle
R. Rapid
VR. Very rigid
CO. Unconsolidated
VWC. Very weakly cemented
WC. Weakly cemented
SC. Strongly cemented
I. Indurated
SE. Slightly hard
VF. Very hard

STICKINESS (SI)

SO. Non sticky
SS. Slightly sticky
S. Sticky
VS. Very sticky

FORM SCS-SOI-232 (Continued)

STRUCTURE		CONSISTENCE		MOTTLES		SURFACE FEATURES		EFFER- VES- CENCE		
G R D	SZ	DRY MOIST OTHER	ST PL E M	A R	C O N	V A M L	R A M C D T N	D L O C	V A M L	
										1
										2
										3
										4
										5
										6
										7
										8
										9
										10

MOTTLE ABUNDANCE CODES

F Few
C Common
M Many

MOTTLE SIZE CODES

1 Fine < 1mm
2 Med 1mm - 5mm
3 Coarse 5 - 10mm
12 Fin. to medium
13 Fine to coarse
23 Coarse to medium

MOTTLE CONTRAST CODES

F Faint
D Distinct
P Prominent
D Distinct
P Prominent

PLASTICITY

PO Non plastic
SP Slightly plastic
P Plastic
VP Very plastic

SURFACE FEATURE

LOCATION CODES (LOC)

P On faces of peds

H On horizontal faces of peds

V On vertical faces of peds

Z On vertical and

horizontal faces of peds

U On upper surfaces of peds or stones

L On lower surfaces of peds or stones

C On tops of columns

M On bottoms of peds

SURFACE FEATURES (KND)

U Coils
B Black stains
D Clay bridging
I Iron stains
Q Noninterlocking spherulodes
L Lime or carbonate coats
M Manganese or iron manganese stains
S Salterans (sand or silt)
X Oxide coats
A Spherulans over curans
C Chalcocite on opal
G Gubinite coats
H Interlocking spherulodes
P Pressure facets
O Organic coats
T Clay films

AMOUNT (AMT)

V Very few
F Few
C Common
M Many

CONTINUITY (CN)

P Patchy
D Discontinuous
C Continuous

DISTINCTNESS (DST)

F Faint
D Distinct
P Prominent

BOUNDARY

DISTINCTNESS TOPOGRAPHY
A Abrupt S Smooth
C Clear W Wavy
G Gradual I Irregular
D Diffuse B Broken

EFFERVESCENCE (EFF)

1 Slightly effervescent
2 Strongly effervescent
3 Violently effervescent
0 Very slightly effervescent

EFFERVESCENCE AGENT CODES

H HCl (Unspecified)
D H2O2 (3.4%)
I HCl (10%)
P H2O2 (Unspecified)

EXTENSION

C Continous
D Discontinuous

HORIZON NOTES

1	
2	
3	
4	
5	
6	
7	
8	
9	
10	

FOR SCS-SOI-232 (Continued)

SAMPLES	CLODS	ROOTS			PORES			CONCENTRATIONS				FIELD MEASURED PROPERTIES		ROCK FRAGMENTS		
		DT	SZ	LOC	SMP	DT	SZ	RND	DT	SMP	SZ	RND	AMOUNT	RND	%	SZ
												P				1
																2
																3
												P				1
																2
																3
												P				1
																2
																3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												ON				3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												OIN				3
												P				1
												OIA				2
												OIN				3

LOG		LOCATION OF ROOTS		SHAPE OF PORES		F1		F2		F3		F4		F5		F6		F7		F8		F9		F10		F11		F12		F13		F14		F15		F16		F17		F18		F19		F20		F21		F22		F23		F24		F25		F26		F27		F28		F29		F30		F31		F32		F33		F34		F35		F36		F37		F38		F39		F40		F41		F42		F43		F44		F45		F46		F47		F48		F49		F50		F51		F52		F53		F54		F55		F56		F57		F58		F59		F60		F61		F62		F63		F64		F65		F66		F67		F68		F69		F70		F71		F72		F73		F74		F75		F76		F77		F78		F79		F80		F81		F82		F83		F84		F85		F86		F87		F88		F89		F90		F91		F92		F93		F94		F95		F96		F97		F98		F99		F100		F101		F102		F103		F104		F105		F106		F107		F108		F109		F110		F111		F112		F113		F114		F115		F116		F117		F118		F119		F120		F121		F122		F123		F124		F125		F126		F127		F128		F129		F130		F131		F132		F133		F134		F135		F136		F137		F138		F139		F140		F141		F142		F143		F144		F145		F146		F147		F148		F149		F150		F151		F152		F153		F154		F155		F156		F157		F158		F159		F160		F161		F162		F163		F164		F165		F166		F167		F168		F169		F170		F171		F172		F173		F174		F175		F176		F177		F178		F179		F180		F181		F182		F183		F184		F185		F186		F187		F188		F189		F190		F191		F192		F193		F194		F195		F196		F197		F198		F199		F200		F201		F202		F203		F204		F205		F206		F207		F208		F209		F210		F211		F212		F213		F214		F215		F216		F217		F218		F219		F220		F221		F222		F223		F224		F225		F226		F227		F228		F229		F230		F231		F232		F233		F234		F235		F236		F237		F238		F239		F240		F241		F242		F243		F244		F245		F246		F247		F248		F249		F250		F251		F252		F253		F254		F255		F256		F257		F258		F259		F260		F261		F262		F263		F264		F265		F266		F267		F268		F269		F270		F271		F272		F273		F274		F275		F276		F277		F278		F279		F280		F281		F282		F283		F284		F285		F286		F287		F288		F289		F290		F291		F292		F293		F294		F295		F296		F297		F298		F299		F300		F301		F302		F303		F304		F305		F306		F307		F308		F309		F310		F311		F312		F313		F314		F315		F316		F317		F318		F319		F320		F321		F322		F323		F324		F325		F326		F327		F328		F329		F330		F331		F332		F333		F334		F335		F336		F337		F338		F339		F340		F341		F342		F343		F344		F345		F346		F347		F348		F349		F350		F351		F352		F353		F354		F355		F356		F357		F358		F359		F360		F361		F362		F363		F364		F365		F366		F367		F368		F369		F370		F371		F372		F373		F374		F375		F376		F377		F378		F379		F380		F381		F382		F383		F384		F385		F386		F387		F388		F389		F390		F391		F392		F393		F394		F395		F396		F397		F398		F399		F400		F401		F402		F403		F404		F405		F406		F407		F408		F409		F410		F411		F412		F413		F414		F415		F416		F417		F418		F419		F420		F421		F422		F423		F424		F425		F426		F427		F428		F429		F430		F431		F432		F433		F434		F435		F436		F437		F438		F439		F440		F441		F442		F443		F444		F445		F446		F447		F448		F449		F450		F451		F452		F453		F454		F455		F456		F457		F458		F459		F460		F461		F462		F463		F464		F465		F466		F467		F468		F469		F470		F471		F472		F473		F474		F475		F476		F477		F478		F479		F480		F481		F482		F483		F484		F485		F486		F487		F488		F489		F490		F491		F492		F493		F494		F495		F496		F497		F498		F499		F500		F501		F502		F503		F504		F505		F506		F507		F508		F509		F510		F511		F512		F513		F514		F515		F516		F517		F518		F519		F520		F521		F522		F523		F524		F525		F526		F527		F528		F529		F530		F531		F532		F533		F534		F535		F536		F537		F538		F539		F540		F541		F542		F543		F544		F545		F546		F547		F548		F549		F550		F551		F552		F553		F554		F555		F556		F557		F558		F559		F560		F561		F562		F563		F564		F565		F566		F567		F568		F569		F570		F571		F572		F573		F574		F575		F576		F577		F578		F579		F580		F581		F582		F583		F584		F585		F586		F587		F588		F589		F590		F591		F592		F593		F594		F595		F596		F597		F598		F599		F600		F601		F602		F603		F604		F605		F606		F607		F608		F609		F610		F611		F612		F613		F614		F615		F616		F617		F618		F619		F620		F621		F622		F623		F624		F625		F626		F627		F628		F629		F630		F631		F632		F633		F634		F635		F636		F637		F638		F639		F640		F641		F642		F643		F644		F645		F646		F647		F648		F649		F650		F651		F652		F653		F654		F655		F656		F657		F658		F659		F660		F661		F662		F663		F664		F665		F666		F667		F668		F669		F670		F671		F672		F673		F674		F675		F676		F677		F678		F679		F680		F681		F682		F683		F684		F685		F686		F687		F688		F689		F690		F691		F692		F693		F694		F695		F696		F697		F698		F699		F700		F701		F702		F703		F704		F705		F706		F707		F708		F709		F710		F711		F712		F713		F714		F715		F716		F717		F718		F719		F720		F721		F722		F723		F724		F725		F726		F727		F728		F729		F730		F731		F732		F733		F734		F735		F736		F737		F738		F739		F740		F741		F742		F743		F744		F745		F746		F747		F748		F749		F750		F751		F752		F753		F754		F755		F756		F757		F758		F759		F760		F761		F762		F763		F764		F765		F766		F767		F768		F769		F770		F771		F772		F773		F774		F775		F776		F777		F778		F779		F780		F781		F782		F783		F784		F785		F786		F787		F788		F789		F790		F791		F792		F793		F794		F795		F796		F797		F798		F799		F800		F801		F802		F803		F804		F805		F806		F807		F808		F809		F810		F811		F812		F813		F814		F815		F816		F817		F818		F819		F820		F821		F822		F823		F824		F825		F826		F827		F828		F829		F830		F831		F832		F833		F834		F835		F836		F837		F838		F839		F840		F841		F842		F843		F844		F845		F846		F847		F848		F849		F850		F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Left justify letters and right justify numbers. Use leading zeros to fill spaces where number entries are used. Enter zero as "0/." All codes are on Form SCS-SOI-232 except for pedon classification and parent material codes which are printed on another sheet. Metric units are specified for this project.

Site Data

Tier Number 1

Series Name

Soil Series Name							

Sample Number

Sample Number				S
St.	County	Unit		u
				b

- St. = State alpha code
- County = 3-digit FIPS county code
- Unit = 3-digit number identify the pedon with a county
- Sub = sub unit alpha code if needed

MLRA

Major Land Resource Areas

MLRA	
	s
	u
	b

Latitude of Sample
Site

Latitude				D
Deg	Min	Sec	i	r
_____	_____	_____	_____	_____

Longitude of Sample
Site

Longitude				D
Deg	Min	Sec	i	r
_____	_____	_____	_____	_____

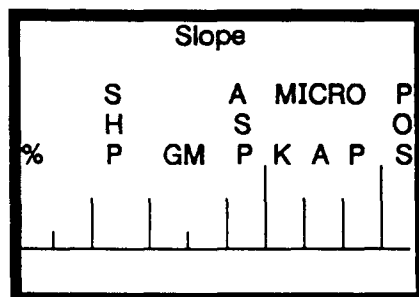
Date

Date		
Mo	Day	Yr
_____	_____	_____

Date = Date pedon was described
Mo = 2-digit code for month
Day = 2-digits, 0 used in left column if one digit
Yr = last 2 digits of the year

Tier Number 2

Slope Characteristics



- % = Slope percent
- SHP = Slope shape - The configuration of the slope
- GM = Geomorphic position code - Specific part of a hillslope or mountain slope, grading from summit areas to lowlands
- ASP = Slope aspect code - Direction slope is facing
- MICRO = Microrelief codes
 - K = Kind - Kind, amount, and pattern of microrelief that includes polypedon described
 - A = Amount in elevation code
 - P = Pattern code - Pattern of the low parts of the microrelief
- POS = Pedon position on slope code - Placement of the pedon site within the segment of the Geomorphic Component

Physiography



- RG = Regional - Landform extending for kilometers about the pedon site
- LOC = Local - Landform in the immediate vicinity of the pedon site

**Pedon
Classification**

PEDON CLASSIFICATION											
O	S	O	G	G	S	GPSC	MIN	R	X	TMP	OTH

O = Order
SO = Suborder
GG = Great group
SG = Subgroup
PSC = Particle size class
MIN = Mineralogy
RX = Reaction
TMP = Temperature
OTH = Other code

Precipitation

Not coded by field crews

PRECIP
C M

Water Table

(NSH p. 603-200)

Water Table		
Depth	K	
CM	D	Month

DEPTH = Depth to top of free water (NA used if no
water table observed)
KD = Kind code
MONTH = Month described

Miscellaneous

L	S	P	D
U	T	M	R

- LU = Land use code - Current use of the land at the pedon site (National Inventory and Monitoring Manual)
- ST = Stoniness class - As defined in Soil Survey Manual (NSH p. 602-60)
- PM = Permeability code - Code for the least permeable horizon excluding the surface horizon (NSH p. 603-19)
- DR = Drainage class code - As indicated in the pedon description (SSM p. 4-32)

Tier Number 3

Elevation

ELEVATION METERS			
0	5	2	0

Parent Material
(Glossary of Landform and Geologic Terms)

Parent Material																B D R K
1				2				3				4				
W	M	ORIG		W	M	ORIG		W	M	ORIG		W	M	ORIG		
0				0				0				0				

- W = Not coded by field crews, 0 in box
- M = Mode of deposition code
- ORIG = Origin of material code
- BDRK = Bedrock fracturing

The Arabic numbers 1, 2, 3, and 4 are for separate types of material that may occur within the profile. They correspond to lithologic discontinuities.

Temperatures

Temperature °C					
Average Air			Average Soil		
ANN	SUM	WINTER	ANN	SUM	WINTER
_____	_____	_____	_____	_____	_____

ANN = Annual
 SUM = Summer
 WINTER = Winter

} Not coded by field crews

Moisture Regime
(MST RGE)
(Soil Taxonomy p. 51)

MST RGE _____

Weather station number
(not coded by field crews)

WEATHER STA NUMBER _____

Tier Number 4
Control Section

CONTROL SECTION C M _____

CONTROL SECTION = upper and lower limits of particle size control section (Soil Taxonomy p. 385)


Water erosion code (ERWA)
(not coded by field crew)

ER WA

RN
OF[illegible]

Flooding (NSH p. 603-40)

FLOODING
FRQ **DUR**



FRQ = Frequency (times/yr)
DUR = Duration - months between which flooding occurs

**Vegetation-Scientific
plant name symbol for
dominant species
(National Handbook of
Plant Names)**

3rd

The major, 2nd, and 3rd fields should include the dominant tree species by order of basal area. For areas that were clearcut since mapping was conducted, use the code CC. Describe the dominant vegetation types prior to the clearcut in the free-form site notes.

Describers' Names and Crew I.D.

CREW I.D.

Tier Number 6

Location Description

Spaces

- 1 - 6 = Watershed I.D.
 - 7 = Dash
 - 8 = Site Number
 - 9 = Dash
- 10 - 12 = Sampling class code. If class only has 2 characters, add a zero (0) before the number, e.g., S9 becomes S09.
- 13 = Dash
- 14 - 16 = Aspect - Determined by the face of the pit described in a perpendicular direction based on magnetic north. If azimuth cannot readily be determined, as in Histosols, use N/A in this field. Use leading zeros.
- 17 = Degree symbol
- 18 to end = Location notes

LOCATION	DESCRIPTION

FREE FORM SITE NOTES

HORIZON DATA

Depth
(SSM p. 4-50)

DEPTH	
UPPER	
LOWER	

Horizon Designation
(SSM p. 4-39)

HORIZON DESIGNATION		
D I S C	MASTER	
	LETTER SUFFIX	

DISC = Discontinuity
(Arabic number)
MASTER LETTER = Master horizon
designation
SUFFIX = Subscript

Thickness (SSM p. 4-50)

AVE = Average thickness of horizon
MAX = Maximum thickness of horizon
MIN = Minumum thickness of horizon

THICK- NESS	
AVE	
MAX	
MIN	

COLORS (Dry and Moist)

DRY COLOR									
L					V	C			
O					A	H			
C	%	HUE		L	R				

There is space for three matrix color entries. Enter the dominant color on upper line (SSM p. 4-62).

LOC = Location code
% = Percent of matrix (leave blank if 100).
HUE = Hue (left justify; a decimal requires a space).
VAL = Value
CHR = Chroma

Hues are coded as 0.

MOIST COLOR									
L					V	C			
O					A	H			
C	%	HUE		L	R				

Texture

(SSM p. 4-52 and
NSH p. 603-198)

TEXTURE									
CLASS					MOD				

CLASS = Class code
MOD = Texture modifier

Structure

STRUCTURE									
G									
R									
D									
SZ					SHP				

GRD = Grade code (SSM p. 4-72)
SZ = Size code (SSM p. 4-99)
SHP = Shape code (SSM p. 4-71)

Consistence
(SSM p. 4-81)

CONSISTENCE									
DRY									
MOIST									
OTHER									
		ST / PL				C			
						E			
						M			

DRY = Dry (1st line left side of field)
MOIST = Moist (2nd line left side of field)
OTHER = Other code (3rd line left side of field)
 (SSM p. 4-83)
ST = Stickiness (1st line middle of the field)
PL = Plasticity (2nd line middle of the field)
CEM = Cementation code (lower right of field)
 (SSM p. 4-79)

Mottles
(SSM p. 4-66)

MOTTLES									
A B		C O N		H U E		V A L		C H R	

AB = Abundance code
SZ = Size code
CON = Contrast code
HUE = Hue (left justify)
VAL = Value
CHR = Chroma

Surface features

SURFACE FEATURES									
K A		D L		H U E		V A L		C H R	

KND = Kind code
AMT = Amount code
CN = Continuity
DST = Distinction code
LOC = Location code
HUE = Hue (left justify)
VAL = Value
CHR = Chroma

Boundary
(SSM p. 4-51)

Distinctness-left
Topography-right

BOUN-	
DARY	

Effervescence
(SSM p. 4-91)

Not coded by field crews

EFFERVES-		
CENCE		
C	A	E
L	G	X

CL = Class code
AG = Agent code
EX = Extent code

Roots
(SSM p. 4-85)

ROOTS		
		L
		O
QT	SZ	C

QT = Quantity code
SZ = Size code
LOC = Location code

Pores
(SSM p. 4-84)

PORES			
SHP	QT	SZ	

SHP = Shape code
QT = Quantity code
SZ = Size code

Concentrations
(SSM p. 4-76)

CONCENTRATIONS				
KND	QT	S	P	SZ
		H		

KND = Kind code
QT = Quantity code
SHP = Shape code
SZ = Size code

FIELD MEASURED PROPERTIES		P E R M	S O I L
KND	AMOUNT		
P			
			++

KND = Kind code
 pH = line one, all horizons
 OA = % Clay, line two, horizon 4-10
 ON = % Sand, line three, horizon 4-10
 AMOUNT = Amount, no decimals
 PERM = Permeability of horizon. Use same codes
 as permeability on page one. Upper line.
 SOIL = Soil moisture code. Lower line.

Rock Fragments
(SSM p. 4-97)

ROCK FRAGMENTS		
K N D	%	S Z
		1
		2
		3

KND = Kind code
 % = Percent by volume
 SZ = Size code

Free
Form
Notes

Sample Codes	Clods

Sample Codes = Sample taken from particular horizon. Same sample code that appears on Label A.
Clods = Number of clods taken from particular horizon (if none, use 0)

LOG

1. Weather - Type of weather i.e., rainy, sunny, and average temperature.
2. Set I.D. - Unique numbers assigned to crews for each day in the field.
3. Understory vegetation
4. Slides - Number of slides corresponding to specific picture from film roll

LOG		
WEATHER		
SET I.D.		
UNDERSTORY VEGETATION -		
SLIDE NO.	pedon face	overstory
	understory	landscape

2.0 Soil Description Codes for Form SCS-SOI-232

2.1 Slope Shape Codes

1 convex 2 plane 3 concave 4 undulating 5 complex

2.2 Geomorphic Position Codes

01 summit crested hills	11 summit interfluv
02 shoulder crested hills	12 shoulder interfluv
22 shoulder headslope	42 shoulder noseslope
03 backslope crested hills	23 backslope headslope
33 backslope sideslope	43 backslope noseslope
24 footslope headslope	34 footslope sideslope
44 footslope noseslope	05 toeslope crested hills
25 toeslope headslope	35 toeslope sideslope
04 footslope crested hills	00 not applicable
	32 shoulder sideslope

2.3 Slope Aspect Codes

1 northeast	2 east	3 southeast	4 south
5 southwest	6 west	7 northwest	8 north

2.4 Microrelief (Micro) Codes

2.4.1 Kind (K)

<u>B</u> = micro depression	<u>M</u> = mound
<u>C</u> = tree-throw feature	<u>R</u> = raised bog
<u>F</u> = frost polygon	<u>T</u> = terracettes
<u>G</u> = gilgai	<u>Z</u> = other (specify in notes)
<u>L</u> = land leveled or smooth	

2.4.2 Variation in elevation (A)

<u>0</u> = minimal	<u>2</u> = 20-50 cm
<u>1</u> = <20 cm	<u>4</u> = 50-100 cm

2.4.3 Pattern (P)

<u>0</u> = none	<u>2</u> = closed depressions
<u>1</u> = linear	<u>3</u> = reticulate (net)

2.5 Pedon Position Codes

1 on the crest	2 on slope and crest	3 on upper third
4 on middle third	5 on lower third	6 on a slope
7 on a slope and depression	8 in a depression	9 in a drainageway

2.6 Regional Landform Codes

A coastal plains	B intermountain basin
E lake plains	F river valley
G glaciated uplands	H glaciofluvial landform
I bolson	K karst
L level or undulating uplands	M mountains or deeply dissected plateaus
N high hills	P piedmonts
R hills	U plateaus or tablelands
V mountain valleys or canyons	

2.7 Local Landform Codes

A fan	B bog
C cuesta or hogback	D dome or volcanic cone
E escarpment	F broad plain
G crater	H abandoned channel
I hillside or mountainside	J moraine
K kamefield	L drumlin
M mesa or butte	N low sand ridge--nondunal
P flood plain	Q playa or alluvial flat
R upland slope	S sand dune or hill
T terrace--stream or lake	U terrace--outwash or marine
V pediment	W swamp or marsh
X salt marsh	Y barrier bar
Z back barrier flat	

2.8 Great Group Codes

ALFISOLS

AAQAL Albaqualf	AUDGL Glossudalf
AAQNA Natraqualf	AUDPA Paleudalf
AAQPN Plinthaqualf	AUSHA Haplustalf
ABOCR Cryoboralf	AUSPN Plinthustalf
ABOGL Glossoboralf	AXEFR Fragixeralf
AUDAG Agrudalf	AXEPA Paleixeralf
AUDFS Fraglossudalf	AAQFR Fragiaqualf
AUDNA Natrudalf	AAQOC Ochraqualf
AUSDU Durustalf	AAQUM Umbraqualf
ASUPA Paleustalf	ABOFR Fragiboralf
AXEDU Durixeralf	ABOPA Paleboralf
AXENA Natrixeralf	AUDFR Fragiudalf
AXERH Rhodoxeralf	AUDHA Hapludalf
AAQDU Duraqualf	AUDTR Tropudalf
AAQGL Glossaqualf	AUSNA Natrustalf
AAQTR Tropaqualf	AUSRH Rhodustalf
ABOEU Eutroboralf	AXEHA Haploxeralf
ABONA Natriboralf	AXEPN Plinthoxeralf
AUDFE Ferrudalf	

ARIDISOLS

DARDU Durargid
DARNT Natrargid
DORCM Camborthid
DORPA Paleorthid
DARHA Haplargid
DARPA Paleargid

DORDU Durorthid
DORSA Salorthid
DARND Nadurargid
DORCL Calciorthid
DORGY Gypsiorthid

ENTISOLS

EAQCR Cryaquept
EAQHY Hydraquept
EAQTR Tropaquept
EFLTO Torrifluvent
EFLUS Ustifluent
EORTO Torriorthent
EORUS Ustorthent
EPSQU Quartzipsamment
EPSUD Udipsamment
EAQFL Fluvaquept
EFLCR Cryofluvent
EFLUD Udifluvent
EORCR Cryorthent

EAQPS Psammaquept
EARAR Arent
EFLTR Tropofluvent
EFLXE Xerofluvent
EORTR Troporthent
EORXE Xerorthent
EPSTO Torripsamment
EPSUS Ustipsamment
EAQHA Haplaquept
EAQSU Sulfaquept
EORUD Udorthent
EPSCR Cryopsamment
EPSTR Tropopsamment
EPSXE Xeropsamment

HISTOSOLS

HFIBO Borofibrist
HFIME Medifibrist
HFOBO Borofolist
HHEBO Borohemist
HHEME Medihemist
HHETR Tropohemist
HSAME Medisaprist
HFICR Cryofibrist
HFISP Sphagnofibrist
HFOCR Cryofolist

HHECR Cryohemist
HHESI Sulfihemist
HSABO Borosaprist
HSATR Troposaprist
HFILU Luvifibrist
HFITR Tropofibrist
HFOTR Tropofolist
HHELU Luvihemist
HHESO Sulfohemist
HSACR Cryosaprist

INCEPTISOLS

IANCR Cryandept
IANEU Eutrandept
IANVI Vitrandepte
IAQFR Fragiaquept
IAQHU Humaquept
IAQSU Sulfaquept
IOCDU Durochrept
IOCFR Fragiochrept
IPLPL Plaggept
ITRHU Humitropept
IUMCR Cryumbrept

IAQTR Tropaquept
IOCDY Dystrochrept
IOCUS Ustochrept
ITRDY Dystropept
ITRSO Sombritropept
IUMFR Fragiumbrept
IANDY Dystrandep
IANPK Placandept
IAQCR Cryaquept
IAQHP Haplaquept
IAQPN Plinthaquept

IUMXE Xerumbrept
IANDU Durandep
IANHY Hydrandep
IAQAN Andaquept
IAQHL Halaquept
IAQPK Palacaquept

IOCCR Cryochrept
IOCEU Eutrochrept
IOCXE Xerochrept
ITREU Eutropept
ITRUS Ustropept
IUMHA Haplumbrept

MOLLISOLS

MALAR Argialboll
MAQCA Calciaquoll
MAQHA Haplaquoll
MBOCA Calciboroll
MBONA Natriboroll
MRERE Rendoll
MUDPA Paleudoll
MBOCR Cryoboroll
MBOPA Paleboroll
MUDAR Argiudoll
MUDVE Vermudoll
MUSDU Durustoll
MUSPA Paleustoll
MXECA Calcixeroll
MXENA Natrixeroll
MAQAR Argiaquoll
MAQDU Duraquoll

MUSCA Calciustoll
MUSNA Natrustoll
MXEAR Argixeroll
MXEHA Haploxeroll
MALNA Natralboll
MAQCR Cryaquoll
MAQNA Natraquoll
MBOAR Argiboroll
MBOHA Haploboroll
MBOVE Vermiboroll
MUDHA Hapludoll
MUSAR Argiustoll
MUSHA Haplustoll
MUSVE Vermustoll
MXEDU Durixeroll
MXEPA Palexeroll

OXISOLS

OAQGI Givvsiaquox
OAQUM Umbraquox
OHUHA Haplohumox
OOREU Eutrorthox
OORSO Sombriorthox
OUSAC Acrustox
OUSH A Haplustox
OAQOC Ochraquox
OHUAC Acrohumox
OHUSO Sombrihumox

OORGI Gibbsiorthox
OORUM Umbriorthox
OUSEU Eutrustox
OUSO Sombriustox
OAQPN Plinthaquox
OHUGI Gibbsihumox
OORAC Acroorthox
OORHA Haplorthox
OTOTO Torrox

SPODOSOLS

SAQCR Cryaquod
SAQHA Haplaquod
SAQTR Tropaquod
SHUFR Fragihumod
SHUTR Tropohumod
SORHA Haplorthod
SAQDU Duraquod
SAQPK Placaquod
SFEFE Ferrod

SHUHA Haplohumod
SORCR Cryorthod
SORPK Placorthod
SAQFR Fragiaquod
SAQSI Sideraquod
SHUCR Cryohumod
SHUPK Placohumod
SORFR Fragiorthod
SORTR Troporthod

ULTISOLS

UAQAL Albaquult
UAQPA Paleaquult
UAQUM Umbraquult
UHUPN Plinthohumult
UUDFR Fragiudult
UUDPN Plinthudult
UUSHA Haplustult
UUSRH Rhodustult
UAQFR Fragiaquult
UHUPA Palehumult
UHUTR Tropohumult
UUDPA Paleudult

UAQPN Plinthaquult
UHUHA Haplohumult
UHUSO Sombrihumult
UUDHA Hapludult
UUDRH Rhodudult
UUSPA Paleustult
UXEHA Haploxerult
UAQOC Ochraqult
UAQTR Tropaquult
UUDTR Tropudult
UUSPN Plinthustult
UXEPA Palexerult

VERTISOLS

VTOTO Torrt
VUSCH Chromustert
VXEPE Pelloxerert
VUDCH Chromudert

VUSPE Pellustert
VUDPE Pelludert
UXECH Chromxerert

2.9 Subgroup Codes

AA Typic
ABO4 Abruptic aridic
AB10 Abruptic haplic
AB16 Abruptic xerollic
AE03 Aeric arenic
AE06 Aeric humic
AE09 Aeric tropic
AE12 Aeric xeric
AL02 Albaquultic
AL08 Albic glossic
AL13 Alfic andeptic
AL16 Alfic lithic
AN01 Andeptic
AN Andic
AN22 Andic ustic
AQ Aqualfic
AQ04 Aqueptic
AQ08 Aquic arenic
AQ16 Aquic duriorthidic
AQ24 Aquic haplic
AQ31 Aquic psammentic
AQ36 Aquultic
AR02 Arenic aridic
AR04 Arenic plinthaquic
AR08 Arenic rhodic
AR14 Arenic umbric
AR18 Arenic ustollic
AR24 Argiaquic xeric

AB Abruptic
AB08 Abruptic cryic
AB14 Abruptic ultic
AE Aeric
AE05 Aeric grossarenic
AE08 Aeric mollic
AE10 Aeric umbric
AL Albaquic
AL04 Albic
AL10 Alfic
AL12 Alfic arenic
AN24 Andaqueptic
AN11 Andeptic glossoboric
AN06 Andic Dystric
AN30 Anthropic
AQ02 Aquentic
AQ06 Aquic
AQ14 Aquic duric
AQ18 Aquic dystric
AQ26 Aquic lithic
AQ34 Aquollic
AR Arenic
AR03 Arenic orthoxic
AR06 Arenic plinthic
AR10 Arenic ultic
AR16 Arenic ustalfic
AR22 Argiaquic
AR26 Argic

AR28 Argic lithic
AR32 Argic vertic
AR36 Aridic calcic
AR50 Aridic pachic
AN03 Andaquic

BO02 Borolfic lithic
BO06 Borollic
BO10 Borollic lithic

CA Calcic
CA06 Calciorthidic
CA20 Cambic
CH06 Chromudic
CR10 Cryic lithic
CU Cumulic
CU04 Cumulic ultic

DU Durargidic
DU08 Durixerollic
DU11 Durochreptic
DU14 Durorthidic xeric
DY03 Dystric entic
DY06 Dystric lithic

EN Entic
EN06 Entic ultic
EP10 Epiaquic orthoxic
EU02 Eutrochreptic

FE Ferrudalfic
FI02 Fibric terric
FL06 Fluventic
FR10 Fragiaquic

GL02 Glossaquic
GL10 Glossic udic
GL14 Glossoboralfic
GR Grossarenic
GR04 Grossarenic plinthic

HA01 Haplaquic
HA02 Haplic
HA07 Haploxerollic
HA12 Hapludollic
HE Hemic
HI Histic
HI06 Histic pergelic
HU Humic
HU05 Humic pergelic
HY Hydric

AR30 Argic pachic
AR34 Aridic
AR42 Aridic duric
AR52 Aridic petrocalcic
BO Boralfic

BO04 Boroalfic udic
BO08 Borollic glossic
BO12 Borollic vertic

CA04 Calcic pachic
CA10 Calcixerollic
CH Chromic
CR Cryic
CR14 Cryic pachic
CU02 Cumulic udic

DU02 Duric
DU10 Durixerollic lithic
DU12 Durorthidic
DY02 Dystric
DY04 Dystric Fluventic
DY08 Dystropeptic

EN02 Entic lithic
EP Epiaquic
EU Eutric
EU04 Eutropeptic

FI Fibric
FL02 Fluvaquentic
FL12 Fluventic umbric
FR18 Fragic

GL04 Glossic
GL12 Glossic ustollic
GL16 Glossoboric
GR01 Grossarenic entic

HA Haplaquodic
HA05 Haplohumic
HA09 Hapludic
HA16 Haplustollic
HE02 Hemic terric
HI02 Histic lithic
HU10 Humaqueptic
HU02 Humic lithic
HU06 Humoxic
HY02 Hydric lithic

LE Leptic
LI01 Lithic
LI06 Lithic ruptic-alfic
LI09 Lithic ruptic-entic
LI13 Lithic ruptic-ultic
LI11 Lithic ruptic-xerorthentic
LI12 Lithic ultic
LI16 Lithic ustic
LI20 Lithic vertic
LI24 Lithic xerollic

MO Mollic

OC Ochreptic
OR Orthidic
OX Oxic

PA Pachic
PA04 Pachic ultic
PA08 Paleustollic

PA20 Paralithic vertic
PE01 Pergelic ruptic-histic
PE04 Petrocalcic
PE08 Petrocalcic ustollic
PE16 Petroferric
PK Placic
PK12 Plaggic
PL04 Plinthic
PS Psammaquentic

QU Quartzipsammentic

RE Rendollic
RU02 Ruptic-alfic
RU11 Ruptic-lithic-entic
RU17 Ruptic-ultic

SA Salorthidic
SA04 Sapric terric
SO04 Sombrihumic
SP02 Sphaginic terric
SU Suflic

AA Typic
TH04 Thapto-histic
TO Torreritic
TO04 Torriorthentic
TO10 Torrox
TR02 Tropeptic

LI Limnic
LI04 Lithic mollic
LI07 Lithic ruptic-argic
LI08 Lithic ruptic-entic-xerollic
LI15 Lithic ruptic-xerochreptic
LI10 Lithic udic
LI14 Lithic umbric
LI18 Lithic ustollic
LI22 Lithic xeric

NA06 Natric

OR01 Orthic
OR02 Orthoxic

PA02 Pachic udic
PA06 Paleorthidic
PA10 Palexerollic

PE Pergelic
PE02 Pergelic sideric
PE06 Petrocalcic ustalfic
PE14 Petrocalcic xerollic
PE20 Petrogypsic
PK10 Plaggeptic
PL Plinthaquic
PL06 Plinthudic
PS02 Psammentic

RH Rhodic
RU09 Ruptic-lithic
RU15 Ruptic-lithic-xerochreptic
RU19 Ruptic-vertic

SA02 Sapric
SI Sideric
SP Sphaginic
SP04 Spodic

TE Terric
TH06 Thapto-histic tropic
TO02 Torrifluventic
TO06 Torripsammentic
TR Tropaquodic
TR04 Tropic

UD01 Udalfic	UD Udertic
UD02 Udic	UD03 Udollic
UD05 Udorthentic	UD10 Udoxic
UL Ultic	UM Umbreptic
UM02 Umbric	US Ustalfic
US02 Ustertic	US04 Ustic
US06 Ustochreptic	US08 Ustollic
US12 Ustoxic	
VE Vermic	VE02 Vertic
XE Xeralfic	XE02 Xerertic
XE04 Xeric	XE08 Xerollic

2.10 Particle Size Codes

002 not used

005 ashy	007 ashy over cindery
008 ashy over loamy	013 ashy over loamy-skeletal
019 ashy over medial	009 ashy-skeletal
003 cindery	006 cindery over loamy
015 skeletal-cindery over medial	
004 cindery over sandy or sandy-skeletal	
114 clayey	122 clayey over fine-silty
116 clayey over fragmental	124 clayey over loamy
120 clayey over loamy-skeletal	
118 skeletal-clayey over sandy or sandy	
056 clayey-skeletal	058 clayey-skeletal over sandy
080 coarse-loamy	086 coarse-loamy over clayey
082 coarse-loamy over fragmental	
084 skeletal-coarse-loamy over sandy or sandy	
088 coarse-silty	094 coarse-silty over clayey
090 coarse-silty over fragmental	
092 skeletal-coarse-silty over sandy or sandy	
126 fine	096 fine-loamy
102 fine-loamy over clayey	098 fine-loamy over fragmental
100 skeletal-fine-loamy over sandy or sandy	
106 fine-silty	112 fine-silty over clayey
108 fine-silty over fragmental	
110 skeletal-fine-silty over sandy or sandy	
036 fragmental	068 loamy
072 skeletal-loamy over sandy or sandy	050 loamy-skeletal

054 loamy-skeletal over clayey	051 loamy-skeletal over fragmental
052 loamy-skeletal over sand	
010 medial	
012 medial over cindery	014 medial over clayey
016 medial over fragmental	018 medial over loamy
020 medial over loamy-skeletal	
022 skeletal-medial over sandy or sandy	
024 medial over thixotropic	011 medial-skeletal
062 sandy	063 sandy or sandy-skeletal
066 sandy over clayey	063 sandy over loamy
044 sandy-skeletal	046 sandy-skeletal over loamy
026 thixotropic	028 thixotropic over fragmental
034 thixotropic over loamy	032 thixotropic over loamy-skeletal
030 thixotropic over sandy or sandy-skeletal	
027 thixotropic-skeletal	134 very fine

2.11 Mineralogy Codes

02 not used	04 calcareous	05 carbonatic
09 chloritic	07 clastic	08 coprogenous
10 diatomaceous	12 ferrihumic	14 ferritic
18 gibbsitic	20 glauconitic	22 gypsic
24 halloysitic	26 illitic	27 illitic (calcareous)
28 kaolinic	30 marly	32 micaceous
34 mixed	35 mixed (calcareous)	37 montmorillonitic
38 (calcareous) montmorillonitic		44 serpentinitic
40 oxidic	42 sepiolitic	
46 siliceous	50 vermiculitic	

2.12 Reaction Codes

02 not used	04 acid	08 dysic	10 euic
12 nonacid	14 noncalcareous		

2.13 Temperature Regime Codes

02 not used	04 frigid	06 hyperthermic	08 isofrigid
10 isohyperthermic	isomesic	14 isothermic	16 mesic
18 thermic			

2.14 Other Family Codes

02 not used	04 coated	05 cracked	06 level
08 micro	12 ortstein		14 shallow
15 shallow and uncoated	17 shallow and coated		
16 sloping	19 orstein shallow		uncoated

2.15 Kind of Water Table Codes

no water table observed	1 flooded	2 perched
3 apparent	4 ground water	5 ponded

2.16 Landuse Codes

A abandoned cropland (>3 yrs)	C cropland
E forest land grazed	F forest land not grazed
G pasture land and native pasture	H horticultural land
I cropland irrigated	L waste disposal land
N barren land	P rangeland grazed
Q wetlands drained	R wetlands
S rangeland not grazed	T tundra
U urban and built-up land	

2.17 Stoniness Class Codes

0 class 0	2 class 2	4 class 4
1 class 1	3 class 3	5 class 5

2.18 Permeability Codes

1 very slow	2 slow	3 moderately slow	4 moderate
5 moderately rapid	6 rapid	7 very rapid	

2.19 Drainage Codes

1 very poorly drained	2 poorly drained
3 somewhat poorly drained	4 moderately well drained
5 well drained	6 somewhat excessively drained
7 excessively drained	

2.20 Parent Material Mode of Deposition Codes

A alluvium	E eolian	H volcanic ash	W loess
S eolian-sand	D glacial drift	G glacial outwash	T glacial till
L lacustrine	M marine	O organic	Y solifluctate
V local colluvium	R solid rock	X residuum	U unconsolidated sediments

2.21 Parent Material Origin Codes

Mixed Lithology

Y0 mixed	Y1 mixed-noncalcareous
Y2 mixed-calcareous	Y3 mixed-lithology, unspecified
Y4 mixed-igneous-metamorphic and sedimentary	Y5 mixed-igneous and metamorphic
Y6 mixedigneous and sedimentary	Y7 mixed-metamorphic and sedimentary

Conglomerate

C0 conglomerate
C2 conglomerate-calcareous

C1 conglomerate-noncalcareous

Igneous

10 igneous
11 igneous-course
13 igneous-intermediate
15 igneous-fine
17 igneous-andesite
19 igneous-ultrabasic

12 igneous-basic
14 igneous-granite
16 igneous-basalt
18 igneous-acid

Metamorphic

M0 metamorphic
M1 gneiss
M3 metamorphic-basic
M5 schist and thyllite
M7 methamorphic-basic
M9 quartzite

M2 metamorphic-acidic
M4 serpentine
M6 metamorphic-acidic
M8 slate

Sedimentary

S0 sedimentary
S1 marl

S2 glauconite

Interbedded Sedimentary

B0 interbedded sedimentary
B2 limestone-sandstone
B4 limestone-siltstone
B6 sandstone-siltstone

B1 limestone-sandstone-shale
B3 limestone-shale
B5 sandstone-shale
B7 shale-siltstone

Sandstone

A0 sandstone
A2 arkosic-sandstone
A4 sandstone-calcareous

A1 sandstone-noncalcareous
A3 other sandstone

Shale

H0 shale
H1 shale-noncalcareous

H2 shale-calcareous

Siltstone

T0 siltstone
T2 siltstone-calcareous

T1 siltstone-noncalcareous

Limestone

L0 limestone

L1 chalk

L3 dolomite

L5 limestone-arenaceous

L7 limestone-cherty

L2 marble

L4 limestone-phosphatic

L6 limestone-argillaceous

Pyroclastic

P0 pyroclastic

P1 tuff

P3 tuff-basic

P5 breccia-acidic

P7 tuff-breccia

P9 pahoehoe

P2 tuff-acidic

P4 volcanic breccia

P6 breccia-basic

P8 aa

Ejecta Material

E0 ejecta-ash

E1 acidic-ash

E3 basaltic-ash

E5 cinders

E7 scoria

E2 basic-ash

E4 andesitic-ash

E6 pumice

E8 volcanic bombs

Organic Materials

K0 organic

K2 herbaceous material

K4 wood fragments

K6 charcoal

K9 other organics

K1 mossy material

K3 woody material

K5 logs and stumps

K7 coal

2.22 Bedrock Fracturing

1 10 cm between fractures

3 45 cm to 1 m between fractures

5 2 m between fractures

2 10 to 45 cm between fractures

4 1 to 2 m between fractures

2.23 Moisture Regime Codes

AR aridic moisture regime

US ustic moisture regime

AQ aquic moisture regime

XE xeric moisture regime

UD udic moisture regime

TO torric moisture regime

PU perudic moisture regime

2.24 Erosion Codes

0 none

1 slight

2 moderate

3 severe

2.25 Runoff Codes

1 none	2 ponded	3 very slow	4 slow
5 moderate	6 rapid	7 very rapid	

2.26 Diagnostic Feature Codes

Epipedon

A anthropic	H histic	mollic
O ochric	P plaggen	U umbric

Horizons

Q albic	R argic	T argillic
C calcic	B cambic	G gypsic
N natric	X oxic	E petrocalcic
J petrogypsic	K placic	Y salic
I sombric	S spodic	V sulfuric

Properties

D durinodes	Z duripan	L lithic contact
W paralithic contact	F fragipan	

2.27 Horizon Codes

Color Location Codes

0 unspecified	1 ped interior	2 ped exterior	3 rubbed or crushed
---------------	----------------	----------------	---------------------

2.28 Texture Classes

C	clay	CIND	cinders
CL	clay loam	COS	coarse sand
COSL	coarse sandy loam	CSCL	coarse sandy clay loam
FM	fragmental material	FS	fine sand
FSL	fine sandy loam	G	gravel
L	loam	LCOS	loamy coarse sand
LFS	loamy fine sand	LS	loamy sand
LVFS	loamy very fine sand	S	sand
SC	sandy clay	SCL	sandy clay loam
SG	sand and gravel	SI	silt
SIC	silty clay	SICL	silty clay loam
SIL	silt loam	SL	sandy loam
VFS	very fine sand	VFSL	very fine sandy loam
ICE	ice or frozen soil	GYP	gypsiferous earth
VAR	variable	CE	coprogenous earth
DE	diatomaceous earth	FB	fibric material
MARL	marl	MPT	mucky peat
MUCK	muck	PDOM	partially decomposed organics

UDOM	undecomposed organics	PEAT	peat
SP	sapric material	HM	hemic material
OPWD	oxide-protected weathered bedrock	UWB	unweathered bedrock
U	unknown texture	IND	indurated
WB	weathered bedrock	CEM	cemented

2.29 Texture Modifiers

BY	bouldery	BYV	very bouldery	BYX	extremely bouldery
ST	stony	STV	very stony	STX	extremely stony
CB	cobbly	CBA	angular cobbly	CBV	very cobbly
CBX	extremely cobbly	CN	channery	CNV	very channery
CNX	extremely channery	CR	cherty	CRC	coarse cherty
CRV	very cherty	CRX	extremely cherty	FL	flaggy
FLV	very flaggy	FLX	extremely flaggy	GR	gravelly
GRF	fine gravelly	GRC	coarse gravelly	GRV	very gravelly
GRX	extremely gravelly	SH	shaley	SHV	very shaley
SHX	extremely shaley	SY	slaty	SYV	very slaty
SYX	extremely slaty	CY	cindery	AY	ashy
SR	stratified	MK	mucky	PT	peaty
GY	gritty	GYV	very gritty	GYX	extremely gritty
RB	rubbly				

2.30 Grade of Structure

0 not used	1 weak	2 moderate
3 strong	4 very strong	5 weak and moderate
6 moderate and strong		

2.31 Size of Structure

EF extremely fine	VF very fine	FF very fine and fine
F fine	FM fine and medium	M medium
MC medium and coarse	CO coarse	VC very coarse
CV coarse and very coarse		

2.32 Structure Shape

PL platy	LP lenticular	PR prismatic
COL columnar	BK blocky	BK angular blocky
SBK subangular blocky	GR granular	CDY cloddy
CR crumb	MA massive	SGR single grain
WEG wedge		

2.33 Dry Consistence

L loose	S soft	SH slightly hard
H hard	VH very hard	EH extremely hard

2.34 Moist Consistence

L loose VFR very friable FR friable FI firm
VFI very firm EFI extremely firm

2.35 Other Consistence

WSM weakly smeary SM strongly smeary MS moderately smeary
B brittle R rigid VR very rigid
CO uncemented VWC very weakly cemented WC weakly cemented
SC strongly cemented I indurated SF slightly fluid
VF very fluid

2.36 Stickiness

SO nonsticky SS slightly sticky S sticky VS very sticky

2.37 Plasticity

PO nonplastic SP slightly plastic P plastic
VP very plastic

2.38 Cementation Agent

H humus I iron L lime S silica X lime and silica

2.39 Mottle Abundance Codes

F few C common M many

2.40 Mottle Size Codes

1 fine (5 mm) 2 medium (5 to 15 mm) 3 coarse (>15 mm)
12 fine to medium 13 fine to coarse 23 medium to coarse

2.41 Mottle Contrast Code

F faint D distinct P prominent

2.42 Surface Features

U coats A skeletal over cutans
B black stains C chalcedony on opal
D clay bridging G gibbsite coats
I iron stains K intersecting slickensides
Q nonintersecting slickensides P pressure faces
L lime or carbonate coats X oxide coats
M manganese or iron-manganese stains O organic coats
S skeletal (sand or silt) T clay films

2.43 Surface Feature Amount Codes

V very few F few C common M many

2.44 Surface Feature Continuity Codes

P patchy D discontinuous C continuous

2.45 Surface Feature Distinctness Codes

F faint D distinct P prominent

2.46 Location of Surface Features

P on faces of peds	M on bottoms of plates
H on horizontal faces of peds	B between sand grains
V on vertical faces of peds	I in root channels or pores
Z on vertical and horizontal faces of peds	T throughout
U on upper surfaces of peds or stones	R on rock fragments
L on lower surfaces of peds or stones	F on faces of peds and in pores
C on tops of columns	N on nodules

2.47 Boundary

A abrupt C clear G gradual D diffuse

2.48 Topography

S smooth W wavy I irregular B broken

2.49 Effervescence

1 slightly effervescent	2 strongly effervescent
3 violently effervescent	0 very slightly effervescent

2.50 Effervescence Agent Codes

H HCl (unspecified)	I HCl (10%)	P H ₂ O ₂ (unspecified)
Q H ₂ O ₂ (3 to 4%)		

2.51 Field Measured Property Kind Codes

2.51.1 For Organic Materials

Column 1	Column 2	
F fiber	B unrubbed	R rubbed
H hemic	W woody	H herbacious
L limnic	S sphagnum	C coprogenous earth
S sapric	D diatomaceous earth	M marly
	F ferrihumic	U humilluvic
	O other	L sulfidic

2.51.2 For Mineral Materials

ON sand OI silt OA clay

2.51.3 pH

pM pH meter (1:1 H ₂ O)	pN pH (0.1 M CaCl ₂)	pH Hellige-Truog
pL Lamotte-Morgan	pB Bromthymol blue	pC Cresol red
pP Phenol red	pT Thymol blue	pS soiltex
pY Ydrion	pG Bromcresol green	pR Chlorophenol red

2.52 Soil Moisture Codes

D dry M moist V very moist W wet

2.53 Quantity (Roots, Pores, Concretions)

VF very few	FF very few to few	F few	FC few to common
CM common to many	C common	M many	

2.54 Size (Roots, Pores, Concretions)

M micro	MI micro and fine	V1 very fine
11 very fine and fine	1 fine	12 fine and medium
2 medium	23 medium and coarse	3 coarse
4 very coarse	5 extremely coarse	13 fine to coarse

2.55 Location of Roots

C in cracks	M in mat at top of horizon
P between peds	S matted around stones
T throughout	

2.56 Shape of Pores

IR interstitial	IE filled with coarse material
IT interstitial and tubular	IF void between rock fragment
TU tubular	TC continuous tubular
TD discontinuous tubular	TE dendritic tubular
TS constricted tubular	VS vesicular
VT vesicular and tubular	TP total porosity

2.57 Kind of Concentrations

B1 barite crystals	B2 soft masses of barite
K2 soft masses of carbonate	K3 carbonate concretions
K4 carbonate nodules	C1 calcite crystals
C2 soft masses of lime	C3 lime concretions
C4 lime nodules	T2 worm casts
T3 insects casts	T4 worm nodules
A2 clay bodies	D1 mica flakes
D2 soft dark	D3 dark concretions
D4 dark nodules	E3 gibbsite concretions
E4 gibbsite nodules	G1 gypsum crystals
G2 masses of gypsum	F1 plinthite segregations
F2 soft masses of iron	F3 iron concretions
F4 ironstone nodules	M1 nonmagnetic shot
M2 soft masses of iron-manganese	M3 iron-manganese concretions
M4 magnetic shot	H1 halite crystals
H2 salt masses	S1 opal crystals
S2 soft masses of silica	S3 silica concretions
S4 durinodes	

2.58 Shape of Concentrations

C cylindrical	D dendritic	O rounded	P plate like
T threads	Z irregular		

2.59 Rock Fragment Kind Codes

Y mixed lithology	O oxide-protected rock	F ironstone
S sedimentary rocks	I igneous rocks	M metamorphic rocks
A sandstone	B mixed sedimentary rocks	L limestone
H shale	T siltstone	E ejecta
K organic fragments	P pyroclastic rocks	R saprolite

2.60 Rock Fragment Size Codes

- | | | |
|----------------|-----------------|------------|
| 1. 20 to 76 mm | 2. 76 to 250 mm | 3. >250 mm |
|----------------|-----------------|------------|

Appendix B

Forms for Reporting Analytical Laboratory Data

The following forms are used for recording raw data and results from the analytical procedures detailed in Sections 3.0 through 16.0 of *Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey* by K. A. Capps, L. J. Blume, G. A. Raab, J. K. Bartz, and J. L. Engels, U.S. Environmental Protection Agency, Las Vegas, Nevada, 1987.

An index of data forms is presented on the following page. Form 101 summarizes data from the preparation laboratory. Form 102 is a shipping form that is used to confirm sample shipment and receipt. Forms 103a and 103b summarize pH, moisture, and particle size analysis results. Forms 109 through 114 contain quality control data. The 200-series forms summarize data that are corrected for both blanks and dilutions. Raw data are recorded on forms 115, 116, 303b, 306, and 308.

Index of Data Forms

Form Number	Title
101	Preparation Laboratory Data
102	Shipping Form
103 (a,b)	Summary of pH and Particle Size Results
109 (a,b,c)	Quality Control: Detection Limits
110 (a,b,c)	Quality Control: Matrix Spikes
111 (a through i)	Quality Control: Replicates
112 (a through h)	Quality Control: Blanks and QCCS
113	Quality Control: Ion Chromatograph Resolution Test
114 (a,b,c)	Quality Control: Standard Additions
115 (a through e)	Sample Weight in Grams
116 (a through h)	Dilution Factors and Dilution Blanks; Solution Concentration; Titer and Normality
204 (a,b,c,d)	Summary of Exchangeable Bases and CEC Results Blank Corrected
205	Summary of Iron- and Aluminum-Extraction Data Blank Corrected
206	Summary of Extractable Nitrate and Sulfate, Exchangeable Acidity, and Exchangeable Aluminum Blank Corrected
207	Summary of Sulfate-Adsorption Isotherm Data Blank Corrected
208	Summary of C, N, S, and Specific-Surface Results Blank Corrected
303b	Summary of Particle Size Analysis Raw Data
306	Summary of BaCl ₂ Exchangeable Acidity Raw Data
308	Summary of C, N, S, and Specific Surface Raw Data

DIRECT/DELAYED RESPONSE PROJECT (DDRP) FORM 101

Batch ID _____

DATE RECEIVED
BY DATA MGT.

____ D ____ D ____ M ____ M ____ Y ____ Y

Crew ID _____

Batch Sent to _____

Date Shipped _____

No. of Samples _____

Set ID _____ Date Sampled _____ Date Received _____ Date Prep Completed _____				_____				_____			
Set ID _____ Date Sampled _____ Date Received _____ Date Prep Completed _____				_____				_____			
Sample No.	Site ID	Sample Code	Set ID	Rock Fragments weight %	Air Dried Moisture weight %	Soil Type M = MIN O = ORG	Inorg Carbon (IC) Y = Yes N = No	Bulk Density G/CC			
01											
02											
03											
04											
05											
06											
07											
08											
09											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											
25											
26											
27											
28											
29											
30											
31											
32											
33											
34											
35											
36											
37											
38											
39											
40											
41											
42											

Signature of Preparation Laboratory Manager: _____

Comments: _____

DIRECT/DELAYED RESPONSE PROJECT (DDRP) SOIL SURVEY
SHIPPING FORM 102

DATE RECEIVED
BY DATA MGT.

Prep Lab ID _____	Date Received _____	D	D	M	M	M	Y	Y
Batch ID _____	Date Shipped _____	_____	_____	_____	_____	_____	_____	_____
Analytical Lab ID _____		_____	_____	_____	_____	_____	_____	_____

Sample Number	Sample (Identify By Check)		Soil Type (Identify By Check)		Inorganic Carbon	Rock Fragments Shipped Check if Yes
	Shipped	Received	Organic	Mineral	Y - Yes N - No	
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						
41						
42						

Signature of Preparation Laboratory Manager: _____
Comments: _____

White - SMO Canary - Analytical with copy to SMO Pink - Analytical with copy to EMSL-LV Gold - Analytical Lab

SUMMARY OF pH AND MOISTURE DATA
DIRECT/DELAYED RESPONSE PROJECT (DDRP) SOIL SURVEY REPORT FORM 103a

Analytical Lab ID _____ Lab Manager's Signature _____
 Batch ID _____ Date Form Completed _____
 Prep Lab Name _____ Date Batch Received _____
 Remarks _____

Sample Number	pH in H ₂ O	pH in 0.01M CaCl ₂	pH in 0.002M CaCl ₂	Moisture, Weight %
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				
31				
32				
33				
34				
35				
36				
37				
38				
39				
40				
41				
42				

PARTICLE SIZE ANALYSIS DATA
DIRECT/DELAYED RESPONSE PROJECT (DDRP) SOIL SURVEY REPORT FORM 103b

Analytical Lab ID _____ Lab Manager's Signature _____
Batch ID _____ Date Form Completed _____
Prep Lab Name _____ Date Batch Received _____
Remarks _____

[illegible]

Direct/Delayed Response Project (DDRP) Soil Survey

Form 109a

Quality Control: Detection Limits

Lab Name _____ Batch ID _____
Lab Manager's Signature _____

Parameter	Reporting Units	Contract-Required Detection Limit	Instrumental Detection Limit	Date Determined (DD MMM YY)
Total S	wt. %	0.010%		
Total N	wt. %	0.050%		
Total C	wt. %	0.050%		
Inorganic C	wt. %	0.010%		
CEC (FIA)	meq/100 g	0.140 mg N/L		
CEC (titration)	meq/100 g	0.010 meq NH ₄ ⁺ *		
<u>Exchangeable Acidity:</u>				
BaCl ₂ -TEA	meq/100 g	0.40 meq*		
KCl	meq/100 g	0.25 meq*		
KCl-Al ³⁺	meq/100 g	0.10 mg/L		

*For titrations, the instrumental detection limit is a calculated value based upon a minimum titration.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 109b

Quality Control: Detection Limits

Lab Name _____ Batch ID _____
Lab Manager's Signature _____

Parameter	Calculated Reporting Units	Contract-Required Instrumental Detection Limit	Instrumental Detection Limit	Date Determined (DD MMM YY)
<u>NH₄OAc Extract:</u>				
Ca ²⁺	meq/100 g	0.050 mg/L		
Mg ²⁺	meq/100 g	0.020 mg/L		
K ⁺	meq/100 g	0.020 mg/L		
Na ⁺	meq/100 g	0.020 mg/L		
<u>NH₄Cl Extract:</u>				
Ca ²⁺	meq/100 g	0.050 mg/L		
Mg ²⁺	meq/100 g	0.020 mg/L		
K ⁺	meq/100 g	0.020 mg/L		
Na ⁺	meq/100 g	0.020 mg/L		
<u>0.002 M CaCl₂ Extract:</u>				
Ca ²⁺	meq/100 g	---*		
Mg ²⁺	meq/100 g	0.020 mg/L		
K ⁺	meq/100 g	0.020 mg/L		
Na ⁺	meq/100 g	0.020 mg/L		
Fe ³⁺	meq/100 g	0.050 mg/L		
Al ³⁺	meq/100 g	0.050 mg/L		

*Report the standard deviation of 10 non-consecutive blank analyses.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 109c

Quality Control: Detection Limits

Lab Name _____ Batch ID _____
Lab Manager's Signature _____

Parameter	Calculated Reporting Units	Contract-Required Instrumental Detection Limit	Instrumental Detection Limit	Date Determined (DD MMM YY)
SO ²⁻ ₄ Adsorption	mg S/L	0.10 mg SO ²⁻ ₄ /L		
SO ²⁻ ₄ (H ₂ O extract)	mg S/Kg	0.1 mg SO ²⁻ ₄ /L		
NO ⁻ ₃ (H ₂ O extract)	mg N/Kg	0.10 mg NO ⁻ ₃ /L		
SO ²⁻ ₄ (PO ³⁻ ₄ extract)	mg S/Kg	0.10 mg SO ²⁻ ₄ /L		
<u>Pyrophosphate Extract:</u>				
Fe ³⁺	wt. %	0.50 mg/L		
Al ³⁺	wt. %	0.50 mg/L		
<u>Acid-Oxalate Extract:</u>				
Fe ³⁺	wt. %	0.50 mg/L		
Al ³⁺	wt. %	0.50 mg/L		
<u>Citrate-Dithionite Extract:</u>				
Fe ³⁺	wt. %	0.50 mg/L		
Al ³⁺	wt. %	0.50 mg/L		

DIRECT/DELAYED RESPONSE PROJECT (DDRP) SOIL SURVEY
FORM 110a

QUALITY CONTROL: MATRIX SPIKES

LAB NAME _____ BATCH ID _____

LAB MANAGER'S SIGNATURE _____

Extractant	1.0 M NH ₄ OAc				1.0 M NH ₄ Cl				0.002 M CaCl ₂						NONE
Parameter	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	Fe, mg/L	Al, mg/L	CEC NH ₄ ⁺ , _____
First Matrix Spike Sample ID: _____															
Sample Result															
Spike Result															
Spike Added															
% Recovery															
Second Matrix Spike Sample ID: _____															
Sample Result															
Spike Result															
Spike Added															
% Recovery															
Third Matrix Spike Sample ID: _____															
Sample Result															
Spike Result															
Spike Added															
% Recovery															

*CEC units are instrument and method dependent: Fill in mg N/L for flow injection analysis or meq for distillation/titration.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 110b

Quality Control: Matrix Spikes

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	Pyrophosphate		Acid-Oxalate		Citrate-Dithionite		KCl
Parameter	Fe, mg/L	Al, mg/L	Fe, mg/L	Al, mg/L	Fe, mg/L	Al, mg/L	Al, mg/L
First Matrix Spike Sample ID:							
Sample Result							
Spike Result							
Spike Added							
% Recovery							
Second Matrix Spike Sample ID:							
Sample Result							
Spike Result							
Spike Added							
% Recovery							
Third Matrix Spike Sample ID:							
Sample Result							
Spike Result							
Spike Added							
% Recovery							

Direct/Delayed Response Project (DDRP) Soil Survey

Form 110bb

Quality Control: Matrix Spikes

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	Deionized			500 mg P/L
Parameter	NO ₃ ⁻ , mg/L	H ₂ O SO ₄ ²⁻ , mg/L	SO ₄ ²⁻ , mg/L	
First Matrix				
Spike Sample ID:				
Sample Result				
Spike Result				
Spike Added				
% Recovery				
Second Matrix				
Spike Sample ID:				
Sample Result				
Spike Result				
Spike Added				
% Recovery				
Third Matrix				
Spike Sample ID:				
Sample Result				
Spike Result				
Spike Added				
% Recovery				

Direct/Delayed Response Project (DDRP) Soil Survey

Form 110c

Quality Control: Matrix Spikes

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Total S, Weight %	Total N, Weight %	Total C, Weight %	Inorganic C, Weight %	
				<2 mm	2-20mm
First Matrix					
Spike Sample ID:					
Sample Result					
Spike Result					
Spike Added					
% Recovery					
Second Matrix					
Spike Sample ID:					
Sample Result					
Spike Result					
Spike Added					
% Recovery					
Third Matrix					
Spike Sample ID:					
Sample Result					
Spike Result					
Spike Added					
% Recovery					

Direct/Delayed Response Project (DDRP) Soil Survey

Form 110d

Quality Control: Matrix Spikes

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Sulfate remaining in solution, mg S/L					
	Initial solution concentration, mg S/L					
	0	2	4	8	16	32
First Matrix						
Spike Sample ID:						
Sample Result						
Spike Result						
Spike Added						
% Recovery						
Second Matrix						
Spike Sample ID:						
Sample Result						
Spike Result						
Spike Added						
% Recovery						
Third Matrix						
Spike Sample ID:						
Sample Result						
Spike Result						
Spike Added						
% Recovery						

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111a

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	pH in H ₂ O	pH in 0.01 M CaCl ₂	pH in 0.002 M CaCl ₂	Specific Surface, m ² /g
Triplicate Sample ID:				
First Replicate Result				
Second Replicate Result				
Third Replicate Result				
Average				
Standard Deviation				NA
% RSD	NA	NA	NA	

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111b

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

[illegible]

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111c

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	1.0 M NH ₄ OAc				
Parameter	Ca, meq/100 g	Mg, meq/100 g	K, meq/100 g	Na, meq/100 g	CEC, meq/100 g
Duplicate Sample ID:					
Sample Result					
Duplicate Results					
% RSD					
Second Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					
Third Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					

Direct/Delayed Response Project (DDRP) Soil Survey

Form 11ld

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	1.0 M NH ₄ Cl				
Parameter	Ca, meq/100 g	Mg, meq/100 g	K, meq/100 g	Na, meq/100 g	CEC, meq/100 g
Duplicate Sample ID:					
Sample Result					
Duplicate Results					
% RSD					
Second Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					
Third Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111e

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	0.002 M CaCl ₂				
Parameter	Ca, meq/100 g	Mg, meq/100 g	K, meq/100 g	Na, meq/100 g	CEC, meq/100 g
Duplicate Sample ID:					
Sample Result					
Duplicate Results					
% RSD					
Second Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					
Third Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111f

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extract	Polyphosphate		Acid-Oxalate		Citrate-Dithionite	
Parameter	Fe, Weight %	Al, Weight %	Fe, Weight %	Al, Weight %	Fe, Weight %	Al, Weight %
Duplicate Sample ID:						
Sample Result						
Duplicate Results						
% RSD						
Second Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						
Third Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						

Direct/Delayed Response Project (DDRP) Soil Survey

Form 111g

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extract	Extractable Nitrate,	Extractable Sulfate,		Exchangeable Acidity		Extractable Al,
	mg N/kg	mg S/kg		meq/100 g		meq/100 g
	H ₂ O	H ₂ O	PO ₄	BaCl ₂	KCl	KCl
Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						
Second Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						
Third Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						

Direct/Delayed Response Project (DDRP) Soil Survey
Form 111h
Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Sulfate remaining in solution, mg S/L					
	Initial solution concentration, mg S/L					
	0	2	4	8	16	32
Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						
Second Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						
Third Duplicate Sample ID:						
Sample Result						
Duplicate Result						
% RSD						

Direct/Delayed Response Project (DDRP) Soil Survey

Form 1111

Quality Control: Replicates

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Total S, Weight %	Total N, Weight %	Total C, Weight %	Inorganic C, Weight %	
				<2 mm	2-20mm
Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					
Second Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					
Third Duplicate Sample ID:					
Sample Result					
Duplicate Result					
% RSD					

Direct/Delayed Response Project (DDRP) Soil Survey

Form 112a

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter		pH in H ₂ O	pH in 0.01M CaCl ₂	pH in 0.002M CaCl ₂
Reagent Blank*				
DL	Theoretical	NA	NA	NA
QCCS	Measured	NA	NA	NA
Low QCCS True Value				
Low QCCS Upper Limit				
Lower QCCS Lower Limit				
Initial				
Continuing				
Continuing				
Continuing				
Continuing				
Final				
High QCCS True Value				
High QCCS Upper Limit				
High QCCS Lower Limit				
Initial				
Continuing				
Continuing				
Final				

*Reagent blank is the solution being added to the soil.

[illegible]

Direct/Delayed Response Project (DDRP) Soil Survey

Form 112c

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant		1.0 M NH ₄ OAc					1.0 M NH ₄ Cl				
Parameter		Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	CEC, *—	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	CEC, *—
Calibration Blank											
Reagent Blank 1											
Reagent Blank 2											
Reagent Blank 3											
DL	Theoretical										
QCCS	Measured										
Low QCCS True Value											
Low QCCS Upper Limit											
Lower QCCS Lower Limit											
Initial											
Continuing											
Continuing											
Continuing											
Continuing											
Final											
High QCCS True Value											
High QCCS Upper Limit											
High QCCS Lower Limit											
Initial											
Continuing											
Continuing											
Final											

*CEC reporting units are instrument and method dependent. Fill in mg N/L for flow injection analysis or meq for distillation/titration.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 112d

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant		0.002 M CaCl ₂					
Parameter		Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	Fe, mg/L	Al, mg/L
Calibration Blank							
Reagent Blank*							
DL	Theoretical						
QCCS	Measured						
Low QCCS True Value							
Low QCCS Upper Limit							
Lower QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Continuing							
Continuing							
Final							
High QCCS True Value							
High QCCS Upper Limit							
High QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Final							

*Analyze 0.002 M CaCl₂ solution that has been extracted through filter pulp.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 112e

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extractant	Phosphate		Acid-Oxalate		Citrate-Dithionite	
Parameter	Fe, mg/L	Al, mg/L	Fe, mg/L	Al, mg/L	Fe, mg/L	Al, mg/L
Calibration Blank						
Reagent Blank*						
DL Theoretical						
QCCS Measured						
Low QCCS True Value						
Low QCCS Upper Limit						
Lower QCCS Lower Limit						
Initial						
Continuing						
Continuing						
Continuing						
Continuing						
Final						
High QCCS True Value						
High QCCS Upper Limit						
High QCCS Lower Limit						
Initial						
Continuing						
Continuing						
Final						

Direct/Delayed Response Project (DDRP) Soil Survey

Form 112f

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter		Extractable Nitrate, mg/L	Extractable Sulfate mg/L		Extractable Acidity, mg/L		Extractable Al, mg/L
Extractant		H ₂ O	H ₂ O	PO ₃ - ₄	BaCl ₂	KCl	KCl
Calibration Blank							
Reagent Blank 1							
Reagent Blank 2							
Reagent Blank 3							
DL	Theoretical						
QCCS	Measured						
Low QCCS True Value							
Low QCCS Upper Limit							
Lower QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Continuing							
Continuing							
Final							
High QCCS True Value							
High QCCS Upper Limit							
High QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Final							

*Reagent blank is the extracting solution.

Direct/Delayed Response Project (DDRP) Soil Survey
Form 112g
Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____
Lab Manager's Signature _____

Parameter	Total S, Weight %	Total N, Weight %	K Factor $\mu\text{V}/\mu\text{g}$	Total C, Weight %	K Factor $\mu\text{V}/\mu\text{g}$	Inorganic C, Weight %	
						<2 m	2-20mm
Calibration Blank			NA		NA		
Reagent Blank*	NA		NA		NA		
DL Theoretical							
QCCS Measured							
Low QCCS True Value							
Low QCCS Upper Limit							
Low QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Continuing							
Continuing							
Final							
High QCCS True Value							
High QCCS Upper Limit							
High QCCS Lower Limit							
Initial							
Continuing							
Continuing							
Final							

Direct/Delayed Response Project (DDRP Soil Survey

Form 112h

Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Specific Surface, m ² /g (at equilibrium)	Day*	Weight of EGME in mg		
			Blank 1	Blank 2	Blank 3
Low QCCS True Value		0			
Low QCCS Upper Limit		1			
Low QCCS Lower Limit		2			
Initial		3			
Continuing		4			
Continuing		5			
Continuing		6			
Continuing		7			
Continuing		8			
Continuing		9			
Continuing		10			
Continuing		11			
Continuing		12			
Continuing		13			
Final		14			
High QCCS True Value		15			
High QCCS Lower Limit		16			
Initial		17			
Continuing		18			
Continuing		19			
Continuing		20			
Continuing		21			
Continuing		22			
Continuing		23			
Continuing		24			
Continuing		25			
Continuing		26			
Continuing		27			
Final		28			

*Measurements may be taken less frequently than daily, but record the results on the day actually performed.

Direct/Delayed Response Project (DDRP) Soil Survey
Form 112i
Quality Control: Blanks and QCCS

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Parameter	Sulfate remaining in solution, mg S/L					
	Initial solution concentration, mg S/L					
	0	2	4	8	16	32
Reagent Blank		N/A	N/A	N/A	N/A	N/A
Low QCCS True Value						
Low QCCS Upper Limit						
Low QCCS Lower Limit						
Initial						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Final						
High QCCS True Value						
High QCCS Lower Limit						
Initial						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Continuing						
Final						

Direct/Delayed Response Project (DDRP) Soil Survey
Form 113

Quality Control: Ion Chromatography Resolution Test

Lab Name _____ Batch ID _____
Lab Manager's Signature _____
Date of Analysis MM/DD/YR _____

IC Make and Model: _____

	Concentration (mg/L)	Peak Area (integrator units)	Peak Height (cm)
SO ₄ ²⁻	_____	_____	_____
PO ₄ ³⁻	_____	_____	_____
NO ₃ ⁻	_____	_____	_____

Column Back Pressure (at max. of stroke): _____ psi

Flow Rate: _____ mL/min

Column Model: _____ Date of Purchase: _____

Column Manufacturer: _____

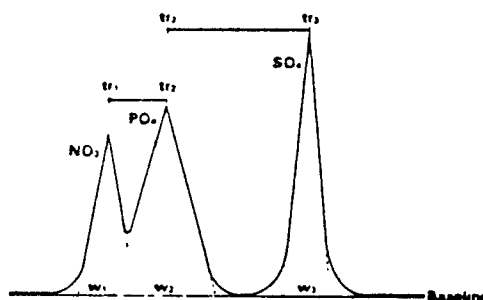
Column Serial No: _____

Precolumn in system _____ Yes _____ No

Percentage Resolution: $100 \times 2(tr_2 - tr_1)/(W_1 + W_2)$ NO₃ - PO₄ _____
 $100 \times 2(tr_3 - tr_1)/(W_1 + W_3)$ NO₃ - SO₄ _____
 $100 \times 2(tr_3 - tr_2)/(W_2 + W_3)$ PO₄ - SO₄ _____

The resolution must be greater than 60%.

Test Chromatogram:



(FACSIMILE)

*Calculations may change if order of elution is different from test chromatogram.

Direct/Delayed Response Project (DDRP) Soil Survey

Form 114a

Quality Control: Standard Additions

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Extract	1.0 M NH ₄ OAc				1.0 M NH ₄ Cl			
Parameter	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L	Ca, mg/L	Mg, mg/L	K, mg/L	Na, mg/L
Original Sample ID:								
Single Response								
Spike Added Concentration								
Sample Spike 1 Response								
Spike 2 Concentration								
Sample Spike 2 Response								
Sample Con- centration for Original Sample (calc.)								

[illegible]

[illegible]

Air Dry Sample Weight in Grams
Direct/Delayed Response Project (DDRP) Soil Survey
Form 115a

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Moisture ^a				Particle Size Analysis ^d	CEC and Exchangeable Cations	
	Dup 1		Dup 2			NH ₄ OAc	NH ₄ Cl
	Air	Oven	Air	Oven			
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
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32							
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34							
35							
36							
37							
38							
39							
40							
41							
42							
Rep 1 ^b			NA	NA			
Rep 2			NA	NA			
Rep 3 ^c			NA	NA			

^aMoisture is performed in duplicate; place one sample weight in each column. First column is air-dry weight, second column is oven-dry weight.

^bReplicates are recorded here; the sample weight recorded by the sample number is repeated as Rep 1.

^cNot all methods require three replicates.

^dOven-dry weight after organic matter removal.

Air Dry Sample Weight in Grams
Direct/Delayed Response Project (DDRP) Soil Survey
Form 115b

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Exchangeable Cations in 0.002 M CaCl ₂	Exchangeable Acidity	
		BaCl ₂	KCl
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
32			
33			
34			
35			
36			
37			
38			
39			
40			
41			
42			
Rep 1 *			
Rep 2			
Rep 3**			

*Replicates are recorded here; the sample weight recorded by the sample number is repeated as Rep 1.
**Not all methods require three replicates.

Air Dry Sample Weight in Grams
Direct/Delayed Response Project (DDRP) Soil Survey
Form 115c

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Extractable Fe and Al			H ₂ O Extractable SO ₄ ²⁻ and NO ₃ ⁻	PO ₄ ³⁻ Extractable SO ₄ ²⁻
	Pyrophosphate	Acid-Oxalate	Citrate-Dithionite		
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					
32					
33					
34					
35					
36					
37					
38					
39					
40					
41					
42					
Rep 1*					
Rep 2					
Rep 3**					

*Replicates are recorded here; the sample weight recorded by the sample number is repeated as Rep 1.

**Not all methods require three replicates.

Air Dry Sample Weight in Grams
Direct/Delayed Response Project (DDRP) Soil Survey
Form 115d

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Sulfate Adsorption Isotherm					
	Initial Solution Concentration, mg S/L					
	0	2	4	8	16	32
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
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26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						
41						
42						
Rep 1*						
Rep 2						
Rep 3**						

*Replicates are recorded here; the sample weight recorded by the sample number is repeated as Rep 1.
**Not all methods require three replicates.

Air Dry Sample Weight in Grams
Direct/Delayed Response Project (DDRP) Soil Survey
Form 115e

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Total S, mg	Total N, mg	Specific Surface, ^a g	Total C, mg	Inorganic C, mg	
					<2 mm	2-20 mm
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
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22						
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24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						
41						
42						
Rep 1 ^b						
Rep 2						
Rep 3 ^c						

^ap₂₀₅ - dry weight.

^bReplicates are recorded here; the sample weight recorded by the sample number is repeated as Rep 1.

^cNot all methods require three replicates.

Exchangeable Basic Cations in NH_4OAc
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116a

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Exchangeable Basic Cations in NH_4OAc													
Sample Number	Solution Recovered in Syringe (mL)	Aliquot Volume (mL)*				Total Dilution Volume (mL)*				Solution Concentration (mg/L)			
		Ca	Mg	K	Na	Ca	Mg	K	Na	Ca	Mg	K	Na
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
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38													
39													
40													
41													
42													

Blank	Total Volume in Sample (mL)	Aliquot Volume in Dilution (mL)	Total Volume of Dilution (mL)	Dilution Blank Concentrations (mg/L)			
				Ca	Mg	K	Na
D-Blank							
D-Blank							
D-Blank							
D-Blank							
D-Blank							
D-Blank							

*Enter U if no dilution is made.

Exchangeable Basic Cations in NH_4Cl
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116b

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Exchangeable Basic Cations in NH_4Cl												
	Solution Recovered in Syringe (mL)	Aliquot Volume (mL)*				Total Dilution Volume (mL)*				Solution Concentration (mg/L)			
		Ca	Mg	K	Na	Ca	Mg	K	Na	Ca	Mg	K	Na
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
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33													
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35													
36													
37													
38													
39													
40													
41													
42													

Blank	Total Volume in Sample (mL)	Aliquot Volume in Dilution (mL)	Total Volume of Dilution (mL)	Dilution Blank Concentrations (mg/L)			
				Ca	Mg	K	Na
D-Blank							
D-Blank							
D-Blank							
E-Blank							
D-Blank							
D-Blank							

*Enter U if no dilution is made.

Cation Exchange Capacity
Dilution Factors and Dilution Reagent Blank Values; Titer and Normality
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116c

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Cation Exchange Capacity (FIA)							Cation Exchange Capacity (Titration)			
	Total Volume in Sample (mL)	Aliquot Volume (mL)*		Total Dilution Volume (mL)*		Solution Conc. mg N/L		NH ₄ OAc		NH ₄ Cl	
		NH ₄ OAc	NH ₄ Cl	NH ₄ OAc	NH ₄ Cl	NH ₄ OAc	NH ₄ Cl	Titer (Volume in mL)	Normality of Titrant	Titer (Volume in mL)	Normality of Titrant
01											
02											
03											
04											
05											
06											
07											
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38											
39											
40											
41											
42											

Blank	Cation Exchange Capacity (FIA)				
	Total Volume in Sample (mL)	Aliquot Volume (mL)	Dilution Volume (mL)	Dilution Conc. (mg N/L)	
				NH ₄ OAc	NH ₄ Cl
D-Blank					
D-Blank					
E-Blank					

*Enter U if no additional is made.

**KCl-Exchangeable Acidity and Extractable Aluminum
Dilution Factors and Dilution Reagent Blank Values; Titer and Normality
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116cc**

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Solution Recovered in Syringe (mL)	KCl-Extractable Al			KCl-Exchangeable Acidity	
		Aliquot Volume (mL)*	Total Dilution Volume (mL)*	Solution Conc. (mg/L)	Titer (Volume in mL)	Normality of Titrant
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
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32						
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37						
38						
39						
40						
41						
42						

Blank	Total Volume in Sample (mL)	KCl-Extractable Al		
		Aliquot Volume (mL)	Total Dilution Volume (mL)	Dilution Conc. (mg/L) KCl
D-Blank				
D-Blank				
D-Blank				

*Enter 0 if no dilution is made.

Exchangeable Basic Cations in CaCl_2
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116d

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

	Exchangeable Basic Cations in CaCl_2												
Sample Number	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b				Total Dilution Volume (mL) ^b				Solution Concentration (mg/L)			
		Ca	Mg	K	Na	Ca	Mg	K	Na	Ca	Mg	K	Na
01													
02													
03													
04													
05													
06													
07													
08													
09													
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13													
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37													
38													
39													
40													
41													
42													

Blank	Total Volume in Sample (mL)	Aliquot Volume in Dilution (mL)	Total Volume of Dilution (mL)	Dilution Blank Concentrations (mg/L)			
				Ca	Mg	K	Na
D-Blank							
D-Blank							
D-Blank							
U-Blank							
D-Blank							
U-Blank							

^aVolume added for extraction.
^bEnter U if no dilution is made.

Exchangeable Fe and Al in CaCl₂
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116e

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Extractable Fe and Al in CaCl ₂								Soil Type Mineral (M) or Organic (O)	Extraction Ratio ^c
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b		Total Dilution Volume (mL) ^b		Solution Conc. (mg/L)				
		Fe	Al	Fe	Al	Fe	Al			
01										
02										
03										
04										
05										
06										
07										
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11										
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38										
39										
40										
41										
42										

Blank	Extractable Fe and Al in CaCl ₂				
	Total Volume in Sample (mL)	Aliquot Volume (mL)	Dilution Volume (mL)	Dilution Conc. (mg/L)	
				Fe	Al
D-Blank					
D-Blank					
D-Blank					
D-Blank					
D-Blank					

^aVolume added for extraction.

^bEnter U if no dilution is made.

^cSoil to solution ratio is expressed as 1:x; enter the value of x.

Exchangeable Fe and Al in Pyrophosphate
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116ee

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Extractable Fe and Al in Pyrophosphate						
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b		Total Dilution Volume (mL) ^b		Solution Conc. (mg/L)	
		Fe	Al	Fe	Al	Fe	Al
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
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38							
39							
40							
41							
42							

Blank	Extractable Fe and Al in Pyrophosphate				
	Total Volume in Sample (mL)	Aliquot Volume (mL)	Dilution Volume (mL)	Dilution Conc. (mg/L)	
				Fe	Al
D-Blank					
D-Blank					
D-Blank					
D-Blank					
D-Blank					
D-Blank					

^aVolume added for extraction.
^bEnter U if no dilution is made.

Exchangeable Fe and Al in Acid-Oxalate
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116f

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Extractable Fe and Al in Acid-Oxalate						
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b		Total Dilution Volume (mL) ^b		Solution Conc. (mg/L)	
		Fe	Al	Fe	Al	Fe	Al
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
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36							
39							
40							
41							
42							

Blank	Extractable Fe and Al in Acid-Oxalate				
	Total Volume in Sample (mL)	Aliquot Volume (mL)	Dilution Volume (mL)	Dilution Conc. (mg/L)	
				Fe	Al
U-Blank					
D-Blank					
U-Blank					
D-Blank					
U-Blank					
D-Blank					

^aVolume added for extraction.
^bEnter U if no dilution is made.

Extratable Fe and Al in Citrate-Dithionite
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116ff

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Extratable Fe and Al in Citrate-Dithionite						
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b		Total Dilution Volume (mL) ^b		Solution Conc. (mg/L)	
		Fe	Al	Fe	Al	Fe	Al
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
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38							
39							
40							
41							
42							

Blank	Extratable Fe and Al in Citrate-Dithionite				
	Total Volume in Sample (mL)	Aliquot Volume (mL)	Dilution Volume (mL)	Dilution Conc. (mg/L)	
				Fe	Al
D-Blank					
D-Blank					
D-Blank					
D-Blank					
D-Blank					
D-Blank					

^aVolume added for extraction.
^bEnter U if no dilution is made.

**Water Extratable Sulfate and Nitrate
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116g**

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	H ₂ O Extractable Nitrate				H ₂ O Extractable Sulfate		
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b	Total Dilution Volume (mL) ^b	Solution Concentration (mg/L)	Aliquot Volume (mL) ^b	Total Dilution Volume (mL) ^b	Solution Concentration (mg/L)
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
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32							
33							
34							
35							
36							
37							
38							
39							
40							
41							
42							

Blank	Total Volume in Sample (mL)	Aliquot Volume in Dilution (mL)	Total Volume of Dilution (mL)	Dilution Blank Concentrations	
				NO ₃ ⁻	SO ₄ ²⁻
U-Blank					
D-Blank					
D-Blank					

^aVolume added for extraction.

^bEnter U if no dilution is made.

Phosphate Extratable Sulfate
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116gg

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	PO ₄ ³⁻ Extractable Sulfate			
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b	Total Dilution Volume (mL) ^b	Solution Concentration (mg/L)
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
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37				
38				
39				
40				
41				
42				

Blank	Total Volume in Sample (mL)	Aliquot Volume in Dilution (mL)	Total Volume of Dilution (mL)	Dilution Blank Concentration (mg/L)
U-Blank				
U-Blank				
U-Blank				

^aVolume added for extraction.
^bEnter U if no dilution is made.

Sulfate Adsorption Isotherms
Dilution Factors and Dilution Reagent Blank Values
Direct/Delayed Response Project (DDRP) Soil Survey
Form 116h

Lab Name _____ Batch ID _____

Lab Manager's Signature _____

Sample Number	Sulfate Adsorption Isotherm																		
	Total Volume in Sample (mL) ^a	Aliquot Volume (mL) ^b						Total Dilution Volume (mL) ^b						Solution Concentration (mg/L)					
		0	2	4	8	16	32	0	2	4	8	16	32	0	2	4	8	16	32
01																			
02																			
03																			
04																			
05																			
06																			
07																			
08																			
09																			
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32																			
33																			
34																			
35																			
36																			
37																			
38																			
39																			
40																			
41																			
42																			

^aVolume added for adsorption.
^bEnter U if no dilution is made.

Summary of Exchangeable Cations in NH₄OAc
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 204a

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Exchangeable Cations in NH ₄ OAc, meq/100g			
	Ca	Mg	K	Na
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
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38				
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41				
42				

Summary of Exchangeable Cations in NH_4Cl
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 204b

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Exchangeable Cations in NH_4Cl , meq/100g			
	Ca	Mg	K	Na
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
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33				
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39				
40				
41				
42				

Summary of Exchangeable Cations in 0.002 M CaCl₂
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 204c

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Exchangeable Cations in 0.002 M CaCl ₂ , meq/100g					
	Ca*	Mg	K	Na	Fe	Al
01						
02						
03						
04						
05						
06						
07						
08						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
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38						
39						
40						
41						
42						

*Reported data may be negative.

Summary of Cations Exchange Capacity (CEC)
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 204d

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	CEC, meq/100g		CEC, meq/100g	
	NH ₄ OAc	NH ₄ Cl	NH ₄ OAc	NH ₄ Cl
01			22	
02			23	
03			24	
04			25	
05			26	
06			27	
07			28	
08			29	
09			30	
10			31	
11			32	
12			33	
13			34	
14			35	
15			36	
16			37	
17			38	
18			39	
19			40	
20			41	
21			42	

Summary of Extractable Iron and Aluminum Data
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 205

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Pyrophosphate Extractable, Weight %		Acid-Oxalate Extractable, Weight %		Citrate-Dithionite Extractable, Weight %	
	Fe	Al	Fe	Al	Fe	Al
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
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16						
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42						

Summary of Extractable Sulfate, Exchangeable Acidity, and
Extractable Aluminum Data, Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 206

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Extractable Nitrate mg N/kg	Extractable Sulfate, mg S/kg		Exchangeable Acidity, meq/100g		Extractable Al, meq/100g
	H ₂ O	H ₂ O	PO ₄ ³⁻	BaCl ₂	KCl	KCl
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
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41						
42						

Summary of Sulfate-Adsorption Isotherm Data
Corrected for Blanks* and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 207

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Sulfate Remaining in Solution, mg S/L					
	Initial Solution Concentration, mg S/L					
	0	2	4	8	16	32
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
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29						
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42						

*Blanks are double-deionized water.

Summary of Total C, N, S, Specific Surface, and Inorganic Carbon Data
Corrected for Blanks and Dilutions
Direct/Delayed Response Project (DDRP) Soil Survey Form 208

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Total S, Weight %	Total N, Weight %	Specific Surface, m ² /g	Total C, Weight %	Inorganic C, Weight %	
					<2 mm	2-20 mm
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
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42						

Particle Size Analysis Raw Data
Direct/Delayed Response Project (DDRP) Soil Survey Form 303b

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Cylinder Volume (mL) _____ Pipet Volume (mL) _____

Weight of Fraction, grams								
Size Class and Particle Diameter (mm)								
Sample Number	Sand							
	Sand (2.0- 0.05)	Clay and Fine Silt (<0.02)	Clay (<0.002)	Very Coarse (2.0- 1.0)	Coarse (1.0- 0.5)	Medium (0.5- 0.25)	Fine (0.25- 0.1)	Very Fine (0.1- 0.05)
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
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41								
42								

Summary of BaCl₂ - Exchangeable Acidity Raw Data
Direct/Delayed Response Project (DDRP) Soil Survey Form 306

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	BaCl ₂ - Exchangeable Acidity	
Extract	Titer (Volume in mL)	Normality of Titrant
01		
02		
03		
04		
05		
06		
07		
08		
09		
10		
11		
12		
13		
14		
15		
16		
17		
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42		

Summary of Total C, N, S, Specific Surface, and Inorganic Carbon Data
Direct/Delayed Response Project (DDRP) Soil Survey Form 308

Analytical Lab ID _____

Date Form Completed _____ Batch ID _____

Date Batch Received _____ Prep Lab Name _____

Lab Manager's Signature _____

Remarks _____

Sample Number	Total S, µg	Total N, µg	Specific Surface, mg EGME		Total C, µg	Inorganic C, µg	
			added	retained		<2 mm	2-20 mm
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
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Appendix C

Plan for Laboratory Audit Samples

1.0 Introduction

Natural audit samples are used for monitoring the analytical laboratories of the Direct/Delayed Response Project (DDRP) Soil Survey. Synthetic audit samples of known composition are not used in this project. The purpose of natural audit samples is to determine within-batch precision and relative intralaboratory and interlaboratory bias and to assure that each laboratory is maintaining the capability to analyze samples satisfactorily. Every effort is made to ensure that the analytical laboratory does not recognize an audit sample as different from a routine sample. Therefore, an audit sample is a double-blind quality assurance (QA) sample; that is, the analytical laboratory does not recognize an audit sample as a QA sample and does not know its composition.

2.0 Source of Laboratory Audit Samples

Because audit samples should have properties similar to those samples undergoing physical, chemical, and mineralogical characterization, six soil samples were chosen to serve as natural audit samples for the soil survey. Four samples from New York were derived from horizons of an Inceptisol, a Histosol, and two Spodosols; these are representative of soils sampled in the northeastern United States. The two samples from Georgia are Ultisols, representative of soils from the southeastern United States.

Specific descriptions below include series name, soil taxonomic class, interval from which the sample was taken, vegetative cover, geomorphic position, and geographic location:

1. Bw - Bice series; Typic Dystrochrept, coarse loamy, mixed, frigid; depth 38 to 96 cm; sugar maple - yellow birch - cherry; convex glacial till upland; Ava (Oneida County), New York; West Avenue Road, 90 m west of cemetery.
2. Oa - Palms series; Terric Medisaprists; depth 25 to 140 cm; open wetland, sphagnum; kettle position; Rome (Oneida County), New York; Tannery Road.
3. Bs - Allagash series; Typic Haplorthod, coarse loamy over sandy, mixed, frigid; depth 36 to 64 cm; sugar maple - beech - yellow birch with balsam inclusions; convex high terrace; Webb (Herkimer County), New York; along upper end of Independence Lake water line.
4. C - Adams series; Typic Haplorthod, sandy, mixed, frigid; depth 0.9 to 9.1 m; sugar maple - beech with black cherry inclusions; terrace; Webb (Herkimer County), New York; 305 m east of Old Forge Airport.
5. A - Hayesville series; Typic Hapludult, clayey, oxidic, mesic; depth 0 to 20 cm; mixed forest; upland, 10 to 25 percent slopes; near Blue Ridge (Fannin County), Georgia.
6. B2t - Hayesville series, Typic Hapludult, clayey, oxidic, mesic; depth 38 to 119 cm; mixed forest; upland, 10 to 25 percent slopes; near Blue Ridge (Fannin County), Georgia.

Bulk soil sample and descriptive information were provided by the Soil Conservation Service in New York and Georgia.

3.0 Characterization of Laboratory Audit Samples

The audit samples are used to monitor laboratories providing physical and chemical data, as well as laboratories providing mineralogical data.

The initial referee laboratories responsible for characterizing the chemical and physical parameters according to the analytical procedures set forth in their contracts with EPA were the Soil Conservation Service National Soil Survey Laboratory in Lincoln, Nebraska, and the Weyerhaeuser Technology Center Analytical Laboratory in Federal Way, Washington.

The referee laboratory responsible for mineralogical characterization was the Soil Conservation Service National Soil Survey Laboratory in Lincoln, Nebraska.

The data obtained from referee laboratories are used to set acceptance windows for single-parameter values reported by analytical laboratories over the course of the soil survey (see Section 12.1).

4.0 Stability of Laboratory Audit Samples

Data generated by the contractor analytical laboratories will be examined to assess possible changes in the chemical parameters of the audit samples with respect to time.

5.0 Logistics

Audit samples prepared at the QA laboratory are packaged to resemble routine samples: audit samples for physical and chemical parameters, in 1-kg lots; those for mineralogical analyses, in 500-g lots. Audit samples are supplied to each preparation laboratory. Without additional processing of the samples, the preparation laboratory inserts the audit samples into batches that are sent to the analytical laboratories.

For physical and chemical parameters, two audit samples of the same type are included in each analytical batch. These are specified by the QA manager or designee. Each analytical batch of up to 39 routine samples and field duplicates also includes one preparation duplicate.

The use of audit samples for mineralogical laboratories is specified in Section 15.0 of this document.

Appendix D

Field Sampling On-Site Evaluation Questionnaires

This appendix contains questionnaires for evaluation of sampling crews in the Northeastern Soil Survey (Fall, 1985) and in the Southern Blue Ridge Province Soil Survey (Spring, 1986).

Field Sampling On-Site Evaluation Questionnaire
Northeastern DDRP Soil Survey

Date: _____ Crew ID: _____
State: _____

Reviewers

Name	Title	Education	Experience
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

I.	<u>Equipment</u>	<u>Yes</u>	<u>No</u>	<u>Comments</u>
	1. Munsell color book (condition)	—	—	
	2. Clinometer (type)	—	—	
	3. Camera (type)	—	—	
	4. Film (type, expiration date)	—	—	
	5. Lens (type)	—	—	
	6. Spades (type)	—	—	

I.	<u>Equipment</u> (continued)	<u>Yes</u>	<u>No</u>	<u>Comments</u>
7.	Augers (type) What is used to sample Histosols?	—	—	
8.	Sieves (size, brand)	—	—	
9.	Compass (type, declination)	—	—	
10.	Measuring tape	—	—	
11.	SCS-232 Form How is it kept dry?	—	—	
12.	Marking pens	—	—	
13.	Saran Ratio Quantity How often is it used? When is it used?	—	—	
14.	Coolers	—	—	
15.	Gel packs	—	—	
16.	Thermometers	—	—	
17.	Maps	—	—	
18.	Aerial photographs	—	—	
19.	Flagging	—	—	
20.	Marker flags	—	—	
21.	Staplers or twist ties	—	—	
22.	Clod boxes (type, condition)	—	—	

I. Equipment (continued)

- | | | |
|-----------------|---|---|
| 23. Clod wire | — | — |
| 24. Clod labels | — | — |

II. Site Selection

- | | | |
|---|---|---|
| 1. Does the crew have a list of sampling classes to be sampled in each watershed? | — | — |
| 2. Does the crew have a map with the five (5) random points marked? | — | — |
| 3. How are distances measured?
If pacing is used, is pacing standardized? | — | — |
| 4. What does the crew use for the starting point or control site? | — | — |
| 5. Does the crew mark the initial random point with a marker flag? | — | — |
| 6. Does the crew leader stay within a 100 square yard area when assessing sampling class? | — | — |
| 7. Does the crew understand vegetation class? | — | — |
| 8. How does the crew decide if the soil type is of the desired sampling class? | — | — |
| 9. On what area is vegetation class determined? | — | — |
| 10. Does the crew have a clear under standing of basal area? | — | — |

II. Site Selection (continued)

- | | |
|---|-----|
| 11. Does the crew leader proceed at 20-foot intervals from the initial random point? | — — |
| 12. Does the crew leader use a compass to determine cardinal direction? | — — |
| 13. Does the crew understand which direction corresponds to the random numbers from 1 to 8? | — — |
| 14. Does the crew have enough copies of the field sampling manual? | — — |
| 15. Are the criteria used in selection of each site entered in the logbook? | — — |
| 16. Is the field logbook neat and legible? | — — |
| 17. Is a pen used for all entries in the logbook? | — — |
| 18. Are entries in the logbook reviewed or checked by other members of the crew? | — — |

III. Sampling and Pedon Description

- | | |
|---|-----|
| 1. Is the pit large enough for description, i.e., 1 meter vertical face? | — — |
| 2. Is loose soil material cleaned from the sides of the pit prior to profile description? | — — |
| 3. Are pit faces examined from the top downward? | — — |

III. Sampling and Pedon Description (continued)

- | | | |
|--|---|---|
| 4. Are horizon boundaries marked before identification? | — | — |
| 5. Are photographs taken after horizons are identified? | — | — |
| 6. Is each horizon studied in the horizontal exposure? | — | — |
| 7. Are the following parameters determined for each horizon? | — | — |
| a) Type | — | — |
| b) Depth | — | — |
| c) Boundary | — | — |
| d) Color | — | — |
| e) Texture | — | — |
| f) Structure | — | — |
| g) Consistence | — | — |
| h) Presence of mottles: | | |
| (1) abundance | — | — |
| (2) size | — | — |
| (3) contrast | — | — |
| 8. Are the following parameters determined for each pedon? | | |
| a) Surface vegetation | — | — |
| b) Rock fragments | — | — |
| c) Presence of roots, pores, etc. | — | — |
| d) Slope and aspect | — | — |
| e) Physiographic region and location | — | — |
| f) Azimuth perpendicular to pedon face | — | — |

III. Sampling and Pedon Description (continued)

- | | | | |
|-----|--|---|---|
| g) | Drainage class | — | — |
| h) | Permeability | — | — |
| i) | Pedon position | — | — |
| j) | Water table | — | — |
| k) | Depth to bedrock | — | — |
| l) | Diagnostic features | — | — |
| m) | Taxonomic classification | — | — |
| n) | Bulk density | — | — |
| 9. | How are photographs taken
(distance, angle, scale)?
By whom? | | |
| 10. | In sampling for bulk density: | | |
| a) | Is an attempt made to obtain
clods from all horizons? | — | — |
| b) | Are clods fist-sized? | — | — |
| c) | Are clods taken in
triplicate? | — | — |
| d) | How are clods dried? | — | — |
| e) | Are clods sufficiently
dipped in the Saran resin? | — | — |
| f) | Are clods labeled correctly? | | |
| | (1) Sample code | — | — |
| | (2) Horizon | — | — |
| | (3) Replicate number | — | — |
| g) | Are clods packed carefully? | — | — |
| 11. | Is NADSS LABEL A filled out
correctly and neatly? | — | — |
| 12. | Is one field duplicate
sampled per day? | — | — |

III. Sampling and Pedon Description (continued)

- | | | |
|---|---|---|
| 13. How is the field duplicate sampled? | — | — |
| 14. Are both plastic and canvas bags labeled? | — | — |
| 15. Are two sample bags completely filled for organic horizons? | — | — |
| 16. Is mineral soil sieved through a 19-mm sieve onto plastic or into a 1-gallon bucket? | — | — |
| 17. Is excess water drained from Histosols? | — | — |
| 18. Are precautions taken to prevent contamination from above and below horizon? | — | — |
| 19. Are sieves and sampling tools cleaned sufficiently between samples? | — | — |
| 20. On SCS Form 232: | | |
| a) Is the day added under sampling date? | — | — |
| b) Is vegetation correctly described in order of tree basal area? | — | — |
| c) Is the CREW ID written in the lower right hand corner of box labeled "DESCRIBERS NAMES"? | — | — |
| d) Are digits 1 through 17 of "LOCATION DESCRIPTION AND FREE FORM SITE NOTES" correct? | — | — |
| 1-6 = site ID | — | — |
| 8 = random point | — | — |
| 10-12 = sampling class | — | — |
| 14-17 = azimuth | — | — |

III. Sampling and Pedon Description (continued)

- e) Are volume estimates of coarse fragments correctly recorded?
- | | | |
|-------------|---|---|
| 2 - 75 mm | — | — |
| 75 - 250 mm | — | — |
| >250 mm | — | — |
- f) Are horizon descriptions legible?
- | | | |
|--|---|---|
| | — | — |
|--|---|---|

Field Sampling On-Site Evaluation Questionnaire

Southern Blue Ridge Province DDRP Soil Survey

General (Page 1 of 1)

Date: _____ State: _____

Crew ID: _____ Site Number: _____

Time of arrival at site: _____ Time of departure: _____

Field Crew:

Name

Audit Team:

Name

Representing

Notes or Comments:

Site Selection (Page 1 of 3)

Item	Used in Field?		
	Yes	No	
Screw auger			
Bucket auger			
Aerial photographs			
Stereoscope			
Compass			
Punch probe			
Spade			
Topographic site map			
Sampling site map			
Random number table			
Other site selection equipment used:			

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Site Selection (Page 2 of 3)

Item	Used in Field?		Comments
	Yes	No	
Does the field crew have the watershed soil map with prioritized starting points?			
Are the procedures detailed in Section 3.0 of the sampling manual followed? If no - note deviations: _____ _____ _____			
Is the starting point marked? How?			
How many compass directions were attempted?			
What were the total number of points necessary to arrive at an acceptable site?			
Were the number of points and the compass direction recorded properly on 232 Form?			
How are the 10-m intervals measured?			
How is the sampling class assessed at each site?			
How is the vegetation class assessed at each site?			

Site Selection (Page 3 of 3)

Item	Used in Field?		
	Yes	No	
Was this site a paired pedon?			
If yes, describe how the second pedon was chosen.			
If yes, is the pedon of the same series?			
If yes, is the pedon of the same sampling class?			
How far was the paired pedon from the routine pedon?			
Are the slope and elevation the same as that of the routine pedon?			

Comments:

Pedon Excavation (Page 1 of 2)

Item	Used in Field?		
	Yes	No	
Shovels			
Spades (sharpshooters)			
Picks/Bars			
Hand pump (Beckenson Gusher*, 16 GPM)			
Posthole digger			
Backhoe			
Other pedon excavation equipment used:			

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Pedon Excavation (Page 2 of 2)

Item	Used in Field?		
	Yes	No	
Is the excavated pit of suitable size (1m x 2M)?			
Does this pit have any water table problems?			
If yes, what was done to control sample contamination?			
Is this an organic soil?			
If yes, how was the soil excavated?			
If yes, what was used to excavate?			

Comments:

Photographic Documentation (Page 1 of 2)

Item	Used in Field?		
	Yes	No	
35-mm camera, automated with flash*			
If the camera is supplied by the crew, what type is it?			
Slide film ASA _____			
Photogray cards*			
Khaki measuring tape [†]			

Other photographic equipment used:

*Supplied by EMSL-Las Vegas

†Supplied by ERL-Corvallis

Photographic Documentation (Page 2 of 2)

Item	Used in Field?		
	Yes	No	
Are the photos taken before destructive profile description is begun?			
Are the horizons delineated with golf tees?			
Is the khaki measuring tape included in the photo?			
Is the photogray card placed at the top of the profile?			
Is it correctly filled out?			
Are slides recorded in the field notebook?			
Are slides recorded on the 232 Form?			
Are the 4 required (minimum) photographs taken:			
pedon face?			
tree canopy?			
understory vegetation?			
landscape/landform?			

Comments:

Pedon Description (Page 1 of 3)

Item	Used in Field?	
	Yes	No
SCS-232 Form*		
Tablet/form holder		
Munsell color chart		
Condition:		
Clinometers		
Compass		
Set for declination?		
What was local declination?		
Hard lens		
Knife, ice pick, or equivalent		
pH kit		
Kind-		
Indicators-		
Is the indicator fresh (<3 months old)?		
Peat sampler (Histosols)		
Flagging*		

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Pedon Description (Page 2 of 3)

Item	Used in Field?		
	Yes	No	
Yellow flag markers*			
Labeling pens* indelible?			
Golf tees			
Other soil description equipment used?			
List			
Is the pit face cleaned before horizons are delineated?			
Is spatial variability assessed not only horizontally but also in three dimensions?			
How?			
Is horizon depth measured from an accurate zero-point at the top of the profile?			
Specifically where?			
Over what horizontal range is horizon thickness determined?			
Who determines color?			
Describer?			

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Pedon Description (Page 2 of 3) Continued

Item	Used in Field?	
	Yes	No
Recorder?		
other?		

*Supplied by EMSL-Las Vegas

†Supplied by ERL-Corvallis

Pedon Description (Page 3 of 3)

Item	Used in Field?		
	Yes	No	
Is the 232 Form filled in completely?			
Is the 232 Form filled in legibly?			
Who recorded 232 form data?			
Is the compass used for azimuth determination corrected for declination?			
What is the declination?			
How was the declination value determined?			
Is the azimuth determined perpendicular to the pedon face?			
Are the codes adequate for all situations encountered for this pedon?			
Were the codes adequate for other pedons?			
Was any of the 232 form filled out before arrival in field?			

Comments:

Soil Sampling (Page 1 of 4)

Item	Used in Field?		
	Yes	No	
20-mm sieve*			
1-gallon plastic bucket			
How many?			
Plastic sheet*			
Brush for cleaning sieve			
What is used to clean the pedon face?			
Plastic inner bags*			
Canvas outer bags*			
Label A*			
Staplers*			
Dust pan			
Hand trowel			
Post hole digger (Histosols only)			
Spatula or putty knife			

Other sampling equipment used:

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Soil Sampling (Page 2 of 4)

Item	Used in Field?		
	Yes	No	
Are all important horizons sampled?			
Was adequate amount of organic horizon material collected?			
Was adequate mineral material collected for each horizon?			
If no, was there a limiting factor?			
What?			
How was the pedon sampled?			
State the order of horizon sampling _____ _____			

Soil Sampling (Page 3 of 4)

Item	Used in Field?		
	Yes	No	
Was the pedon sampled in such a way as to avoid contamination?			
If no, give a detailed explanantion:			
Was each horizon sampled into a dustpan			
Was the sampled sieved according to proctocol?			
Were any horizons split for sampling?			
Specify:			
Were they >30 cm thick (above 1m)			
Were they >60 cm thick? (below 1m)			
Were the sample bags labeled correctly?			
Were the canvas bags labeled correctly?			
How were the sample bags closed?			

Soil Sampling (Page 4 of 4)

Item	Used in Field?		
	Yes	No	
Were any problems or concerns identified in the field sampling methods?			
If yes, provide a detailed explanantion:			
Was the field duplicate taken?			
Was the field duplicate properly labeled?			
How was the field duplicate taken?			
How were the two samples for paired pedons collected?			
Were alternate trowelsful used?			
Were rock fragment size classes determined correctly?			

Comments:

Clod Sampling (Page 1 of 2)

Item	Used in Field?		
	Yes	No	
Saran*			
Mixture ratio	NA	NA	
acetone for thinning?			
What is the saran stored in?	NA	NA	
Hairnets*			
Plastic bags*			
Clod box*			
Labels*			
on the clod			
on the box			
Describe system for drying clods.	NA	NA	
Comments:			

Other clod sampling equipment used:

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Clod Sampling (Page 2 of 2)

Item	Used in Field?		
	Yes	No	
Were 3 clods obtained from each horizon sampled?			
If no, which horizons had no corresponding clod samples?			
Why?			
Were clods fist sized?			
If no, is there any explanation?			
Are clods dipped once in Saran?			
If more dips are required, is it noted?			
Is the clod-drying set-up adequate?			
If no, explain			
Are clods labeled correctly?			
Were clods placed correctly in the clod box (i.e., No. 1 in upper left, etc.)			
Are replicate numbers assigned?			

Comments:

Sample Transport (Page 1 of 3)

Item	Used in Field?		
	Yes	No	
Backpacks			
Styrofoam coolers*			
Gel-packs*			
Are there any leaking problems?			
Thermometers*			

Other sample transport equipment used?

* Supplied by EMSL-Las Vegas

† Supplied by ERL-Corvallis

Sample Transport (Page 2 of 3)

Item	Used in Field?		
	Yes	No	
How are samples carried from the site to the vehicle?			
How are clod boxes carried?			
Were all samples accounted for upon arrival at the vehicle?			
Were coolers available?			
With gel-pacs?			
What was the temperature in the cooler?			
Were samples to be transported to the preparation laboratory that evening?			
If not, how were they kept cool until delivery?			
Were there any problems in the past with sample bags breaking?			
Were there any problems with contamination due to gel-pack leakage?			
Were there any problems with sample cross-contamination?			

Sample Transport (Page 3 of 3)

Item	Used in Field?		
	Yes	No	
Was all field equipment accounted for at the end of sampling?			
Was the pit closed?			
Was the pit marked?			
Was the field notebook filled in?			
Was the field notebook legible?			

Comments:

Summary (Page 1 of 1)

Summary Comments:

Areas of Concern:

Concerns that should be reported to sampling task leader (with suggested resolution, if appropriate):

Appendix E

Preparation Laboratory On-Site Evaluation Questionnaire

The following questionnaire is completed to provide documentation of an on-site evaluation. Generally, a preparation laboratory is evaluated prior to receiving samples to assess the ability of the laboratory, in terms of personnel, facilities, and equipment, to process soil samples successfully. A second evaluation is made after sample processing is underway. At the time of the second evaluation, adherence to protocol is evaluated, and specific problems are addressed.

Preparation Laboratory On-Site
Evaluation Questionnaire
DDRP Soil Survey

General (Page 1 of 2)

Date _____

Laboratory: _____

Street Address: _____

Mailing Address (if different from above): _____

City: _____

State: _____

Laboratory Telephone Number: () _____

Laboratory Director: _____

Laboratory Quality Assurance Officer: _____

Type of Evaluation: _____

Contract Number: _____

Contract Title: _____

General (Page 2 of 2)

Personnel Contacted:

Name

Title

[illegible]

Laboratory Evaluation Team:

Name

Title

[illegible]

.....

Laboratory Organizational Chart

Organization and Personnel (Page 2 of 3)

Laboratory Personnel

Position	Name	Academic Training*	Special Training	Years Experience†
----------	------	--------------------	------------------	-------------------

*List highest degree obtained and specialty. Also list years toward a degree.
†List only experience directly relevant to task to be performed.

Organization and Personnel (Page 3 of 3)

Item	Yes	No	Comments
Do personnel assigned to this project have the appropriate educational background to successfully accomplish the objectives of the program?			
Do personnel assigned to this project have the appropriate level and type of experience to successfully accomplish the objectives of this program?			
Is the organization adequately staffed to meet project commitments in a timely manner?			
Was the Laboratory Manager available during the evaluation?			
Was the Quality Assurance Supervisor available during the evaluation?			
Do the laboratory personnel observe safety regulations?			
Are the following available: Lab coats?			
Goggles?			
Gloves?			
Aspirators?			
Is there a laboratory dress code?			
If there is a dress code, is it enforced?			
Who will be responsible for splitting preparation duplicate samples? _____			
Who will be responsible for receiving audit samples? _____			

Laboratory Manager (Page 1 of 1)

Item	Yes	No	Comments
Does the laboratory manager have his/her own copy of the Field Sampling Manual and the Laboratory Methods Manual?			
Before filling out Form 102, does the laboratory manager: Review data values on Form 101?			
Review raw data in lab notebooks?			
Check for adequate and accurate ID of QC sample?			
Does the laboratory manager have forms 101 and 102 on file?			

Procedural Questions:

Who is responsible for assuring that Form 102 is contained in each box shipped to the analytical laboratory? _____

How many copies of the forms are filed by the preparation laboratory? _____

Standard Operating Procedures (Page 1 of 1)

Item	Yes	No	Comments
Does the laboratory have a standard operating procedures (SOP) manual?			
Is the SOP manual followed in detail?			
Does the SOP manual contain quality control practices?			
Does each analyst/technician have a copy of the SOP manual?			
Does the SOP manual deviate from the procedures required by this project?			
If the SOP manual does deviate, are the deviations documented in written form?			
Does each analyst/technician have a copy of all methods and procedures required by this project?			

Comments:

Laboratory Facilities (Page 1 of 3)

When touring the facilities, give special attention to (1) the overall appearance of organization and neatness, (2) the proper maintenance of facilities and instrumentation, and (3) the general adequacy of the facilities to accomplish the required work.

Item	Yes	No	
Does the laboratory appear to have adequate work-space for sample drying?			
Does the laboratory appear to have adequate work-space for sample preparation (sieving, crushing)?			
Does the laboratory have a source of distilled/deionized/demineralized water?			
Is the analytical balance located away from draft and areas subject to rapid temperature changes?			
Has the balance been calibrated within the past year by a certified technician?			
Is the balance checked with a class S standard weight before each use, and is the result recorded in a logbook?			
Are exhaust hoods provided that allow adequate workspace within?			
Is the laboratory clean and organized?			
Are contamination-free work areas provided?			
Are adequate cold storage facilities provided for sample storage?			
Are all samples stored in cold storage (4 °C) when not in use?			

Laboratory Facilities (Page 2 of 3)

Item	Yes	No	Comment
Is the temperature of the cold storage facilities recorded daily in a logbook?			
Is there a temperature gauge on the outside of each cold storage unit that measures the temperature of that unit?			
Are the stored samples tightly closed?			
Are there any open samples stored in the storage units?			
Is there any food stored in the units?			
Are there any reagents stored in the units?			
Are all chemicals dated upon receipt and thrown away when shelf life is exceeded?			
Are chemical waste disposal procedures/policies adequate?			
Is the laboratory secure?			

Laboratory Facilities (Page 3 of 3)

Item	Available		Comments
	Yes	No	
Gas			
Lighting			
Compressed air			
Electrical services			
Hot and cold water			
Laboratory sink			
Ventilation system			
Hood space			
Cabinet space			
Storage space (m2)			
Shared space			

Comments:

General Equipment (Page 1 of 1)

Item	Equipment			Condition/Age			Comments
	Quantity	Make	Model	Good	Fair	Poor	
Balance, analytical							
Balance, top-loading							
Class "S" weights							
Balance table							
NBS-calibrated thermometer							
Distilled/Deionized water							
Drying oven							
Drying surfaces							
Drying containers/trays							
Riffle splitter							

Comments:

Soil Preparation Process (Page 1 of 1)

Equipment	Available		Comments
	Yes	No	
Drying surfaces			
Wooden rolling pin			
Crushing tray or surface			
2-mm sieve, US 10 std. mesh, sq. hole			
Jones-type riffle splitter (or comparable equipment)			

Procedural Questions:

How is cross-contamination between samples in the drying area avoided? _____

Are there separate workspaces for sample drying and for sample preparation? _____

How are riffle splitters and sieves cleaned between samples? _____

Is drying area removed from reagent storage? _____
reagent use? _____

Are labels kept with drying samples? _____ How? _____

How is the moisture-content sample removed? _____

Is the moisture-content sample returned to the bulk sample? _____

Qualitative Test for Inorganic Carbon and Handling of Rock Fragments
(Page 1 of 1)

Equipment	Available		Comments
	Yes	No	
Porcelain spot plate			
DI water in squeeze bottle or eyedropper			
Microscope (10X or higher power)			
4 N HCL			
Test soil spiked with 5% CaCO_3			
Test soil spiked with 5% $\text{CaMg}(\text{CO}_3)_2$			

Procedural Questions:

How are rock fragments saved from the sieving process? _____

Is this analysis physically removed from the sieving and soil-drying processes? _____

How are rock fragments from a positive test labeled? _____

_____ stored? _____

How are rock fragments from a negative test disposed of? _____

Comments:

Sample Archiving and Shipping (Page 1 of 1)

How are archived samples labeled? _____

stored? _____

Is there a systematic storage procedure? Explain. _____

Is a map or key showing the location of archived samples readily available?

Are archived samples easily retrieved? _____

Are sample identifications permanent and legible? _____

Is there a designated sample custodian? If yes, name. _____

Are the sample custodian's procedures and responsibilities documented? If yes,
where? _____

Are sample numbers cross-referenced with field data and filed? _____

where? _____

Comments:

Summary (Page 1 of 2)

Item	Yes	No	Comments
Do responses to the evaluation indicate that project and supervisory personnel are aware of QA and its application to the project?			
Do project and supervisory personnel place positive emphasis on QA/QC?			
Has responses with respect to QA/QC aspects of the project been open and direct?			
Has a cooperative attitude been displayed by all project and supervisory personnel?			
Have any QA/QC deficiencies been discussed during evaluation?			
Is the overall QA adequate to accomplish the objectives of the project?			
Have corrective actions recommended during previous evaluations been implemented?			
Are any corrective actions required? If so, list in detail below and on following page.			

Summary (Page 2 of 2)

Summary comments and corrective actions:

Appendix F
Facsimile of
Instructions for Pre-Award Performance
Evaluation Samples

Instructions accompany the pre-award performance evaluation samples that are sent to potential contractor laboratories.

In the instructions, three references are made to exhibits of the Invitation for Bid (IFB). The corresponding references are indicated below:

- 1) "Exhibit B" is Appendix B of this document.
- 2) "Exhibit D" is derived from the *Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey* by K. A. Cappel, L. J. Blume, G. A. Raab, J. K. Bartz, and J. L. Engels, U.S. Environmental Protection Agency, Las Vegas, Nevada, 1987.
- 3) "Exhibit E" is Section 10.0 (Internal Quality Control) of this document and Section 2.0 of Cappel et al. (1987).

2.0 Direct/Delayed Response Project Soil Survey Pre-Award Performance Evaluation Samples

Instructions

Enclosed are two 1-kg soil samples to be used in the evaluation of contractor laboratories interested in participating in the Direct/Delayed Response Project Soil Survey, sponsored and conducted by the U.S. Environmental Protection Agency.

Upon receipt, check the contents of this package to ensure that both containers are present and intact. Call the Quality Assurance Manager immediately in case of missing items, spillage, or questionable condition of the pre-award samples.

Each sample is to be analyzed for all parameters according to the methods described in Exhibit D. All quality control (QC) procedures specified in Exhibit E must be followed. Duplicate sample analyses are required for each parameter, with the exception that triplicate samples are required for surface. Matrix spike analyses are required for all parameters except particle size, pH, and specific surface. Replicate and matrix spike analyses may be performed on either soil sample. Initial, continuing, and final quality control calibration samples, as well as reagent and calibration blanks, are required for the parameters indicated on forms 112 a through g. Instrumental detection limits must be determined and reported for each parameter as indicated on forms 109a through c.

Sample data and QC results must be submitted on enlarged copies of DDRP forms 103 through 113 as specified in Exhibit B. Copies of associated raw data and documentation of instrumental detection limits must be submitted. The complete data package must be received by both data recipients within 25 calendar days of sample receipt.

On-site evaluations will be scheduled immediately after successful completion and scoring of the pre-award performance evaluation samples. Prior to the on-site evaluation, a preliminary questionnaire will be sent. This will include a request for fully documented standard operating procedures. This questionnaire must be completed before the on-site evaluation and will be discussed at that time.

Data Recipients:

Lockheed-EMSCO Attn: DDRP QA Manager
Flamingo Executive Park, Suite 200
1050 East Flamingo Road
Las Vegas, NV 89119

U.S. Environmental Protection Agency
Contract Laboratory Program
Sample Management Office Attn: DDRP
300 North Lee Street
Alexandria, VA 22314

Appendix G

Pre-Award Performance Evaluation Scoring Sheet

Data from bidding laboratories are evaluated according to the criteria described on the scoring sheet. A successful laboratory scores at least 80 percent overall for the categories of quantification, quality assurance, and reporting and deliverables.

Direct/Delayed Response Project Soil Survey
Pre-Award Performance Evaluation Scoring Sheet

Laboratory: _____

Date: _____

Quantitation: _____

QA/QC: _____

Sample 1: _____

Deliverables: _____

Sample 2: _____

Note: Samples will be two of 1, 2, 3, or 4.

Sample 3: _____

Sample 4: _____

Total Score
(Maximum = 200 points)

Part I. Quantitation

A. Parameters

- 1) pH in 0.01 M CaCl₂ and DI H₂O: number of parameters within acceptance criteria x 10/2*.
- 2) CEC (NH₄OAc): number of parameters within acceptance criteria x 20/5*.
- 3) CEC (NH₄Cl): number of parameters within acceptance criteria x 20/5*.
- 4) Fe and Al (in oxalate, citrate-dithionite, and pyrophosphate extracts): number of parameters within acceptance criteria x 18/6*.
- 5) Lime and Aluminum Potentials (pH, K, Na, Mg, Ca, Fe, Al in 0.002 M CaCl₂): number of parameters within acceptance criteria x 21/7*.

Possible Points	Points Awarded (Samples)				Total Score
	1	2	3	4	
10					
20					
20					
18					
21					

*Number of parameters analyzed.

(continued)

Laboratory: _____

Date: _____

Part I. Quantitation (continued)

- 6) Specific surface: number of parameters within acceptance criteria x 8/1*.
- 7) Particle Size (percent sand, silt, and clay): number of parameters within acceptance criteria x 6/3*.
- 8) Exchangeable Acidity (BaCl₂-TEA and HCL): number of parameters within acceptance criteria x 8/2*.
- 9) Extractable Sulfate (DI water and PO³⁻, soluble): number of parameters within acceptance criteria x 12/2*.
- 10) Sulfate Adsorption (6 point isotherm): number of parameters within acceptance criteria 30/6*.
- 11) Total Sulphur: number of parameters within acceptance criteria x 4/1*.
- 12) Total Organic Carbon: number of parameters within acceptance criteria x 4/1*.
- 13) Inorganic Carbon: number of parameters within acceptance criteria x 4/1*.
- 14) Total Nitrogen: number of parameters within acceptance criteria x 4/1*.
- 15) Extractable Al (in KCL): number of parameters within acceptance criteria x 5/1*.

Possible Points	Points Awarded (Samples)				Total Score
	1	2	3	4	
8					
6					
8					
12					
30					
4					
4					
1					
4					
5					

*Number of parameters analyzed.

Laboratory: _____

Date: _____

Part II. Quality Assurance

A. Reagent Blank Analyses:

1. All parameters less than IDL.
2. One parameter at more than IDL.
3. Two parameters at more than IDL.
4. Three or more parameters at more than IDL.

B. Quality Control Check Sample:

1. All verifications within acceptance criteria.
2. One or more verifications outside acceptance criteria.

C. Matrix Spike Analyses:

1. All percent recoveries within acceptance criteria or analyzed by Method of Standard Additions.
2. Percent recoveries outside acceptance criteria and not corrected by Method of Standard Additions.

D. Duplicate Sample Analyses:

1. All RSD within acceptance criteria.
2. One or two parameters outside acceptance criteria.
3. Three or four parameters outside acceptance criteria.
4. Five or more parameters outside acceptance criteria.

Possible Points	Points Awarded (Samples)				Total Score
	1	2	3	4	
3					
2					
1					
0					
5					
0					
2					
0					
3					
2					
1					
0					

(continued)

Laboratory: _____

Date: _____

Part II. Quality Assurance (continued)

E. Detection Limits:

1. All instrumental detection limits within acceptance criteria.
2. One or more outside acceptance criteria.
3. Two or more outside acceptance criteria.

Possible Points	Points Awarded (Samples)				Total Score
	1	2	3	4	
4					
2					
0					

Part III. Reporting and Deliverables

Possible Points

- | | |
|--|---|
| A. Data results submitted in acceptance format on standard forms. | 4 |
| B. Quality assurance/quality control data supplied in acceptable format. | 2 |
| C. Raw data supplied. | 2 |
| D. Tabulated instrument detection limits and associated blank data supplied. | 2 |
| E. Validation of results submitted with signature of Laboratory Manager. | 2 |
-

Appendix H

Analytical Laboratory On-Site Evaluation Questionnaire

The following questionnaire is completed to provide documentation of an on-site evaluation. An analytical laboratory is evaluated prior to the award of a contract to assess the ability of the laboratory, in terms of personnel, facilities, and equipment, to analyze soil samples successfully. A second evaluation is made after sample analysis is under way. At the time of the second evaluation, adherence to protocol is evaluated, and specific problems are addressed.

Analytical Laboratory On-Site
Evaluation Questionnaire
DDRP Soil Survey

General (Page 1 of 2)

Date _____

Laboratory: _____

Street Address: _____

Mailing Address (if different from above): _____

City: _____

State: _____ Zip: _____

Laboratory Telephone Number: () _____

Laboratory Director: _____

Laboratory Quality Assurance Officer: _____
(Quality Control Chemist)

Type of Evaluation: _____

Contract Number: _____

Contract Title: _____

Title

[illegible]

Title

[illegible]

Organization and Personnel (Page 1 of 3)

Laboratory Organizational Chart

Organization and Personnel (Page 2 of 3)

Laboratory Personnel

Position	Name	Academic Training*	Special Training	Years Experience†
----------	------	--------------------	------------------	-------------------

*List highest degree obtained and specialty. Also list years toward a degree.

†List only experience directly relevant to task to be performed.

Organization and Personnel (Page 3 of 3)

Item	Yes	No	Comment
Do personnel assigned to this project have the appropriate educational background to successfully accomplish the objectives of the program?			
Do personnel assigned to this project have the appropriate level and type of experience to successfully accomplish the objectives of this program?			
Is the organization adequately staffed to meet project commitments in a timely manner?			
Does the laboratory Quality Assurance Supervisor report to senior management levels?			
Was the Project Manager available during the evaluation?			
Were chemists and technicians available during the evaluation?			
Was the Quality Assurance Supervisor available during the evaluation?			

Laboratory Manager (Page 1 of 1)

Item	Yes	No	Comment
Does the laboratory manager have his/her own copy of the standard operating procedures?			
Does the laboratory manager have his/her own copy of the instrument performance data?			
Does the laboratory manager have his/her own copy of the latest monthly QC plots?			
Is the laboratory manager aware of the most recent control limits?			
Does the laboratory manager review the following before reporting data:			
a. The data itself?			
b. The quality control data sheet with analyst notes?			
c. The general instrument performance and routine maintenance reports?			

Standard Operating Procedures (SOP) (Page 1 of 1)

Item	Yes	No	Comment
Does the laboratory have a standard operating procedure (SOP) manual?			
Is the SOP manual followed in detail?			
Does the SOP manual contain quality control practices?			
Does each analyst/technician have a copy of the SOP manual?			
Does the SOP manual deviate from the procedures required by the project?			
If the SOP manual does deviate, are the deviations documented in written form?			
Does each analyst/technician have a copy of all methods and procedures required by this project?			
Are plots of instrumental accuracy and precision available for every analysis?			
Are detection limit data tabulated for each analysis?			

Laboratory Facilities (Page 1 of 4)

When touring the facilities, give special attention to: (1) the overall appearance of organization and neatness, (2) the proper maintenance of facilities and instrumentation, (3) the general adequacy of the facilities to accomplish the required work.

Item	Yes	No	Comment
Does the laboratory appear to have adequate work-space (6 linear meters of unencumbered bench space per analyst)?			
Does the laboratory have a source of distilled/demineralized water?			
Is the specific conductance of distilled/demineralized water routinely checked and recorded?			
Are the analytical balances located away from draft and areas subject to rapid temperature changes?			
Has the balance been calibrated within one year by a certified technician?			
Is the balance checked with a class S standard before each use and recorded in a logbook? Have technician demonstrate how this is done.			
Are exhaust hoods provided to allow efficient work with volatile materials?			
Have the hoods been checked for operating efficiency? How often is this done?			
Is the laboratory maintained in a clean and organized manner?			

Laboratory Facilities (Page 2 of 4)

Item	Yes	No	Comment
Are contamination-free work areas provided for the handling of toxic materials?			
Are adequate facilities provided for separate storage of samples, extracts, and standards, including cold storage?			
Is the temperature of the cold storage units recorded daily in logbooks?			
Are chemical waste disposal policies/procedures adequate?			
Are contamination-free areas provided for trace level analytical work?			
Can the laboratory supervisor document that trace-free water is available for preparation of standards and blanks?			
Do adequate procedures exist for disposal of waste liquids for the ICP and AA spectrometers?			
Do adequate procedures exist for disposing of liquid and solid wastes?			
Is the laboratory secure?			
Are all chemicals dated on receipt and thrown away when shelf life is exceeded?			
Are all samples stored in the refrigerator between analyses?			
Are acids and bases stored in separate areas?			
Are hazardous, combustible, and toxic materials stored safely?			

Laboratory Facilities (Page 3 of 4)

Item	<u>Available</u>		<u>Comments</u> (where applicable, cite system, QC check, adequacy of space)
	Yes	No	
Gas			
Lighting			
Compressed air			
Vacuum system			
Electrical services			
Hot and cold water			
Distilled water			
Laboratory sink			
Ventilation system			
Hood space			
Cabinet space			
Storage space (m ²)			
Refrigerated storage (4°C)			

Laboratory Facilities (Page 4 of 4)

Comments on Laboratory Facilities

Equipment General (Page 1 of 2)

Item	Equipment			Condition/Age			Comments
	# of units	Make	Model	Good	Fair	Poor	
Balance, analytical							
1							
2							
3							
Balance, top loader							
Class "S" weights							
Balance table							
NBS-calibrated thermometer							
Desiccator							
Distilled water							
Double deionized, distilled/deionized, or double distilled water							
Glassware							
1 Beakers							
2 Erlenmeyer flasks							
3 Sedimentation cylinders							
4 Graduated cylinders							

Equipment General (Page 1 of 2)

Item	Equipment			Condition/Age			Comments
	# of units	Make	Model	Good	Fair	Poor	
Glassware (cont.)							
5 Fleakers							
6 Other							
Drying ovens							
Hot plates							
Water bath							
Centrifuge							
Vortex mixer							
Eppendorf pipets (or equivalent)							
Reciprocating shaker							

Comments:

Moisture Content

Item	Manufacturer	Model	Installation Date	Comments
Balance, ± 0.01 g				
Convection ovens				
Item	Available	Quantity	Type	Comments
Thermometers				
0 to 200 °C				
Weighing containers				
Desiccant				
Desiccator				

Comments: _____

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Moisture Content (Page 2 of 2)

Question	Yes	No	NA	Comments
Is the balance calibrated weekly?				
Do the thermometers have a range of -20 to 200 °C?				
Are thermometers calibrated (with barometric correction) at the boiling and freezing points at least once every 3 months?				
Is the oven temperature checked and recorded daily?				
Is the oven temperature calibrated at least monthly?				
Are organic soil samples dried at the specified temperature?				
Are replicates of each sample prepared and run?				
Are mineral soil samples dried at the specified temperature?				
Are two separately calibrated ovens used, one for organic and one for mineral soils?				
If only one oven is used, is at least 24 hours allowed for the oven to stabilize at the new temperature?				
Is sample-drying time extended as specified in the procedure?				
Are calculations correctly performed, and are at least 5% (or 2 per batch) checked by hand?				

Partical Size Analysis

Item	Manufacturer	Model	Installation Date	Comments
Hot plate or block digester				
Analytical balance, 0.1 mg				
Shaker, horizontal reciprocating (120 oscillations/min.)				
Sieve shaker, 1.25 cm vertical and lateral movement)				
Complete sieve set with receiving pan				
Automatic pipets				
Shaw pipet rack				
Motor-driven stirrer				

Partical Size Analysis (Page 2 of 5)

Item	Available	Quantity	Type	Comments
Thermometer 10 to 50 °C				
Erlenmeyer flask or Fleaker 300ml				
Pasteur-Chamberlain filter candles (fineness "F")				
1-L Sedimentation cylinders				
Insulation covering				
Hand-driven stirrer				
Shaw pipet rack equivalent				
Ringstand				
Clamp				
Volumetric pipet, 25 mL				
Evaporating dishes				
Waterproof marker or paint-pen				
Weighing bottles 90-mL wide-mouth				
Desiccator				

Partical Size Analysis (Page 3 of 5)

Chemical	Quantity	Grade	Expiration Date	Comments
Hydrogen peroxide (H ₂ O ₂) 30 to 35%				
Dessicant: Phosphorus pentoxide (P ₂ O ₅)				
Sodium carbonate (Na ₂ CO ₃)				
Sodium Hexameta phosphate (NaPO ₃) ₆				

Comments: _____

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Particle size Analysis (Page 4 of 5)

Question	Yes	No	NA	Comments
Is analysis performed on mineral horizons only?				
Is the organic matter removed as specified before proceeding?				
Are chemicals reagent grade or better?				
Is heat applied after organic matter is visibly destroyed to remove excess H ₂ O ₂ ?				
Is reciprocating shaker calibrated once every 6 months if no gauge is included (every year with gauge)?				
Is the 500 stroke per minute (1.25 cm vertical and lateral oscillator) shaker calibrated once every 6 months?				
Are pipets calibrated monthly, gravimetrically on a calibrated balance?				
Are the specified methods used for separating sand, silt, and clay?				
Is a standard sand, silt, clay "soil" used as a control?				
Is the water temperature checked during sedimentation to determine when to take a sample?				
Are the specified procedures followed during sedimentation?				
Is note made of which sedimentation table is used to determine sampling depth and time?				

Particle size Analysis (Page 5 of 5)

Question	Yes	No	NA	Comments
Are weights for each mineral fraction correctly recorded and calculated?				
Are calculations correctly performed, and are at least 5% (or 2 per batch) checked by hand?				

pH Determination

Item	Manufacturer	Model	Installation Date	Comments
Digital pH meter				
Combination electrodes, non-gel type				
Item	Available	Quantity	Type	Comments
Thermometer				
Beakers, 50 mL				
Stirrers				
QCCS standard				
Chemical	Quantity	Grade	Expiration Date	Comments
Calcium Chloride (CaCl ₂)				
Calcium hydroxide (Ca(OH) ₂)				
Chloroform (CHCl ₃) or Thymol (C ₁₀ H ₁₄ O)				
Hydrochloric acid (HCL)				
National Bureau of Standards (NBS) buffers				
Potassium Biphthalate (KHC ₈ H ₄ O ₄)				
Potassium chloride (KCl)				

pH Determination (Page 2 of 3)

Question	Yes	No	NA	Comments
Are chemicals reagent grade or better?				
Is the air-dried soil stored in sealed containers?				
Is the pH meter digital to ± 0.01 (and ± 1 mv)?				
Does the pH meter have internal temperature compensation to ± 0.5 °C?				
Is the combination electrode a non-gel type?				
Is the combination electrode of the recommended style with retractable sleeve junction?				
Are the buffers calibrated daily to ± 0.01 pH units?				
Is the pH meter:				
calibrated before samples are analyzed				
checked every batch as stated in methods				
Is the temperature compensation manual or internal?				
Are equilibrium times required for standards checked, to see if electrode response is slowing?				
Is a spare combination electrode available and properly stored?				
Is manufacturer recommended warm-up time allowed before samples are run?				

pH Determination (Page 3 of 3)

Question	Yes	No	NA	Comments
Are pH meters placed away from drafts and areas of rapid temperature change?				
Are the specified between-sample procedures followed?				
Are pH units equipped with programmable sampling times?				
If yes above, are they used in this analysis?				
Are electrodes properly stored and maintained?				
Are the QC results plotted in real time?				
What is the QCCS sample?				
Is the QCCS solution analyzed first and thereafter as called for in the methods?				
Are a QCCS and duplicate sample included in each run?				
Is the quality control data reviewed by the analyst before deciding whether to release the data for reporting?				

Total Carbon and Total Nitrogen

Item	Manufacturer	Model/Grade	Installation Date	Comments
CHN analyzer				
Mill - Hammer, ball or other				
Thermal detector				
Recording system				
60-mesh sieve				
Balance (.1mg)				
Convection oven				
Desiccator				
Heat resistant vials				
Item	Available	Quantity	Type	Comments
Natural gas				
Helium gas (He) 99.995+%				
Oxygen gas (O ₂) 99.99+%				
Air source (pressurized)				
Acetanilide NBS standard				

Total Carbon and Total Nitrogen (Page 2 of 4)

Item	Available	Quantity	Type	Comments
Alumina wool (blank)				
Forceps				
Bunsen burner				
Tamping device				

Comments: _____

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Total Carbon and Total Nitrogen (Page 3 of 4)

Question	Yes	No	NA	Comments
Are compressed gases of required purity?				
Is the pressure in each gas cylinder checked before each use?				
Is the gas flow periodically checked and recorded during operation?				
Are extra cylinders of gas available in the laboratory?				
Is one- or two-day delivery of compressed gas available?				
Is distributor willing/able to replace contaminated gas IMMEDIATELY?				
Has contaminated gas ever been delivered by this distributor previously?				
Are balances away from drafts and areas of sudden temperature changes?				
Is the hammermill properly maintained?				
Is the acetanilide of NBS origin?				
Is alumina wool of sufficient purity?				
Is alumina wool pretreated according to the procedures?				
Are specified procedures followed while working with the alumina wool?				
Is the thermal conductor properly calibrated with the acetanilide standard, an alumina wool blank, and one or more in-house soil standards?				
Do the in-house standards meet the specifications of the procedure for 10% relative standard deviation?				

Total Carbon and Total Nitrogen (Page 4 of 4)

Question	Yes	No	NA	Comments
Is the instrument recalibrated whenever the system is opened?				
Is the instrument recalibrated whenever traps, scrubbers, or combustion or reduction tubes are changed the oxygen or helium is changed, or gas system otherwise modified?				
Are vials properly handled during a run?				
Are most components left on to prevent warm-up problems?				
If not, is the manufacturer specified warm-up time allowed before samples are run?				
Are at least 5% (2 per batch) of the calculations check manually?				
Are calculations performed correctly?				

Inorganic Carbon

Item	Manufacturer	Model	Installation Date	Comments
Coulometer				
Mineral carbon apparatus				
Item	Available	Quantity	Type	Comments
Acid dispenser automatic repipet adjustable to 2 mL				
Weighing boats mineral carbon free				
Heating unit				
Coulometer accessories, manufacturers recommended				
Chemical	Quantity	Grade	Expiration Date	Comments
Sulfuric acid (H ₂ SO ₄)				
Calcium carbonate (CaCO ₃)				

Inorganic Carbon (Page 2 of 4)

Chemical	Quantity	Grade	Expiration Date	Comments
Hydrochloric acid (HCl)				
Potassium hydroxide (KOH)				
Silver sulfate (Ag ₂ SO ₄)				
Hydrogen peroxide (H ₂ O ₂ -30%)				
Potassium iodide (KI)				
Stannous chloride (SnCl ₂)				
Ferrous sulfate (FeSO ₄)				
Anti-foam agents				

Comments:

Inorganic Carbon (Page 3 of 4)

Question	Yes	No	NA	Comments
Is the inorganic carbon (IC) test run only if a positive test for carbonates is found?				
Is the IC test run on both the soil (<2mm) and rock fragment (2 to 20mm) fractions?				
Are duplicates of each sample used?				
Is sample weight based on the expected carbonate content?				
Is the amount of soil used equivalent to 1 to 3 mg of mineral carbon?				
Are samples weighed into a weighing boat before being placed into the sample tube?				
If the sample is placed directly into the sample tube, is the tube first cleared of residual acid?				
Are all accessory tubes and materials inspected daily?				
Is the acid dispenser calibrated daily so that approximately 2 mL of acid are delivered?				
Are standards containing a known weight of carbonate (QCCS) used with each run?				
Is the system checked daily for leaks?				
Is the temperature of the heating unit checked daily?				

Inorganic Carbon (Page 4 of 4)

Question	Yes	No	NA	Comments
Is the temperature low enough so the scrubber is not overloaded?				
Is the sample allowed to purge until the coulometer gives a relatively steady reading?				
Are the times required for the reaction recorded for blanks, samples, and standards?				
Are calculations performed correctly, and are at least 5% (2 per batch) checked by hand?				

Total Sulfur

Item	Manufacturer	Model	Installation Date	Comments
SO ₂ Analyzer				
Detector				
Temperature regulator				
Recorder				
Analytical balance				
Item	Available	Quantity	Type	Comments
Crucibles				
Gas trap				
Dust trap				
Moisture trap				
Catalytic oxidants				
Forceps				
standards				
oxygen				
Chemical	Quantity	Grade	Expiration Date	Comments
Chemicals as called for in manufacturer's method				

Comments: _____

Total Sulfur (Page 2 of 4)

Question	Yes	No	NA	Comments
Is the detector checked and calibrated at least weekly?				
Is the temperature regulator on the detector unit stable to ± 0.5 °C?				
Is the instrument checked at least weekly for both correct temperature and stability?				
Are the crucibles used able to withstand heat repeatedly?				
Are crucibles handled according to manufacturer's specifications in order to avoid contamination?				
Do the crucibles produce low blank values (based on manufacturer's ratings)?				
Are all traps checked before each run to see if they are working properly?				
Are the traps changed on a scheduled basis and more frequently if needed?				
Is the SO ₂ analyzer away from drafts and areas of rapid temperature changes?				
Is the purity of the oxygen gas equal or greater than specified by the manufacturer?				
Will the distributor replace contaminated gas or deliver new gas within one day?				
Is an extra cylinder of oxygen available?				
Is the oxygen pressure monitored periodically during a run?				

Total Sulfur (Page 3 of 4)

Question	Yes	No	NA	Comments
If used, are catalytic oxidizers of sufficient purity?				
Are standards NBS-traceable?				
Are standards used before, during, and after each run?				
Is the balance away from drafts and areas of rapid temperature change?				
Are soil samples adequately ground?				
Is the required amount of soil used for the expected values of sulfur?				
Have manufacturer's recommendations or other procedural modifications been approved by the QA manager or designee?				
If the titration method of detection is used:				
-Is the buret checked for accuracy gravimetrically at least monthly?				
-Are reagents of a quality equal to or exceeding manufacturer's specifications?				
-Are sufficient quantities of chemicals available?				
-Are manufacturer's specified or recommended standards used before, during, and after a run to assure accuracy?				

Total Sulfur (Page 4 of 4)

Question	Yes	No	NA	Comments
Is a QC sample run with each batch?				
Is the titrator restandardized when any changes are made in the system, or when irreproducible results occur?				
Are detection limits tested before and after each run?				
Is the detection limit determined according to protocol?				

Cation Exchange Capacity

Item	Manufacturer	Model	Installation Date	Comments
Mechanical extractor				
Flow injection analyzer				
Titration apparatus				
Reciprocating shaker				
Item	Available	Quantity	Type	Comments
Steam distillation unit				
Digestion tubes 250mL				
Kjeldahl flasks 800mL				
Analytical filter pulp				
Disposable syringes 60 mL				
Rubber tubing connectors				
Linear polyethylene bottles, 25 mL				

Cation Exchange Capacity (Page 2 of 5)

Chemical	Quantity	Grade	Expiration Date	Comments
Glacial acid ($\text{HC}_2\text{H}_3\text{O}_2$)				
Ammonium hydroxide (NH_4OH)				
Ammonium acetate (NH_4OAC)				
Ammonium chloride (NH_4Cl)				
Ethanol ($\text{CH}_3\text{CH}_2\text{O}$), 95%				
Nessler's reagent				
Potassium iodide (KI)				
Mercuric iodide (HgI_2)				
Sodium hydroxide (NaOH)				
Sodium chloride (NaCl)				
Antifoam				
Hydrochloric acid (HCl)				

Cation Exchange Capacity (Page 3 of 5)

Chemical	Quantity	Grade	Expiration Date	Comments
Sodium carbonate (Na_2CO_3)				
Methyl orange indicator				
Boric acid				
Zinc, granular				
Phenol ($\text{C}_6\text{H}_6\text{O}$)				
Potassium sodium tartrate ($\text{KNaC}_4\text{H}_4\text{O}_6$) • $4\text{H}_2\text{O}$				
Sodium citrat ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ • $2\text{H}_2\text{O}$)				
Sodium nitroferricya- nide ($\text{Na}_3\text{Fe}(\text{CN})_5 \text{NO}_3$ • $2\text{H}_2\text{O}$)				
Sodium hypochlorite (NaOCl)				

Comments: _____

Cation Exchange Capacity (Page 4 of 5)

Question	Yes	No	NA	Comments
Are the chemicals reagent grade or better?				
Are dilute standards prepared and calibrated daily?				
Are working standards prepared and calibrated at least weekly?				
Are reagents stored properly to prevent premature decomposition?				
Are hazardous chemicals used strictly under the hood?				
Is an antifoam agent available for use?				
Is all glassware cleaned and stored as specified?				
Does the flow injection analyzer (FIA) have the correct interference filter?				
Are the pump lines inspected for wear before each run?				
Is the heat bath of the FIA calibrated monthly and checked before each run?				
Are the pump tubes all of the correct type for the reagents and method in use?				
Are all peripherals such as printer, plotter, and disk drives functional and, in the case of recorders and plotters, calibrated before each run?				
Is the shaker used for organic samples calibrated every six months or less along with general maintenance?				

Cation Exchange Capacity (Page 5 of 5)

Question	Yes	No	NA	Comments
Is the auto analyzer (distillation/titration) calibrated for titration before each run?				
Are the condensation facilities of the distillation apparatus inspected before each run?				
Are all calculations performed correctly, and are at least 5% being checked by hand?				
Is the mechanical extractor calibrated for extraction time?				
Is the calibration of the mechanical extractor checked at least monthly?				
Are the specified size, type, and grade of disposable syringes used with the extractor?				
Is the tubing checked frequently and replaced when needed?				
Is the filter pulp washed before the extraction is performed?				
Are all procedures involving the extraction followed precisely according to the statement of work?				
Are three blanks carried through to record mean and standard deviation?				

Exchangeable Basic Cations

Item	Manufacturer	Model/Grade	Installation Date	Comments
Flame atomic absorption spectrometer				
Inductively coupled plasma emission spectrometer				
Chemical	Quantity	Grade	Expiration Date	Comments
Argon				
Acetylene gas (C ₂ H ₄)				
Natural gas (CH ₄)				
Hydrochloric acid (HCl)				
Nitric acid (HNO ₃)				
Calcium carbonate (CaCO ₃)				
Magnesium oxide (MgO)				
Potassium chloride (KCl)				
Sodium chloride (NaCl)				
Lanthanum oxide (La ₂ O ₃)				

Exchangeable Basic Cations (Page 2 of 3)

Chemical	Quantity	Grade	Expiration Date	Comments
Cesium chloride (CsCl)				
Lithium chloride (LiCl)				
Lithium nitrate (LiNO ₃)				
QCCS				
Calcium (Ca ²⁺)				
Magnesium (Mg ²⁺)				
Potassium (K ⁺)				
Sodium (Na ⁺)				

Comments: _____

Extractable Bases (Page 3 of 3)

Question	Yes	No	NA	Comments
Is the analytical instrument cleaned and adjusted before and after each run?				
Is the power source secure, that is, protected against line fluctuation?				
Are standards made in a matrix as close as possible to that of the extract?				
If the lanthanum oxide method is used, is the La_2O_3 added to the samples and standards?				
Are the pHs of the samples and standards approximately identical?				
Are all chemicals of analytical reagent grade or better?				
Are chemicals used for standards traceable to NBS standards?				
Does the laboratory have copies of Methods for Chemical Analysis of Water and Wastes, and Standard Methods 14th edition or access to them?				
Are all calculations performed correctly, and are at least 5% (2 per batch) checked manually?				
Fill out pertinent section in back of appendix:				
Flame atomic absorption spectrometer				
Inductively coupled plasma spectrometer				
Flame photometer				

Lime and Aluminum Potential

Item	Manufacturer	Model/Grade	Installation Date	Comments
Flame atomic absorption spectrometer				
Inductively coupled plasma emission spectrometer				
Flame atomic emission spectrometer				
Mechanical extractor				
Item	Available	Quantity	Type	Comments
Reciprocating shaker				
Disposable syringes, 60 mL				
Rubber tubing connectors				
Analytical filter pulp				
Linear poly- ethylene bottle (25 and 50 mL)				

Lime and Aluminum Potential (Page 2 of 4)

Chemical	Quantity	Grade	Expiration Date	Comments
Calcium chloride (CaCl ₂)				
Hydrochloric acid (HCl)				
NBS traceable standards:				
Calcium (Ca ²⁺)				
Magnesium (Mg ²⁺)				
Potassium (K ⁺)				
Sodium (Na ⁺)				
Iron (Fe ³⁺)				
Aluminum (Al ³⁺)				

Comments:

Lime and Aluminum Potential (Page 3 of 4)

Question	Yes	No	NA	Comments
Is the reciprocating shaker calibrated every six months or less in addition to general maintenance?				
What is the QC source?				
Is the QC solution analyzed first and as called for in the methods?				
Are the QC results plotted in real time?				
Is the quality control data reviewed by the analyst before deciding whether to release the data for reporting?				
Are the results for Ca reported after adjusting for the CaCl_2 extraction solution?				
Are results reported based on oven-dry soil weight?				
Are all calculations correctly performed, and are 5% (2 per batch) checked manually?				
Fill out the pertinent section(s) in back of appendix:				
Flame atomic absorption				
Inductively coupled plasma				
Flame photometry				

Lime and Aluminum Potential (Page 4 of 4)

Question	Yes	No	NA	Comments
Is the mechanical extractor calibrated for extraction time?				
Is the calibration of the mechanical extractor checked at least monthly?				
Are the specified size, type, and grade of disposable syringes used with the extractor?				
Is the tubing checked frequently and replaced when needed?				
Is the filter pulp washed before the extraction is performed?				
Are all procedures involving the extraction followed precisely according to the statement of work?				
Are three blanks carried through to record mean and standard deviation?				

Extractable Iron and Aluminum

Item	Manufacturer	Model/Grade	Installation Date	Comments
Flame atomic absorption spectrometer				
Inductively coupled plasma emission spectrometer				
Centrifuge				
Mechanical extractor				
Item	Available	Quantity	Type	Comments
Reciprocating shaker				
Repipet				
Automatic pipet				
Buret				
60 mL polypropylene syringes				
Filter pulp				
250 mL polypropylene centrifuge bottles				
Fleakers				
Volumetric pipet				
Volumetric flasks				

Extractable Iron and Aluminum (Continued)

Chemicals	Quantity	Grade	Expiration Date	Comments
Sodium pyro-phosphate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$)				
Sodium hydroxide (NaOH)				
pH buffers, pH = 7 and 10				
Phosphoric acid (H_3PO_4)				
Superfloc 16				
Sodium Dithionite ($\text{Na}_2\text{S}_2\text{O}_4$)				
Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot x\text{H}_2\text{O}$)				
Ammonium oxalate ($\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$				
Oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$)				
pH buffers, pH = 4 and 2				
Nitric acid (HNO_3)				

Comments: _____

Extractable Iron and Aluminum (Page 3 of 5)

Question	Yes	No	NA	Comments
Sodium Pyrophosphate and Citrate-Dithionite Method				
Are the proper type of polypropylene 250 mL centrifuge tubes used?				
Is the reciprocating shaker calibrated yearly if it possesses a speed gauge, every 6 months if not?				
Is the centrifuge calibrated yearly if it possesses a speed gauge, every 6 months if not?				
Are standards made up in the same expected matrix as are the extracts?				
Are the chemicals reagent grade or better?				
Is the extract promptly stored at 4 °C?				
Is analysis performed for Fe and Al within 24 hours of extraction?				
Are the calculations carried out correctly and are at least 5% (2 per batch) checked by hand?				
Fill out pertinent section in back of appendix:				
Flame atomic absorption				
Inductively coupled plasma				

Extractable Iron and Aluminum (Page 4 of 5)

Question	Yes	No	NA	Comments
<u>Acid-Oxalate Extraction</u>				
Is the shaker used for organic samples calibrated every six months or less along with general maintenance?				
Is an antifoam agent available for use?				
Are component reagents properly mixed to provide the final reagent?				
Is the correct filter pulp used?				
Is the extractor covered for the overnight extraction?				
Is the extract promptly stored at 4 °C?				
Is analysis performed for Fe and Al within 48 hours of extraction?				
Are the calculations carried out correctly and are at least 5% (2 per batch) checked by hand?				
Fill out pertinent section in back of appendix:				
Flame atomic absorption				
Inductively coupled plasma				

Extractable Iron and Aluminum (Page 5 of 5)

Question	Yes	No	NA	Comments
Is the mechanical extractor calibrated for extraction time?				
Is the calibration of the mechanical extractor checked at least monthly?				
Are the specified size, type, and grade of disposable syringes used with the extractor?				
Is the tubing checked frequently and replaced when needed?				
Are all procedures involving the extraction followed precisely according to the statement of work?				
Are three blanks carried through to record mean and standard deviation?				

Extractable Sulfate and Nitrate

Item	Manufacturer	Model	Installation Date	Comments
Balance, ± 0.01 g				
Ion chromatograph				
Automated injection system				
Filtration apparatus				
Centrifuge				
Vortex mixer				
Reciprocating shaker				
Item	Available	Quantity	Type	Comments
100-mL centrifuge tubes with screw caps				
Volumetric flasks				
0.20 μ m pore size membrane filters				
Volumetric pipets				

Extractable Sulfate and Nitrate (Page 2 of 4)

Chemicals	Quantity	Grade	Expiration Date	Comments
Monobasic sodium phosphate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$)				
Sodium carbonate (Na_2CO_3)				
Sodium hydroxide (NaOH)				
Sulfuric acid (H_2SO_4)				
Magnesium sulfate (MgSO_4)				
Sodium nitrate (NaNO_3)				

Extractable Sulfate and Nitrate (Page 3 of 4)

Question	Yes	No	NA	Comments
Is the ion chromatograph maintained according to manufacturer's specifications?				
Are manufacturer recommendations for optimum IC sensitivity used?				
Are chemicals reagent grade or better?				
Are the phosphate and sulfate concentrations low enough so they elute separately? Are dilutions made if not?				
Are all the proper accessories maintained on the IC?				
-anion separation column?				
-micro-membrane suppressor (anion separation) column?				
Is the optional automatic injection system used?				
Are all solutions made fresh when needed?				
-0.40M NaCO ₃				
-0.0020M Na ₂ CO ₃ /0.002M NaOH				
-other				
-stock resolution standard				
-working resolution standard				
-sulfate and nitrate calibration				

Extractable Sulfate and Nitrate (Page 4 of 4)

Question	Yes	No	NA	Comments
Are conversion operations performed correctly for:				
-meq/L to mg/L SO ₄ ? (1 meq = 48.0288 mg)				
-mg/L to meq/L SO ₄ ? (1 meq = 0.02082 meq)				
-SO ₄ to S? (1 mg SO ₄ = .3338 mg S)				
-S to SO ₄ ? (1 mg S = 2.9962 mg SO ₄)				
Are recording instruments calibrated before each use?				
Is a pump stroke noise supressor or pressure gauge used to stabilize pressure?				
Is resolution high enough so no startover is required?				
Are peak heights/areas recorded in a logbook?				
If peaks are not sharp and symmetric, is an approved method for peak area determination used?				
Is peak area determined by microprocessor? If yes, which methods and formulas does it use?				
Are calibration curves constructed according to manufacturer's recommendations?				
Is the flow rate checked gravimetrically with time for consistency of flow?				
Is the rate of flow calibrated before each batch is run?				

Sulfate Adsorption Isotherms

Item	Manufacturer	Model	Installation Date	Comments
Balance, ± 0.01 g				
Ion chromatograph				
Filtration apparatus				
Centrifuge				
Reciprocating shaker				

Item	Available	Quantity	Type	Comments
Centrifuge tubes with screw caps, 100 or 50mL				
0.20 μ m pore size membrane filters				
Volumetric pipets 50mL				

Chemical	Quantity	Grade	Expiration Date	Comments
Magnesium sulfate (MgSO_4)				

Comments: _____

Sulfate Adsorption Isotherms (Page 2 of 2)

Question	Yes	No	NA	Comments
Are $MgSO_4$ adsorption solutions correctly prepared?				
Are the adsorption solutions calibrated for accuracy before being used in the run?				
Are the working standards made fresh daily?				
Is the deionized water sent through the 0.20 μ m membrane filter?				
Is the correct amount of soil (oven-dried weight) used?				
Are methods of analysis by ion chromatography the same as used in extractable sulfate procedure?				
Are the correct conversion factors used as in the extractable sulfate procedure?				
Are all calculations performed correctly, and are at least 5% (2 per batch) checked?				

Exchangeable Acidity

Item	Manufacturer	Model	Installation Date	Comments
Mechanical extractor				
pH meter				
Automatic titrator				
pH electrode				
Reciprocating shaker				
Item	Available	Quantity	Type	Comments
Pipettors, adjustable to 25 mL				
Eppendorf pipets, 5 mL and 5 L				
Titration (Erlen- meyer) 250 and 125 mL				
Linear polyethylene bottles 25 mL				
Volumetric flasks				
Drying tube				
Diluter				
Tubes, 25 mL glass				
Stirring rods				
Syringes 60 mL				

Exchangeable Acidity (Page 2 of 5)

Chemicals	Quantity	Grade	Expiration Date	Comments
Ascarite				
Barium chloride (BaCl ₂ •2H ₂ O)				
Triethanolamine (N(CH ₂ CH ₂ OH) ₃)				
Sodium hydrox- ide (NaOH)				
Sulfuric acid (H ₂ SO ₄)				
Hydrochloric acid (HCl)				
Potassium chloride (KCl)				
Methyl orange indicator				
Nitric acid (HNO ₃)				
Phenolphthalein				
NBS-traceable buffers, pH=4, 7, and 10				
Primary alumi- num standard				

Comments: _____

Exchangeable Acidity (Page 3 of 5)

Question	Yes	No	NA	Comments
<u>BaCl₂ - TEA Extraction</u>				
Is the buffer solution protected from CO ₂ ?				
Are the syringes prepared according to protocol?				
Is the pH calibration the same as for the pH procedure (comment on any exceptions)?				
Is the automatic titrator calibrated gravimetrically before each batch?				
Is the pH endpoint of the automatic titrator calibrated to 4.60?				
Are at least 5% of the calculations checked manually?				
Are calculations performed correctly?				
<u>KCl Extraction</u>				
Are the prepared solutions CO ₂ free?				
Are the solutions protected against atmospheric CO ₂ ?				
Are syringes prepared according to protocol?				
Is the titrator calibrated gravimetrically before each batch?				
Are all samples titrated to the same color (or pH = 8.4) endpoint?				
Is aliquot for aluminum determination acidified immediately?				
Is aluminum determined by ICP?				

Exchangeable Acidity (Page 4 of 5)

Question	Yes	No	NA	Comments
Are all aspects of aluminum determination correct according to Lime and Aluminum Potential procedure?				
<u>Common</u>				
Is the reciprocating shaker calibrated every six months or less in addition to general maintenance?				
Is the auto analyzer (distillation/titration) calibrated for titration before each run?				
Are titration results calculated, and are 5% hand checked?				
Is the 25-mL pipetter calibrated gravimetrically daily (if the adjustable type) and at least weekly if a fixed volume?				
Is the dilutor calibrated and checked gravimetrically before each run?				
Is the same amount of filter pulp used with each sample?				
Is the filter pulp washed before use?				
Is the specified number of blanks run for each batch?				

Exchangeable Acidity (Page 5 of 5)

Question	Yes	No	NA	Comments
<u>Common (Continued)</u>				
Are chemicals of reagent grade or better?				
Fill out pertinent section in back or appendix:				
Is the mechanical extractor calibrated for extraction time?				
Is the calibration of the mechanical extractor checked at least monthly?				
Are the specified size, type, and grade of dispos- able syringes used with the extractor?				
Is the tubing checked frequently and replaced when needed?				
Are all procedures involving the extraction followed precisely according to the statement of work?				
Are three blanks carried through to record mean and standard deviation?				

Specific Surface

Item	Manufacturer	Model	Installation Date	Comments
Analytical balance ±0.1 mg				
Vacuum desiccator				
Item	Available	Quantity	Type	Comments
Vacuum pump				
Drying tube for EGME trap				
Syringe, 1 mL				
Chemical	Quantity	Grade	Expiration Date	Comments
Calcium chloride (CaCl ₂) anhydrous				
Ethylene glycol mono- ethyl ether (EGME) reagent grade				
Phosphorus pentoxide (P ₂ O ₅)				
Item	Manufacturer	Type	Grade	Comments
Quality control cali- bration samples				

Specific Surface (Page 2 of 2)

Question	Yes	No	NA	Comments
Is specific surface determined only on mineral soils?				
Is the balance calibrated at least weekly?				
Is the balance located away from areas of sudden temperature changes and drafts?				
Is the soil sufficiently dried by vacuum over P ₂ O ₅ ?				
Is sufficient EGME used to cover and coat all surfaces of the soil samples?				
Is the standard surface area material suitable for the EGME method?				
Are weighings performed daily until three successive daily weights are within 1 mg EGME/gram soil?				
Are calculations performed correctly, and are at least 5% (2 per batch) checked manually?				

Flame Atomic Absorption Spectroscopy

Question	Yes	No	NA	Comments
For which methods is this instrument used?				
Is the burner head cleaned and adjusted for each run?				
Is the burner head cleaned frequently when solutions of high ionic strength are analyzed?				
Is DI water or cleaning solution aspirated both before and after a run?				
Is the nebulizer cleaned at least weekly?				
Is the correct flame type used for determination of each element?				
Is the acetylene of specified purity?				
Is gas pressure monitored during a run?				
Are filters used to remove water and oil from the compressed air?				
Is constant air pressure maintained? How?				
Is the wavelength optimized before a run?				
Is the slit width correctly set for the desired element?				
Is the optical system aligned at least every 6 months? With a major realignment every 12 months?				

Flame Atomic Absorption Spectroscopy (Continued)

Question	Yes	No	NA	Comments
Are the lamp and instrument allowed adequate time to warm up before use?				
-Lamp time (30-60+ minutes)?				
-Instrument time (constant if possible)?				
-Flame time (5+ minutes)				
Is the unit adequately vented?				
Is tubing inspected before each run?				

Inductively Coupled Plasma Emission Spectroscopy

Question	Yes	No	NA	Comments
For which methods is this instrument used?				
Is the tubing inspected before each run?				
Are the electrodes replaced as instructed by the manufacturer or more frequently?				
Is the instrument adequately vented?				
Is the instrument in a temperature controlled room?				
Is ample time allowed for the instrument to warm up?				
Are standards calibrated both alone and as part of a multi-element matrix?				
Is the UV-IR shielding in place?				
Is an adequate supply of the carrier gas present?				
Are manufacturer operating procedures followed?				
On multi-element units, are alternate wavelengths used when necessary to avoid interference?				

Flame Photometry (Flame Atomic Emission)

Question	Yes	No	NA	Comments
For which methods is this instrument used?				
Are the correct filters used for each element?				
Is the pressure of the gases monitored during a run?				
Is the oxygen supply of 99.95% purity or higher?				
Is the fuel supply of sufficient purity and of constant pressure?				
Is the aspirator cleaned before and after each run?				
Is a rinse solution of DI water (or wash solution) used between samples to prevent salting-up of the aspirator?				
Is the unit given adequate time to warm up before use?				
Is the unit calibrated before use?				
Is the aspirator/nebulizer unit inspected daily for proper seating and function?				
Is the unit placed away from areas of drafts and sudden temperature changes?				

Documentation/Tracking (Page 1 of 1)

Item	Yes	No	Comments
Is a sample custodian designated? If yes, name of sample custodian _____.			
Are the sample custodian's procedures and responsibilities documented? If yes, where are these documented?			
Is sample tracking performed via paper or computer?			
Are written standard operating procedures (SOPs) developed for receipt of samples? If yes, where are they documented?			
Are written standard operating procedures (SOPs) developed for compiling and maintaining sample document files? If yes, where are they documented?			
Are samples stored under refrigeration? At what temperature?			
After completion of the analysis are the samples properly stored for six months or until laboratory personnel are told otherwise?			
Are magnetic tapes stored in a secure area?			

Analytical Methodology (Page 1 of 2)

Item	Yes	No	Comments
Are the required methods used?			
Is there any unauthorized deviation from contract methodology?			
Are written analytical procedures provided to the analyst?			
Are reagent grade or higher purity chemicals used to prepare standards?			
Are fresh analytical standards prepared at a frequency consistent with good QA?			
Are reference materials properly labeled with concentrations, date of preparations, and the identity of the person preparing the sample?			
Is a standard preparation and tracking logbook maintained?			
Do the analysts record bench data in a neat and accurate manner?			
Is the appropriate instrumentation used in accordance with the required protocol(s)?			

Comments on Analytical Methods and Practices

Quality Control (Page 1 of 3)

Item	Yes	No	Comments
Does the laboratory maintain a quality control manual?			
Does the manual address the important elements of a QC program, including the following:			
a. Personnel?			
b. Facilities and equipment?			
c. Operation of instruments?			
d. Documentation of procedures?			
e. Procurement and inventory practices?			
f. Preventive maintenance?			
g. Reliability of data?			
h. Data validation?			
i. Feedback and corrective action?			
j. Instrument calibration?			
k. Record keeping?			
l. Internal audits?			

Quality Control (Page 2 of 3)

Item	Yes	No	Comments
Are QC responsibilities and reporting relationships clearly defined?			
Have standard curves been adequately documented?			
Are laboratory standards traceable?			
Are quality control charts maintained for each routine analysis?			
Do QC records show corrective action when analytical results fail to meet QC criteria?			
Do supervisory personnel review the data and QC results?			
Does the QC chemist have a copy of the standard operating procedures?			
Does the QC chemist have a copy of the instrument performance data?			
Does the chemist have a copy of the latest QC plots?			
Is the QC chemist aware of the most recent control limits?			
Does the QC chemist prepare a blind audit sample once per week?			
Does the QC chemist routinely review and report blank audit data to the laboratory manager?			

Quality Control (Page 3 of 3)

Item	Yes	No	Comments
Does the QC chemist update control limits and obtain new control charts once per batch?			
Are all QC data (e.g., control charts, regression charts, QC data bases) up to date and accessible?			
Are minimum detection limits calculated as specified?			
Is QC data sheet information reported to the analyst?			

Data Handling (Page 1 of 2)

Item	Yes	No	Comments
Does data clerk check all input to the computer for accuracy?			
Are calculations checked by another person?			
Are calculations documented?			
Does strip chart reduction by on-line electronic digitization receive at least 5% manual spot checking?			
Are data from manually interpreted strip charts spot-checked after initial entry?			
Do the laboratory records include the following information:			
Sample identification number			
Sample type			
Date sample received in laboratory			
Date of analysis			
Analyst			
Result of analysis (including raw analytical data)			
Recipient of the analytical data			

Data Handling (Page 2 of 2)

Item	Yes	No	Comments
Does the laboratory follow required sample tracking procedures from sample receipt to discard?			
Does the data clerk routinely report quality control data sheet information to the analyst?			
Does the data clerk submit quality control data sheet information to the laboratory manager, along with the analytical data to be reported?			
Do records indicate corrective action taken?			
Are provisions made for data storage for all raw data, calculations, quality control data, and reports?			
Are all data and records retained for the required amount of time?			
Are computer printouts and reports routinely spot-checked against laboratory records before data are released?			

Summary (Page 1 of 2)

Item	Yes	No	Comments
Do responses to the evaluation indicate that project and supervisory personnel are aware of QA and its application to the project?			
Do project and supervisory personnel place positive emphasis on QA/QC?			
Have responses with respect to QA/QC aspects of the project been open and direct?			
Has a cooperative attitude been displayed by all project and supervisory personnel?			
Does the organization place the proper emphasis on quality assurance?			
Have any QA/QC deficiencies been discussed before leaving?			
Is the overall quality assurance adequate to accomplish the objectives of the project?			
Have corrective actions recommended during previous evaluations been implemented?			
Are any corrective actions required? If so, list the necessary actions below.			

Summary Comments and Corrective Actions

Appendix I

Facsimile of the Data Package Completeness Checklist

The Data Package Completeness Checklist was developed to serve three related functions:

- To give the contractor analytical laboratory a concise listing of what is required in the data package.
- To give the data recipients a check-off listing to inventory the contents of the data package.
- To serve as an index to the handwritten data file.

Two references are made to the invitation for bid (IFB). The corresponding references in this document are indicated below:

- (1) "Ex. C, pg. C-2" is Table 9-3, Required Minimum Detection Limits, Expected Ranges, and Intralaboratory Relative Precision Goal.
- (2) "Ex. E, Table 1" is Table 9-4, Summary of Internal Quality Control.

(continued)

- [illegible]

Note: Checklist must be included in the data package.

Appendix J

Forms for Reporting Mineralogical Laboratory Data

The following forms are used for recording data from the mineralogical procedures.

|||

[illegible]

Direct/Delayed Response Project (DDRP) Soil Survey Form 401

Data from Oriented Pipet Mounts

Analytical Lab ID: _____ Batch No.: _____

Analyst: _____ Date Received: _____

Date Completed: _____

Lab Manager's Signature: _____

Treatment: (circle one)

Mg - sat. A D GLY

K - sat. A D 110°C 350°C 550°C

°20	d(Å)	I/Ii	Mineral Name	JCPDS Card Number	hkl	Response to treatment

Weight from Section 17.10.5 _____g freeze-dried <0.002-mm material.

Direct/Delayed Response Project (DDRP) Soil Survey Form 402a

Chemical Composition of Materials by Wavelength-dispersive XRF

Analytical Lab ID: _____ Batch No.: _____

Analyst: _____ Date Received: _____

Date Analyzed: _____

Lab Manager's Signature: _____

Sample Number: _____

Elements Major	Oxide	Concentration		2σ error	I/I(b)	Detection Limit
		Elemental, wt%	Oxide, wt%			
Sodium	Na ₂ O					
Potassium	K ₂ O					
Rubidium	Rb ₂ O					
Magnesium	MgO					
Calcium	CaO					
Strontium	SrO					
Aluminum	Al ₂ O ₃					
Silicon	SiO ₂					
Phosphorus	P ₂ O ₅					
Iron*	Fe ₂ O ₃					
Manganese	MnO ₂					
Titanium	TiO ₂					
Total		NA		NA	NA	NA

* The iron value represents both the +2 and +3 states of iron.

Comments:

Direct/Delayed Response Project (DDRP) Soil Survey Form 402b

Chemical Composition of Materials by Wavelength-Dispersive XRF

Analytical Lab ID: _____ Batch No.: _____

Analyst: _____ Date Received: _____

Date Analyzed: _____

Lab Manager's Signature: _____

Sample Number: _____

Minor and Trace Elements		Concentration		2 σ error	I/I(b)	Detection Limit
		Elemental, wt% or ppm	Oxide, wt% or ppm			
Sulfur	S					
Chloride	Cl					
Barium	Ba					
Lead	Pb					
Nickel	Ni					
Copper	Cu					
Cobalt	Co					
Chromium	Cr					
Zinc	Zn					
Uranium	U					
Thorium	Th					
Zirconium	Zr					
Niobium	Nb					
Cerium	Ce					

Direct/Delayed Response Project (DDRP) Soil Survey Form 403
Pertinent Geometry and Instrument Settings Specific to the System

Analytical Lab ID: _____

SEM Machine Name: _____

Operator: _____ EDXRF Machine Name: _____

Lab Manager's Signature: _____

1. X-ray detector to specimen fixed angle and azimuth _____
2. X-ray detector to specimen distance _____
3. X-ray detector active area _____
4. X-ray detector window _____
5. Specimen tilt angle and tilt azimuth _____
6. Specimen to SEM pole piece working distance (adjusted on the electron beam axis to the main constant for every spectral collection).

7. SEM operating voltage: _____
8. SEM beam current ($\pm 10\%$): _____
9. SEM spot size: _____
10. SEM scan rate (preferred as fast as possible): _____
11. Specimen area fluoresced: _____; volume excited: _____
12. Magnification: _____; full frame or partial field: _____
13. Spectral acquisition time (dead-time corrected): _____
14. Spectrometer pulse shaping time constant: _____
electron volts/channel: _____
15. Average absorbed current: _____
16. Average input count rate: _____

Direct/Delayed Response Project (DDRP) Soil Survey Form 404
Comments on Observations, Photographs, and Areas of Analysis

Analytical Lab ID: _____ Batch No: _____

Analyst: _____ **Date Received:** _____

Date Completed _____

Lab Manager's Signature:_____

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins, text, or other markings on the paper.

Direct/Delayed Response Project (DDRP) Soil Survey Form 405
SEM Photograph and Chemical Composition of Minerals

Analytical Lab ID: _____ Batch No.: _____

Analyst: _____ Date Received: _____

Date Completed: _____

Lab Manager's Signature: _____

Clay Mineral:	yes	no	(circle one)	
Light Mineral:	yes	no	(circle one)	
Heavy Mineral:	yes	no	(circle one)	If yes, include:
Wt % Heavy Minerals	_____			Wt % Light Minerals _____
Sample Number:	_____			Mineral Name: _____

Magnification: _____

Composition: (Attach spectrum to the back of this sheet.)

Appendix K

Mineralogical Laboratory On-Site Evaluation Questionnaire

The following questionnaire is completed to provide documentation of an on-site evaluation. A mineralogical laboratory is evaluated prior to the award of a contract to assess the ability of the laboratory, in terms of personnel, facilities, and equipment, to analyze soil samples successfully. A second evaluation is made after sample analysis is underway. At the time of the second evaluation, adherence to protocol is evaluated, and specific problems are addressed.

Mineralogical Laboratory On-Site
Evaluation Questionnaire
DDRP Soil Survey

General (Page 1 of 2)

Date _____

Laboratory: _____

Street Address: _____

Mailing Address (if different from above): _____

City: _____

State: _____ Zip _____

Laboratory Telephone Number (): _____

Laboratory Director: _____

Laboratory Quality Assurance Officer: _____

Type of Evaluation: _____

Contract Number: _____

Contract Title: _____

General (Page 2 of 2)

Personnel Contacted:

Name

Title

[illegible]

Laboratory Evaluation Team:

Name

Title

[illegible]

Laboratory Organizational Chart

Organization and Personnel (Page 2 of 3)

Laboratory Personnel

Position	Name	Academic Training*	Special Training	Years Experience†
----------	------	--------------------	------------------	-------------------

*List highest degree obtained and specialty. Also list years toward a degree.
†List only experience directly relevant to task to be performed.

Organization and Personnel (Page 3 of 3)

Item	Yes	No	Comments
Do personnel assigned to this project have the appropriate educational background to successfully accomplish the objectives of the program?			
Do personnel assigned to this project have the appropriate level and type of experience to successfully accomplish the objectives of this program?			
Is the organization adequately staffed to meet project commitments in a timely manner?			
Was the project manager available during the evaluation?			
Was the Quality Assurance Supervisor available during the evaluation?			
Does the laboratory QA supervisor report to senior management levels?			
Were chemists and technicians available during the evaluation?			

Laboratory Manager (Page 1 of 1)

Item	Yes	No	Comments
Does the laboratory manager have his/her own copy of the standard operating procedures?			
Does the laboratory manager have his/her own copy of the instrument performance data?			
Does the laboratory manager have his/her own copy of the latest monthly QC plots?			
Is the laboratory manager aware of the most recent control limits?			
Does the laboratory manager review the following before reporting data:			
a. The data itself?			
b. The quality control data sheet with analyst's notes?			
c. The general instrument performance and routine maintenance reports?			

Standard Operating Procedures (Page 1 of 1)

Item	Yes	No	Comments
Does the laboratory have a standard operating procedure (SOP) manual?			
Is the SOP manual followed in detail?			
Does the SOP manual contain quality control practices?			
Does each analyst/technician have a copy of the SOP manual?			
Does the SOP manual deviate from the procedures required by this project?			
If the SOP manual does deviate, are the deviations approved for this project and documented in written form?			
Does each analyst/technician have a copy of all methods and procedures required by this project?			
Are plots of instrument accuracy and precision available for every analysis?			
Are detection limit data tabulated for each analysis?			

Laboratory Facilities (Page 1 of 3)

When touring the facilities, give special attention to (1) the overall appearance of organization and neatness, (2) the proper maintenance of facilities and instrumentation, and (3) the general adequacy of the facilities to accomplish the required work.

Item	Yes	No	Comment
Does the laboratory appear to have adequate work-space (6 linear meters of unencumbered bench space per analyst)?			
Is the specific conductance of deionized water routinely checked and recorded?			
Have the hoods been checked for operating efficiency? How often is this done?			
Are the analytical balances located away from draft and areas subject to rapid temperature changes?			
Has the balance been calibrated within one year by a certified technician?			
Is the balance checked with a class S standard weight before each use, and is the result of the check recorded in a logbook? (Have technician demonstrate how this is done.)			
Are exhaust hoods provided that allow efficient work with volatile materials?			
Is the laboratory clean and well organized?			

Laboratory Facilities (Page 2 of 3)

Item	Yes	No	Comment
Are contamination-free work areas provided for the handling of toxic materials?			
Are adequate facilities provided for separate storage of samples, extracts, and standards, including cold storage?			
Is the temperature of the cold storage units recorded daily in logbooks?			
Are chemical-waste disposal policies/procedures adequate?			
Are contamination-free areas provided for trace-level analytical work?			
Can the laboratory supervisor document that water is used for preparation of standards and blanks?			
Are all chemicals dated upon receipt and thrown away when shelf life is exceeded?			
Do adequate procedures exist for disposal of waste liquids from the ICP and AA spectrometers?			
Do adequate procedures exist for disposal of liquid and solid wastes?			
Is the laboratory secure?			
Are all samples stored in the refrigerator between analyses?			
Are acids and bases stored in separate areas?			
Are hazardous, combustible, and toxic materials stored safely?			

Laboratory Facilities (Page 1 of 3)

Item	Available		Comments (where applicable, cite system, QC check, adequacy of space)
	Yes	No	
Gas			
Lighting			
Compressed air			
Electrical services			
Hot and cold water			
Laboratory sink			
Ventilation system			
Hood space			
Cabinet space			
Storage space (m ²)			
Vacuum system			
Deionized water			
Refrigerated storage (4°C)			

Comments on Laboratory Facilities

Equipment, General (Page 1 of 2)

Item	Equipment			Condition/Age			Comments
	Quantity	Make	Model	Good	Fair	Poor	
Balance, analytical (1)							
(2)							
(3)							
Balance, top-loader							
Class "S" weights							
Balance table							
NBS-calibrated thermometer							
Double-deionized (DDI) water source or equivalent system							
Desiccator							
Glassware (1) Beakers							
(2) Vacuum flasks							
(3) Fritter funnels							
(4) Graduated cylinders							

Equipment, General (Page 2 of 2)

Item	Equipment			Condition/Age			Comments
	Quantity	Make	Model	Good	Fair	Poor	
Glassware (cont.) (5) Fleakers							
(6) Other							
Riffle-splitter, Jones-type							
Muffle Furnace (0°-600°C)							
Wiggle bug mixer							
Ultrasound water bath							
Automated mortar and pestle							
Pulverizer							
Reciprocating shaker							

Comments: _____

Equipment for SQXRD

Item	Manufacturer	Model	Installation Date	Comments
International No. 2 centrifuge with a No. 240 head, or equivalent				
Centrifuge tubes, plastic, 100mL				
Centrifuge tubes, glass, 50 mL				
X-ray powder diffraction unit with Cu-radiation tube, an x-, y- plotter, solid state pulse height analyzer, peak area integration capability, rotating and oscillating stage, diffraction pattern library, and data analysis software				
Eye dropper or pipet				
Desiccator				
Freeze-dryer				
Ring-and-puck pulverizer, titanium carbide, or equivalent equipment				
Convection oven				
Syringes, plastic, 10 mL				
Screen, 80-mesh				

Reagents and Consumables for SQXRD

Chemical	Quantity	Grade	Expiration Date	Comments
Scribe				
Reference minerals (list those used)				
Calibration standard (specify)				
Sodium Hexameta- phosphate [$\text{Na}(\text{PO}_3)_6$]				
Magnesium chloride (MgCl_2)				
Ethanol ($\text{C}_2\text{H}_5\text{OH}$)				
Methanol (CH_3OH)				
Silver nitrate (AgNO_3)				
Potassium chloride (KCL)				
Ethylene glycol ($\text{CH}_2\text{OHCH}_2\text{OH}$)				
Linde semiconductor grade $\alpha\text{-Al}_2\text{O}_3$, corundum, 1 micron				
Cation exchange resin Rexyn 101 (H) or equivalent				
Silica gel				
Hydrogen peroxide (H_2O_2)				

Reagents and Consumables for SQXR

Chemical	Quantity	Grade	Expiration Date	Comments
Dialysis tubing				
Sodium acetate (NaC ₂ H ₃ O ₂)				

Equipment for XRF

Item	Manufacturer	Model	Installation Date	Comments
Simultaneous wave-length-dispersive X-ray fluorescence spectrometer				
Hydraulic press capable of producing pressure of 5 T/in ²				
Pellet die				
Desiccator				

Reagents and Consumables for XRD

Chemical	Quantity	Comments
Microcellulose powder		
Desiccant		
Calibration standards made from CCRMP-, NBS-, or USGS-certified rock standards		

Equipment for SEM/EDXRF

Item	Manufacturer	Model	Installation Date	Comments
Scanning electron microscope with 200- to 300-angstrom resolution in the secondary electron mode				
Gold/palladium sputter coater with argon diffusion chamber				
Energy-dispersive X-ray fluorescence analytical unit and software (or equivalent) which can interface with SEM				
Separatory funnel, 250 mL				
Fritted funnel, 50 mL				
Sieves, 60-mesh and 270-mesh				
Polaroid camera				

Comments: _____

Reagents and Consumables for SEM/EDXFR

Chemical	Quantity	Grade	Expiration Date	Comments
Gold/palladium wire, metal for coating specimens				
Film, 35-mm or 4x5- inch format				
Film, Polaroid Type 55				
Certified microprobe mineral and rock standards				
Sodium polytungstate, reagent grade, density = 2.95				
Filter paper, Whatman No. 1				
Carbon specimen mount				
Silver conducting paint				

Comments: _____

Documentation/Tracking (Page 1 of 1)

Item	Yes	No	Comments
Is a sample custodian designated? If yes, name of sample custodian _____.			
Are the sample custodian's procedures and responsibilities documented? If yes, where are these documented?			
Is sample tracking performed via paper or computer?			
Are written standard operating procedures (SOPs) developed for receipt of samples? If yes, where are they documented (e.g., laboratory manual, written instructions)?			
Are written standard operating procedures (SOPs) developed for compiling and maintaining sample document files? If yes, where are they documented?			
After completion of the analysis, are the samples correctly stored for 6 months or until laboratory personnel are told otherwise?			
Are magnetic tapes stored in a secure area?			

Analytical Methodology (Page 1 of 2)

Item	Yes	No	Comments
Are the specified methods used?			
Is there any unauthorized deviation from contract methodology?			
Are written analytical procedures provided to the analyst?			
Are reagent grade or higher purity chemicals used to prepare standards?			
Are fresh analytical standards prepared at a frequency as specified in the methods manual?			
Are reference materials properly labeled with concentrations, date of preparations, and the identity of the person preparing the sample?			
Is a standard preparation and tracking logbook maintained?			
Do the analysts record bench data in a neat and accurate manner?			
Is the appropriate instrumentation used in accordance with the required protocol(s)?			

Analytical Methodology (Page 2 of 2)

Comments on Analytical Methods and Practices

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Quality Control (Page 1 of 2)

Item	Yes	No	Comments
Does the laboratory maintain a quality control manual?			
Does the manual address the important elements of a QC program, including the following:			
a. Personnel?			
b. Facilities and equipment?			
c. Operation of instruments?			
d. Documentation of procedures?			
e. Procurement and inventory practices?			
f. Preventive maintenance?			
g. Reliability of data?			
h. Data validation?			
i. Feedback and corrective action?			
j. Instrument calibration?			
k. Record keeping?			
l. Internal audits?			

Quality Control (Page 2 of 2)

Item	Yes	No	Comments
Are QC responsibilities and reporting relationships clearly defined?			
Are laboratory standards traceable?			
Are quality control charts maintained for each routine analysis?			
Do QC records show corrective action when analytical results fail to meet QC criteria?			
Do supervisory personnel review the data and QC results?			
Does the QC analyst have his/her own copy of the standard operating procedures?			
Does the QC officer have his/her own copy of the instrument performance data?			
Does the QC officer have his/her own copy of the latest QC plots?			
Is the QC officer aware of the most recent control limits?			
Does the QC officer obtain control limits and obtain new control chart plots once per batch?			
Are all QC data (e.g., control charts, regression charts, QC data bases) up to date and accessible?			
Are minimum detection limits calculated as specified?			
Is information on QC data sheet reported to the analyst?			

Data Handling (Page 1 of 2)

Item	Yes	No	Comments
After data are input into the computer, does data clerk check all data for accuracy?			
Are calculations checked by another person?			
Are calculations documented?			
Does strip chart reduction by on-line electronic digitization receive at least 5% manual spot checking?			
Are data from manually interpreted strip charts spot-checked after initial entry?			
Do the laboratory records include the following information:			
Sample identification number			
Sample type			
Date sample received in laboratory			
Date of analysis			
Analyst			
Result of analysis (including raw analytical data)			
Recipient of the analytical data			

Data Handling (Page 2 of 2)

Item	Yes	No	Comments
Does the laboratory follow required sample tracking procedures from sample receipt to discard?			
Does the data clerk routinely report quality control data sheet information to the analyst?			
Does the data clerk submit quality control data sheet information to the laboratory manager, along with the analytical data to be reported?			
Do records indicate corrective action taken?			
Are provisions made for data storage for all raw data, calculations, quality control data, and reports?			
Are all data and records retained for the required amount of time?			
Are computer printouts and reports routinely spot-checked against laboratory records before data are released?			

Summary (Page 1 of 2)

Item	Yes	No	Comments
Do responses to the evaluation indicate that project and supervisory personnel are aware of QA and its application to the project?			
Do project and supervisory personnel place positive emphasis on QA/QC?			
Have responses with respect to QA/QC aspects of the project been open and direct?			
Has a cooperative attitude been displayed by all project and supervisory personnel?			
Does the organization place the proper emphasis on quality assurance?			
Have any QA/QC deficiencies been discussed (before the audit team leaves)?			
Is the overall quality assurance adequate to accomplish the objectives of the project?			
Have corrective actions that were recommended during previous evaluations been implemented?			
Are any corrective actions required? If so, list the necessary actions below.			

Summary (Page 2 of 2)

Summary Comments and Corrective Actions

Appendix L

Mineralogical Data Package Completeness Checklist

The mineralogical data package completeness checklist that follows was developed to (1) give the contractor mineralogical laboratory a concise listing of what is required in the data package; (2) give the data recipients a check-off listing to inventory the contents of the data package; and (3) to serve as an index to the handwritten data file.

Mineralogical Data Package Completeness Checklist

Lab Name: _____ **Batch ID:** _____

Lab Manager's Signature: _____ **Date:** _____

1. Any major difficulties during analysis have been discussed with the QA Manager or designee.

2. a. For SQXRD the patterns obtained according to the procedures described in the methods manual are included:

- NBS silicon powder standards, 3 patterns
- Randomly oriented powder mount of the multiphase standard; one for every batch of samples
- Randomly oriented powder mount of <2-mm fraction with standard
- Oriented Mg-sat. AD
- Oriented Mg-sat. gly
- Oriented K-sat. AD
- Oriented K-sat. 110°C
- Oriented K-sat. 350°C
- Oriented K-sat. 550°C
- Randomly oriented powder mount of <0.002-mm fraction with standard.

- b. Required forms (400-405) are submitted.**

- c. Lab name, batch number, prep lab name, lab manager's signature, date form completed, and date batch received are recorded on all forms.

- d. Correct number of samples were analyzed and the results for each parameter are tabulated.

Note: Checklist must be included in the data package.

[illegible]

Appendix M

Example Verification Report

The verification report summarizes the review of the data for each analytical batch. It also documents the required actions, e.g., confirmation and reanalysis requests and flagging of data.

Northeastern DDRP Soil Survey
Verification Report

Batch ID: _____
Analytical Laboratory: _____
Preparation Laboratory: _____
Audit Horizon Type(s): _____
Audit Pair(s): _____
Preparation Pair(s): _____
Field (Sampling) Pair(s): _____
Organic Samples: _____
Missing Samples: _____

Date Data Package Received:	__/__/__	
Date Data Package Evaluated (Initial):	__/__/__	By: _____
Date Evaluation Letter Sent:	__/__/__	By: _____
Date Laboratory Response Received:	__/__/__	By: _____
Date Reanalysis Request Sent:	__/__/__	By: _____
Date Verified (First Pass):	__/__/__	By: _____
Date Verification Tape Sent to ORNL (First Pass):	__/__/__	By: _____
Date Verified (Final):	__/__/__	By: _____
Date Verification Tape Sent to ORNL (Final):	__/__/__	By: _____

I. Outstanding Issues - Contractor Analytical Laboratory

The following items that are identified as missing should be resubmitted and problems should be resolved before verification is completed:

A. General (forms 102-108)

1. Required forms have been submitted.
2. Laboratory name, batch ID, preparation laboratory name, laboratory manager's signature, date form completed, and date batch received are included on all forms.
3. Correct data qualifiers (tags) were used as needed (see Table 1).

B. Data examination (forms 103-108)

1. Check that audit pairs are within established control criteria.
2. Estimate %RSD for all paired QA samples for each parameter, and record in Table 3.
3. Check the internal consistency of the data.
 - a. Form 103a: pH, $H_2O > 0.002 > 0.01$.
 - b. Form 103b: sand + silt + clay = 100 ± 0.2 .
 - c. Form 104d: CEC $NH_4OAc > CEC NH_4Cl$.
 - d. Form 106: Ext. Sulfate, $H_2O < PO_4$.
 - e. Form 106: Exch. Acidity, $BaCl_2 > KCl$.
 - f. Form 107: Sulfate Isotherms are $0 < 2 < 4 < 8 < 16 < 32$. Adsorption solution is within 5% of the theoretical value.
 - g. Form 104c: Extraction ratio is 1:2 for mineral samples and 1:10 or 1:25 for organic samples.
 - h. Forms 103b and 108: For particle size analysis and specific surface, organic samples are reported as a U.

C. General (forms 109-116)

1. Required forms have been submitted.
2. Laboratory name, batch ID, and laboratory manager's signature are included on all forms.

D. Data examination (forms 109-116)

1. Forms 109a-c: Detection Limits
 - a. Check that instrumental detection limits (IDL) and associated dates of determination are tabulated. IDL should be updated monthly for each parameter.
 - b. IDL should be less than or equal to the contract-required detection limit (CRDL) for each parameter.
2. Form 110a-c: Matrix Spikes
 - a. Identify samples used for spiking.
 - b. Check that percent recovery for matrix spikes is reported for each parameter required.

- c. Check that percent recovery is calculated correctly (recalculate at least three per page).
- d. Check that percent recovery is $100 \pm 15\%$ for each parameter; if it is not, then spiking must be repeated on two different samples.
- e. Verify that the level of spike is 10 times the CRDL or equal to the endogenous level, whichever is greater.
- f. Check that the sample used for Total S, N, and C is not an organic sample for each batch.

3. Form 111a-i: Replicates

- a. Replicate precision results are reported for each parameter. For pH and specific surface, triplicates are determined.
- b. Correct equation is used to calculate %RSD (degrees of freedom equal $n-1$).
- c. %RSDs are 0-10% (except on fractionated sand and silt).

4. Forms 112a-h: Blanks and QCCS

- a. Calibration blanks, reagent blanks, and detection limit (DL) QCCS are reported where required.
- b. Calibration and reagent blanks should be less than or equal to the CRDL.
- c. Form 112g: K-factors are reported correctly.
- d. Form 112h: Three high EGME blanks are reported correctly.
- e. DL QCCS theoretical values are approximately 2 to 3 times the CRDL, and the measured values are within 20% of the theoretical value.
- f. QCCS true values are approximately in the midrange of the reported sample values or of the calibration curve.
- g. Initial, continuing, and final QCCS values are within upper and lower control limits.

5. Form 113: Ion Chromatography

- a. IC resolution test results are reported.
- b. Resolution value exceeds 60%.
- c. Peaks are clean on chromatogram(s).
- d. At least one chromatogram is provided for each day of operation for each instrument.

6. Form 114: Standard Additions

- a. Standard additions are performed and results are reported when matrix spike results do not meet contractual requirements.

7. Forms 115a-e: Air Dry Sample Weights

- a. The air-dried soil weight is reported for each parameter, except for particle-size analysis (oven dried) and specific surface ($P_2 O_5$ wt. = oven dried).
- b. Weights are reported correctly (see Table 2).
- c. Form 115a: One sample is determined in triplicate for moisture and specific surface.
- d. Duplicates are reported correctly.

8. Forms 116a-h: Dilution Factors

- a. Total sample volume, aliquot volume, total dilution volume, dilution concentrations, and dilution blanks are recorded for each sample.

E. Forms 200: Blank-corrected data

1. Required forms 204-208 have been submitted.
2. Laboratory name, batch ID, preparation laboratory name, manager's signature, and date batch received are included on all forms.
3. Correct number of samples were analyzed, and the results for each parameter are tabulated.

F. Forms 300: Raw Data

1. Required forms 303b-308 have been submitted.
2. Laboratory name, batch ID, preparation laboratory name, laboratory manager's signature, and date batch received are included on all forms.
3. Correct number of samples were analyzed, and the results for each parameter are tabulated.

G. Reporting units are correct on the following forms (see Table 4):

1. 103-108
2. 109: Detection Limits
3. 110: Matrix Spikes
4. 111: Replicates
5. 112: Blanks and QCCS
6. 115: Air Dry Sample Weights
7. 116: Dilution Factors/Concentration
8. 200: Blank-Corrected Data
9. 300: Raw Data

Table 1. Analytical Laboratory/Field Data Qualifiers (TAGS)

Data Qualifier	Indicates
A	Instrument unstable.
B	Redone, first reading not acceptable.
F	Result outside criteria with consent of QA Manager.
G	Result obtained from method of standard additions.
J	Result not available; insufficient sample volume shipped to laboratory.
L	Results not available due to interference.
M	Result not available; sample lost or destroyed by laboratory.
N	Result outside QA criteria.
P	Result outside criteria, but insufficient volume for reanalysis.
R	Result from reanalysis.
S	Contamination suspected.
T	Container broken.
U	Result not required by procedure; unnecessary.
X	No sample.
Y	Available for miscellaneous comments.
Z	Result from approved alternate method.

Table 2. Required Sample Weights for the Analytical Parameters

Parameter	Replicates	Weights		Air (AD) or Oven Dried (OD)
		Mineral	Organic	
Moisture Content	3	10±0.01 g	same	AD
Particle Size Analysis	2	Should be oven dried wt. after removal of organics and soluble salts (±0.001 g). NA for organics.		
Cation Exchange Capacity and Exchange Cations	2	2.50±0.01 g	same	AD
CaCl ₂ Cations	2	10.00 g (or 1.6 g for absorbent organics)	4.00 g	OD*
Exchangeable Acidity	2	2.00	same	
BaCl ₂			(can use 1.00 g for organics)	
KCl	2	2.50	same	OD*
Ext. Fe and Al:				
Pyrophosphate	2	2.00 g±0.001 g	same	AD
Acid - Oxalate	2	0.500 g±0.001 g	same	AD
Citrate Dithionite	2	4.00 g±0.01 g	same	AD
Extractable Sulfate	2	4.00 g±0.01 g	same	AD
Sulfate Adsorption Isotherms	2	5.00 g±0.01 g	same	OD*
Total Carbon and Total Nitrogen	2	30 g±0.001 mg	(weight will vary considerably with varying instrumentation)	AD
Total Sulfur	2	0.500 g±0.001 g	(will vary with 30 mg instrumentation and organic content)	AD
Specific Surface	3	4 g AD soil into desiccator for 2 days then weigh P ₂ O ₅ 0.500 g soil to ±0.1 mg. NA for organics.		

Weight for organic samples may be reduced by one half or more if necessary for CEC, Extractable Cations, and Exchangeable Acidity.
Air-dried equivalent to oven dried weight of soil = (grams oven-dried soil desired)

$$\frac{1.000 - \% \text{ moisture}}{100}$$

Table 3. %RSD Chart for Replicate QA Samples

Key: A - Audit Pair

P - Preparation Pair

F - Field Pair

	A1	A2	P	F1	F2	F3	F4	F5	F6	F7	F8	F9
pHH ₂ O												
pH01CaCl ₂												
pH002CaCl ₂												
Sand												
silt												
Clay												
VCSand												
CSand												
MSand												
FSand												
VFSand												
CSilt												
FSilt												
Exchangeable Bases:												
Ca, NH ₄ OAc												
Mg, NH ₄ OAc												
K, NH ₄ OAc												
Na, NH ₄ OAc												
Ca, NH ₄ Cl												
Mg, NH ₄ Cl												
K, NH ₄ Cl												
Na, NH ₄ Cl												
Ca, CaCl ₂												
Mg, CaCl ₂												
K, CaCl ₂												
Na, CaCl ₂												
Fe, CaCl ₂												
Al, CaCl ₂												
CEC, NH ₄ OAc												
CEC, NH ₄ Cl												
Fe, Pyro												
Al, Pyro												

(continued)

Table 3. (Continued)

Key: A - Audit Pair

P - Preparation Pair

F - Field Pair

[illegible]

Table 4. Required DDRP Soil Reporting Units

Direct/Delayed Response Project Soil Reporting Units									
Data Type									
Parameter	Forms 103-108 Reporting Forms	Form 109 Detection Limits	Form 110 Matrix Spikes	Form 111 Replicates	Form 112 Blanks and QCCS	Form 115 Air-Dry Sample wt.	Form 116 Dilution Concent.	Form 200 Blank Corrected	Form 300 Raw Data
Partical Size	wt%	NA	NA	wt%	wt%	grams	NA	NA	grams
Exchangeable Cations	meq/100 g	mg/L	mg/L	meq/100 g	mg/L	grams	mg/L	meq/100	mg/L
Cation Exchange Capacity									
FIA	meq/100 g	mg N-NH ₄ /L	mg N-NH ₄ /L	meq/100 g	meq/L	grams	mg N-NH ₄ /L	meq/100 g	mg N-NH ₄
Titration	meq/100 g	meq NH ₄ /L	meq NH ₄ /L	meq/100 g	meq NH ₄ /L	grams	meq NH ₄ /L	meq/100 g	meq NH ₄
Extractable Fe and Al	wt%	mg/L	mg/L	wt%	mg/L	grams	mg/L	wt%	mg/L
Extractable SO ₄	mg S/kg	mg SO ₄ /L	mg SO ₄ /L	mg S/kg	mg S/L	grams	mg S/L	mg S/kg	mg SO ₄ /L
Exchangeable Acidity	meq/100 g	meq/L	NA	meq/100 g	meq	grams	NA	meq/100 g	meq
KCl-Extractable Al	meq/100 g	NA	NA	meq/100 g	mg/L	grams	mg/L	meq/100 g	mg/L
SO ₄ Isotherms	mg S/L	mg SO ₄ /L	NA	mg S/L	mg S/L	grams	mg S/L	mg S/L	mg S/L
Total S, N, and C	wt%	wt%	wt%	wt%	wt%	grams	NA	wt%	ug
Specific Surface	m ² /g	NA	NA	m ² /g	mg EGME (blanks) m ² /g (QCCS)	grams	NA	m ² /g	mg EGME

II. Sample Data Review

A. The reported sample data (were, were not) complete. The following suspect sample results should be confirmed by the contractor analytical laboratory:

<u>Parameter</u>	<u>Sample Number</u>	<u>Form Number</u>	<u>Date Requested</u>	<u>Date Confirmed</u>	<u>Reason for Confirmation</u>
------------------	--------------------------	------------------------	---------------------------	---------------------------	------------------------------------

B. Sample analysis (was, was not) complete based on data submitted. Reanalysis is recommended for the following suspect samples:

<u>Parameter</u>	<u>Sample Number</u>	<u>Date Requested</u>	<u>Date Submitted</u>	<u>Reason for Reanalysis</u>
------------------	--------------------------	---------------------------	---------------------------	----------------------------------

III. QA Data Review

Data for the following parameters and samples were not acceptable based on the following:

- A. For a routine/field pair, a preparation pair, or an audit pair with one or both concentrations greater than 10 times the CRDL, the duplicate precision was not within the expected criteria. The maximum expected %RSD was exceeded for the following parameters:

<u>Parameter</u>	<u>Replicate Sample Type</u>	<u>Reported %RSD</u>	<u>Contract-Required Maximum %RSD</u>	<u>Explanation</u>
------------------	----------------------------------	--------------------------	---	--------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged with the appropriate parameter flag D1, D2, or D5-D8.

- B. Audit sample data were not within the expected performance range of the audit windows.
The following audit samples were outside the expected range:

<u>Parameter</u>	<u>Audit Horizon Type</u>	<u>Reported Value</u>	<u>Expected Range</u>	<u>Explanation</u>
------------------	-------------------------------	---------------------------	---------------------------	--------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged using the appropriate parameter flag N0/, N1, or N2.

IV. QC Data Review

- A. If the Instrumental detection limit (IDL) reported on Form 109 exceeded the CRDL, the integrity of the following sample values that are reported at less than 10 times the CRDL could be in question:

<u>Parameter</u>	<u>Sample Number</u>	<u>Reported Concentration</u>	<u>Reported IDL</u>	<u>CRDL</u>	<u>Explanation</u>
------------------	--------------------------	-----------------------------------	-------------------------	-------------	--------------------

NOTE: Only samples with concentrations less than 10 times the CRDL for the affected parameters listed above should be flagged with the sample flag L1.

- B. Matrix spike recovery reported on Form 110 should be $100 \pm 15\%$. If it is not, two different samples should be run. If the recovery for one or both samples is not within $100 \pm 15\%$, standard additions must be performed. Spike concentrations must be equal to 10 times the CRDL or equal to the endogenous level, whichever is greater.

<u>Parameter</u>	<u>Sample Result</u>	<u>Spike Level</u>	<u>10 Times CRDL</u>	<u>Contract- Required Spike Level</u>	<u>Percent Recovery</u>
------------------	--------------------------	------------------------	--------------------------	---	-----------------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged with the appropriate parameter flag S0/ or S1.

- C. Replicate precision data reported on Form 111 should be 10% or less. If initial replicate precision was outside the criterion, an additional replicate must be analyzed as required by the contract. The 10% RSD criterion is applicable only when the mean of the duplicate analyses exceeds the CRDL by a factor of 10.

<u>Parameter</u>	<u>Reported %RSD</u>	<u>Program Calculated %RSD</u>	<u>Explanation</u>
------------------	--------------------------	--	--------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged with the appropriate parameter flag D3 or D4.

D. Blanks and QCCS reported on Form 112:

- 1. Calibration and reagent blanks:** If either blank was greater than the CRDL and contributed more than 50% to the sample concentrations, then list contaminated samples:

<u>Parameter</u>	<u>Sample Number</u>	<u>% Concentration</u>	<u>Explanation</u>
------------------	----------------------	------------------------	--------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged with the sample flag B3 or the appropriate parameter flag B4, B5, or B7.

2. Quality control calibration sample (QCCS) analyses: List those QCCSs not within contractual requirements. Were sufficient QCCS run?

<u>Parameter</u>	<u>Reported Value</u>	<u>Required Range</u>	<u>Number of QCCS Runs Performed</u>	<u>Number of QCCS Runs Required</u>	<u>Explanation</u>
------------------	-----------------------	-----------------------	--------------------------------------	-------------------------------------	--------------------

3. Detection Limit (DL) QCCS (DL QCCS) analyses: for those theoretical DL QCCS concentrations that exceeded 2 to 3 times the CRDL, the measured concentration of DL QCCS should be within 20% of the theoretical concentration.

<u>Parameter</u>	<u>Theoretical Value</u>	<u>Measured Concentration</u>	<u>CRDL</u>	<u>Explanation</u>
------------------	--------------------------	-------------------------------	-------------	--------------------

NOTE: All samples in the batch for the affected parameters listed above should be flagged with the appropriate parameter flags Q1-Q4.

E. The following air-dry sample weights reported on Form 115 were not within contractual requirements:

<u>Parameter</u>	<u>Reported Value</u>	<u>Contract-Required Sample Weight</u>	<u>Explanation</u>
------------------	---------------------------	--	--------------------

NOTE: Only samples affected for the parameters listed above should be flagged with the sampling flag W0.

F. The following dilution factors, total sample volumes, aliquot volumes, total dilution concentrations were not reported correctly on Form 116:

<u>Parameter</u>	<u>Reported Value</u>	<u>Contract- Required Value</u>	<u>Explanation</u>
------------------	---------------------------	-------------------------------------	--------------------

G. Summarize requests for confirmation of data or reanalysis of samples on Form 500 (see page 21).

V. Summary of Flagged Data

All QC data (matrix spikes, replicates, calibration blanks, reagent blanks, QCCS, IDL, air-dry sample weights, and dilution factors) and paired QA data (preparation duplicates, field duplicates, and audits) were not within contractual or expected criteria for the samples and the associated parameters listed below:

(Parameter Flags: B4-B7, D1-D8, K0-K4, N0, N1, N2, Q1-Q4, S0, and S1) (Sample Flags: A0, B3, L1, M0, W0, X0, X1, and X2)

List parameter flags and the affected parameters for this batch:

Round _____

Page: _____ of _____

VI. Summary of Modifications

Pre-verification_____ (Additions/Deletions of Numerical/Flag Transactions)

Post-verification_____ To be applied to the Raw Data Set by Lockheed-EMSCO QA Staff

[illegible]

***See Table 4**

Appendix M
Revision 0
Date: 3/87
Page 22 of 28

Table 4. Datatype and Subtype Definitions

Datatype	Subtype	
1	Blank	
2	Blank	
3	Blank	
M	SPR	Spike result
M	SPA	Spike added
M	REC	Recovery, percent
M	SAR	Sample result
R	REP1	Replicate 1
R	REP2	Replicate 2
R	REP3	Replicate 3 (not required for all parameters)
R	AV1	Average
R	RSD	
W	Blank	Weights

VII. Modifications (Additions and Deletions) to be made to a copy of the Raw Data Set by ORNL Staff

Batch ID	Form Number	Sample ID	Watershed ID	Variable Name	Original Value	New Value	Comments	Date Changes Applied at ORNL	
									By

VIII. Summary of Outstanding Issues Addressed to Sample Management Office
Regarding 15% Withholding

Batch ID: _____

Sample Number	Parameter	Flag Used	Cause of Exception	Reason For Recommendation of Penalty or Waiver	Lockheed- EMSCO Recommendation*	EPA Approval

*Possible recommendations: P = Penalty or W = Waiver

Table 5. Data Qualifiers for the Verification of Analytical Data (FLAGS)

Miscellaneous

AO* Value missing

Generated by Appropriate Blank Exception Program

B3* Internal (laboratory) calibration or reagent blanks are $>2x$ CRDL and contribute $>50\%$ to the sample concentrations in the batch.

B4** Potential negative sample bias based on internal (laboratory) blank data.

B5** Calibration blank >1.05 x reagent blank.

Generated by Duplicate Precision Exception Program

D1** Field duplicate precision exceeded the maximum expected percent relative standard deviation (%RSD), and either the routine or the duplicate value was ≥ 10 x CRDL.

D2** Field duplicate precision exceeded the maximum expected %RSD, and both the routine and duplicate sample concentration was ≥ 10 x CRDL.

D3** Internal (laboratory) replicate precision exceeded the maximum contract required %RSD, and either the routine or the duplicate sample concentration was ≥ 10 x CRDL.

D4** Internal (laboratory) replicate precision exceeded the maximum contract required both the routine and duplicate sample concentrations were ≥ 10 x CRDL.

D5** Preparation duplicate precision exceeded the maximum expected %RSD and either routine or the duplicate value was ≥ 10 x CRDL.

D6** Preparation duplicate precision exceeded the maximum expected %RSD and both the routine and the duplicate sample concentrations were ≥ 10 x CRDL.

D7** Audit duplicate precision exceeded the maximum expected %RSD, and either of the audit sample concentrations was ≥ 10 x CRDL.

D8** Audit duplicate precision exceeded the maximum expected %RSD, and both audit pair concentrations were ≥ 10 x CRDL.

(continued)

Table 5. (Continued)

Generated for Known Relationships of Sulfur Isotherms

K0** Elemental parameter out of range; used for total C, N, and S only.

K1** Organic soil (total C 20-60%) and $\text{SO}_4\text{H}_2\text{O} > 1.05 \times \text{SO}_4\text{PO}_4$.

K2** Mineral soil (total C 0-20%) and $\text{SO}_4\text{H}_2\text{O} > 1.05 \times \text{SO}_4\text{PO}_4$.

K3** Organic soil: $1,000 \times \text{Total S} < \text{SO}_4\text{PO}_4$ or $\text{SO}_4\text{H}_2\text{O}$.

K4** Mineral soil: $3,000 \times \text{Total S} < \text{SO}_4\text{PO}_4$ or $\text{SO}_4\text{H}_2\text{O}$.

Generated by Detection Limit Exception Program

L1* Instrumental detection limit (IDL) exceeded contract-required detection limit (CRDL) and sample concentration was $< 10 \times \text{CRDL}$.

Miscellaneous

M0* Value was obtained by using a method that is unacceptable according to the contract.

Generated by Audit Check Program

N0** Audit sample value exceeded upper control limit.

N1** Audit sample value was below lower control limit.

N2** Audit sample value exceeded control limits; and sample preparation procedure is suspect.

Generated by QCCS Exception Program(s)

Q1** Quality control calibration sample (QCCS) was above contractual criteria.

Q2** QCCS was below contractual criteria.

Q3** Insufficient number of QCCSs were measured.

Q4** Detection limit QCCS was not 3 CRDL and measured DL QCCS value was not within 20% of the theoretical concentration.

Generated by Matrix Spike Program

S1** Percent recovery of matrix spike was above contractual criteria ($100 \pm 15\%$).

S2** Percent recovery of matrix spike was below contractual criteria ($100 \pm 15\%$).

Table 5. (Continued)

Miscellaneous

W0* Air dry sample weight was not within contractual requirement.

Miscellaneous (not to be included in any statistical analyses)

X0* Invalid but confirmed data based on QA/QC data review.

X1* Invalid but confirmed data - potential gross contamination of sample or parameter.

X2* Invalid but confirmed data - potential sample switch.

* Sample Flag: Flag the affected parameter for the affected samples only.

** Parameter Flag: Flag the affected parameter for ALL samples in the batch (the assumption is that QA/QC represents all samples in the batch).