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Pennsylvania State University

Soil Characterization Laboratory

Methods Manual

Second Edition

by

Nelson C. Thurman
Edward J. Ciolkosz
and
Robert R. Dobos

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TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

Using The Manual

The methods described in this manual are used by the Pennsylvania State University Soil Characterization Laboratory to analyze and characterize the soils of Pennsylvania. This manual has the following two major objectives:

(1) To document the procedures used in the Soil Characterization Laboratory, and provide a reference for consistency in analysis.

(2) To provide sufficient detail for each method so that newcomers in the laboratory (graduate students, hourly-wage workers, etc.) will be able to understand and perform the analyses.

All newcomers should first become familiar with the general laboratory procedures and conduct (Chapter 2). Safety, courtesy to others, and quality control are essential to the operation of the laboratory and the successful completion of laboratory analyses. The analyst should also be familiar with the equipment used in the laboratory. Operating procedures for laboratory equipment are provided in the procedures or in appendices.

In addition to detailed steps for analysis, each chapter includes background information on the procedure, a list of required materials, equipment, and reagents, and an overview of the procedure. The list of materials, equipment, and amount of reagents needed allows the analyst to plan ahead to ensure that sufficient materials are on hand to complete the characterization. The procedure overview includes an estimate of the time required for the analysis so that the analyst can effectively budget the time in his or her schedule.

Results of the laboratory analyses are entered into a soil characterization database system (Ciolkosz et al., 1992). This system includes a program that converts the raw data into final laboratory data. The laboratory input forms (Appendix A) are tailored for the database system. The calculation program is illustrated in Appendix B and each chapter provides an explanation of the calculations used to obtain final laboratory data.

Soil Characterization at the Pennsylvania State University

The Pennsylvania State University has been involved in soil characterization since 1957. Prior to 1957, three counties (Lancaster in 1955, Chester in 1956, and Erie in 1956) were sampled and characterized by the Soil Conservation Service (SCS). Since 1957, soil characterization in Pennsylvania has been a joint SCS-Penn State program. Soil sites are selected and described jointly, while the sampling and analyses are done by the staff of the Penn State Soil Characterization Laboratory. The soil characterization data (profile and site descriptions, and physical, chemical and mineralogical data) have been published for all the sites sampled except for recent years (see Table 1.1 for a listing of all published data).

Table 1.1. Listing of published soil characterization data for Pennsylvania.

Date	Reference
1968	Characteristics, Interpretations and Uses of Pennsylvania Soils: Dauphin County. Petersen et al. Penn State Agr. Expt. Sta. Prog. Rept. 306.
1969	Characteristics, Interpretations and Uses of Pennsylvania Soils: Northampton County. Cunningham et al. Penn State Agr. Expt. Sta. Prog. Rept. 295.
1970	Characteristics, Interpretations and Uses of Pennsylvania Soils: Warren County. Ciolkosz et al. Penn State Agr. Expt. Sta. Prog. Rept. 306.
1970	Characteristics, Interpretations and Uses of Pennsylvania Soils: Huntingdon County. Ranney et al. Penn State Agr. Expt. Sta. Prog. Rept. 300.
1971	Characteristics, Interpretations and Uses of Pennsylvania Soils: Armstrong County. Cunningham et al. Penn State Agr. Expt. Sta. Prog. Rept. 316.
1972	Characteristics, Interpretations and Uses of Pennsylvania Soils: Bradford County. Ranney et al. Penn State Agr. Expt. Sta. Prog. Rept. 320.
1972	Characteristics, Interpretations and Uses of Pennsylvania Soils: Bedford County. Ciolkosz et al. Penn State Agr. Expt. Sta. Prog. Rept. 323.
1972	Characteristics, Interpretations and Uses of Pennsylvania Soils: Bucks County. Petersen et al. Penn State Agr. Expt. Sta. Prog. Rept. 324.
1972	Characteristics, Interpretations and Uses of Pennsylvania Soils: Butler County. Cunningham et al. Penn State Agr. Expt. Sta. Prog. Rept. 326.
1972	Laboratory Characterization Data and Field Descriptions of Selected Pennsylvania Soils. Cunningham et al. Penn State Agronomy Series No. 25 (All data prior to publication of the Agr. Expt. Sta. Series).
1974	Characteristics, Interpretations and Uses of Pennsylvania Soils Developed From Cherty Limestone Material. Ciolkosz et al. Penn State Agr. Expt. Sta. Prog. Rept. 341.
1974	Characteristics, Interpretations and Uses of Pennsylvania Soils Developed From Colluvial Materials. Cunningham et al. Penn State Agr. Expt. Sta. Prog. Rept. 344.
1976	Characteristics, Interpretations and Uses of Pennsylvania Soils Developed From Redbeds and Calcareous Materials. Ciolkosz et al. Penn State Agr. Expt. Sta. Prog. Rept. 355.
1977	Characteristics, Interpretations and Uses of Pennsylvania Soils Developed From Acid Shale. Cunningham et al. Penn State Agr. Expt. Sta. Prog. Rept. 362.
1983	Characteristics, Interpretations and Uses of Pennsylvania Minesoils. Ciolkosz et al. Penn State Agr. Expt. Sta. Prog. Rept. 381.

In addition, the data for Lancaster, Chester and Erie Counties have been published by the SCS (SCS, 1974). Presently there are no plans to publish data acquired since the above-listed publications. The data in these publications and all subsequent and future data will be a part of the Penn State Soil Characterization Database. This system, described by Ciolkosz and Thurman (1992), is a very user-friendly computerized database. Site and profile descriptions, as well as the physical, chemical and mineralogical data, are included within the database.

Revisions to the Laboratory Manual

This manual supersedes other manuals published by the Laboratory (Ciolkosz and Pletcher, 1974; Ciolkosz et al., 1988). Additional soil characterization sampling and analysis methods are available in Soil Survey Investigations Staff (1991), Page et al. (1982), Klute (1986), Singer and Janitzky (1986), and Franzmeier et al. (1977). Some methods and procedures have been changed or discontinued during the past 37 years. Table 1.2 gives a listing of these changes.

Standard Samples

Standard samples have been run as a part of the routine flow of samples through the characterization laboratory since 1980. One standard sample is included with each sample characterization run. The data for these standards are presented by Ciolkosz and Cronce (1986) and Ciolkosz and Dobos (1991).

Table 1.2. Changes In Soil Characterization Methods (See Ciolkosz and Pletcher, 1974, and Ciolkosz et al., 1988, for details of these methods).

Method	Change
Core bulk density	Discontinued: used on soils sampled prior to 1967; for soils sampled in 1966 cores and one clod were taken. Core bulk density data from 1955-1962 were on a moist basis and were not corrected for rock fragments. All bulk density data since 1966 are on a moist (1/3 atm) basis and are corrected for rock fragments.
Available water	All 1/3-atm water data from 1955-1965 were from cores and were not corrected for rock fragments. Core 1/3-atm water data were corrected for rock fragments in 1966. All data since 1966 are on clods and and corrected for rock fragments.
Exchangeable Ca in calcareous soils	Discontinued: used on soils sampled prior to 1966.
Cation determinations in calcareous soils	Discontinued: used on soils sampled prior to 1966.
Mehlich method for cation determinations	Discontinued: used on soils sampled prior to 1966.
Cation exchange capacity (CEC)	The pH 7.0 NH_4OAc method has been used for some CEC analyses.

Table 1.2. Changes In Soil Characterization Methods (continued).

Method	Change
KCl extractable Al	Yuan method used on soils sampled prior to 1966. Colorimetric method used 1966-1991. Since 1992, Al is determined by atomic absorption.
Nitrogen, Kjeldahl	Discontinued: used on soils sampled prior to 1973.
Organic carbon, Walkley-Black method	Discontinued, except for soils high in carbonates; used on all soils sampled prior to 1967; from 1967 to 1980 the Fisher induction furnace method was used; since 1980 the Leco furnace method has been used.
CBD-extractable Fe	Fe was analyzed by titration on soils sampled prior to 1966, and by the o-phenanthroline colorimetric method between 1966 and 1991. Since 1992, Fe is determined by atomic absorption.
CBD-extractable Al and Mn	Not analyzed on samples prior to 1992.
Field pH (portable meter)	Discontinued: lab data included field measurements of pH with a portable pH meter from 1958-1970. Since 1970, only colorimetric field pH measurements are made; these are included in the pedon description.
Weight basis	All data prior to 1964 was based on air-dry weight of the soil; oven-dry weights are now used.
Method of extraction	The mechanical vacuum extractor has been used for extracting cations, KCl extractable aluminum, and exchangeable acidity since 1980.
Soil monoliths	Discontinued; used in the past in conjunction with characterization (see Ciolkosz and Pletcher, 1974).
Percolation tests	Discontinued; procedure, data summarized by Matelski (1975).
Very fine sand	Since 1974 the very fine sand fraction (100-50 um) has been separated into very fine sand 1 (100-74 um) and very fine sand 2 (74-50 um) fractions.

References

Ciolkosz, E. J., and R. C. Cronce. 1986. Pennsylvania State University soil characterization laboratory standard sample data summary. Penn State Univ. Agron. Series No. 87.

Ciolkosz, E. J., R. C. Cronce, and R. R. Dobos. 1988. Pennsylvania State University soil characterization laboratory manual. Penn State Univ. Agron. Series No. 101.

Ciolkosz, E. J., and R. R. Dobos. 1991. Pennsylvania State University soil characterization laboratory data summary for standard samples. Penn State Univ. Agron. Series No. 112.

Ciolkosz, E. J., and R. M. Pletcher. 1974. Laboratory procedures of the Pennsylvania State University soil characterization laboratory. Penn State Univ. Agron. Series No. 36.

Ciolkosz, E. J., and N. C. Thurman. 1992. The Penn State soil characterization database system. Penn State Univ. Agron. Series (In preparation).

Ciolkosz, E. J., N. C. Thurman, and R. R. Dobos. 1992. Penn State University soil characterization database system. Penn State Univ. Agron. Dept.

Franzmeier, D. P., G. C. Steinhardt, J. R. Crum, and L. D. Norton. 1977. Soil characterization in Indiana: 1. Field and laboratory procedures. Purdue Univ. Research Bull. No. 943.

Klute, A. (editor). 1986. Methods of soil analysis. Part 1. Physical and mineralogical methods. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Matelski, R. P. 1975. The percolation rate of Pennsylvania soils for septic tank drainage fields. Penn State Agr. Expt. Sta. Prog. Rept. 345.

Page, A. L., R. H. Miller, and D. R. Keeney (editors). 1982. Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Singer, M. J. and P. Janitzky (editors). 1986. Field and laboratory procedures used in a soil chronosequence study. U.S. Geol. Sur. Bull. 1648.

SCS. 1974. Soil survey laboratory data and descriptions for some soils of Pennsylvania. USDA Soil Conservation Service. Soil Surv. Invest. Rept. No. 27.

Soil Survey Investigations Staff. 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rept. No. 42. Version 1.0. National Soil Survey Center. Lincoln, NE.

CHAPTER 2

FIELD SAMPLING AND SAMPLE PREPARATION

Site Selection

The key to successful soil characterization is in site selection and sample collection. The purpose of any soil sample is to obtain information about the characteristics of a soil (SSIS, 1991). The site should be representative of the soil or feature being studied and be located away from roads, fence rows, old farmsteads, and any other features that may cause aberrant soil properties (SSIS, 1991).

Sampling schemes include duplicate or paired pedons located 1.5 to 30 km (1 to 20 mi) apart in different mapping delineations, transects across the landscape, satellite pedons, and random pedons selected from the same or different mapping units. The sampling scheme used will depend on the nature of the study and the tests being undertaken. No attempt is made to discuss these schemes in detail here. The reader is referred to Mausbach and Wilding (1991), Petersen and Calvin (1986), and SSIS (1991) for more detail on sample selection and statistical analysis.

Descriptions of the site and the soil are essential in understanding and interpreting the data. See SSS (1990, 1991) for conventions in soil and site descriptions.

Sampling Pedons

Sampling Materials and Equipment

1. Backhoe, shovels, or other means to excavate a pit. (While a bucket auger or probe can be used, it may be difficult at times to determine whether the sample is truly representative of a particular horizon. Whenever possible, sample from a pit large enough to expose an entire pedon.)
2. Sample bags, 4 liter plastic, with tags and labels.
3. Sieve, 19 mm (3/4 in) openings.

Sampling Procedure (adapted from SSIS, 1991)

Sample freshly dug pits or, if the excavation has already been opened, cut back to expose a fresh soil face. The pit should be at least 1 by 2 m across and extend either (a) 2 to 3 m deep, (b) into the C horizon if it is deeper, or (c) to bedrock if it is shallower. Describe the exposed soil and mark horizons for sampling. Some pedons have cyclic variations that are larger than the pit and may require supplemental borings or excavations for a complete evaluation.

Care must be taken in describing soils which have weathered rock in the lower part of the profile. Observations in soils weathered from schist rock in the Piedmont of southeast Pennsylvania found that the sidewalls of pits dug with a backhoe were disturbed by the bucket as it dragged across the face of the pit. The resultant morphology of the lower part of the soil differed significantly in the sidewalls than in the headwall of the pit.

Sample each horizon, working from the bottom of the pit to the surface to minimize contamination. First sample clods from the center of the horizon for bulk density and related fabric analyses first (see Chapter 7 for the sampling procedure). Next take a 3- to 5-kg bulk sample (fill approximately three-fourths of a 4 liter bag) representative of the horizon.

In laterally uniform pedons, sample a 30- to 50-cm wide vertical section. For horizons that are discontinuous or vary greatly in thickness or degree of expression, collect samples from different parts of the pedon or pit face. Estimate the proportions of any contrasting soil materials, noting this in the pedon description, and sample the components separately, if reasonable. Split large horizons (>30 cm thick in the upper pedon or >60 cm in the lower pedon) into arbitrary subhorizons, considering soil classification requirements in locating subhorizon boundaries.

Sampling Soils with Rock Fragments >19 mm (3/4 in) in Diameter

Rock fragments are particles 2 mm or larger in diameter that are not attached to bedrock. Unlike the term "coarse fragments," which excludes stones and boulders larger than 254 mm (10 in) in diameter, rock fragments include all sizes that have horizontal dimensions less than the size of a pedon. The largest size rock fragment that can be included in a sample depends on the size of the sample. As a general rule of thumb, a sample should be 100 to 200 times the size of the largest particle sampled. For a 3- to 5-kg sample, the largest size rock fragment sampled is 19 mm (3/4 in).

Record in the pedon description and on Page 1 of the Lab Input Form (see Appendix A) the volume percentage estimates of rock fragments >254 mm (10 in), 76 to 254 mm (3 to 10 in), 19 to 76 mm (3/4 to 3 in), and 2 to 19 mm (2 mm to 3/4 in) in diameter. Volume estimates of the <2 mm fractions may also be recorded. Sieve out all rock fragments >19 mm in diameter and bag the <19 mm field sample.

Labelling Samples

Mark the depth, horizon symbol (e.g., Ap), and pedon code number on 2 tags. Put one tag in the bag and tie the second tag to the outside of the bag. Label clods with a tag clipped to the hairnet. The pedon code number (e.g., S92-PA-014-010-01) includes the year sampled (92), state code (PA), county number from Table 2.1 (14), pedon number within the county(10), and horizon number in numeric order from the surface down(01). All organic (O) horizons are included in the numbering sequence.

Laboratory Preparation

Soil samples are air-dried, sieved to <2-mm, mixed to homogenize the soil, and subsampled for analysis. The remainder of the sample is stored. The 2 to 19 mm rock fragments are separated and weighed for later calculations (Chapter 6). This process creates a lot of dust and is done in a preparation room separate from the analytical laboratory. Wear a dust mask, eye protection (goggles), and protective clothing while working in the dusty environment.

Materials and Equipment

1. Drying trays and brown paper for spreading and drying samples
2. Wooden rolling pin and rubber roller
3. Sieves, <4.7 mm and <2 mm
4. Balance
5. Sample splitter
6. Sample cartons, pint and gallon size

Table 2.1. County numbers used in the pedon sample code numbers for Pennsylvania.

No.	County	No.	County	No.	County
1	Adams	24	Elk	47	Montour
2	Allegheny	25	Erie	48	Northampton
3	Armstrong	26	Fayette	49	Northumberland
4	Beaver	27	Forest	50	Perry
5	Bedford	28	Franklin	51	Philadelphia
6	Berks	29	Fulton	52	Pike
7	Blair	30	Greene	53	Potter
8	Bradford	31	Huntingdon	54	Schuylkill
9	Bucks	32	Indiana	55	Snyder
10	Butler	33	Jefferson	56	Somerset
11	Cambria	34	Juniata	57	Sullivan
12	Cameron	35	Lackawanna	58	Susquehanna
13	Carbon	36	Lancaster	59	Tioga
14	Centre	37	Lawrence	60	Union
15	Chester	38	Lebanon	61	Venango
16	Clarion	39	Lehigh	62	Warren
17	Clearfield	40	Luzerne	63	Washington
18	Clinton	41	Lycoming	64	Wayne
19	Columbia	42	McKean	65	Westmoreland
20	Crawford	43	Mercer	66	Wyoming
21	Cumberland	44	Mifflin	67	York
22	Dauphin	45	Monroe		
23	Delaware	46	Montgomery		

Procedure

Spread the field samples out on trays (or sheets of brown paper) and air-dry at 25-35°C (normally 3 to 7 days). Roll the sample with a wooden rolling pin to break up clods, taking care not to destroy rock fragments (use a rubber roller for easily-crushed rock fragments). Pass the rolled sample through sieves to separate the <2 mm, 2 to 4.7 mm, and 4.7 to 19 mm fractions. Continue rolling and sieving until only rock fragments that do not slake in water or sodium metaphosphate dispersant remain on the sieves. Weigh each size fraction and record the weights on page 1 of the Lab Input Form. These weights will be used to calculate the percentages of the various fractions (Chapter 6).

Thoroughly mix and pass the <2-mm fraction through a sample splitter, collecting enough to fill a properly-labelled pint-size sample carton. This subsample will be used for laboratory analyses. Place the remainder of the sample, along with the bagged rock fragments, in a labelled gallon carton for storage. Label the pint and gallon cartons with the soil number (e.g., S92-PA-014-010-01), depth and horizon.

References

Mausbach, M. J. and L. P. Wilding. 1991. Spatial variability of soil and landforms. SSSA Spec. Publ. No. 28. Soil Sci. Soc. of Amer. Madison, WI.

Petersen, R. G., and L. D. Calvin. 1986. Sampling. p. 33-51 in A. Klute (ed.). Methods of soil analysis. Part 1. Physical and mineralogical methods. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Soil Survey Investigations Staff (SSIS). 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rpt. No. 42. Version 1.0. National Soil Survey Center. Lincoln, NE.

Soil Survey Staff (SSS). 1990. Keys to soil taxonomy. SMSS Tech. Monograph No. 19. 4th ed. Virginia Polytech. Inst. and State Univ. Blacksburg, VA.

Soil Survey Staff (SSS). 1991. Soil survey manual (draft). USDA Soil Conservation Service. Washington, DC.

CHAPTER 3

GENERAL LABORATORY PROCEDURES AND CONDUCT

Introduction

The Soil Characterization Laboratory serves several functions. The laboratory is committed to assisting graduate students and faculty in basic soils research, characterizing soils in conjunction with the Cooperative Soil Survey, and advancing the knowledge of soil science through various activities within the laboratory. This commitment includes making the laboratory facilities available to graduate students and faculty who need to do certain basic soil characterization analyses and performing various analyses for others. The amount of assistance given, however, is determined at any particular time by the existing workload priorities and available resources.

Safety, courtesy to others, and quality control are essential to the operation of the laboratory and successful completion of laboratory analyses. These general guidelines and procedures are intended to ensure the safety of all persons who use the facilities, the efficient functioning of the laboratory, and the maintenance of quality control. Adherence to these rules of "laboratory etiquette" are particularly critical when several persons may be utilizing the facilities simultaneously. Failure to do so will result in a loss of laboratory privileges (such a decision, made by the laboratory director as approved by the soil characterization project leader, can be made at any time, with or without prior notice).

The following guidelines of common sense conduct for safety, consideration and cooperation are expected of people working in the laboratory. The list is not all-inclusive and further guidelines for conduct may be added as situations arise.

General Considerations

1. For scheduling purposes, notify the laboratory director at least one (1) week in advance of when you wish to do work in the laboratory. Provide information on the number of samples and type of analyses involved, the estimated duration of laboratory utilization, and the purpose and use of the data to be generated.

2. The laboratory is available only for those basic soil characterization procedures that are described in this manual and are impractical to do elsewhere.

3. Determine what chemicals, reagents, and materials will be required to complete the analyses. Check the available inventory and order needed supplies through the laboratory director. Plan ahead to allow enough time for the supplies to arrive.

4. Items in the laboratory may be borrowed only with the consent of the laboratory director (a sign-out system is used).

5. Return all tools, equipment, glassware, chemicals, and other items to their proper place immediately after you finish using them.

6. Include sufficient time in your schedule to clean-up the same day. Do not leave a dirty work space or dirty glassware - you are responsible for your own clean-up.

7. Work study or hourly workers are not available to assist graduate students in the lab unless directed to do so by the laboratory director.

Safety

1. Know where the first aid kit, showers, eye-wash station, fire extinguisher, and other safety items are located before you need them.

2. Be aware of the particular safety hazards of the chemicals (printed on the containers) and equipment (check the operating manual) used in the analyses and take all appropriate precautions. Inspect equipment before using to make sure it is in good repair. Replace any damaged parts before using.

3. Always wear rubber gloves, a protective apron, and an eye and face shield when handling acids or bases. Thoroughly wash hands after handling reagents.

4. Mix all reagents that involve acids, bases, volatiles, or other noxious chemicals under a fume hood. Never leave reagents open in the general work area.

5. Clean up any spills immediately. For spills involving concentrated reagents or toxic chemicals, notify the laboratory director and University safety personnel immediately. Use water and sodium bicarbonate to neutralize spilled acids.

6. Always use a pipette bulb to draw reagents into a pipette. Pipetting by mouth may be hazardous: acids and bases can cause severe internal damage if swallowed; metal salts are toxic and may be fatal if ingested. Similarly, keep all food and drink away from the work area to prevent contamination.

7. Wear protective gloves and use tongs, and heat-resistant holders when working around an oven, hot plate, hot water bath, or burner. Keep flammable materials away from an open flame.

Soil Samples

1. Process (mixing, sieving, grinding, splitting) all bulk samples in a preparation room and not in the laboratory. Any procedure that creates dust will be done in a separate processing room or under an exhaust hood.

2. Clearly label all samples (see Chapter 2).

3. Place the samples in a temporary storage space assigned by the laboratory director. Remove the samples once the analyses are completed. Samples may be stored in the lab for over 1 month while not being analyzed only with the consent of the lab director and only as space allows).

4. To keep the work space and equipment open for use, take samples out of the storage area only when you are weighing them out for an analysis. Store samples that are pending analysis or between treatments in an area designated by the laboratory director.

Laboratory Reagents and Chemicals

1. Remove chemicals and reagents from the cabinets only as required to prepare reagents or perform an analysis. Return the chemicals to storage as soon as you finish using them. Chemicals and reagents are generally stored in alphabetical order and should be returned their proper place.

2. Notify the director when any chemical appears to be in short supply so that it can be restocked for future use.

3. Use only distilled and deionized water (D&D H_2O) to prepare reagents, perform analyses, and provide a final rinse for glassware. Use deionizing columns to provide a supply of D&D H_2O with less than 2 ppm salts (as NaCl), adjusting the flow rates through the column to meet this water quality standard.

4. Label all mixed reagents, standard solutions, etc., to identify exactly what it is, who prepared it, and when it was prepared. Keep the solutions in a designated storage area, taking them out only when needed and putting them away in a timely manner.

5. When the analysis is complete, properly dispose of all mixed reagents and clean and put away the containers.

6. To avoid potential contamination, never return any reagent or chemical to its original container once it has been removed.

Labware

1. Remove labware from the storage cabinet or drawer only as needed. Labware may be kept in a temporary storage area only when it is being utilized.

2. Use a non-permanent magic marker or grease pencil to mark glassware. Do not mark on the rough circles or leave permanent markings on the glassware. Use a marking tape to label plasticware; avoid marking directly on the plasticware.

3. Clean and put away all labware immediately after the analysis is completed (within one day). Remove all markings and tape. Clean the labware with a brush in hot soapy water. Rinse three times with hot tap water and then three times with D&D H_2O. Use an HCl solution to remove stains, films, or precipitates. After cleaning, soak pipettes, extract storage bottles, and glassware that will be used for sensitive chemical analysis in 0.5 to 1 N HCl and rinse the acid-washed labware three times with D&D H_2O. Place the clean labware on a drying rack and put away immediately after drying. Unwashed or partially cleaned glassware should not be left behind.

4. Discard permanently stained or streaked, cracked or broken glassware.

5. Wash the mechanical extractor tubes in a separate sink to prevent contamination with the silicon lubricant. Leave the label markings on the tubes and clean them in the manner as you would clean other labware.

Equipment

1. Before using any equipment in the laboratory, become familiar with the operating procedure. Only use the equipment for its intended use.

2. Inspect and maintain all equipment being used for analysis. This includes replacing damaged, worn or broken parts, cleaning, oiling and lubricating machinery, greasing stopcocks, etc. Repair and/or report any equipment breakage to the laboratory director immediately.

3. Clean the equipment with a damp sponge immediately after use. Clean any spills immediately.

4. Shut off all equipment not in use. Relevel balances, set to zero and shut off after cleaning. Leave the pH meter on standby/off. Turn off all gases and bleed the pressure from the lines.

5. Keep liquid or other spillable materials away from any piece of electronic equipment.

Workspace

1. Use only the area(s) in the laboratory designated for a particular analysis.

2. Clean up all spills, broken glass, paper, soil, etc. immediately.

3. At the end of each day, clean the work space with soapy water and a sponge, and mop the area if necessary.

4. Rinse and clean the sink after use.

5. Keep all cabinet and cupboard doors closed.

6. Do not tamper with, rearrange, discard or otherwise disturb anything in the laboratory that does not belong to you.

Quality Control

1. Pay close attention to the procedures and follow the steps closely. A major source of error comes from inconsistencies in reagent preparation and sample treatments between runs. Variability between different analysts for the same procedure can be minimized if each analyst closely adheres to the procedure as written.

2. Always use clean glassware and materials. When in doubt, clean them again.

3. Run a laboratory standard soil sample with each run and compare the results with published data (Ciolkosz and Dobos, 1991). Repeat any runs in which the standard falls outside a standard deviation of the published mean.

Reference

Ciolkosz, E. J., and R. R. Dobos. 1991. Pennsylvania State University soil characterization laboratory data summary for standard samples. Penn State Univ. Agron. Series No. 112.

CHAPTER 4

MOISTURE CORRECTION FACTOR

Background and Theory

Soil properties are typically expressed on the basis of an oven-dry weight. Since air-dry samples are analyzed, a moisture correction factor is used. For some procedures, such as bulk density, moisture retention, and particle size, the oven-dry weight is determined as a part of the process. In other procedures, particularly the chemical analyses, a correction factor must be calculated.

A representative sample of air-dry soil is weighed, dried at 105°C for 24 hr, and weighed again to obtain the oven-dry weight. The air-dry weights of samples used in subsequent analyses can be converted to an oven-dry weight by using the appropriate moisture correction factor.

Materials and Equipment

1. Balance, sensitive to 0.001 g
2. Oven
3. Drying cans

Procedure

1. Weigh the drying can and record the CAN WEIGHT to the nearest 0.001 g on page 6 of the Laboratory Data Input Form (see Appendix A).

2. Add a representative sample (10 to 20 g) of <2-mm, air-dry soil, recording the can plus air-dry soil weight (CAN + ADSOWT) to the nearest 0.001 g.

3. Dry the soil plus can at 105°C for a minimum of 24 hr.

4. Remove the oven-dry samples and cool them in a dessicator.

5. After the samples have reached room temperature, record the can plus oven-dry soil weight (CAN + ODSOWT) to the nearest 0.001 g.

Calculations

1. The moisture correction factor (MC) is calculated by adding the moisture content of the air-dry soil, expressed as a fraction of the oven-dry weight, to 1:

$$MC = 1 + \frac{\text{(air-dry soil wt. - oven-dry soil wt.)}}{\text{oven-dry soil wt.}}$$

2. The air-dry soil weight is divided by the moisture correction factor (MC) to obtain the oven-dry weight.

3. Calculations for the moisture correction factor and air-dry to oven-dry conversions are included in the soil characterization laboratory database program (Appendix B).

CHAPTER 5

PARTICLE SIZE, <2 mm -- PIPETTE METHOD

Background and Theory

The distribution of sand, silt, and clay in the soil affects many important soil properties and behavior, including water and gas movement in the soil, water and nutrient retention, and erodibility. Particle size distribution is a powerful tool in the study of soil genesis and morphology, particularly in determining the potential existence of argillic horizons and the continuity of parent materials.

Organic matter, which binds together soil particles, is destroyed and the soil particles are dispersed. Sand-sized particles are separated by sieving and silt- and clay-sized particles are determined by the pipette method. Stokes' Law -- particles of similar density but different size will settle out of suspension at different rates -- provides the basis for the pipette method (Day, 1965; Tanner and Jackson, 1947). The settling rate, V, (cm/sec) calculated by Stokes' Law is:

$$V = \frac{X^2 g(P_S - P_L)}{18m}$$

where X is the particle diameter (cm), g is the acceleration due to gravity (cm/sec^2), P_S is the particle density (g/cm^3), P_L is the liquid density (g/cm^3), and m is the liquid viscosity (dyne sec/cm^2). Thus all silt-sized particles in a suspension will have dropped below the sample depth after a certain period of time, leaving only clay-sized particles in their original concentration. The assumptions and limitations of Stokes' Law can be found in most soil physics textbooks.

The pipette analysis should be carried out in a vibration-free room with a constant temperature. Vibrations and temperature fluctuations will affect the settling rate of particles, leading to errors in determination.

The percentages of total clay (<2 um); fine (2-5 um), medium (5-20 um), and coarse (20-50 um) silt; and very coarse (1.0-2.0 mm), coarse (0.5-1.0 mm), medium (0.25-0.5 mm), fine (0.10-0.25), and very fine (0.05-0.07 and 0.07-0.10 mm) sand are routinely determined in the soil characterization laboratory. Although the coarse (0.2-2um) and fine (<0.2 um) clay contents are not routinely determined, a procedure is included for this separation. Fine clay data can be helpful in determining the presence of illuviated clay and, thus, an argillic horizon (SSIS, 1992).

Materials and Equipment

1. Fleakers, 300 ml, tared to nearest 0.01 g, stoppered lids and watch glass covers
2. Hot plate
3. Filter candles, Pasteur-Chamberlain, fineness "F"
4. Filtering apparatus (Figure 5.1)
5. Oven
6. Balance, capable of reading to nearest 0.001 g
7. Reciprocating shaker
8. Sedimentation cylinders, 1000 ml
9. Pipette weighing bottles, polypropylene, 60-70 ml, tared to nearest 0.001 g

10. Sieve, 300 mesh (0.046 mm), 76-mm (3 in) diameter brass, with a funnel mounted on a ring stand
11. Lowry pipette, 25 ml automatic, with vacuum and rack (Fig. 5.2)
12. Nest of 76-mm diameter sieves, Nos. 18 (1.0 mm), 35 (0.5 mm), 60 (0.25 mm), 140 (0.105 mm), 200 (0.074 mm), and 300 (0.046 mm)
13. Sieve shaker
14. Beakers, 250 ml, or weighing pans, tared to nearest 0.01 g

Chemicals and Reagents

1. Hydrogen peroxide (H_2O_2), 30%.
2. Hydrogen peroxide (H_2O_2), 10%. Mix one part 30% H_2O_2 with two parts H_2O.
3. Sodium acetate (NaOAc) buffer (pH 5.0), only for samples with a pH $\geq$ 6.0. Mix 136 g of $NaOAc \cdot 3H_2O$ and 27 ml of glacial acetic acid (HOAc) per liter. Adjust to pH 5.0 with either HOAc or NaOAc.
4. Dispersing agent (sodium hexametaphosphate solution). Mix 35.7 g of sodium metaphosphate ($NaPO_3$) and 7.9 g of sodium carbonate (Na_2CO_3) per liter.
5. Use distilled and deionized H_2O throughout the procedure.

Overview of the Procedure

Step	Result	Approx. Time
Treat with H_2O_2	Destroys organic matter binding soil	4-6 hr
Candle/ filter samples	Removes dissolved minerals, organic matter	2-3 hr
Dry in oven, weigh	Oven-dry soil wt.	overnight
Add dispersing agent, shake	Disperses soil particles	overnight
Pass sample through a 300-mesh sieve	Separates sand from silt, clay	1-2 hr
Pipette <20 um, <5 um, <2 um fractions	Samples silt, clay fractions	full day (1 hr/ pipetting)
Dry sand, pipette samples in oven	Removes water	overnight
Weigh dried pipette samples	Oven-dry silt, clay wts.	1 hr
Sieve sands, weigh fractions	Sand fractions	3-4 hr

A typical sample run consists of 11 samples and 1 standard (12 total). Allow 1 to 2 days for the pretreatment to remove organic matter and dissolved minerals. The samples are dispersed overnight and the pipetting procedure takes a full day for one run. The sand fractions can be sieved at any time after drying.

Separation of the fine clay (<0.2 um) fraction consists of centrifuging an aliquot of the silt and clay suspension. Allow an additional 2 to 3 hours after the final pipetting for this step (this can be done the following day).

Removing Organic Matter

1. Weigh 10 g of air-dry soil into a pre-weighed fleaker (record as FLK WT on page 2 of the Laboratory Data Input Form; see Appendix A).

2. For O and A horizons with a pH of 6.0 or higher add about 20 ml of pH 5.0 NaOAc buffer. This step is not necessary for soils with lower pH values or for subsoils.

3. Add 25 ml of 10% H_2O_2 to the sample and cover the fleaker with a smooth watchglass. Let the sample sit without heating until the initial reaction ceases.

4. Heat the samples to 80-90°C on a hot plate. When the reaction stops bubbling, add 25 ml of 10% H_2O_2. Cover with a watch glass. Remove any samples that begin to boil over from the heat, and use a glass rod or water from a squirt bottle to break up the bubbles.

5. After the reaction subsides, add 5 ml of 30% H_2O_2 to the fleaker. Continue to heat the samples until the reaction subsides, taking care that no samples boil over.
   NOTE: For problem samples, treat the sample in a larger beaker and then transfer the sample to the fleaker after the initial violent reaction.

6. Continue this treatment until the reaction subsides. B and C horizons (except Bh horizons) will not require additional treatment. A horizons may require 1 to 2 additional treatments (until lighter in color).

Removing Dissolved Mineral and Organic Components

1. Clean all soil particles from the watchglass and the sides of the fleaker with a rubber policeman and H_2O.

2. Fill the fleakers half-full with H_2O and place in the filtering apparatus. Place the filter candles into the fleakers and fill the fleaker up to its neck with H_2O.
   NOTE: Soak the candles beforehand to wet the ceramic material. This speeds up the initial candling.

3. Apply a vacuum to the filter candles as follows (see Figure 5.1):
   a. Turn on the vacuum source and open the vacuum valve.
   b. Turn the 3-way flow valve so that the direction arrow points toward the collection bottle and away from the candling rack.
   c. Connect the filter candles to the vacuum and open the candle valves.
   d. Continue to apply suction with the vacuum until approximately 50 ml of H_2O is left in the fleaker (rates will vary with texture and condition of the candle) and then close the valve to that fleaker.

4. After candling, backflush the samples to remove soil from the filter candle:
   a. Fill the wash bottle at least 1/3 full with water.
   b. Adjust the wash regulator to 12.5 cm of Hg (5 psi). Do not exceed 5 psi or the hoses may blow off.
   c. Turn the 3-way flow valve so that the flow direction arrow is pointing toward the candles.
   d. Open the candle valve for one sample and allow water to be forced through until the candle "sweats" to the top.
   e. Use water and rubber policeman to dislodge soil from the candle. Once the candle is clean, close the candle valve and backflush the next sample.

Figure 5.1. Filtering apparatus.

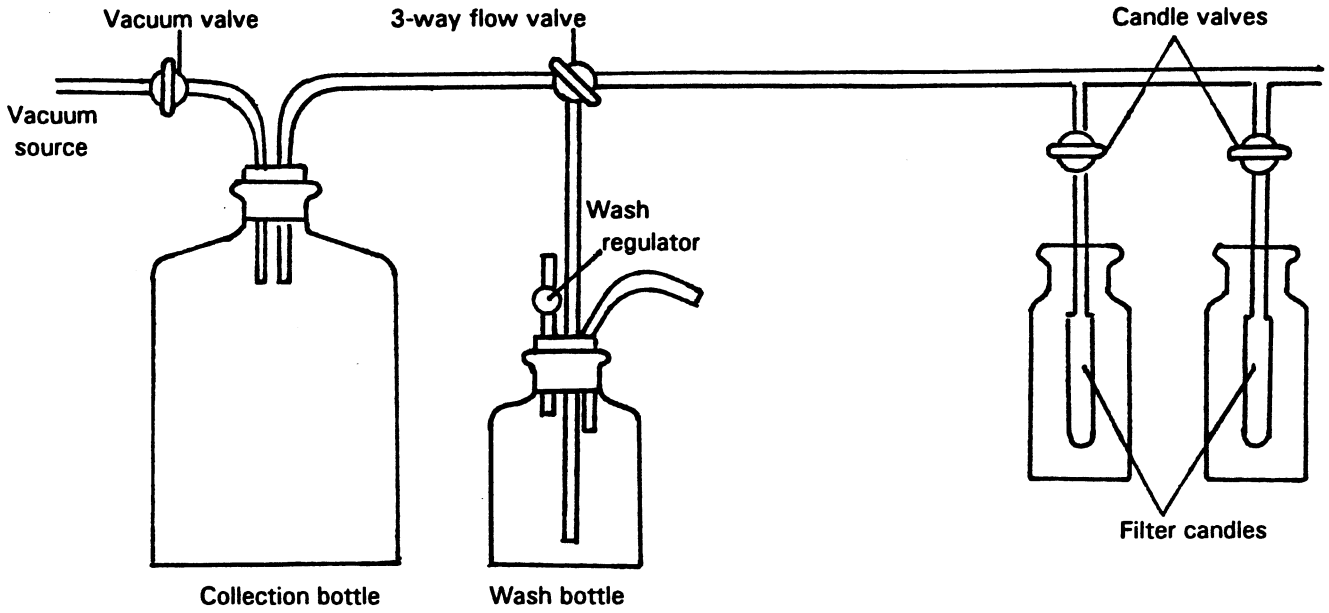
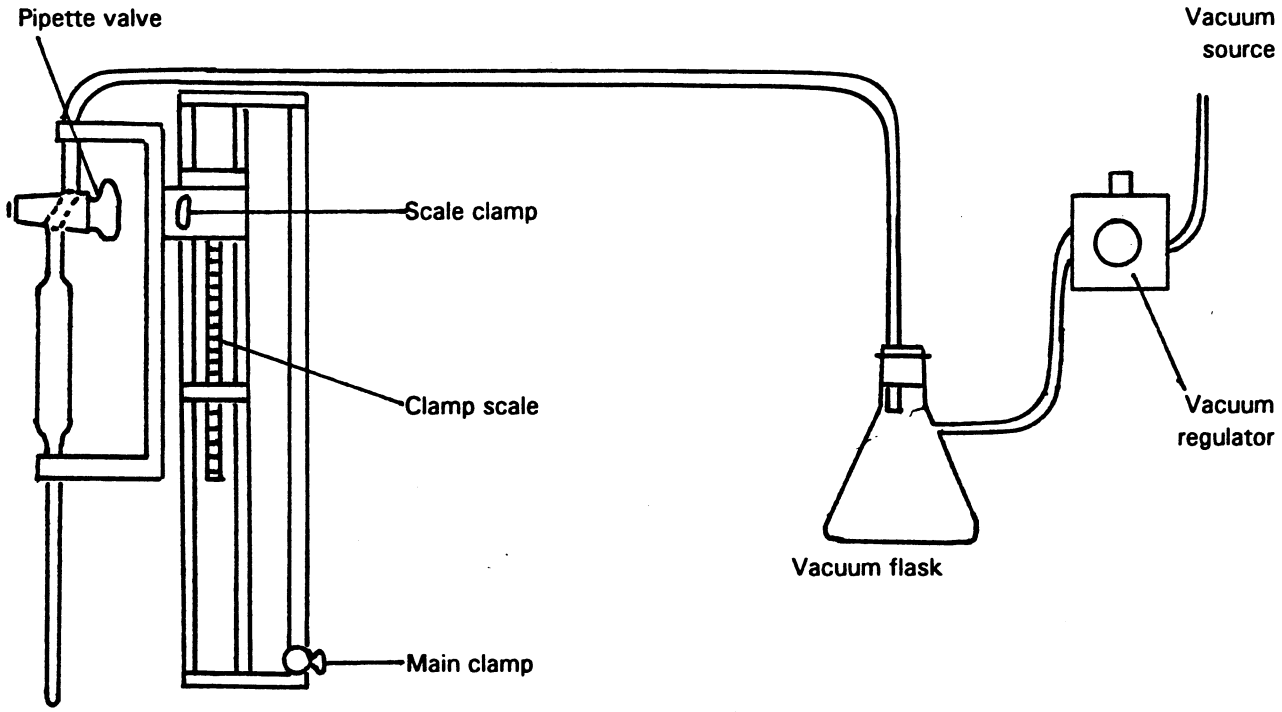


Figure 5.2. Pipetting apparatus.



Lowry pipette

6. Reverse the direction of the 3-way flow valve so that the flow direction arrow points towards the collection bottle. Repeat the candling (steps 2 through 4) and backflush (step 5) process at least two more times, stirring the sample before candling. The sample should be dispersed and not settle out of suspension when fresh water is added to the fleaker. Do not refill the fleaker after the last backflush.

7. Remove the samples from the candling rack and clean the soil from the fleaker neck and sides (easier done now than after drying in the oven).

8. Dry the samples at 105°C for 12 hours after the H_2O has evaporated.

Dispersing the Sample

1. Cool the oven-dry samples in a desiccator, and weigh them to the nearest 0.01 g. Record the fleaker plus soil weight (FLK PSOWT) on the Lab Input Form.

2. Add 10 ml of dispersing agent to each fleaker, fill them to the 200 ml mark with distilled water, and cap tightly with a rubber fleaker lid.

3. Shake the samples overnight (minimum 6.5 hr) at low speed (120 oscillations/min) on a reciprocating shaker. Load 12 fleakers end to end (4 wide, 3 long), tightly securing the samples with the adjustable bar (fleakers that are not fastened securely may come loose and break during shaking).

Separating the Sand From the Silt and Clay Fractions

1. Remove the samples from the shaker (an early start is recommended since the pipette procedure takes all day to run from start to finish).

2. Place a 300-mesh sieve in a large glass funnel on a rack and place a numbered 1000-ml sedimentation cylinder underneath the funnel.

3. Shake the sample in the fleaker, remove the lid and rinse the soil from the lid into the sieve in the funnel. Pour the remainder of the sample onto the sieve, rinsing the fleaker with water to wash out all of the sample.

   NOTE: Wet the sieve beforehand to allow water to pass through more readily. If the water does back up, hold the sieve firmly in the funnel and tap on the side of the funnel.

4. Wash the sample on the 300 mesh sieve with water until all of the <300-mesh sample passes through (water passing through the sieve will be clear) and fill the cylinder to the 1000 ml mark.

5. Cover the cylinder with a watch glass and move it to the pipetting rack for analysis.

6. Transfer the sand retained on the 300-mesh sieve into a 250-ml beaker by inverting the sieve and rinsing the sand into the beaker. Allow the sand to settle and pour off the clear water.

7. Dry the sand and beakers overnight at 105°C. The dry sands can be sieved at any time (See Sieving the Sand Fractions).

8. Continue until all of the samples are sieved. The silt and clay fractions will now be in the sedimentation cylinders ready to pipette.

Preparing For the Pipette Procedure

1. Before pipetting the samples, you should become familiar and comfortable with the pipette apparatus (Figure 5.2) and the procedure. Practice with water samples until you can quickly raise and lower the pipettes, operate the two way valve on the Lowry pipette, and adjust the rate at which the pipette takes up the sample. You should be able to perform the rapid sequence of steps involved in pipetting the samples in a smooth, error-free manner.

2. Adjust the rate at which the pipette draws up a sample (refer to Figure 5.2):
   a. Open the vacuum source and adjust the vacuum regulator to 10 centibars.
   b. Close all pipette valves.
   c. Open the valve of one pipette and draw water from a beaker, measuring the time it takes to fill the pipette. Adjust the vacuum until the pipette draws a 25-ml sample in about 12 sec.

3. Position the sedimentation cylinders so that each cylinder is accessible by a pipette. Keep a watch glass on top of each cylinder except when stirring or pipetting.

4. Adjust the sample depth to 10 cm on the clamp scale.

5. Keep an extra cylinder of water nearby to rinse the stirrer between samples.

6. Fill a small beaker (50-100 ml) with acetone and a second beaker with water to rinse the pipette between samples.

7. Record the temperature of a cylinder containing water (equilibrated to the temperature of the pipette room) to determine the settling times for the <20-um and <5-um fractions and the sampling depth for the <2-um fraction (Table 5.1).
   NOTE: Use a worksheet similar to Table 5.2 to note the temperature and settling times and set up a schedule for stirring and pipetting (5-minute intervals for stirring and sampling work well) and keep track of sample and pipette bottle numbers.

8. For each sample, weigh three weighing bottles to the nearest 0.001 g (this should be done in advance) and record the <20-um (BWT 20UM), <5-um (BWT 5UM), and <2-um (BWT 2UM) bottle weights on the Lab Input Form.
   NOTE: The plastic bottles will crack over time with continued oven-drying. Inspect the bottles and discard any that show signs of cracks.

9. Pipette 10 ml of dispersing agent into each of three pre-weighed bottles. Dry the bottles overnight at 105°C, cool in a desiccator, and weigh them to determine the weight of the sodium hexametaphosphate in the bottles. This weight is divided by 40 (dilution factor equal to a 25-ml aliquot from a 1000-ml solution) to determine weight of the dispersing solution in each sample aliquot. The average of the three values is recorded as BLANK WT on the Lab Input Form.

Timing the Pipetting Sequence

The pipetting sequence of lowering the pipette into the suspension, taking a sample, pouring the sample and a rinse into a weighing bottle, and cleaning the pipette is described for one sample. During the initial pipetting (<20 um sample), you are faced with an overlap in steps, stirring with one hand while adjusting the pipette and sampling with the other. Advanced planning and practice are essential.

Table 5.1. Sampling times for 20-um and 5-um fractions, 10-cm sampling depth, and depths for 2-um fractions, 6.5 hr sample time.

Temp. °C	20 um time min : sec	5 um time min : sec	2 um depth (cm)	Temp. °C	20 um time min : sec	5 um time min : sec	2 um depth (cm)
15.0	5 : 23	86 : 23	7.17	23.0	4 : 27	71 : 10	8.77
15.2	5 : 22	85 : 57	7.21	23.2	4 : 26	70 : 49	8.81
15.4	5 : 20	85 : 31	7.25	23.4	4 : 24	70 : 31	8.85
15.6	5 : 19	85 : 03	7.29	23.6	4 : 23	70 : 12	8.89
15.8	5 : 17	84 : 37	7.33	23.8	4 : 22	69 : 53	8.93
16.0	5 : 15	84 : 11	7.37	24.0	4 : 21	69 : 32	8.97
16.2	5 : 14	83 : 46	7.41	24.2	4 : 20	69 : 14	9.01
16.4	5 : 12	83 : 20	7.45	24.4	4 : 18	68 : 55	9.05
16.6	5 : 11	82 : 56	7.49	24.6	4 : 17	68 : 37	9.09
16.8	5 : 09	82 : 32	7.53	24.8	4 : 16	68 : 18	9.14
17.0	5 : 08	82 : 11	7.57	25.0	4 : 15	67 : 58	9.18
17.2	5 : 06	81 : 49	7.61	25.2	4 : 14	67 : 41	9.22
17.4	5 : 05	81 : 25	7.65	25.4	4 : 13	67 : 23	9.26
17.6	5 : 05	81 : 02	7.69	25.6	4 : 12	67 : 05	9.30
17.8	5 : 02	80 : 38	7.73	25.8	4 : 10	66 : 46	9.34
18.0	5 : 00	80 : 14	7.77	26.0	4 : 09	66 : 27	9.39
18.2	4 : 59	79 : 50	7.81	26.2	4 : 08	66 : 10	9.43
18.4	4 : 58	79 : 28	7.86	26.4	4 : 07	65 : 53	9.47
18.6	4 : 56	78 : 04	7.89	26.6	4 : 06	65 : 36	9.51
18.8	4 : 55	78 : 40	7.93	26.8	4 : 05	65 : 18	9.56
19.0	4 : 54	78 : 18	7.97	27.0	4 : 04	64 : 59	9.60
19.2	4 : 52	77 : 55	8.01	27.2	4 : 03	64 : 43	9.64
19.4	4 : 51	77 : 32	8.05	27.4	4 : 02	64 : 25	9.69
19.6	4 : 49	77 : 10	8.09	27.6	4 : 00	64 : 08	9.73
19.8	4 : 48	76 : 48	8.13	27.8	3 : 59	63 : 51	9.77
20.0	4 : 47	76 : 26	8.16	28.0	3 : 58	63 : 35	9.81
20.2	4 : 45	76 : 03	8.20	28.2	3 : 57	63 : 17	9.86
20.4	4 : 44	75 : 41	8.25	28.4	3 : 56	63 : 01	9.90
20.6	4 : 42	75 : 19	8.29	28.6	3 : 55	62 : 45	9.95
20.8	4 : 41	74 : 58	8.32	28.8	3 : 54	62 : 29	9.99
21.0	4 : 40	74 : 36	8.36	29.0	3 : 53	62 : 13	10.03
21.2	4 : 38	74 : 14	8.41	29.2	3 : 52	61 : 56	10.07
21.4	4 : 37	73 : 52	8.45	29.4	3 : 51	61 : 40	10.12
21.6	4 : 36	73 : 32	8.49	29.6	3 : 50	61 : 24	10.16
21.8	4 : 34	73 : 10	8.53	29.8	3 : 49	61 : 09	10.20
22.0	4 : 33	72 : 51	8.57	30.0	3 : 48	60 : 54	10.25
22.2	4 : 32	72 : 29	8.61	30.2	3 : 47	60 : 38	10.29
22.4	4 : 31	72 : 10	8.65	30.4	3 : 46	60 : 23	10.33
22.6	4 : 29	71 : 49	8.69	30.6	3 : 45	60 : 08	10.38
22.8	4 : 28	71 : 39	8.73	30.8	3 : 45	59 : 52	10.42

Table 5.2. Example worksheet for the pipetting procedure, 12-sample run at 23.6°C, with (a) 4-minute and (b) 5-minute sampling intervals.

Date 5/14/92
Temp (am), °C 23.6
Temp (pm), °C 23.6

<20 um time 4:23
< 5 um time 1:10:12
< 2 um depth 8.89 cm

(a) Pipetting sequence with a 4-minute interval between starting times.

Sample No.	Begin Stirring	Stop Stirring	Lower Pipette	<20 um time	<5 um time	<2um time
1	8:59 am	9:00	9:03:23	9:04:23	10:10:12	3:30 pm
2	9:03	9:04	9:07:23	9:08:23	10:14:12	3:34
3	9:07	9:08	9:11:23	9:12:23	10:18:12	3:38
4	9:11	9:12	9:15:23	9:16:23	10:22:12	3:42
5	9:15	9:16	9:19:23	9:20:23	10:26:12	3:46
6	9:19	9:20	9:23:23	9:24:23	10:30:12	3:50
7	9:23	9:24	9:27:23	9:28:23	10:34:12	3:54
8	9:27	9:28	9:31:23	9:32:23	10:38:12	3:58
9	9:31	9:32	9:35:23	9:36:23	10:42:12	4:02
10	9:35	9:36	9:39:23	9:40:23	10:46:12	4:06
11	9:39	9:40	9:43:23	9:44:23	11:50:12	4:10
12	9:43	9:44	9:47:23	9:48:23	11:54:12	4:14

(b) Pipetting sequence with a 5-minute interval between starting times.

Sample No.	Begin Stirring	Stop Stirring	Lower Pipette	<20 um time	<5 um time	<2um time
1	8:59 am	9:00	9:03:23	9:04:33	10:12:51	3:30 pm
2	9:04	9:05	9:08:23	9:09:33	10:17:51	3:35
3	9:09	9:10	9:13:23	9:14:33	10:22:51	3:40
4	9:14	9:15	9:18:23	9:19:33	10:27:51	3:45
5	9:19	9:20	9:23:23	9:24:33	10:32:51	3:50
6	9:24	9:25	9:28:23	9:29:33	10:37:51	3:55
7	9:29	9:30	9:33:23	9:34:33	10:42:51	4:00
8	9:34	9:35	9:38:23	9:39:33	10:47:51	4:05
9	9:39	9:40	9:43:23	9:44:33	10:52:51	4:10
10	9:44	9:45	9:48:23	9:49:33	10:57:51	4:15
11	9:49	9:50	9:53:23	9:54:33	11:02:51	4:20
12	9:54	9:55	9:58:23	9:59:33	11:07:51	4:25

Table 5.2 illustrates a 12-sample pipetting sequence for 23.6°C with 4 and 5 minute starting intervals. With a 4-minute interval, you will be lowering the pipette to the 10-cm depth with one hand while stirring a second with the other. You will finish stirring in time to take the sample. With the 5-minute interval, you will be taking the sample with one hand while stirring a second sample with the other. The 4-minute interval allows sufficient time to take the sample, add a rinse to the bottle, and wash the pipette, provided you work quickly; the 5-minute interval allows more time between sampling. Choose the interval with which you feel most comfortable. After the first pipetting, the samples will not have to be stirred again.

Pipetting the <20 um Fraction

1. Stir the sample in the sedimentation cylinder for approximately 1 minute.
   NOTE: If the suspensions have been sitting for >24 hours, stir each sample for 6 to 8 minutes before starting the process.
   NOTE: Stir the samples with an up and down motion. Avoid circular motions which may cause a whirlpool that affects particle settling.

2. Record the time at which you stop stirring (this is the starting settlement time). The <20-um settling time (10-cm sample depth) from Table 5.1 is added to the starting time to determine the sampling time.

3. Lower the pipette with the main clamp (Fig. 5.2) so that the tip touches the top of the water in the cylinder. One minute before the sampling time, lower the pipette to the 10-cm depth with the scale clamp. Do not take the sample yet.

4. At the appropriate sampling time, open the pipette valve and draw a sample into the pipette. Close the valve and raise the pipette out of the cylinder.

5. Transfer the contents of the pipette into a labeled pipette weighing bottle. Add one rinse of water to the bottle.

6. Dry the pipette by rinsing with acetone, returning the acetone to its beaker (the acetone can be reused until it gets cloudy). Open the pipette valve to draw in air. Close the valve when the drops of acetone inside the pipette evaporate.

7. Continue stirring (rinse the stirrer between samples) and pipetting until each cylinder has been sampled.
   NOTE: To prevent potential contamination, cover the sedimentation cylinders with a watch glass except during stirring or pipetting.

8. Dry the pipetted samples overnight at 105°C, cool in a desiccator, and weigh them to the nearest 0.001 g. Record the oven-dry bottle plus <20-um sample weight (BP 20UMWT) on the Lab Input Form.

Pipetting the <5 um Fraction

NOTE: The pipetting procedure is exactly the same as that for the <20-um fraction, except that the samples are not stirred again.

1. Lower the tip of the pipette until it touches the top of the suspension. Approximately 1 minute before pipetting time (as determined from Table 5.1), lower the pipette to a 10-cm depth below the top of the liquid.

2. Pipette and rinse as described in Steps 4 to 6 above. Repeat until all cylinders have been sampled.

3. After this second pipetting, shut off the vacuum pump (the <2-um pipetting is not for several hours).

4. Dry the pipetted samples overnight at 105°C, cool in a desiccator, and weigh them to the nearest 0.001 g. Record the oven-dry bottle plus <5-um sample weight (BP 5UMWT) on the Lab Input Form.

Pipetting the <2-um Fraction

NOTE: For the <20-um and <5-um fractions, the sample depth is kept constant (10 cm) and the sample time is determined based on the cylinder water temperature. For the <2-um fraction, a sample time (6.5 hr) is selected and the sample depth is determined based on the water temperature.

1. Adjust the depth on the pipette clamps as indicated in Table 5.1. If the temperature has changed since the first pipettings, use the average of the temperatures to determine the sampling depth.

2. Turn on the vacuum and take the pipettings exactly the same way as with the earlier fractions, omitting the stirring step.

3. Dry the pipetted samples overnight at 105°C, cool in a desiccator, and them weigh to the nearest 0.001 g. Record the bottle plus oven-dry <2-um sample weight (BP 2UMWT) on the Lab Input Form.

Separating the Fine Clay (<0.2-um) Fraction

NOTE: Separating the fine clay (<2-um) fraction from total clay is not part of the routine soil characterization analyses. However, because the fine clay content is useful in soil genesis studies, these steps are included. The procedure is based on Jackson (1969) as described by SSIS (1991). The calculations for fine clay at the end of this chapter are not part of the soil characterization database program (Appendix B).

1. After completing the <20-um, <5-um, and <2-um fractions, stir the sample thoroughly for 5 minutes to bring the soil particles into suspension. Allow the suspension to settle for 15 min.

2. Pour the suspension into a 500-ml centrifuge bottle to a level that is 13 cm from the center of rotation. Measure the 13-cm distance from the center of rotation as follows:
   a. Place the centrifuge bottle in a holder in the centrifuge.
   b. With the centrifuge bottle and holder extended horizontally, measure a 13-cm distance out from the center of the centrifuge (this will be approximately equivalent to the top of the bottle holder).
   c. Mark this distance on the centrifuge bottle.

3. Stopper the bottle and shake the suspension. Remove the stoppers and load the centrifuge bottles into the centrifuge. Make sure that the loads are evenly balanced in the centrifuge.

4. Record the temperature of the suspension in the bottle. This will be used to determine the centrifuge time (Table 5.3).

5. The centrifuge time is calculated from an equation modified from Stokes' law (Jackson, 1969):

$$t_{min} = (63.0 \times 10^8 \times n \times \log(rs^{-1})) / (R^2 \times D^2 \times dp)$$

where

t_{min} = centrifuge time, minutes
n = viscosity, poises (Table 5.3)
s = distance from center of rotation to liquid surface (13 cm)
r = distance from center of rotation to sample depth (13 + 3 cm)
R = centrifuge rpm (1500)
D = particle diameter (0.2 um)
dp = difference in specific gravity between particles and liquid (Table 5.3)

Table 5.3. Centrifuge time for the <0.2-um clay fraction (SSIS, 1991).

Temp. (°C)	Viscosity (n)	Spec. grav. diff. (dp)	Cent. time (min)
20	0.01005	1.502	42.2
21	0.00981	1.502	41.2
22	0.00958	1.502	40.3
23	0.00936	1.502	39.2
24	0.00914	1.503	38.4
25	0.00894	1.503	37.6
26	0.00874	1.503	36.7
27	0.00855	1.503	35.9
28	0.00836	1.504	35.1
29	0.00818	1.504	34.3
30	0.00801	1.504	33.6

6. Centrifuge at exactly 1500 rpm for the time period determined from Table 5.3.

7. After centrifuging, lower a lowry pipette to a 3-cm depth in the supsension and follow the pipetting procedure to withdraw a 25-ml aliquot. Transfer the aliquot and a rinse to a pre-weighed pipette weighing bottle.

8. Dry the samples overnight at 105°C, cool in a dessicator, and weigh them to the nearest 0.001 g.

Sieving the Sand Fractions

1. Remove the sands from the oven and cool them in a dessicator.

2. Assemble two complete nests of sieves, cleaning the sieves with a fine-haired brush and inspecting for holes or rips. Replace damaged sieves.

3. Use a rubber policeman and a fine-haired brush to transfer the sand sample from the beaker to the top sieve.

4. Place the top on nest of sieves and secure the sieves in the sieve shaker. Shake the sieves for 3 minutes, transferring another sample to the second nest of sieves while the first is shaking.

   NOTE: If the sieves are not clamped squarely into the shaker, the nest may loosen and fly apart while shaking.

5. After the 3-min period, remove the sieves from the shaker, place a second nest of sieves on the shaker and begin shaking for 3 minutes.

6. Separate the sieves and weigh each sand fraction as follows:
   a. Place a container, such as a paper carton lid turned top-side down, on top of the balance and tare to 0.00 g with the lid.
   b. Brush the sand out of the No. 18 sieve (use a fine-haired brush to transfer all of the sand from the sieve). Record the very coarse sand weight (VCSANDWT) to the nearest 0.01 g on page 3 of the Lab Data Input Form.
   c. Do not discard the sand or re-tare the balance. Instead, add the sand from the No. 35 sieve to the container and record the cumulative sand weight (CSANDWT) to the nearest 0.01 g on the Lab Data Input Form.
   d. Continue until each sand fraction is added to the container and weighed. The final weight, VFSAND2WT, will be the total weight of all of the sand fractions.
   e. Note any sample collected on the bottom pan. If more than 0.05 g passed through the sieves into the pan, then the original wet-sieving into the sedimentation cylinder was incomplete.

7. Discard the collected sands (unless mineralogical analyses are planned), put the empty container back on the balance, tare to 0.00 g, and continue with the next sample.

Calculations

NOTE: The percentages of total clay, silt fractions and total silt, sand fractions and total sand, and the soil texture are calculated in the soil characterization database program (see Appendix B for the computer program).

Percentages of the different size fractions

1. The organic-free, oven-dry soil weight (SOIL WT) is the difference between the oven-dry soil and fleaker weight (FLK PSOWT) and the fleaker weight (FLK WT).

2. Percent total clay is the weight of the <2-um size fraction divided by the total soil weight and multiplied by 100:
   a. Wt. of clay (<2 um) in sample = wt. of pipetted aliquot [(sample + beaker wt.) - beaker wt. - blank wt.] multiplied by the dilution factor (25-ml aliquot from 1000 ml suspension = 40X):

      TCLAY WT = (BP 2UMWT - BWT 2UM - BLANK WT) x 40

   b. Pct. total clay (TCLAY) = (TCLAY WT / SOIL WT) x 100

3. Percent fine (<0.2 um) and coarse (0.2 - 2 um) clay are calculated as follows:
   a. Pct. fine clay = wt. of pipetted aliquot [(sample + bottle wt.) - bottle wt. - blank wt.] x 40 (25-ml aliquot from 1000 ml suspension) x 100
   b. Pct coarse clay = Pct. total clay - Pct. fine clay

4. Percent silt is the sum of the coarse (20 - 50 um), medium (5 - 20 um), and fine (2 - 5 um) silt fractions divided by the total soil weight and multiplied by 100:

   a. Wt. of fine silt (2 - 5 um) = wt. of the <5-um fraction minus the wt. of clay (TCLAY WT):

      5UM WT = (BP 5UMWT - BWT 5UM - BLANK WT) x 40
      FSILT WT = 5UM WT - TCLAY WT

   b. Wt. of medium silt (5 - 20 um) = wt. of the <20-um fraction minus the wt. of the <5-um fraction:

      20UM WT = (BP 20UMWT - BWT 20UM - BLANK WT) x 40
      MSILT WT = 20UM WT - 5UM WT

   c. Pct. fine silt (FSILT) = (FSILT WT / SOIL WT) x 100

   d. Pct. medium silt (MSILT) = (MSILT WT / SOIL WT) x 100

   e. Pct. coarse silt (20 - 50 um) is calculated by subtracting the percent of the other fractions from 100 percent:

      CSILT = 100 - (TCLAY + FSILT + MSILT + TSAND)
      [TSAND is calculated in the next step]

   f. Pct. total silt (TSILT) = FSILT + MSILT + CSILT

5. Percent sand is the sum of the percentages of each sand size fraction (weight of sand fraction divided by total sand weight and multiplied by 100):

   a. Pct. very coarse (1.0 - 2.0 mm) sand (VCSAND) =
      (VCSANDWT / SOIL WT) x 100

   b. Pct. coarse (0.5 - 1.0 mm) sand (CSAND) =
      (CSANDWT / SOIL WT) x 100

   c. Pct. medium (0.25 - 0.5 mm) sand (MSAND) =
      (MSANDWT / SOIL WT) x 100

   d. Pct. fine (0.10 - 0.25 mm) sand (FSAND) =
      (FSANDWT / SOIL WT) x 100

   e. Pct. very fine (0.07 - 0.10 mm) sand (VFSAND1) =
      (VFSAND1WT / SOIL WT) x 100

   f. Pct. very fine (0.05 - 0.07 mm) sand (VFSAND2) =
      (VFSAND2WT / SOIL WT) x 100

   g. Total very fine (0.05 - 0.10 mm) sand =
      VFSAND1 + VFSAND2

   h. Pct. total sand (TSAND) = VCSAND + CSAND + MSAND + FSAND + VFSAND1 + VFSAND2

Textural Classes

Texture is the relative proportion of the sand, silt, and clay . The ranges for each textural class are defined below (Soil Survey Staff, 1975):

1. Sands (s): $\geq$85% sand; % silt plus 1.5 times % clay is $\leq$15.
   a. Coarse sand (cos): $\geq$25% very coarse and coarse sand, and <50% of any other single grade of sand.
   b. Sand (s): $\geq$25% very coarse, coarse, and medium sand, and <50% of either fine or very fine sand.
   c. Fine sand (fs): $\geq$50% fine sand; or <25% very coarse, coarse, and medium sand and <50% very fine sand.
   d. Very fine sand (vfs): $\geq$50% very fine sand.

2. Loamy sands (ls): upper limit of 85 to 90% sand, with % silt plus 1.5 times % clay $\geq$15; lower limit of $\geq$70 to 85% sand, with % silt plus 2 times % clay $\leq$30.
   a. Loamy coarse sand (lcos): $\geq$25% very coarse and coarse sand, and <50% of any other one grade of sand.
   b. Loamy sand (ls): $\geq$25% very coarse, coarse, and medium sand, and < 50% of either fine or very fine sand.
   c. Loamy fine sand (lfs): $\geq$50% fine sand; or <25% very coarse, coarse, and medium sand and <50% very fine sand.
   d. Loamy very fine sand (lvfs): $\geq$50% very fine sand.

3. Sandy loams (sl): $\leq$20% clay, with % silt plus 2 times % clay >30, and $\geq$52% sand; or <7% clay, <50% silt, and 43 to 52% sand.
   a. Coarse sandy loam (cosl): $\geq$25% very coarse and coarse sand and <50% of any other one grade of sand.
   b. Sandy loam (sl): $\geq$30% very coarse, coarse, and medium sand, but <25% very coarse sand, and <30% of either very fine or fine sand.
   c. Fine sandy loam (fsl): $\geq$30% fine sand and <30% very fine sand (or) 15 to 30% very coarse, coarse, and medium sand.
   d. Very fine sandy loam (vfsl): $\geq$30% very fine sand; or >40% fine and very fine sand, at least half of which is very fine sand, and <15% very coarse, coarse, and medium sand.

4. Loam (l): 7 to 27% clay, 28 to 50% silt, and <52% sand.

5. Silt loam (sil): $\geq$50% silt and 12 to 27% clay, or 50 to 80% silt and <12% clay.

6. Silt (si): $\geq$80% silt and <12% clay.

7. Sandy clay loam (scl): 20 to 35% clay, <28% silt, and $\geq$45% sand.

8. Clay loam (cl): 27 to 40% clay and 20 to 45% sand.

9. Silty clay loam (sicl): 27 to 40% clay and <20% sand.

10. Sandy clay (sc): $\geq$35% clay and $\geq$45% sand.

11. Silty clay (sic): $\geq$40% clay and $\geq$40% silt.

12. Clay (c): $\geq$40% clay, <45% sand, and <40% silt.

References

Day, P. R. 1965. Particle fractionation and particle size analysis. p. 545-567 In C. A. Black (Ed.). Methods of soil analysis. Part 1. Physical and mineralogical properties. Agronomy no. 9. Amer. Soc. Agron. Madison, WI.

Jackson, M. L. 1969. Soil chemical analysis -- advanced course. 2nd edition. Univ. of Wisconsin, Madison, WI.

Kilmer, V. J. and L. T. Alexander. 1949. Methods of making mechanical analyses of soils. Soil Sci. 68:15-24.

Soil Survey Staff. 1975. Soil taxonomy: a basic system of soil classification for making and interpreting soil surveys. USDA Agric. Handbook No. 436. U.S. Govt. Printing Off., Washington, D.C.

Soil Survey Investigations Staff (SSIS). 1991. Soil survey laboratory methods manual. USDA SCS Soil Survey Invest. Rpt. No. 42. Version 1.0. National Soil Survey Center. Lincoln, NE.

Tanner, C. B., and M. L. Jackson. 1947. Nomographs of sedimentation times for soil particles under gravity or centrifugal acceleration. Soil Sci. Soc. Amer. Proc. 12:60-65.

CHAPTER 6

ROCK FRAGMENTS, VOLUME TO WEIGHT CONVERSIONS

Background

Rock fragments are particles >2 mm in diameter that are not attached to bedrock. Weight estimates of various size fractions (2-5, 5-20, 20-75, 75-250, and >250 mm, or close approximations, are common sizes) are used in soil interpretations (Soil Survey Staff, 1991).

Data on the rock fragment content in the soil is usually a combination of volume estimates and field and laboratory weighings. Volume estimates (pct.) are made for rock fragments >254 mm (10 in), 76 to 254 mm (3 to 10 in), and 19 to 76 mm (3/4 to 3 in). Volume estimates may also be made for the 4.7 to 19 mm (3/16 to 3/4 in) and 2 to 4.7 mm fractions. Rock fragments >19 mm are sieved from the sample (see Chapter 2) and the 4.7 to 19, 2 to 4.7, and <2 mm fractions are sieved and weighed in the laboratory.

The calculations described in this chapter determine weight percentages for each size fraction from the original volume estimates and weight determinations. Bulk density of the <2 mm fraction (see Chapter 7) and rock fragment density (assumed to be 2.45 g/cm^3 unless measured) are needed for the conversions.

Calculations

NOTE: These volume to weight conversions are calculated by the soil characterization database program (see Appendix B).

1. Collect the following information (See Chapters 2 and 7) on page 1 of the Lab Input Form (see Appendix A):

   a. >254 mm volume est., pct ($1VP_{>254\ mm}$)
   b. 76 - 254 mm volume est., pct. ($2VP_{254\text{-}76\ mm}$)
   c. 19 - 76 mm volume est., pct. ($3VP_{76\text{-}19\ mm}$)
   d. 4.7 - 19 mm sample wt., g ($4WT_{19\text{-}4.7\ mm}$)
   e. 2 - 4.7 mm sample wt., g ($5WT_{4.7\text{-}2\ mm}$)
   f. <2 mm sample wt., g ($WT_{<2mm}$)
   g. Bulk density, 1/3 atm. water content, <2 mm, g/cm^3 ($BDM_{<2mm}$)
   h. Rock fragment density, 2.45 g/cm^3, unless measured ($BD_{>2mm}$)

2. Convert the weights of the <19 mm fraction to volume (divide by the density):

   a. $VOL_{<2\ mm} = WT_{<2mm} / BDM_{<2mm}$
   b. $VOL_{4.7\text{-}2\ mm} = 5WT_{4.7\text{-}2\ mm} / 2.45$ (or $BD_{>2mm}$)
   c. $VOL_{19\text{-}4.7\ mm} = 4WT_{19\text{-}4.7\ mm} / 2.45$ (or $BD_{>2mm}$)
   d. $VOL_{<19\ mm} = VOL_{<2\ mm} + VOL_{4.7\text{-}2\ mm} + VOL_{19\text{-}4.7\ mm}$

3. Subtract the percent volume estimates of the >19 mm fractions from 100 to get the percent volume of the <19 mm fraction:

   a. Vol. pct. <19 mm ($VP_{<19\ mm}$) = $100 - (1VP_{>254\ mm} + 2VP_{254\text{-}76\ mm} + 3VP_{76\text{-}19\ mm})$

4. Convert the percent volume estimates of the >19 mm fractions to volume by multiplying the volume of the <19 mm fraction by the ratio of the volume percent, >19 mm, to the volume percent, <19 mm:

   a. $1VOL_{>254\ mm} = VOL_{<19\ mm} \times (1VP_{>254\ mm} / VP_{<19\ mm})$
   b. $2VOL_{76\text{-}254\ mm} = VOL_{<19\ mm} \times (2VP_{76\text{-}254\ mm} / VP_{<19\ mm})$
   c. $3VOL_{19\text{-}76\ mm} = VOL_{<19\ mm} \times (3VP_{19\text{-}76\ mm} / VP_{<19\ mm})$

5. Convert the calculated volumes to weight estimates by multiplying the volume of each fraction by the density:

   a. $1WT_{>254\ mm} = 1VOL_{>254\ mm} \times 2.45$ (or $BD_{>2mm}$)
   b. $2WT_{76\text{-}254\ mm} = 2VOL_{76\text{-}254\ mm} / 2.45$ (or $BD_{>2mm}$)
   c. $3WT_{19\text{-}76\ mm} = 3VOL_{19\text{-}76\ mm} / 2.45$ (or $BD_{>2mm}$)

6. Add the measured and calculated weights to get the total soil weight (TSOILWT):

   a. $TSOILWT = 1WT_{>254\ mm} + 2WT_{76\text{-}254\ mm} + 3WT_{19\text{-}76\ mm} + 4WT_{4.7\text{-}19\ mm} + 5WT_{2\text{-}4.7\ mm} + WT_{<2\ mm}$

7. Divide the weights by the total soil weight to get the estimated weight pct:

   a. $Wt.\ pct._{>254\ mm} = (1WT_{>254\ mm} / TSOILWT) \times 100$
   b. $Wt.\ pct._{76\text{-}254\ mm} = (2WT_{76\text{-}254\ mm} / TSOILWT) \times 100$
   c. $Wt.\ pct._{19\text{-}76\ mm} = (3WT_{19\text{-}76\ mm} / TSOILWT) \times 100$
   d. $Wt.\ pct._{4.7\text{-}19\ mm} = (4WT_{4.7\text{-}19\ mm} / TSOILWT) \times 100$
   e. $Wt.\ pct._{2\text{-}4.7\ mm} = (5WT_{2\text{-}4.7\ mm} / TSOILWT) \times 100$
   f. $Wt.\ pct._{<2\ mm} = (WT_{<2\ mm} / TSOILWT) \times 100$

References

Soil Survey Investigations Staff. 1991. Soil survey laboratory methods manual. USDA SCS Soil Survey Invest. Rpt. No. 42. Version 1.0. National Soil Survey Center, Lincoln, NE.

Soil Survey Staff. 1991. Soil survey manual (draft). U.S. Dept. of Agric. Soil Conservation Service. Washington, DC.

CHAPTER 7

BULK DENSITY, COEFFICIENT OF LINEAR EXTENSIBILITY, AND WATER DETERMINATIONS

Background and Theory

Bulk density is the ratio of soil mass to soil volume, expressed in Mg m^{-3} (preferred SI units) or g cm^{-3} (numeric values are the same for both units). It is used to estimate soil porosity (when particle density is known), convert soil water data from a weight to a volume basis, and estimate the weight of a large volume of soil. Bulk density affects soil water movement and root penetration and growth. Bulk density is also an indicator of fragipan horizons in soils.

Coefficient of linear extensibility (COLE) is the cube root of the ratio of moist (1/3 atm, 33 kPa) to oven-dry (105°C) bulk density. COLE is an estimate of the shrink-swell potential (susceptibility to volume change) of soil upon wetting and drying and is related to the type and amount of clay minerals present in the soil. Papers by Franzmeier and Ross (1968), Grossman et al. (1968), and Holmgren (1968) provide further discussion on COLE.

Water determinations in the laboratory have been used to estimate the plant available water in the soil. "Plant available" water is usually defined as that water held between matrix potentials of 1/3 and 15 atmosphere (33 and 1500 kPa). This data should be viewed with caution in relating to field conditions (see Gardner, 1971). A more detailed discussion of water retention, "available" water, and soil-water properties can be found in Baver et al. (1972), Richards (1965), and Peters (1965).

Because soil volume changes with changes in water content, bulk density and COLE are determined at standard water contents (1/3 atm. and oven-dry). Bulk density, COLE, and 1/3 atm (33 kPa) water retention are determined on undisturbed soil clods. Water retention at 15 atm (1500 kPa) is determined using <2-mm soil samples.

Bulk density as measured by clods will generally be slightly higher than the whole-soil bulk density because inter-clod pores are excluded in the measurements. Whole-soil bulk density is more difficult to measure.

Materials and Equipment

1. Field (sampling and collecting clods):
   a. Saran coating solution. Dissolve 1 part (wt) Dow Saran F220 (or S310) resin in 4 parts (wt) acetone or methyl-ethyl ketone. The solvents are volatile and should be mixed under a hood or in a well-ventilated area away from open flames or sparks. Stir with a non-sparking mechanical stirrer until the resin is dissolved. Store in a metal container, such as a gallon paint can. A second outside container will reduce fumes and minimize spillage.
   b. Nylon hair nets, aluminum tags, paper clips.
   c. String, rope, drying rack and/or stands to suspend and dry clods.
   d. Bags, packing material and boxes for transporting clods.

2. Bulk density, water retention, COLE determinations:
   a. Saran coating solution and clod drying rack.
   b. Power saw, bench-mounted, with masonry blade.
   c. Shallow pans to soak clods (leveled garbage can lids work well).
   d. Celite 545, technical grade, Fisher Sci. Co.

e. Ceramic pressure plates, with a minimum bubbling pressure (maximum pressure the plate will tolerate without allowing air to bubble through the pores) of at least 1/3 atm.
f. Pressure plate extractor apparatus, with pressure regulator.
g. Balance and stand, capable of weighing clods suspended from the bottom.
h. Screen container to hold rock fragments suspended from the balance.
i. Oven.
j. 2 mm sieve, 20 cm (8 in) diam., for rock fragment determination.
k. Soap solution (200 g Alconox in 8 liters of H_2O).
l. Drying pans, 9 x 5 cm (3.5 x 2 in).
m. Pressure membrane extraction apparatus, with regulators (Fig. 6.3).
n. Pressure membranes with a minimum bubbling pressure of 15 atm pressure.
o. Source of regulated pressure (nonflammable nitrogen is used).

Overview of the Procedure

Step	Result	Approx. Time
Bulk density, 1/3 atm (33 kPa) water retention, COLE		
Sample clods, coat w/ saran	Prepares clods for transport	1 day
Coat clods again in lab, cut flat surface	Prepares clods for analysis	1 day
Soak in water	Saturates clods with excess H_2O	2 days
Apply 1/3 atm press. to soils	Adjusts clod H_2O content	3-4 days
Coat cut clod face w/ saran	Prevents clod H_2O loss	1 day
Weigh clods in air and suspended in H_2O	Moist (1/3 atm) wt., vol.	30 $\pm$ min.
Dry at 105°C	Removes H_2O	2 days
Weigh dry clods in air and suspended in H_2O	Oven-dry wt., vol.	30 $\pm$ min.
Remove rock fragments, weigh in air and H_2O	Correct density for rock fragments	3 days
15 atm (1500 kPa) Water Retention		
Soak samples in water	Saturates with excess H_2O	1 days
Apply 15 atm press. to soils	Adjusts soil H_2O content	3-5 days
Weigh samples	15-atm moist wt.	20 - 30 min.
Dry at 105°C	Removes H_2O	1 day
Weigh dry soil	Oven-dry wt.	10-20 min.

A typical run for bulk density, 1/3 atm (33 kPa) water retention and COLE consists of 10 to 12 clods. Depending on the availability of extractors, several simultaneous runs can be made. Approximately 30 to 36 clods can be analyzed, including rock fragment corrections, in a 40-hour work week.

A run for 15 atm (1500 kPa) water retention includes 14 samples plus a standard. With two pressure membrane extractors available, 28 samples can be analyzed in a week.

Sampling and Preparing Clods

1. Carefully remove 3 clods (fist size, equidimensional if possible, 100-300 g) from each soil horizon. Remove smeared edges caused by a shovel or knife.

2. Place the clod in a hair net, tie the end in a knot, and attach a paper clip with a metal tag labelled to show the county, site and horizon numbers, horizon, depth, and clod letter A, B, or C (e.g., 014-020-02, E, 10-20 cm, clod A).

3. Dip the clod quickly into the saran solution and hang it on a drying rack or rope with the paper clip.

4. Place the dry clods (no longer tacky) in bags and carefully pack them in a box for transport to the lab. Put all clods from the same horizon in one bag.

5. Unpack the clods in a well-ventilated area at the lab, dip them again in saran solution, and hang them on a rack to dry.

6. Make a flat surface on the clod by cutting a thin slice off one side with a bench power saw equipped with a masonry blade. If the clod is not equidimensional, cut along the long dimension to maximize clod contact with water and minimize the distance water will travel while the clod is soaking.

   NOTE: Be careful when using the power saw. It can cut fingers as easily as clods!

Determining Bulk Density, 1/3 Atm (33kPa) Water, COLE

1. Place porous ceramic plates in shallow soaking pans (garbage can lids work well) and cover the plates with 2 to 4 cm of water. Check for leaks by holding the plate under water and blowing into the tube to inflate the rubber backing slightly. If bubbling occurs, replace the plate.

2. Cover the plate with a 1- to 2-cm thick layer of celite. Allow the celite to wet up and spread it evenly across the plate.

3. Mix a celite-water paste and fill in the voids and irregular areas of the cut surface of the clods. Place the clods cut side down into the celite on the plate making sure good contact is made between the clod and plate (10 to 12 clods fit on a plate).

4. Add water to a level 2 to 5 cm above the bottom of the clods. Soak the clods, adding additional water as necessary, until the tops of the clods are noticeably soft (this takes approximately 2 days, longer for dense horizons).

5. Place the plates with clods in the pressure extractor and connect the outlet tube to the fitting on top of the plate. Apply 1/3 atm pressure (5 psi or 25 cm of Hg) by opening the air valve on the pressure regulator and regulating the pressure with the

fine adjustment so that the Hg in the manometer (glass utube) rises to a 25-cm height differential (12.5 cm above equilibrium level).

6. Continue the pressure until no more water is extracted (3 to 4 days for most samples). Pressure plates in good condition should yield 120 ml of H_2O in the first 30 min, slowing in rate with time. An overhand loop tied in the plastic outlet tube aids in observing the rate of outlet flow.

7. When the outlet tubes stop dripping, shut off the pressure, remove the clods and clean off the celite. Under a hood, dip the cut side of the clod in the saran solution and dry for about 5 minutes on a hanging rack. Do not allow the clods to touch because they will stick together. Before hanging the clods, line the bottom of the hood with disposable paper to catch any saran that drips from the clod.

8. Weigh the clods on a top-loading balance (or suspended from below the balance). Tare the balance beforehand by adjusting to 0.0 with a tag, hair net and paper clip. Record the clod weight (moist) in air (WTMAIR) on page 4 of the Laboratory Data Input Form (see Appendix 1).

9. Suspend the clod from the bottom of the balance and raise a large beaker of water from below until the clod is completely submerged, but not touching the sides or bottom of the beaker. Record the clod weight (moist) in water (WTMWAT) on the input form. The coated clods will absorb water to some extent and, consequently, a quick reading is imperative (do not wait for the balance to stop oscillating).

   NOTE: In this fashion, the clod volume is determined by subtracting the weight of the clod suspended in water below the balance from the clod weight in air (WTMAIR - WTMWAT). An alternative method for determining the clod volume is to place the container of water on top of the balance and suspend the clod in water using string and a ring stand. The weight of the clod suspended in water on top of the balance, in g, is equal to the volume of the clod, in cm^3.

10. Place the clods on a sheet of aluminum foil (this prevents the clods from sticking to the shelves) in an oven and dry them for approximately 2 days at 105°C.

11. Cool the clods in the oven until the saran coat no longer sticks to the foil or to other clods. Remove and place the clods in a desiccator (this prevents the oven-dry clods from absorbing moisture from the air).

12. Repeat steps 8 and 9 on the oven-dry clods. Record the clod weight (oven-dry) in air (WTODAIR) and in water (WTODWAT) on the input form.

Correcting For Rock Fragments

NOTE: If the rock fragment content of the soil is 2% or less, a correction is not needed. Otherwise fragments are separated from the soil in the clods, their density is determined, and the bulk density is adjusted accordingly.

1. Cut the excess hairnet from the clods and place the clods in labelled beakers (use various sized beakers to match the sizes of clods). Remove the metal tag and tape it to a clod hot plate form (Figure 7.1). It is also a good idea to record the information from the metal tag on the clod form below the tag. Place the beakers on the hot plate in the same positions as indicated on the hot plate form.

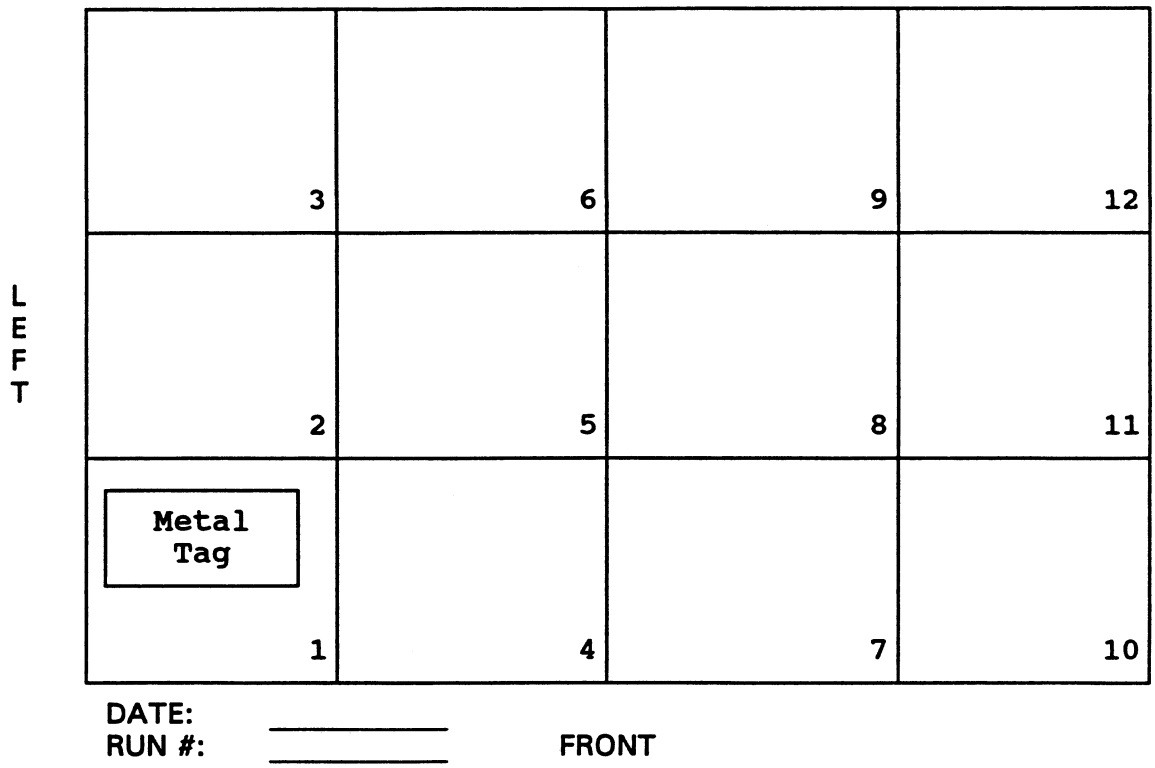


Figure 7.1. Clod Hot Plate Form

2. Burn off as much saran as possible from the clods by turning the hot plate to high under a hood. Cover the beakers with an aluminum foil hood to keep the beaker temperature as hot as possible. Leave the clods on the hot plate overnight.

3. Shut off the hot plate and allow the beakers to cool. Return the metal tag to the beaker, add 50 ml of soap solution (make sure the beakers are cool so they don't break), and enough water to cover the clod. Let the clods slake for 1 to 3 hours.

4. Wash the material from the beaker through a large diameter (20 cm) 2-mm sieve and collect the waste material in a bucket (a double sedimentation system with a small bucket or large beaker inside of a larger bucket is recommended to catch the sediment). Use a large rubber stopper and additional water to separate the rock fragments (>2 mm) from the fine earth (<2 mm) and unburned and charred saran.

5. Transfer the rock fragments and metal tag to a drying can and dry them in an oven overnight at 105°C.

6. Remove the oven-dry samples and allow them to cool (a desiccator is not necessary for the rock fragments).

7. Tare a metal can to 0.0 g on a top-loading balance and transfer the rock fragments to the tared can for weighing. Record the rock fragment weight in air (RFWTAIR) on the input form. Return the fragments to their original can and repeat this step for each sample.

8. Attach the screen container for weighing rock fragments to the bottom of the balance and tare to 0.0 g. (Pre-soak the screen for 1 hr before it is used).

9. Empty the rock fragments from the first sample onto the screen container suspended from the bottom of the balance. Raise a beaker of water to submerge the fragments in water and record the rock fragment weight suspended in water (RFWTWAT) on the input form. Repeat this step for each sample.

NOTE: To speed the process, re-tare the balance without discarding the fragments and continue with the subsequent samples. Empty the rock fragments only when the screen becomes full.

Determining 15 Atmosphere Water Retention

1. Pre-soak a porous cellulose membrane overnight in water. Inspect the membrane closely for pin holes or weak areas and discard damaged or worn membranes.

2. Place the membrane on the pressure membrane extractor, smoothing out wrinkles and ensuring that no soil is trapped between the extractor and the membrane (this could lead to a puncture when high pressure is applied). Place an O-ring between the pressure ring and the membrane and clamp down the ring. Pack moist tissues around the inside edge of the extractor to keep soil from getting between the ring and the membrane.

3. Place rubber retaining rings on the membrane (11 around the edge, 4 in the center) and fill the rings to the top with soil. Pack the soil slightly ensure firm contact with the membrane. Use an extractor diagram (Figure 7.2) to note the location of each sample. Note the county, site, horizon, and can number on the form.

4. Gently pour water onto the membrane between the samples until the water level is midway up on the rubber rings. Recheck them in a couple of hours to ensure that sufficient water remains to completely saturate the samples in the extractor. Cover the extractor to prevent evaporation and let samples soak overnight. Be sure enough water is in the extractor so that the membrane does not dry out.

5. Remove the excess water and stray soil from between the samples with a vacuum flask suction tube.

6. Place an O-ring and a rubber diaphragm on top of the pressure ring and close the lid. Tighten the lid anchor bolts to 10 foot-pounds with a torque wrench, alternating from one side to the other until all bolts are torqued to the same pressure.

7. Set up the valves to apply pressure (refer to Figure 7.3). At the start, the tank, gas regulator, and pressure release valves should all be closed. The Hg bypass valves should be open so that no gas goes through the U-shaped Hg tube. The line valves leading to the extractors should also be open. The gas regulator valve must be closed before the tank valve is opened to prevent a sudden burst of pressure which may disturb the samples or damage the membrane.

8. Open the tank valve. Slowly open the gas regulator valve. Slowly add pressure to the extractors until a pressure of 220 psi is reached.

NOTE: A hissing sound or air bubbles coming from the extractor outlet tube indicate a leak or punctured membrane. If this happens, shut down the system, drain off the pressure, and fix the leak. Great care in the initial assembly and loading of the extractor will minimize such leaks.

Figure 7.2. Extractor Diagram Showing Sample Locations.

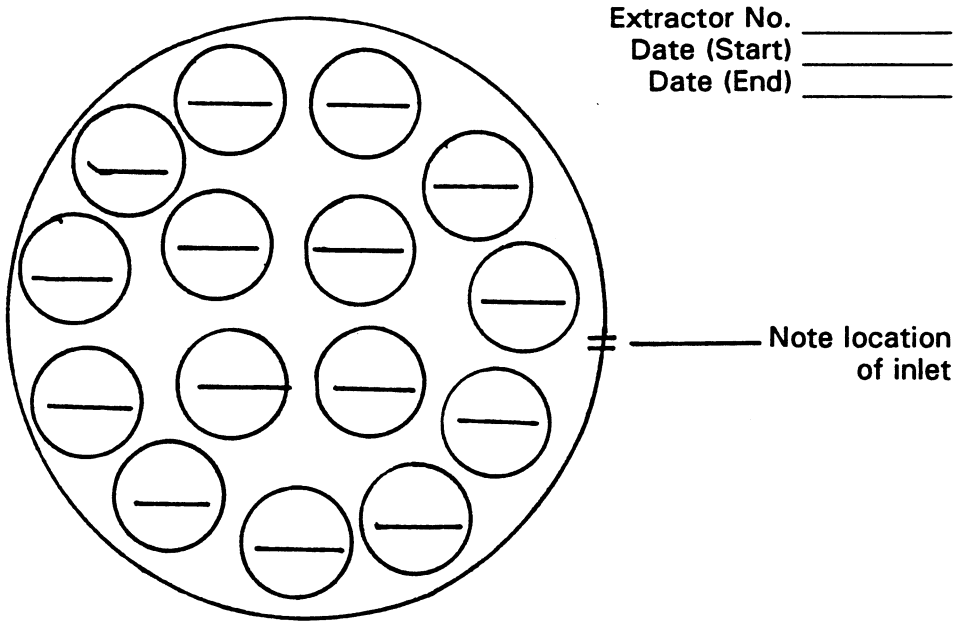
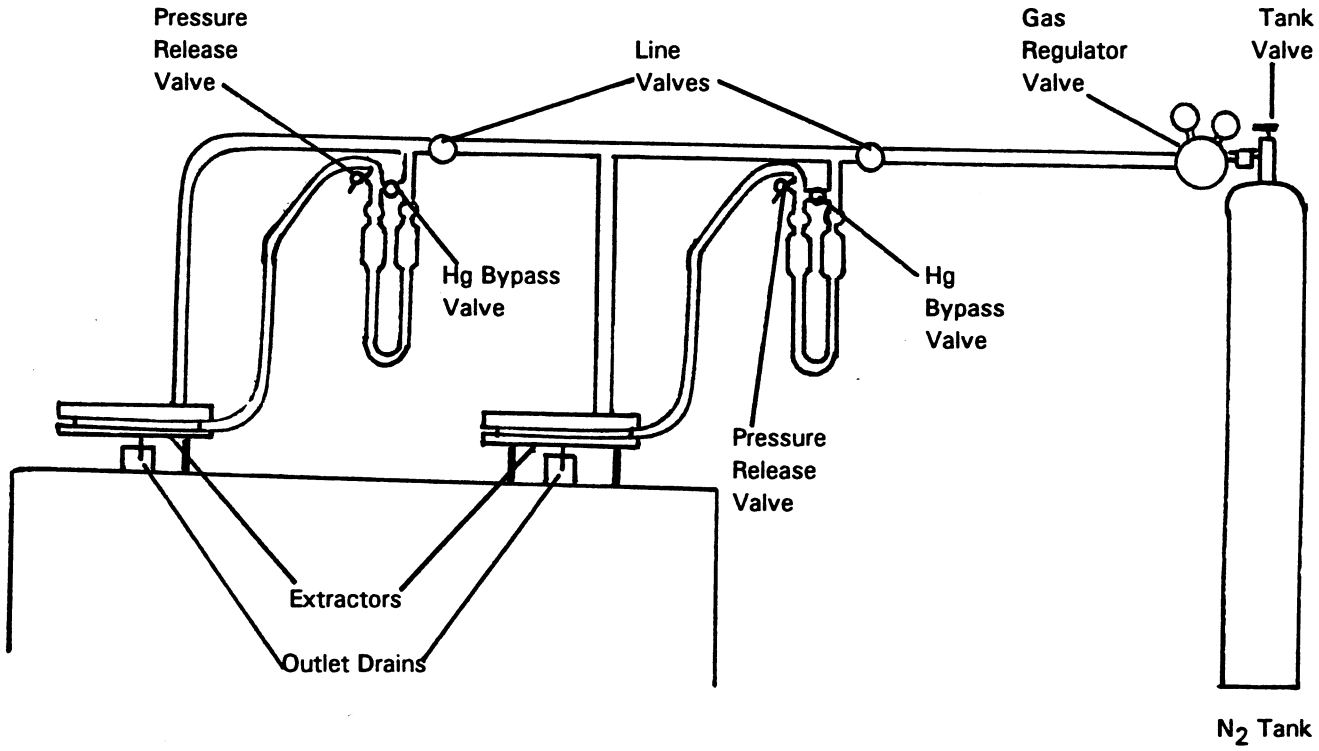


Figure 7.3. Diagram of the 15-Atmosphere Pressure Extraction Apparatus.



9. The pressure must be adjusted for samples high in clay (clay, silty clay, sandy clay textures) to prevent the soil from curling and shrinking away from the membrane as it dries. This is done as follows:
   a. After the first flush of water has drained (usually 2 to 3 hours), close the Hg bypass valve.
   b. Open the pressure release valve slightly just long enough (a few seconds) to re-route the gas through the Hg in the U-shaped tube and then close the valve again. The gas passing through the Hg will make a bubbling sound.
   c. Readjust the pressure to 225 psi on the gas regulator.

   The gas coming into the side of the extractor is diverted through the Hg tube, resulting in a slightly higher pressure on the upper diaphragm than within the extractor chamber. The diaphragm presses down on the samples, ensuring good contact with the cellulose membrane.

10. Maintain the pressure at 225 psi until water no longer comes from the outlet (usually within 3 days, but may be longer for high clay samples). When water flow ceases, remove the outlet tube from the water collection beaker (to prevent a backflow when pressure is released) and shut off the pressure at the gas tank valve. Open the Hg bypass valve and the pressure release valve to drain off the pressure. Open the extractor lid.

11. Remove the samples from the extractor with a spatula and place them in preweighed drying cans (record the CAN WT on the input form). Immediately place a lid on the can (to prevent moisture loss) and weigh the sample. Record the can plus 15-atm soil weight (CAN + 15AMWT) on the input form.

12. Dry the samples overnight at 105°C, removing the can lid (place under or wedge into the can) so that the sample is open.

13. Remove the samples from the oven, close the lids, and cool them in a desiccator. Weigh the samples and record the can plus oven-dry soil weight (CAN + 15AODWT) on the input form.

Calculations

NOTE: The calculations described below are performed by the soil characterization database program (Appendix B).

1. Bulk density is the ratio of the oven-dry clod weight to the volume of the clod at either 1/3-atmosphere or oven-dry water content. Because volume changes with moisture content will affect density, the moisture content at which the measurement is taken should be reported along with the bulk density value. When rock fragments are present, the rock fragment weight and volume are subtracted from the clod weight and volume to obtain the bulk density of the <2-mm soil fraction.

BD_m = Clod $Wt._d$ / Clod $Vol._m$

BD_d = Clod $Wt._d$ / Clod $Vol._d$

$BD_{<2,m}$ = (Clod $Wt._d$ - Rock Frag. $Wt._d$) / (Clod $Vol._m$ - Rock Frag. Vol.)

$BD_{<2,d}$ = (Clod $Wt._d$ - Rock Frag. $Wt._d$) / (Clod $Vol._d$ - Rock Frag. Vol.)

where:

BD = bulk density, in g/cm^3 or Mg/m^3

Clod Vol. = clod wt.$_{\text{suspended in air}}$ - clod wt.$_{\text{suspended in water}}$

Rock Frag. Vol. = rock frag. wt.$_{\text{suspended in air}}$ - rock frag. wt.$_{\text{suspended in water}}$

$_m$ = moist (1/3 atmosphere) soil

$_d$ = oven-dry soil

$_{<2}$ = <2-mm fraction, corrected for rock fragments

NOTE: If the clod is suspended in a container of water on top of the balance, then the weight of the clod suspended in water, in g, is equal to the volume of the clod, in cm^3.

2. The coefficient of linear extensibility (COLE) is the fractional change in the linear dimension of a clod as it goes from a moist (1/3 atm) to a dry (oven-dry) state. Because the calculations use the change in bulk density (BD), a three-dimensional measurement, the cube root is taken.

$$COLE = (L_m - L_d) / L_d = (L_m/L_d) - 1 = (BD_d/BD_m)^{1/3} - 1$$

where:

L_m = length of moist clod

L_d = length of dry clod

BD_m = moist bulk density

BD_d = dry bulk density

If the clods contain rock fragments, then a rock fragment conversion factor is used:

$$COLE = [\ (R_m \times BD_m/BD_d) + (1 - R_m)\ ]^{-1/3} - 1$$

where:

R_m = Vol. <2-mm material, moist / Vol. total clod, moist

3. Water retention is the fractional change in water content from a moist (1/3 or 15 atm) to an oven-dry state. It is expressed as an oven-dry weight percent.

$$WR_{1/3} = [\ (\text{Clod Wt.}_{1/3} - \text{Clod Wt.}_d) / \text{Clod Wt.}_d\ ] \times 100$$
$$WR_{15} = [\ (\text{Soil Wt.}_{15} - \text{Soil Wt.}_d) / \text{Soil Wt.}_d\ ] \times 100$$

where:

WR = water retention, % (wt.)

$_{1/3}$ = 1/3 atmosphere water retention

$_{15}$ = 15 atmosphere water retention

$_d$ = oven-dry

4. The water retention difference (WRD) is calculated as the difference in water retention between 1/3 and 15 atmospheres of pressure.

$$WRD = WR_{1/3} - WR_{15}$$

5. Porosity, the ratio of pore volume to total soil volume (expressed as a percent) can be calculated from the bulk density (BD) and particle density (PD, measured directly or assumed to be 2.65 g/cm^3).

$$\text{Porosity (\%)} = (\text{Vol.}_{\text{pores}} / \text{Vol.}_{\text{ttl. soil}}) \times 100$$

where:

$Vol._{ttl.\ soil} = Mass_{ttl.\ soil} / BD$

$Vol._{pores} = Vol._{ttl.\ soil} - Vol._{soil\ solids}$

$Vol._{soil\ solids} = Mass_{ttl.\ soil} / PD$

substituting into the above equation and simplifying:

$$\text{Porosity (\%)} = [\ 1 - (BD/PD)\ ] \times 100$$

References

Baver, L. D., W. H. Gardner, and W. R. Gardner. 1972. Soil Physics. Fourth edition. John Wiley and Sons, Inc. New York.

Brasher, B. R., D. P. Franzmeier, V. Valassis and S. E. Davidson. 1966. Use of saran resin to coat natural soil clods for bulk density and water retention measurements. Soil Sci. 101:108.

Franzmeier, D. P., and S. J. Ross, Jr. 1968. Soil swelling: Laboratory measurement and relation to other soil properties. Soil Sci. Soc. Amer. Proc. 32:573-577.

Gardner, W. R. 1971. Laboratory measurement of available soil water. Soil Sci. Soc. Amer. Proc. 35:852.

Grossman, R. B., B. R. Brasher, D. P. Franzmeier, and J. L. Walker. 1968. Linear extensibility as calculated from natural-clod bulk density measurements. Soil Sci. Soc. Amer. Proc. 32:570-573.

Holmgren, G. G. S. 1968. Nomographic calculation of linear extensibility in soils containing fragments. Soil Sci. Soc. Amer. Proc. 32:568-570.

Peters, D. B. 1965. Water availability. p. 279-285 in C. A. Black (Ed.). Methods of soil analysis. Part 1. Physical and mineralogical properties. Agron. No. 9. Amer. Soc. Agron. Madison, WI.

Reitemeier, R. F. and L. A. Richards. 1944. Reliability of the pressure membrane method for extraction of soil solution. Soil Sci. 57: 119-135.

Richards, L. A. 1949. Methods of measuring soil moisture tension. Soil Science 68:95-112.

Richards, L. A. 1965. Physical condition of water in soil. p. 128-152 in C.A. Black (Ed.). Methods of soil analysis. Part 1. Physical and mineralogical properties. Agron. No. 9. Amer. Soc. Agron. Madison, WI.

CHAPTER 8

REACTION (Soil pH)

Background and Theory

Water ionizes slightly into H^+ and OH^-. The pH of a solution is a measure of the H^+ activity, expressed as the negative logarithm (base 10). The pH will range from 1 (acid) to 14 (basic), with 7 being neutral.

A glass electrode sensitive to the activity of the hydrogen ion measures the pH of a solution. The H^+ activity causes an electrical potential which is measured by a very sensitive potentiometer, the pH meter. A reference electrode is needed to complete the electrical circuit.

Soil pH is important in soil fertility, chemistry, and classification. A low soil pH can indicate phytotoxic conditions (high aluminum concentration in solution) and a high pH can indicate low availability of micronutrients. Soil pH values can also be used as an indicator of base saturation.

Factors that can affect the measurement of soil pH include (1) sample drying, (2) soil/solution ratio, (3) equilibration with CO_2, (4) soluble salt content of the soil, and (5) equipment errors such as liquid-junction potential and suspension effects (Jackson, 1958; McLean, 1982; Peech, 1965). Drying hastens certain reactions and pH should be taken on air-dry samples for uniformity (Jackson, 1958). Equilibration with CO_2 can decrease the pH of alkaline soils but has practically no effect on acid soils (McLean, 1982). Soil pH decreases with increasing soluble salt content, and is generally lower in hot, dry seasons than in cool, wet seasons (McLean, 1982; Peech, 1965).

Peech (1965) recommends measuring soil pH in 0.01 M $CaCl_2$ solution because it approximates the electrolyte concentration of nonsaline soil solutions, and thereby more nearly represents soil pH under field conditions. The $CaCl_2$ solution also minimizes differences due to soil/solution ratios, seasonal variations, varying salt contents, and liquid-junction potential. Measured soil pH values will generally be lower in 0.01 M $CaCl_2$ than in H_2O, and still lower in 1 N KCl solution.

Materials and Equipment

1. pH meter with Ag/AgCl pH and saturated calomel reference electrodes
2. Paper souffle cups, 1 to 2 oz size
3. Glass stirring rod

Reagents

1. Distilled/deionized water.
2. $CaCl_2$ solution, 0.01 M 1.47 g of $CaCl_2 \cdot 2H_2O$ per liter.
3. KCl solution, 1 N. 74.50 g/l of KCl per liter.
4. Buffer solutions, pH 4 and 7 (pH 10, if alkaline soils are anticipated). Refrigerated buffers must be brought to room temperature prior to use.
5. Amount of reagents needed: 10 ml of each reagent is required per sample. A typical run consists of 10 to 15 samples.

Overview of the Procedure

The soil and the appropriate solution are mixed at a ratio of 1:1 (wt:vol) and then allowed to equilibrate for 30 to 60 min. The pH of the soil slurry is measured with a properly-calibrated pH meter. Samples are usually weighed in triplicate for determination in each solution. To minimize the potential for contamination, determine pH first in H_2O, then in 0.01 M $CaCl_2$, and finally in 1 N KCl.

A typical sample run of 30 to 45 readings (10 to 15 different samples) can be determined within 2 to 3 hours, from addition of solution to reading. Allow extra time for weighing (approximately 30 min.) and calibration of the pH meter (5 min.).

Operating and Calibrating the pH Meter

1. Before using the pH meter, make sure that the reference electrode is filled with saturated KCl solution (Orion 90-00-01 or Fisher So-P-138 potassium chloride reference electrode filling solution).
2. Lower the sleeve or remove the plug on the reference electrode during analysis. Replace the sleeve or plug when finished to keep the electrode from drying out.
3. Adjust the temperature control to the room, reagent, and sample temperature.
4. At the beginning and after each measurement, rinse the electrodes with H_2O and wipe them dry with a kimwipe tissue.
5. Place the pH meter on stand-by whenever the electrodes are out of solution. Select operate only when the electrodes are in solution or in the soil slurry.
6. Calibrate the pH meter using standard buffer solutions:
   a. Lower the electrodes into the pH 7.00 buffer, allow the reading to stabilize and adjust the calibration knob until the meter reads 7.00.
   b. Next place the electrodes in the pH 4.00 (use pH 10.00 if alkaline soil samples are being analyzed) buffer, allow the reading to stabilize, and adjust the slope knob so that meter reads 4.00.
   c. Repeat these steps until no adjustment is necessary.
   d. Check the calibrations occasionally (every 8 to 10 readings) and adjust as needed.

Procedure

1. For each sample, weigh out three sets of 10 $\pm$ 0.5 g of air dried soil into separate labeled paper souffle cups.

2. To one set of samples, add 10 ml of H_2O; to a second set, add 10 ml of 0.01 M $CaCl_2$; and to the third set add 10 ml of 1.0 N KCl.

3. Stir the samples occasionally during the equilibration period (30 to 60 minutes), cleaning the glass stirring rod between samples to avoid contamination. Stir the samples again just prior to the measurement.

4. Lower the electrodes into the soil slurry and record the pH to one decimal place. Record the values as pH LAB (WATER, KCL, CACL2) on page 5 of the Laboratory Data Input Form (Appendix A).

   NOTE: Some drift in the reading may occur immediately after immersion of the electrode in the sample. This is most pronounced in pH in H_2O and with soils having high pH values. In contrast, acid samples determined in KCl usually equilibrate quite rapidly.

References

Jackson, M.L. 1958. Soil chemical analysis. Prentice-Hall. Englewood, NJ.

McLean, E. O. 1982. Soil pH and lime requirement. p. 199-224 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. 2nd Edition. Soil Sci. Soc. of Amer. Madison, WI.

Peech, M. 1965. Hydrogen-ion activity. P. 914-926 in C. A. Black et al. (Ed). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. Amer. Soc. of Agron. Madison, WI.

CHAPTER 9

CALCIUM CARBONATE EQUIVALENT (ACID NEUTRALIZATION METHOD)

Background and Theory

The amount of neutralizing bases, expressed in equivalents of calcium carbonate ($CaCO_3$), in a soil is measured by determining how many equivalents of HCl a sample can neutralize. The soil sample is reacted with standardized acid and the excess acid is titrated with a standardized base. This information is used to estimate the amount of leaching that has occurred in carbonate-containing soils. The procedure is also used to determine the neutralization potential of minesoils and overburdens (Sobek et al., 1978). This is not a routine analysis in the Soil Characterization Lab. Only soil samples with a pH of 7.1 or higher are analyzed.

Materials and Equipment

1. Erlenmeyer flasks, 250 ml, with small watchglasses
2. Balance, sensitive to 0.001 g
3. Pipettes, 25 and 10 ml
4. Steam hot plate
5. Magnetic stirrer
6. Burette, 50 ml
7. pH meter

Reagents

1. HCl, 1 N, standardized (certified grade).

2. NaOH, 1 N, standardized (certified grade). Use an ascarite tube to protect from CO_2 in the air. Standardize by titrating 50 ml of 1 N NaOH solution to a pH 7.00 endpoint with 1 N (certified) HCl. The actual normality is calculated as:

$$N_{NaOH} = (N_{HCl} \times Vol_{HCl}) / Vol_{NaOH}$$

3. Phenolphthalein indicator solution

Overview of the Procedure

After standardized acid is added, the soil sample is heated on a hot plate for 30 min, diluted with water and heated to boiling to drive off CO_2 (CO_2 will react with NaOH to precipitate Na_2CO_3, thus producing an erroneous endpoint). The excess (unreacted) acid is measured by titrating to a neutral endpoint with a standardized base. The normality of the NaOH should be determined each day.

A typical sample run contains 2 sets of 12 samples and can be analyzed in 4 to 6 hours, allowing time for to weigh out samples and clean the glassware.

Procedure

1. Weigh out a 5.00 g sample of <2-mm soil ground to pass through a <0.25 mm (fine sand size) sieve. Duplicates for each soil are recommended. Use 2.00 g for soils known to be high carbonates and 1.00 g for limestone samples.

2. Transfer the samples to 250 ml Erlenmeyer flasks.

3. Add 25 ml of standardized 1 N HCl.
   NOTE: 1 g of pure $CaCO_3$ will require 20 meq of acid for neutralization. $Ca(OH)_2$ or CaO will require more.

4. Heat the samples on a hot plate under a hood for 30 minutes. Cover the flasks with a small watch glass to minimize HCl loss. All reactive material should be neutralized within this time.

5. Add approximately 100 ml of distilled water to bring the solution to a convenient volume for titration.

6. Heat to boiling to drive off CO_2 and cool.

7. Add 3 drops of phenolphthalein indicator, place the flask on a magnetic stirrer with a lighted top, and titrate to a faint pink endpoint with standardized 1 N NaOH. The pink color of the endpoint should persist for 30 seconds after thorough swirling of the sample. The clay will start to flocculate near the endpoint. Record the volume of NaOH used in the titration.
   NOTE: As an alternate to the phenolphthalein indicator, titrate to a pH 7.00 endpoint using a pH meter.

Calculations

1. Calculations are done by hand and the results are entered into the soil characterization database lab data file for storage.

2. HCl neutralized (meq) = (ml HCl added x N of HCl) - (ml NaOH added x N of NaOH)

3. $$\% \text{ Equiv. } CaCO_3 = \frac{\text{meq HCl neutralized} \times 100}{20 \text{ meq/g } CaCO_3 \times \text{wt. of soil}}$$

4. Use the moisture correction factor to convert soil wt. from air-dry to oven-dry basis.

References

Allison, L. E. and C. D. Moodie. 1965. Carbonate. p. 1379-1396 in C. A. Black (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. no. 9. Amer. Soc. Agron. Madison, WI.

Sobek, A. A., W. A. Schuller, J. R. Freeman, and R. M. Smith. 1978. Field and laboratory methods applicable to overburdens and minesoils. EPA-600/2-78-054. U.S. Environmental Protection Agency, Cincinnati, OH.

CHAPTER 10

ORGANIC CARBON AND TOTAL SULFUR (LECO FURNACE)

The Leco induction furnace uses the heat generated by the coupling of metallic accelerators to oxidize carbon (C) and sulfur (S) in soils. The oxygen (O_2) that passes over the sample combines with C to produce CO_2 or with S to produce SO_2. These gases are collected and the amount liberated is determined.

Carbon (C) is the chief element in soil organic matter (48 to 58% by weight) and has been used to estimate the amount of organic matter present in the soil (Nelson and Sommers, 1982). Factors to convert from organic C to organic matter vary among soils, horizons, and type of organic matter. Results are best left in the form of organic C rather than as an estimate of organic matter.

The Leco induction furnace oxidizes the C in the soil, which reacts with O_2 to form CO_2. The CO_2 is absorbed in an ascarite bottle, which is then weighed to determine the amount of CO_2 that was absorbed. This method overestimates the organic C content in calcareous soils and in minesoils which contain coal and carboniferous fragments. Organic C in calcareous soils is analyzed by the modified Walkley-Black method (Chapter 11).

The percentage of total sulfur (S) in a soil or minesoil sample can be accurately determined by heating the sample to approximately 1600 degrees in an induction furnace. During combustion, O_2 passed over the sample combines with the liberated S to form SO_2. The SO_2 is passed through a dilute HCl solution containing potassium iodide (KI), starch, and potassium iodate (KIO_3). Free iodine, formed from the reaction between KIO_3, KI and HCl, gives the solution a dark blue color in the presence of starch. The SO_2 takes up iodine, reducing the blue color. A titrator automatically adds KIO_3 until enough free iodine is formed to return the solution to its original dark blue color. The percent total S is calculated from the amount of KIO_3 used in the titration.

Before starting the analysis, read the sections on setting up, operating, shutting down, and cleaning the induction furnace.

Setting Up The Furnace (Refer to Figure 10.1)

1. Plug the furnace into a 110-volts AC source and turn on both grid switches.
2. Turn the grid tap switch to the "med" position.
3. Allow the furnace to warm up for 5 minutes.
4. Check all gas fittings and tubing from the O_2 tank to the furnace, titrator and absorption bottles for cracks or leaks and replace as needed.
5. Inspect the filter column on top of the furnace. It should be packed, from bottom to top, with glass wool, ascarite, glass wool, magnesium perchlorate, and glass wool. When 3/4 of the ascarite turns white, replace the material in the column.
6. Inspect and clean the glass combustion tube located inside the combustion chamber. Replace cracked or broken tubes.
7. For carbon analysis, clamp shut the O_2-flow tube to the sulfur side of the furnace; for sulfur, clamp shut the tube leading to the carbon side.
8. Open the main valve on the oxygen tank.
9. Open the small regulator valve 1/2 turn counter-clockwise and regulate the flow until the float ball is between the marks on the gas flow meter. The O_2 flow will need to be readjusted just before the sample is ignited. To check for small leaks, coat all fittings with a soap solution and look for bubbles.

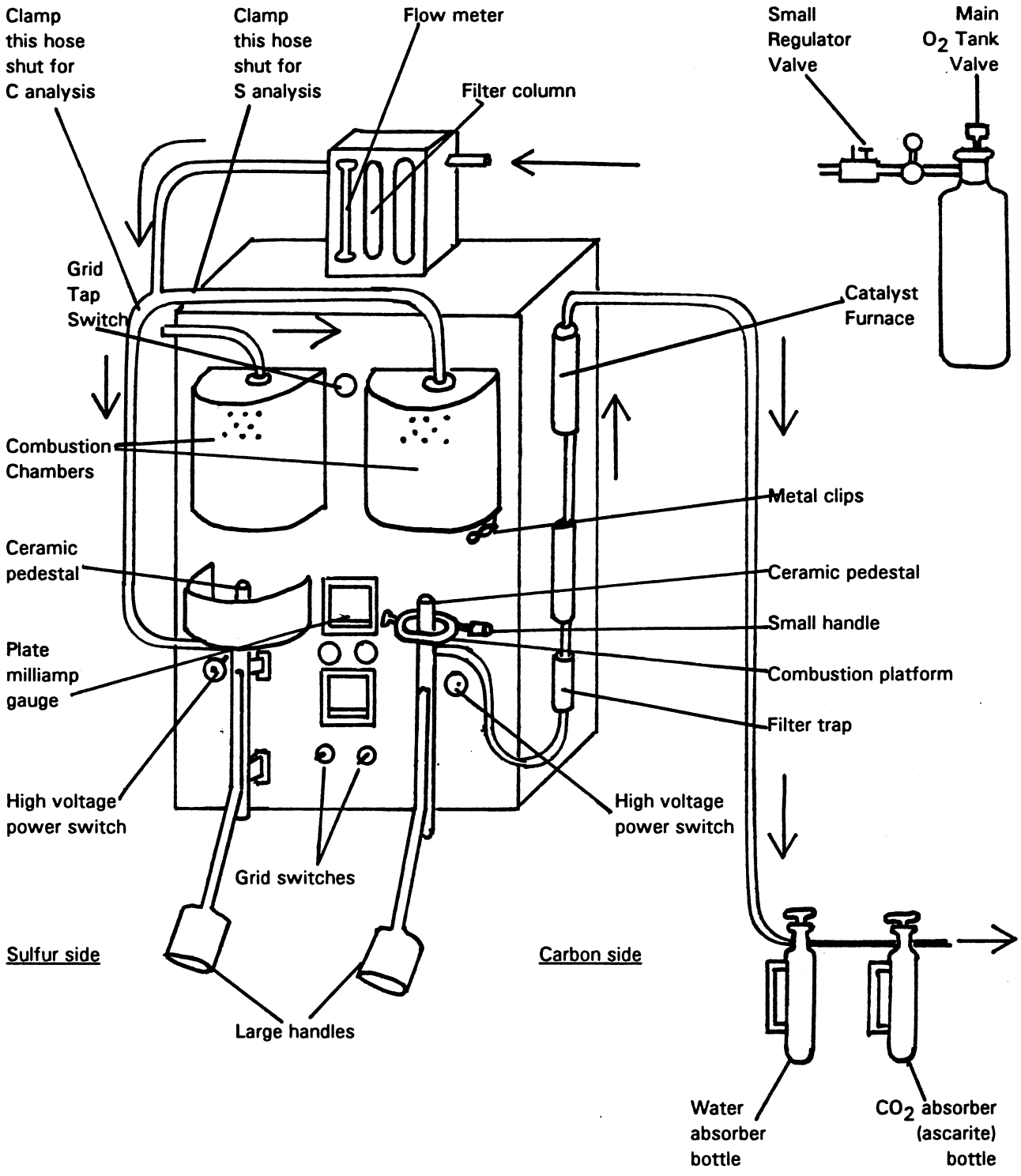


Figure 10.1. Leco Furnace for organic carbon and sulfur

10. Check to make sure that the catalyst furnace on the right side is hot. Do this by putting your hand near the furnace without touching it. If it is not hot, remove it and replace it.

Notes On Operating The Furnace

1. Always use tongs to handle hot crucibles and lids.
2. The catalyst furnace on the right side of the furnace gets hot. Do not touch it.
3. When analyzing C, make sure the stopcocks on the absorption bottles are open when the combustion platform is raised. Otherwise, the outflow line or the stopcocks may blow out.
4. The plate-milliam gauge needle may stick at the maximum reading. If so, tap the gauge to release it.
5. If the crucible sticks inside the glass combustion tube, loosen the crucible by tapping the tube upward with the platform, taking care not to break the tube.
6. Samples that are not mixed well may not burn properly If the burned sample has a cinder look instead of a smooth concave surface, rerun with a fresh sample.
7. The combustion tube, combustion platform area, the filter trap, and the rubber tubing that connects the combustion tube to the filter trap should be cleaned after approximately 20 samples.
8. Use jet type glass combustion tubes (Leco No. 550-122).
9. When 3/4 of the ascarite in any of the tubes turns white, replace with fresh ascarite.
10. If the standard for C is noticeably higher than expected, or if the water absorber bottle appears wet, add fresh magnesium perchlorate.
11. If the standard for C or S is noticeably low, check all seals, tubing, collector bottles, and titrators to insure proper seals throughout the system.

Shutting Down The Furnace

1. Turn off both grid switches and unplug the furnace.
2. Close the main valve on the O_2 tank and allow the pressure to bleed off. After the pressure has bled off, close the small regulator valve on the O_2 tank.
3. Make sure both collecting bottles used in the C analysis are closed.
4. If the titrator is used, turn the titrate-endpoint switch to manual, drain the titrating vessel and rinse twice with dilute HCl. Refill the burette and put a watchglass on top of titrating vessel. Turn off and unplug the titrator.

Cleaning The Furnace

1. Shut down the furnace and allow it to cool for 5 minutes.
2. Remove the ceramic pedestal with tongs and clean it.
3. Release the metal clip at the top of the glass combustion tube and lower the tube.
4. Remove and clean the red rubber ring from the bottom to the glass combustion tube and brush the inside of the tube until it is clean.
5. Replace the tube, making sure the rubber ring is flush with the bottom of the tube.
6. Clean the cloth screen and the glass cup of the filter trap.
7. Remove, clean, and replace the rubber hose leading from the combustion platform to the filter trap.
8. Inspect and replace the ceramic pedestal if needed.

ORGANIC CARBON

Materials and Equipment

1. Leco induction furnace (Fig. 10.1) with gas absorption bottles
2. Ascarite (to absorb CO_2), magnesium perchlorate (to absorb H_2O), and glass wool to pack absorption bottles
3. Balance, sensitive to 0.0001 g
4. Ceramic crucibles and lids with a 10-mm hole in the center
5. Iron chip accelerator (Leco No. 501-077), with scoop
6. Tin metal accelerator (Leco No. 501-076), with scoop
7. Tongs and cleaning brushes

Overview of the Procedure

A small sample of soil is mixed with iron and tin in a ceramic crucible. This mixture is heated in the furnace with O_2. The resulting CO_2 is collected in a bottle containing ascarite, which absorbs CO_2. The amount of gas absorbed is determined by weighing the bottle before and after analysis.

Each sample set consists of 2-3 standards and 17-18 samples. Allow 30 min. for sample preparation, 15 min. to set up the furnace, 60-75 min. to run the samples, and 30-45 min. to clean the furnace between runs.

Procedure

1. Place an empty crucible on the balance and tare it to 0.000 g. Weigh 1 g of soil into the crucible, recording the weight (SOIL WEIGHT) to the nearest 0.001 g on page 6 of the Laboratory Data Input Form (Appendix A).

2. Mix one heaping scoop of iron chips into the soil sample, stirring once (any more and the iron may settle to the bottom).

3. Add one scoop of tin metal <u>onto</u> the surface (do not mix) and cover the crucible with a perforated lid.

4. Set up the Leco furnace to analyze carbon. As indicated at the beginning of this chapter, make sure the gas tube leading to the sulfur side of the furnace is clamped shut (see Figure 10.1).

5. Weigh the CO_2 absorption (ascarite) bottle to the nearest 0.0001 g (use a kimwipe or gloves to avoid fingerprints) and record the bottle weight (BWT) on the input form.

6. Attach the bottle to the end of the outflow tube (insert about 1/2 inch) and open the stopcocks on both absorption bottles.

7. Place the crucible on the ceramic pedestal. Raise the combustion platform up to the combustion chamber with the large handle and, using the small handle, rotate the platform to secure it on the metal clips. Do not depress the high voltage power switch yet.

8. Re-adjust the oxygen flow rate with the small regulator valve until the float ball is between the marks on the flow meter.

9. Move the large handle to the right until the high voltage power switch is depressed. Let the sample burn for about 5 minutes, or until the plate milliamp gauge stabilizes at 300-350 milliamps.

10. Release the high voltage switch by moving the large handle 3/4 way to the vertical slot. Rotate the combustion platform away from the metal clips using both handles. Next lower the combustion platform using the large handle.

11. Close the stopcock on the ascarite bottle, remove it and weigh it to the nearest 0.0001 g. Record the bottle plus CO_2 weight (BPCO2WT) on the lab form.

12. Continue with each sample until all are analyzed and then shut down the furnace.

Calculations

1. Organic C is derived by (a) converting the weight of CO_2 absorbed to the weight of C and (b) dividing the weight of C by the total soil weight and multiplying this by 100 to convert to a percentage:

   a. CO_2 wt (CO2WT) = OCBPCO2WT - OCBWT
   C wt. = CO2WT x 0.2727

   b. Pct. organic C = (C wt / OCSOILWT) x 100

TOTAL SULFUR

Materials and Equipment

1. Leco induction furnace (Fig. 10.1) with titrator
2. Iron chip accelerator (Leco No. 501-077) with scoop
3. Tin metal accelerator (Leco No. 501-076) with scoop
4. Balance, sensitive to 0.001 g
5. Low sulfur ceramic crucibles with glass frit lids

Chemicals and Reagents

1. Dilute HCl (1.5%). Dilute 30 ml of concentrated HCl to 2 liters with H_2O.
2. Potassium iodate (KIO_3) titrating solution. Dissolve 0.4444 g of KIO_3 in H_2O and dilute to 1 liter.
3. Starch solution. Dissolve 2 g of Arrowroot starch in 50 ml of H_2O. Add this solution to 150 ml of boiling H_2O and stir while the solution boils for 7-10 minutes. The solution will be cloudy but should not settle. Cool to room temperature and add 6 g of potassium iodide (KI). Stir until dissolved and transfer into the polyethylene starch dispenser.

Procedure

1. Place an empty crucible on the balance and tare to 0.000 g. Weigh 1.000 g of soil into the crucible, recording the weight to the nearest 0.001 g.

2. Spread 1 1/2 to 2 scoops of iron chips evenly across the surface of the soil sample.

3. Spread 1 scoop of tin metal evenly on top of the iron accelerator and cover the crucible with a glass frit lid.

4. Set up the Leco furnace to analyze sulfur. Make sure the gas tube leading to the carbon side of the furnace is clamped shut.

5. Set up the titrator as follows:
   a. Connect the HCl and KIO_3 solution bottles.
   b. Turn on the titrator main switch.
   c. Fill the burette to the zero line: Cover the air hole on the front of the titrator and gently press the rubber bulb. Do not force the solution out of the top of the burette.
   d. Rinse the titrating vessel by twice filling it half full with HCl and draining it.
   e. Fill the titrating vessel to the marked line with HCl and add 5 ml of starch solution.
   f. With the O_2 flowing, raise the pedestal stage (without a sample) to bubble O_2 through titrating chamber.
   g. Turn the titrate switch to endpoint (titrator automatically establishes a blue color) and allow the level of KIO_3 in the burette to stabilize before disengaging the stage.
   h. Turn the switch to titrate, refill the burette to zero, and drop the stage on the furnace.

6. Place the crucible with glass frit on the pedestal and gently raise the stage into position for combustion.

7. As the sample burns the plate current ammeter will rise to (or above) 400. At this level, most of the sulfur will be burned off. The plate current ammeter will then drop to approximately 300.

8. After the plate current ammeter has dropped to 300, check the burette reading. When the reading remains unchanged for one minute, the ignition is complete. Record the burette reading (the burette is calibrated in fractions of a liter).

9. Lower the stage and refill the burette to zero. Carefully remove the burned sample with forceps and place it on a heat resistant surface.

10. Continue analyzing samples until until the solution in the titrating vessel reaches the HCl entrance hole. Drain the vessel and follow parts d through g in step 3 to rinse and re-set the titrator.

11. Continue until all samples are analyzed and then shut down and clean the furnace and titrator.

Calculations

1. Total % S depends on the amount of KIO_3 titrated (burette reading as a fraction of a liter), the concentration of KIO_3 (g/l) and the weight of soil analyzed (g), and is calculated as:

$$\% S = \frac{\text{Burette reading (l) x KIO3 conc (g/l)}}{444.4 \times \text{wt. soil (g)}} \times 100$$

2. For a concentration of 0.444 g KIO_3 per liter and a 1.000 g soil sample, %S is calculated by multiplying the burette reading (l) by 1.

3. Total S has been used to estimate the maximum potential acidity (MPA), expressed in tons of $CaCO_3$ per acre required for neutralization, from a minesoil or overburden sample. This assumes that all of the S in the sample is in the form of pyrite (Sobek et al., 1978). If other forms of S are present, then this calculation will overestimate the MPA.

$$\text{MPA (T/Ac } CaCO_3 \text{ equiv.)} = \% S \times 31.25$$

References

Leco Corporation. 1974. Instruction Manual, Induction Furnace. Leco Corporation; St. Joseph, MI.

Leco Corporation. 1975. Instruction Manual, Titrators. Leco Corporation, St. Joseph, MI.

Nelson, D. W., and L. E. Sommers. 1982. Total carbon, organic carbon, and organic matter. pp. 539-579 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Sobek, A. A., W. A. Schuller, J. R. Freeman, and R. M. Smith. 1978. Field and laboratory methods applicable to overburdens and minesoils. EPA-600/2-78-054. U.S. Environmental Protection Agency, Cincinnati, OH.

CHAPTER 11

ORGANIC CARBON CONTENT (MODIFIED WALKLEY-BLACK METHOD)

Theory

The Penn State Soil Characterization lab uses the Leco furnace to analyze the organic carbon (C) content of most soils (see Chapter 10). The modified Walkley-Black method is used for soils that have a $CaCO_3$ equivalent (Chapter 9) of 1% or higher. The C in the $CaCO_3$ will also be released during heating in the Leco furnace, causing an overestimation of organic C. A comparative study on four standard samples which had no excess carbonates found no difference between the Leco furnace and Walkley-Black methods (Table 11.1).

In the modified Walkley-Black method, organic matter is oxidized by the dichromate ion ($Cr_2O_7^{2-}$), with sulfuric acid (H_2SO_4) added to provide heat for the reaction. The unreacted $Cr_2O_7^{2-}$ is then determined by titration with a standard Fe^{2+} solution (ferrous sulfate or ferrous ammonium sulfate), and the quantity of oxidizable matter is calculated from the amount of $Cr_2O_7^{2-}$ reduced (Nelson and Sommers, 1982). The organic C is not completely oxidized in this method and a recovery factor of 1.3 is added to the calculations. Nelson and Sommers (1982) report that recovery factor varies among soils and should be determined experimentally for a particular group of soils. Otherwise, results from this procedure should be considered as approximate or semiquantitative estimates of organic C.

The presence of oxidizable and reducible constituents, especially Cl^-, Fe^{2+} (overestimations), and MnO_2 (underestimations), can cause interference errors with the procedures (Nelson and Sommers, 1982).

Safety: Always wear protective clothing (aprons, gloves, etc.) and eye protection (safety glasses) when handling H_2SO_4 and $Cr_2O_7^{2-}$ solution (read Chapter 3). Prepare all reagents and add H_2SO_4 to the samples under a hood.

Materials and Equipment

1. Erlenmeyer flasks, 500 ml wide-mouth
2. Burette, 10 ml
3. Burette, 50 ml
4. Bottle-top dispensers, 2, min. 20-ml capacity, to dispense conc. H_2SO_4 and H_3PO_4
5. Graduated cylinder, 100 to 250 ml capacity
6. Magnetic stirrer and stirrer bars
7. pH meter with platinum electrode for mV reading (for alternate titration method)

Reagents

1. Potassium dichromate ($K_2Cr_2O_7$), 1 N. 49.04 g of $K_2Cr_2O_7$ (dried at 105° C) per liter.
2. Concentrated (96%) sulfuric acid (H_2SO_4), reagent grade.
3. Concentrated (85%) phosphoric acid (H_3PO_4), reagent grade.
4. Ferroin indicator, 0.025 M.
5. Ferrous sulfate ($FeSO_4$), 0.5 N. Dissolve 140 g of $FeSO_4 \cdot 7H_2O$ in distilled H_2O, add 80 ml of concentrated H_2SO_4, cool, and dilute to 1 liter. Make this reagent fresh daily. Standardize daily by titrating against 10 ml of 1 N $K_2Cr_2O_7$ (this can be done using the blank -- see calculations). Store in a dark bottle away from light.

Table 11.1. Organic carbon data for soil characterization standard samples.

Standard	Flask	Weight	ml $FeSO_4$	%Org. C
Gilpin	1	0.5014	-----	-----
Ap	5	0.5010	16.10	1.60
02-01-01	9	0.5029	16.40	1.48
	14	0.5044	16.20	1.57
	18	0.5045	15.95	1.65
	23	0.5034	15.60	1.79
	27	0.5052	16.00	1.63
	28	0.5023	16.10	1.60
	Mean and Standard Deviation (Walkley-Black)			1.62 ± 0.09
	Mean and Standard Deviation (Leco Furnace)*			1.68 ± 0.03
Gilpin	4	2.0062	17.35	0.28
Bt	8	2.0051	17.30	0.28
02-01-02	12	2.0073	17.40	0.27
	13	2.0041	17.30	0.28
	19	2.0041	17.40	0.27
	22	2.0050	17.45	0.27
	25	2.0051	17.50	0.26
	31	2.0040	17.40	0.27
	Mean and Standard Deviation (Walkley-Black)			0.27 ± 0.01
	Mean and Standard Deviation (Leco Furnace)*			0.30 ± 0.02
Hagerstown	2	0.5046	15.90	1.67
Ap	6	0.5025	16.10	1.56
14-01-01	10	0.5057	15.95	1.65
	16	0.5040	16.30	1.52
	20	0.5027	15.90	1.68
	24	0.5018	16.25	1.54
	29	0.5020	16.00	1.64
	32	0.5036	16.00	1.64
	Mean and Standard Deviation (Walkley-Black)			1.61 ± 0.06
	Mean and Standard Deviation (Leco Furnace)*			1.65 ± 0.06
Hagerstown	3	2.0093	19.10	0.10
Bt	7	2.0048	19.00	0.11
14-01-04	11	2.0045	19.10	0.10
	15	2.0010	19.05	0.11
	17	2.0013	19.10	0.10
	21	2.0097	19.15	0.10
	26	2.0034	18.70	0.14
	30	2.0064	19.10	0.10
	Mean and Standard Deviation (Walkley-Black)			0.11 ± 0.01
	Mean and Standard Deviation (Leco Furnace)*			0.17 ± 0.06
Blank #1			20.10	
Blank #2			20.20	

* See Ciolkosz and Cronce (1986) for mean and standard deviation data for the Leco Furnace method.

Overview of the Procedure

Step	Result	Approx. Time
Grind sample to fine-sand size	Increases surface area for reaction	1/2 day per 20-24 samples
Add $K_2Cr_2O_7$, H_2SO_4, stir, stand for 30 min.	Oxidizes organic matter	90 min. (incl. weighing)
Add H_2O	Stops reaction	5 min.
Add H_3PO_4, Ferroin indicator, titrate with $FeSO_4$	Determines amount of unreacted $Cr_2O_7^{2-}$	2 hr

A typical run consists of 21 samples, 1 standard, and 2 blanks. For a large number of samples, do the grinding all at once. A run, from weighing samples through titration to cleaning glassware, can be completed within a day.

Procedure

1. Grind a representative sample (approximately 15 g) of soil with a mortar and pestle to fine-sand (<0.25 mm). Do not grind or sieve soil in the laboratory work space.

2. Depending on the probable organic matter content, weigh 0.5, 1.0 or 2.0 g of ground-soil into a 500 ml Erlenmeyer flask, recording the weight to the nearest 0.0001 g. Use 0.5 g for horizons with a high organic matter content, such as an Ap; 1.0 g for horizons with a medium organic matter content, such as E, Bh and Bhs; and 2.0 g for horizons with a low organic matter content, such as B and C.

3. Add 10 ml of 1 N $K_2Cr_2O_7$ to each sample with a 10-ml burette (volume is critical).

4. Rapidly add 20 ml of concentrated H_2SO_4, using a bottle-top dispenser to minimize the potential for spillage. Because fumes and violent bubbling may occur when the acid is first added, this step should be done under a hood. Swirl the mixture for one minute, taking care not to splash soil particles out of the solution. Let the sample stand on an asbestos sheet for 30 minutes, swirling occasionally.

5. Add 100 - 150 ml of distilled water to stop the reaction.

6. Add 10 ml of concentrated H_3PO_4, using a bottle-top dispenser to minimize the potential for spillage.

7. Add 4 drops of ferroin indicator.

8. Place the flask on a magnetic stirrer and titrate with $FeSO_4$ dispensed from a 50 ml burette. Titrate the blanks first.

   NOTE: The color will change from yellowish-green to green to forest green to blue-green and finally to black/dark green (The color is best seen on a white background without the stirrer light). For most samples, the end-point occurs within 5 to 10 drops after the blue-green color appears. The change from dull blue-green to black green is abrupt (one drop). For 2-g samples, the endpoint may be more gray-green than black, but the color change is still abrupt.

9. Record the amount of titrant used.
   NOTE: If more than 8 ml of $K_2Cr_2O_7$ is consumed in the reaction, reduce the sample size to 0.25 g or increase the volume of $K_2Cr_2O_7$ added.

10. As an alternate to the colorimetric endpoint (steps 6 - 8), titrate the samples with $FeSO_4$ to an endpoint of 630 mV using a pH meter equipped with a platinum electrode.

Calculations

1. Calculate the N of the $FeSO_4$ solution used (take the average of the two blanks):

   N of $FeSO_4$ = (N of $K_2Cr_2O_7$ x ml of $K_2Cr_2O_7$ used) / ml of $FeSO_4$ used

   = 10 / ml of $FeSO_4$ used

2. Calculate the organic C content, as a percent (%OC):

   $$\%OC = (B - S) / W \times N \text{ of } FeSO_4 \times 0.30 \times 1.333$$

   where

   B = ml of $FeSO_4$ used in the blank
   S = ml of $FeSO_4$ used in the sample
   W = sample weight, g
   0.30 = meq wt. of C, g, x 100%
   1.333 = recovery factor for organic C (assumes 77% of C is oxidized)

References

Ciolkosz, E. J. and R. C. Cronce. 1986. Pennsylvania State University Soil Characterization Laboratory Standard Sample Data Summary. Penn State Univ. Agron. Series No. 87.

Nelson, D. W., and L. E. Sommers. 1982. Total carbon, organic carbon, and organic matter. p. 539-579 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

CHAPTER 12

EXTRACTABLE BASES AND CATION EXCHANGE CAPACITY

Background and Theory

The cation exchange capacity (CEC) is a measure of the amount of exchangeable cations held on the exchange complex (negatively-charged surfaces of soil particles, predominantly clay and organic matter). The major basic cations found on the exchange complex of Pennsylvania soils are Ca, Mg, K, and Na. The sum of these extractable basic cations and the total acidity of the soil (Chapter 14) gives an estimate of the total CEC in a soil. Base saturation is the proportion of the exchange complex that retains basic cations.

Extractable bases, CEC, acidity, and base saturation are of interest in soil chemistry, fertility, classification, and genesis investigations. CEC reflects other important soil properties such as the amount and types of clay minerals present and the amount of organic matter present. Base saturation is used in soil classification to distinguish between Alfisols and Ultisols, mollic and umbric epipedons, and eutro- and dystro- criteria (SSS, 1975). The total and relative amounts of the basic and acidic cations are also indicators of the pedogenic processes, such as weathering, eluviation, and illuviation, that have occurred in the soil.

Ammonium acetate (NH_4OAc, 1N, pH 7.0) is the most widely accepted reagent used for extracting bases in nonarid soils (Lanyon and Heald, 1982; Rhoades, 1982). The soil is combined with an excess of NH_4OAc and NH_4^+ ions replace Ca^{2+}, Mg^{2+}, Na^+, and K^+ on the exchange complex. Buffering the extraction at pH 7.0 reduces potential variability due to factors such as the pH dependent charge and the solubility of pH-dependent soil constituents.

The Ca^{2+}, Mg^{2+}, Na^+, and K^+ extracted with 1N NH_4OAc are assumed to be exchangeable bases present on the soil exchange complex. Thomas (1982) warns that this procedure will not give exact results for exchangeable K in soils dominated by mica or vermiculite or for exchangeable Ca in soils containing free $CaCO_3$ or gypsum ($CaSO_4$). However, he notes that no other method gives satisfactory results either. With careful control of the conditions of extraction and analysis, this procedure can provide useful information on extractable bases and cation exchange capacity for the soils of Pennsylvania.

CEC can be measured directly (Rhoades, 1982; SSIS, 1991) or calculated as the sum of the extractable bases and total acidity. Base saturation is the sum of the extractable bases divided by the CEC, expressed as a percentage of the CEC.

Materials and Equipment

1. Mechanical vacuum extractor (see Appendix D for set-up and operation).
2. 60-ml plastic syringes and syringe barrels: 1 sample tube, 1 reservoir tube, and 1 extraction syringe per sample.
3. Filter pulp, ash free.
4. Volumetric flasks, 50-ml.
5. Volumetric flasks, 100-ml, and pipettes, assorted sizes, for making calibration standards.
6. Dilutor, automatic digital.
7. Atomic absorption spectrometer (AAS) (see Appendix C for set-up and operation).

Reagents

NOTE: Use distilled and deionized water to prepare all reagents. Prepare all reagents under a hood (see Chapter 3 for safety precautions).

1. Extracting the basic cations:
   a. Ammonium acetate (NH_4OAc), pH 7.0, 1 N. 58 ml of glacial acetic acid (99.7% HOAc) and 70 ml of ammonium hydroxide (NH_4OH, 28-30% as NH_3) and per liter. Add HOAc to half of the final volume of H_2O and then slowly add the NH_4OH while mixing. Cool, dilute to volume with H_2O, and adjust the pH to 7.0 ($\pm$.05) with HOAc or NH_4OH.

2. Analyzing the basic cations (AAS):
   a. Lanthanum (La) solution, 1%. Dissolve 11.73 g of La_2O_3 in 50 to 100 ml of H_2O. Slowly add 250 ml of concentrated HCl while mixing until the La_2O_3 is dissolved and dilute to 1 liter with H_2O.
   NOTE: La is added to the extracts to minimize interference effects on Ca and Mg due to Al, Si, PO_4 and SO_4 (Lanyon and Heald, 1982).
   b. Ca and Mg standard stock solution. To a 100-ml volumetric flask, add 10 ml of 1000 ppm Ca standard reference solution, 1 ml of 1000 ppm Mg standard reference solution, and dilute to volume with H_2O. This stock solution contains 200 ppm Ca and 20 ppm Mg.
   c. Ca and Mg working standards. Pipette 0, 2, 5, 8, and 10 ppm of the standard stock solution into separate 100-ml volumetric flasks, add 4 ml of NH_4OAc leaching solution, and dilute to volume with 1% La solution. The resulting working standards contain 0, 2, 5, 8, and 10 ppm Ca and 0, 0.2, 0.5, 0.8, and 1.0 ppm Mg. Make these standards fresh daily.
   d. K and Na standard stock solution. To a 100-ml volumetric flask, add 10 ml of 1000 ppm K reference solution, 2 ml of 1000 ppm Na reference solution, and dilute to volume with H_2O. This stock solution contains 100 ppm K and 20 ppm Na.
   e. K and Na working standards. Pipette 0, 2, 5, 8, and 10 ppm of the standard stock solution into separate 100-ml volumetric flasks and dilute to volume with the NH_4OAc leaching solution. The resulting working standards contain 0, 2, 5, 8, and 10 ppm K and 0. 0.4, 1, 1.6, and 2 ppm Na. Make these standards fresh daily.

3. Minimum Reagents Needed Per Run (24 Samples + 1 Blank):
   a. 1800 ml of NH_4OAc.
   b. 500 ml of La solution for working standards.
   c. 250 ml of La solution for initial extract dilutions (more may be needed for additional dilutions).

Overview of the Procedure

Step	Result	Approx. Time
Add 10 ml of NH_4OAc, stir, soak overnight	Replaces bases on exchange site with NH_4^+ (bases go into solution)	Overnight
Extract with additional NH_4OAc	Removes bases from soil, collecting in extract	3 hr

Step	Result	Approx. Time
Collect extract, bring to volume	Prepares extract for analysis	30 - 45 min
Dilute extracts with La solution	Eliminates Ca, Mg interferences, dilutes to analytical range	30 min (24 samples)
Prepare standards, calibrate AAS, analyze samples	Concentration of bases in ppm	30 min for std. prep.; 50-60 samples/hr for each base

An extraction run contains 23 samples and 1 standard (24 total). A blank containing the same proportion of reagents used in the extraction and dilutions should be prepared with each run (use the same reagents used in that particular run). The procedure lends itself to one extraction run per day: samples are prepared for extraction in the late afternoon, soaked overnight, and extracted the next morning, leaving time in the afternoon to prepare for another extraction. The extracts can be analyzed immediately or stored in a refrigerator to be run together in one large batch on the atomic absorption spectrometer.

Extracting the Basic Cations

1. Prepare the mechanical vacuum extractor as described in Appendix D.

2. Add 4.00 g of soil to the middle sample syringe. Record the CECSOILWT on page 7 of the Laboratory Data Input Form (Appendix A).

3. Add 10 ml of NH_4OAc solution to each sample, stir with a glass stirring rod (do not to disturb the filter pulp) and allow to sit overnight.

4. Extract at a 30-minute setting until the extracting solution is drawn to within approximately 0.5 cm of the top of the soil sample.

5. Place the top (reservoir) syringes on the extractor and add 35 ml of the NH_4OAc extracting solution. Continue the extraction at a 3-hour setting.

6. When the extraction is complete, remove the lower syringes from the extractor, and dispense the leachate into a 50 ml volumetric flask. Rinse the syringe with approximately 5 ml of NH_4OAc, add the rinse to the flask, and bring to volume with NH_4OAc.

7. If the samples are not going to be analyzed immediately, store them in polyethylene storage bottles and refrigerate. Allow the samples to come to room temperature before diluting or analyzing.

Analyzing Ca and Mg By Atomic Absorption

1. Use the digital dilutor to dilute 0.4 ml of sample extract with 9.6 ml of 1% lanthanum solution (10 ml total volume). Save the undiluted sample extract for K and Na analysis and also in case a different dilution factor is needed for the Ca and Mg analysis. The 25X dilution factor should be satisfactory for most samples.

2. Follow the instructions in Appendix C to prepare the AAS to analyze Mg and Ca by atomic absorption. Adjust the burner head using the Mg standards (Appendix C).

3. Calibrate the AAS with the standard Mg solutions (readings in ppm) and determine the concentration of Mg in the samples. Check the standard concentrations periodically to ensure against drift. Record the sample concentration (MG CEC PPM).

   NOTE: Subtract the blank concentration from the sample concentration before entering on the Lab Input Form or note the blank concentration and subtract it prior to entering the data into the computer.

4. Dilute any sample with a concentration greater than the highest standard and reanalyze it on the AAS. Record any additional dilution factors (MG CEC DF) on the input form. If the sample concentration is below the detection limit (Appendix C), use the undiluted extract and analyze it at a lower dilution factor.

5. After all samples are analyzed for Mg, calibrate the AAS with Ca standards and repeat steps 3 and 4 for the analysis of Ca.

Analyzing For K and Na By Atomic Emission

1. Prepare the AAS to analyze K by atomic emission (Appendix C). Calibrate the AAS with the standard solutions (readings in ppm) and determine the concentration of K in the undiluted sample extracts. Check the standard concentrations periodically to ensure against drift.

   NOTE: Subtract the blank concentration from the sample concentration before entering on the Lab Input Form or note the blank concentration and subtract it prior to entering the data into the computer.

2. If any undiluted sample extract has a concentration greater than the highest standard, dilute it and re-analyze. Record any dilution factor (K CEC DF) on the input form.

3. Repeat steps 1 and 2 for the analysis of Na.

Calculations

1. The general formula for calculating the concentration of extractable bases, in meq/100 g, or cmol (+)/kg, is:

$$\text{Base, meq/100 g} = \frac{\text{ppm sample} \times \text{ml extract} \times 1000 \times \text{dilution factor} \times 100}{1{,}000{,}000 \times \text{g soil} \times \text{equiv. wt. of base}}$$

   where,

ppm sample	= ppm in sample extract - ppm in blank
ml extract	= initial extract volume (50 ml)
g soil	= oven-dry soil weight (air-dry soil wt. / moisture correction)
dilution factor	= initial dilution (25X for Ca, Mg; 1X for Na, K) + additional dilutions
equiv. wt.	= 20.05 for Ca, 12.15 for Mg, 39.1 for K, 23 for Na

2. This equation is simplified (incorporating the constants, extract volume, initial dilution factor, and equivalent weight of base) in the computer calculations for each extractable base (Appendix B).

3. CEC is calculated as the sum of the extractable bases plus total acidity:

   CEC (meq/100 g) = NH_4OAc-extractable (Ca + Mg + K + Na)
   + $BaCl_2$-TEA extractable Acidity

4. Base saturation is the sum of extractable bases divided by the CEC and multiplied by 100 to express the value as a percentage.

References

Isaac, R. A., and J. D. Kerber. 1971. Atomic absorption and flame photometry techniques and uses in soil, plant, and water analysis. p. 17-37 in L. M. Walsh (Ed.). Instrumental methods for analysis of soils and plant tissue. Amer. Soc. of Agron., Soil Sci. Soc. of Amer. Madison, WI.

Lanyon, L. E., and W. R. Heald. 1982. Magnesium, calcium, strontium, and barium. p. 247-262 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Rhoades, J. D. 1982. Cation exchange capacity. p. 149-157 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Soil Survey Investigations Staff (SSIS). 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rept. No. 42. National Soil Survey Center. Lincoln, NE.

Soil Survey Staff (SSS). 1975. Soil taxonomy: A basic system of soil classification for making and interpreting soil surveys. USDA-SCS Agric. Handbook 436. U.S. Govt. Print. Office, Washington, DC.

Thomas, G. W. 1982. Exchangeable cations. p. 159-165 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

CHAPTER 13

EXTRACTABLE ALUMINUM (KCl METHOD)

Background and Theory

Aluminum (Al) is the most abundant metallic element in soils and the earth's crust (only O and Si are more abundant). It is a major component of most common inorganic soil particles. Aluminosilicates such as feldspars, micas, and clay minerals are the most common primary and secondary minerals. Nonsilicate Al minerals, such as gibbsite, also occur in soils.

Chemically-active forms of Al include exchangeable Al^{3+} and di-and mono-valent hydroxy-Al cations, organic complexes, amorphous coatings and particles, and hydroxy-Al polymers (Barnhisel and Bertsch, 1982). The solubility of Al is greatly affected by the pH of the soil: it increases as the pH decreases. At pH's below 5.5, Al in soluble forms of $Al(H_2O)_6^{3+}$ or di- or mono-valent hydroxy-Al cations tends to dominate the exchange complex. Soluble Al is toxic to many plants. In acid soils, the beneficial effect of liming is often the precipitation of Al in an insoluble form.

Exchangeable (or extractable) Al ions are displaced by an unbuffered neutral salt solution (1N KCl). This extractable Al has been equated to "active" acidity or immediate lime requirement while $BaCl_2$-TEA extractable acidity (see Chapter 14) represents total "potential" acidity (Thomas, 1982). The displaced Al is analyzed by atomic absorption spectrometer (AAS). Prior to 1992 a colorimetric method was used. The colorimetric method (Appendix E) converted the extracted Al to an ionic state with acid and heat. The color is developed with the reagent aluminon. The amount of Al determined by the AAS and the colorimeter methods are comparable (Thurman and Ciolkosz, 1992).

This method is only used on samples that have a pH (in H_2O) of 6.5 or lower. Samples that have a pH of higher than 6.5 do not have any appreciable amounts of 1N KCl extractable aluminum.

Materials and Equipment

1. For Al Extraction:
   a. Mechanical vacuum extractor (see Appendix D for set-up and operation).
   b. 60-ml plastic syringes and syringe barrels: 1 sample tube, 1 reservoir tube, and 1 extraction syringe per sample.
   c. Filter pulp, ash free.
   d. Volumetric flasks, 50-ml.

2. For Al Determination:
   a. Volumetric flasks, 100-ml, and assorted pipettes for making standards.
   b. Dilutor, digital automatic.
   c. Atomic absorption spectrometer (see Appendix C for set-up and operation).

Reagents

1. For Al Extraction:
   a. KCl, 1 N. 74.5 g of KCl per liter. A minimum of 700 ml of KCl is needed per run (24 samples, 1 blank, 5 standards).

2. For Al Determination:
   a. Al stock standard, 200 ppm. Pipette 20 ml of 1000 ppm of Al reference solution to a 100-ml volumetric flask and dilute to volume with H_2O.
   b. Al working standards. Pipette 0, 1, 3, 5 and 10 ml of the Al stock standard into separate 100-ml volumetric flasks, add 40 ml of 1 N KCl solution to each flask, and dilute to volume with H_2O. The resulting working standards contain 0, 2, 6, 10, and 20 ppm of Al. Prepare the working standards fresh daily.

Overview of the Procedure

Step	Result	Approx. Time
Extract with KCl	Removes extractable Al, collecting in extract	3-4 hr., including weighing
Collect extract, dilute to volume	Prepares extract for analysis	30 min.
Prepare AAS standards, calibrate AAS, and analyze samples	Provides Al concentration in ppm	30 min for std. prep.; 50-60 samples/hr for AAS

A typical extraction run consists of 23 samples, 1 standard sample (24 total), and 1 blank. An entire extraction run, including analysis, is possible in a day. Extracts can be stored (refrigerated) to be analyzed as one large batch on the AAS.

Extraction of Al

1. Prepare the mechanical vacuum extractor as described in Appendix D.

2. Add 2.00 g of soil to the middle sample syringe, recording the soil weight (ALKCLSOWT) on Laboratory Data Input Form page 5 (Appendix A).

3. Rinse any soil from the sides of the syringe with 1 N KCl and add 1 N KCl to a mark of 5 ml above the soil surface (read the volume from the side of the syringe). Stir with a glass rod (do not disturb the filter pulp). Attach the reservoir syringe and fill it with an additional 15 ml of 1 N KCl.
   NOTE: Keep the soil weight:extractant volume ratio consistent (1:10). For different soil weights, adjust the extractant volume accordingly.

4. Extract at a 6-hour setting until the KCl solution is within 0.5 cm of the sample surface (approximately 2 hours). Add 20 ml of H_2O to the reservoir syringe, and finish extracting at a 45-min. setting.

5. Transfer the extract to a 50-ml volumetric flask and make to volume with H_2O.

Al Determination by Atomic Absorption Spectrometry (AAS)

1. Prepare the AAS to analyze Al by atomic absorption (Appendix C). Calibrate the AAS with the standard solutions (readings in ppm) and determine the concentration of Al in the samples. Check the standard concentrations periodically to ensure against drift. Record the sample concentration on the Lab Input Form.

2. Dilute any sample with a concentration greater than the high standard and reanalyze on the AAS. For most samples, an additional dilution will not be necessary. Dilution factors of 2X or 5X may be needed for some soil samples.

Calculations

1. The general formula for calculating the concentration of extractable Al is:

$$\text{Al, meq/100 g} = \frac{\text{ppm sample x ml extract x 1000 x d.f. x 100}}{\text{1,000,000 x g soil x equiv. wt.}}$$

where,

ppm sample	= ppm in sample extract
ml extract	= initial extract volume (50 ml)
g soil	= oven-dry soil weight (air-dry soil wt. / moisture correction)
d.f.	= dilution factor
equiv. wt.	= 9 for Al

2. This equation is simplified (incorporating constants, extract volume, soil weight, and equivalent weight of Al) in the Soil Characterization Laboratory Database computer calculations (Appendix B):

$$\text{Al, meq/100 g} = \frac{\text{(ppm sample x d.f.) x 50}}{\text{(2.00 / MC) x 90}}$$

NOTE: In the colorimetric method (Appendix E), an initial 25X dilution factor was incorporated into the calculations for Al. This 25X factor no longer appears in the Database Computer Program (Appendix B) and must be entered manually to use the program with the colorimetric method.

References

Barnhisel, R. and P. M. Bertsch. 1982. Aluminum. p. 279-300 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Soil Survey Investigations Staff. 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rept. No. 42. National Soil Survey Center. Lincoln, NE.

Thomas, G. W. 1982. Exchangeable cations. p. 159-165 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9 (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Thurman, N. C., and E. J. Ciolkosz. 1992. A comparison of soil characterization laboratory methods: CBD-extractable Fe, Al, and Mn; KCl-extractable Al; and exchangeable acidity. Penn State University Agronomy Series (in preparation).

CHAPTER 14

EXCHANGEABLE ACIDITY -- $BaCl_2$-TEA EXTRACTION

Background and Theory

Exchangeable acidity as determined by this method represents the total titratable acidity due to exchangeable H and Al ions, and to weakly dissociated acidic groups on clay minerals and organic matter (Peech, 1965; Thomas, 1982). H in the weakly dissociated acidic groups (the pH-dependent exchange capacity) cannot be exchanged for cations of neutral salt solutions (Peech, 1965). As a result, buffered $BaCl_2$-TEA generally extracts more acidity than unbuffered KCl (Chapter 13). Some refer to the KCl acidity as "active" acidity and to the $BaCl_2$-TEA acidity as the total potential acidity (Thomas, 1982). This determination constitutes the basis of the most quantitative lime requirement methods.

Soil acidity is determined at a pH near 8.3, which is the maximum pH obtainable with $CaCO_3$ (Thomas, 1982). The Ba^{2+} replaces H^+ and Al^{3+} ions on exchange sites and weakly dissociated acidic groups, and triethanolamine (TEA), a weak base, neutralizes the acidity and buffers the solution. The high concentration of Ba^{2+} not only replaces the exchangeable Al^{3+}, but also increases the extent of hydrolysis of the absorbed Al^{3+} and the degree of dissociation of the acidic groups on the clay surface (pH-dependent). Exchangeable acidity is determined by back-titrating the unneutralized TEA in the leachate with HCl.

Materials and Equipment

1. Mechanical vacuum extractor (see Appendix D for set-up and operation).
2. 60-ml plastic syringes and syringe barrels: 1 sample tube, 1 reservoir tube, and 1 extraction syringe per sample.
3. Filter pulp, ash free.
4. Erlenmeyer flasks, wide-mouth, 250 ml.
5. Burette, 50-ml.
6. Magnetic stirrer.

Reagents

1. CO_2-free H_2O: Remove CO_2 by boiling distilled/deionized H_2O, cooling it, and storing it in a bottle equipped with an ascarite tube to absorb CO_2 from the air.

2. Extracting solution, 0.5N $BaCl_2$ - 0.055N triethanolamine (TEA), pH 8.0: Dissolve 488.84 g of $BaCl_2 \cdot 2H_2O$ in 7 liters of CO_2-free H_2O in an 8-liter Pyrex bottle. Add 16 ml of concentrated (96%) HCl and then 59 ml (65.62 g) of concentrated (7.49N) TEA. Dilute to 8 liters with CO_2-free H_2O. Mix well and adjust the pH to 8.00 ± 0.02 with HCl or TEA. This solution is 0.03 N with respect to the free base. Connect the air-inlet tube to an ascarite tube to prevent absorption of CO_2.

3. Mixed indicator solution: Dissolve 0.22 g of bromcresol green and 0.075 g of methyl red in 96 ml of 95% ethanol containing 3.5 ml of 0.1N NaOH.

4. Hydrochloric acid (HCl), 0.040 N: Add 27 ml of conc. (96%) HCl to 7 liters of H_2O and dilute to 8 liters with H_2O.

   NOTE: Standardize the HCl with THAM [Tris (Hydroxymethyl) Aminomethane] as follows: Weigh out 0.1500 g (to nearest 0.0001 g) of THAM into a 150 ml beaker. Add 25 ml deionized water and 4

drops bromcresol green-methyl red indicator. Titrate with the HCl to a faint pink endpoint. Repeat 3 times and average the results.

$$N \text{ of HCl} = \frac{\text{milliequivalent wt. of base (THAM)}}{\text{ml of HCl used in titration}} = \frac{\text{(Wt. of THAM, g)/0.12114}}{\text{ml of HCl used in titration}}$$

Record the normality of the acid (EXACID NORM) on page 7 of the Laboratory Data Input Form (Appendix A).

4. Minimum Reagents Needed Per Run (23 Samples, 1 Standard, 2 Blanks):
   a. 1300 ml of Extracting Solution
   b. 1000 to 1200 ml of HCl, depending on the acidity of the soils.

Overview of the Procedure

Step	Result	Approx. Time
Add 20 ml of extracting solution, soak overnight	Replaces acidity with Ba^{2+} and neutralizes it with TEA	Overnight
Extract, add 25 ml of extracting solution, complete extraction	Collects unneutralized TEA for back-titration	4 hr
Add indicator, titrate blank, samples with HCl	Determines the amount of unneutralized TEA	2 hr

For a typical extraction run (23 samples, 1 standard sample, 2 blanks), the samples are soaked overnight, and the extraction and titration are completed the next day.

Extraction

1. Prepare the mechanical vacuum extractor as described in Appendix D.

2. Place 2.00 g of soil evenly on top of the filter pulp. If the soil is high in organic matter, a smaller sample size may be used.

3. Add 20 ml of $BaCl_2$-TEA extracting solution to the soil, rinsing any soil from the sides of the syringe. Stir the samples, taking care not to disturb the filter pulp, and let them stand overnight.

4. Extract at a 30-minute setting until the solution is within 0.5 - 1.0 cm of the top of the sample.

5. Add 25 ml of $BaCl_2$-TEA extracting solution to the upper reservoir syringe. Extract at a 3.5 to 4 hour setting until the all of the solution is drawn through the sample.

6. Remove the lower syringe and, being careful not to lose any of the extract, press the plunger until the liquid is at the top of the syringe.

7. Lower the syringe into a beaker of $BaCl_2$-TEA and dilute the extract to 50 ml. Transfer the extract to a 250-ml beaker.

8. Prepare two blanks, each containing 50 ml of $BaCl_2$-TEA extracting solution.

Titration

1. Add 6 drops of the indicator solution to the blanks and to the samples.

2. Titrate the blanks and the samples with the standardized HCl (approximately 0.040N) to a faint pink endpoint (pH 5.1). Use an average of the blanks for the calculations.

   NOTE: The indicator will change colors from aqua/green to faint purple and finally to pink. For some soils, the endpoint will fade upon standing due to the slow dissolution of $Al(OH)_3$, which increases the pH. Ignore the fading.

3. Record the amount of acid, in ml, required to titrate the blanks (EXACID BLML, average) and the samples (EXACID SOML) on page 7 of the input form.

Calculations

1. Exchangeable acidity (EA) is calculated as follows (see Appendix B for the computer program):

   $$EA\ (meq/100\ g) = [(ml\ HCl_{Blank} - ml\ HCL_{Sample})/g\ sample\ wt.] \times N\ of\ HCl \times 100$$

   where

   g sample wt. = oven-dry sample wt. (air-dry sample wt. / moisture correction factor)

References

Peech, M. 1965. Exchange acidity. p. 905-913 in C. A. Black et al. (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. Amer. Soc. of Agron. Madison, WI.

Thomas, G. W. 1982. Exchangeable cations. p. 159-165 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

CHAPTER 15

CBD-EXTRACTABLE IRON, ALUMINUM AND MANGANESE

Background and Theory

Iron is an abundant element in soils. A large portion of this iron may be present in the form of iron oxides (which include oxides and hydroxides), coatings on minerals, cementing agents, and discrete particles (Olson and Ellis, 1982; Schwertmann and Taylor, 1989). Iron oxide minerals produce the red and brown colors common in well-drained soils. Iron oxides are important in soil classification, genesis, and chemistry investigations (Olson and Ellis, 1982; Sposito, 1989). Relative age estimates of soils use iron oxide data since the amount of iron oxides increases as weathering time increases.

The citrate-bicarbonate-dithionite (CBD) extraction described by Jackson (1969) is used. Iron oxides are reduced and removed from the soil (brought into solution) with sodium dithionite. The citrate chelates the extracted iron, aiding in the removal, and the bicarbonate buffers the pH at an optimal level (Mehra and Jackson, 1960). Because iron oxides are removed with minimal destruction of silicate clay minerals, the CBD extraction is also used to prepare soils for clay mineralogy analysis (Chapter 16).

The CBD method selectively extracts organically-complexed iron (Fe) and aluminum (Al), non-crystalline Fe and Al hydrous oxides, amorphous aluminosilicates, and crystalline Fe hydrous oxides (Wada, 1989). Crystalline Al hydrous silicates, allophane, and imogolite are not extracted. Manganese occurs in soils in the form of oxides and hydroxides (predominantly amorphous), organic complexes, and co-precipitates with Fe hydrous oxides (Gambrell and Patrick, 1982; McKenzie, 1989). The forms of extractable Mn should be similar to those of Fe.

The National Soil Survey Laboratory uses a modification of the CBD method that omits the bicarbonate buffer (SSIS, 1991). Both methods produce similar results (Thurman and Ciolkosz, 1992).

The concentration of Fe in the extract is determined by atomic absorption spectrometry (AAS). A colorimetric procedure, in which the extracted Fe is complexed with o-phenanthroline and the solution transmittance is determined at 510 nm on a spectrometer, was used in the Soil Characterization Laboratory until 1992. Both AAS and colorimetry yield similar results for Fe_2O_3 content (Thurman and Ciolkosz, 1992). The colorimetric procedure is described in Appendix E.

Materials and Equipment

1. For Fe, Al and Mn Extraction:
   a. Centrifuge tubes, 100 ml plastic, marked to indicate a 10-cm depth of liquid.
   b. Water bath.
   c. Centrifuge.
   d. Vibrating stirrer.
   e. Volumetric flasks, 500 ml.

2. For Fe, Al, and Mn Determination by AAS:
   a. Dilutor, automatic or manual, calibrated.
   b. Storage bottles, 60-ml wide-mouth polyethylene, with caps.
   c. Volumetric flasks, 100 ml, and assorted pipettes for preparing standards.
   d. Atomic absorption spectrometer (AAS).

Reagents

1. For Fe, Al and Mn Extraction:
   a. Sodium citrate ($Na_3C_6H_5O_7$), 0.3M, - sodium bicarbonate ($NaHCO_3$), 1N, buffer. 78.2 g of $Na_3C_6H_5O_7 \cdot 2H_2O$ and 9.3 g of $NaHCO_3$ per liter. Adjust the pH to 7.3 with citric acid.
   NOTE: Approximately 1.25 g of granular citric acid ($C_6H_8O_7 \cdot H_2O$) lowers the pH of 2 liters of buffer solution from 8.4 to 7.3.
   b. Sodium dithionite powder ($Na_2S_2O_4$), low in Fe.
   c. Sodium chloride (NaCl) solution, saturated. Approximately 357 g of NaCl per liter.

2. For Fe, Al, and Mn Determination by AAS:
   a. Stock standard. To a 100-ml volumetric flask, add 10 ml of 1000 ppm Fe reference solution, 20 ml of 1000-ppm Al reference solution, 5 ml of 1000-ppm Mn reference solution, 10 ml of the citrate-bicarbonate buffer. Dilute to 100 ml with H_2O.
   b. Working standards. Pipette 0, 1, 3, 4, and 6 ml of the stock standard into separate 100-ml volumetric flasks and dilute to volume with H_2O. The resulting working standards contain 0, 1, 3, 4, and 6 ppm of Fe; 0, 2, 6, 8, and 12 ppm Al; and 0, 0.5, 1.5, 2, and 3 ppm Mn. Prepare a 20 ppm Al standard by pipetting 10 ml of the stock standard into a 100-ml volumetric and make to volume. Prepare the working standards fresh daily.

3. Minimum Reagents Needed Per Run (16 Samples):
   a. 4000 ml of citrate-bicarbonate buffer.
   b. $70 \pm$ g of $Na_2S_2O_4$.
   c. 320 ml of saturated NaCl solution.

Overview of the Procedure

Step	Result	App. Time
Extraction:		
Add citrate-bicarbonate buffer, dithionite	Reduces Fe, Al and Mn, brings them into solution	1 hr.
Wash w/ NaCl, buffer (twice), collect supernatant	Extracts Fe, Al and Mn for analysis	1 hr.
Repeat the extraction steps	Ensures complete extraction of Fe, Al, Mn	2 hr.
Determination:		
Prepare standards, dilute samples for Fe, analyze samples for Fe, Al, Mn	Provides Fe, Al, and Mn concentrations in ppm	30 min for standards; 30-40 samples/hr dilution; 50-60 samples/hr for AAS

A typical extraction run consists of 15 samples plus 1 standard sample (16 total). Time estimates do not include weighing out the samples (allow approximately 30 minutes). More than one extraction run is possible in a day. Extracts for AAS analysis can be stored (refrigerated) to run as one large batch.

Procedure For Fe, Al and Mn Extraction

1. Weigh 3.00 g of air-dry soil into a 100-cm centrifuge tube.

2. Add 25 ml of citrate-bicarbonate buffer solution to the sample and stir for 30 seconds. Add an additional 25 ml of buffer and stir again. Heat the samples to 75 to 80°C in a water bath.

3. Remove the samples from the water bath, stir to bring all of the soil into suspension (use a glass rod if necessary), and add approximately 2 g of $Na_2S_2O_4$ to the sample with a scoop or dipper. Stir slowly until the initial reaction subsides, and then stir vigorously for a minimum of 60 seconds. Heat the samples in the water bath for an additional 10 to 15 minutes, stirring occasionally.
   NOTE: Jackson (1969) warns that heating the mixture above 80°C may cause black FeS to form.

4. Remove the samples from the water bath, add 10 to 15 ml of saturated NaCl solution, stir, and centrifuge at 1500 - 2000 rpm for 5 minutes (until the supernatant is clear). Pour the supernatant into a 500-ml volumetric flask, taking care not to include any soil.

5. Add 35 ml of citrate-bicarbonate buffer to the sample in the centrifuge tube, stir, and centrifuge. Pour the supernatant into the same 500-ml volumetric flask. Repeat this wash step a second time, also collecting the supernatant in the flask.

6. Repeat steps 2 through 5 a second time, collecting the supernatant in the same volumetric flask.
   NOTE: This is sufficient for complete removal of Fe oxides from most soils. However, if a red or brown color persists in the soil, repeat steps 2 through 5 again, collecting the supernatant in the same flask.

7. Add 1 to 2 g of $Na_2S_2O_4$ to the supernatant in the flask (this eliminates the yellow ferric color that often develops with time) and dilute to 500 ml with H_2O.

Atomic Absorption Spectrometry For Fe, Al, and Mn Determination

NOTE: If the sample extracts are not going to be analyzed within a day or two of the extraction, transfer an aliquot of the extracts into labeled polyethylene storage bottles and store them in a refrigerator. Allow the samples to come to room temperature before further analysis.

1. To analyze for Fe, dilute the extracts at a ratio of 1 part extract to 49 parts H_2O (50X) with an automatic dilutor. Analyze Mn and Al in the undiluted extracts.

2. Calibrate the AAS with the working standard Fe solutions (readings in ppm) and determine the concentration of Fe in the samples. Check the standard concentrations periodically to ensure against drift. Record the sample concentration (FECBDPPM) on the Lab Input Data Form page 5 (Appendix A).

3. Dilute any sample with a concentration greater than the high standard and reanalyze it. Record any additional dilution factor (FECBDDF) on the input form.
   NOTE: If the sample concentration is below the lower detection range (0.1 ppm Fe), re-check the undiluted extract. If no reading is obtained, extract again, using a larger sample of the soil.

4. Analyze the extracts for Al and Mn following the same procedure outlined in steps 3 and 4 above. Record concentrations (ALCBDPPM, MNCBDPPM) and any dilution factors (ALCBDDF, MNCBDDF) on the input form.

Calculations

1. Percent Fe and Fe_2O_3 are calculated from ppm Fe as follows:

$$\%Fe = \frac{\text{extract vol (ml) x df(1) x df(2) x ppm Fe}}{\text{oven-dry sample wt. (g) x 10,000}}$$

where: extract vol. = 500 ml
df(1) = initial dilution factor of 50 (1 part extract to 50 parts)
df(2) = additional dilution factor (FECBDDF)
ppm Fe = ppm Fe from AAS analysis (FECBDPPM)
sample wt. = air-dry wt. (3.00 g) / moisture correction factor (MC)

$$\%Fe_2O_3 = \%Fe \times 1.43$$

2. Percent Al and Mn are calculated as follows:

$$\%Al \text{ or } Mn = \frac{\text{extract vol. x df x ppm Al or Mn}}{\text{oven-dry sample wt. x 10,000}}$$

3. The equations are simplified in the Soil Characterization Lab Database computer program (Appendix B) by incorporating the constant factors into the calculations as follows:

$$\%Fe_2O_3 = \frac{\text{500 x 50 x df(2) x ppm Fe x 1.43}}{\text{(air-dry wt. / MC) x 10,000}}$$

$$\%Fe_2O_3 = (\text{FECBDPPM x FECBDDF x 3.574}) / (3.00 / MC)$$

$$\%Al \text{ or } Mn = \frac{\text{500 x df x ppm Al or Mn}}{\text{(air-dry wt. / MC) x 10,000}}$$

$$\%Al = (\text{ALCBDPPM x ALCBDDF x 0.05}) / (3.00 / MC)$$
$$\%Mn = (\text{MNCBDPPM x MNCBDDF x 0.05}) / (3.00 / MC)$$

References

Gambrell, R. P., and W. H. Patrick, Jr. 1982. Manganese. p. 313-322 in A. L. Page, R. H. Miller, and D. R. Keeney (ed). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Jackson, M. L. 1969. Soil chemical analysis -- advanced course. 2nd edition. Publ. by the author, Dept. of Soil Science, University of Wisconsin, Madison, WI 53706.

McKenzie, R. M. 1989. Manganese oxides and hydroxides. p. 439-465 in J. B. Dixon and S.B. Weed (ed). Minerals in soil environments. 2nd ed. SSSA Book Series, no. 1. Soil Sci. Soc. Amer., Madison, WI.

Mehra, O. P., and M. L. Jackson. 1960. Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate. Seventh Natl. Conf. on Clays and Clay Minerals. pp. 317-327.

Olson, R. V., and R. Ellis, Jr. 1982. Iron. p. 301-312 in A. L. Page, R. H. Miller, and D. R. Keeney (ed). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Sposito, G. 1989. The chemistry of soils. Oxford University Press. New York.

Schwertmann, U., and R. M. Taylor. 1989. Iron oxides. p. 379-438 in J. B. Dixon and S.B. Weed (ed). Minerals in soil environments. 2nd ed. SSSA Book Series, no. 1. Soil Sci. Soc. Amer., Madison, WI.

Thurman, N. C., and E. J. Ciolkosz. 1992. A comparison of soil characterization laboratory methods: CBD-extractable Fe, Al, and Mn; KCl-extractable Al; and exchangeable acidity. Penn State University Agronomy Series (in preparation).

Wada, K. 1989. Allophane and Imogolite. p. 1051-1087 in J. B. Dixon and S.B. Weed (ed). Minerals in soil environments. 2nd ed. SSSA Book Series, no. 1. Soil Sci. Soc. Amer., Madison, WI.

CHAPTER 16

CLAY PREPARATION AND X-RAY ANALYSIS

Background and Theory

Clay minerals influence many soil characteristics relating to chemistry, fertility, soil-plant interactions, physics, classification, and interpretations. Clay surfaces are the site of many chemical reactions. Soils high in kaolinite have a much lower nutrient-holding capacity than soils high in montmorillinite. Soils with a prevalence of montmorillinite will have a high shrink-swell potential that could damage building foundations.

Initial clay preparation consists of removing the cementing agents (organic matter and oxides), dispersing the soil, and separating and collecting the clay minerals. Organic matter is destroyed (oxidized) with sodium hypochlorite (bleach). Iron (and other) oxides which may bind together particles are removed using the citrate-bicarbonate-dithionite extraction described in Chapter 15. The CBD extraction removes iron oxides with minimal disturbance of the clay minerals (Mehra and Jackson, 1960).

Once collected, the clay samples are saturated with magnesium (Mg^{2+}) or potassium (K^{+}), transferred to a slide, and analyzed by X-ray diffraction.

Clay mineralogy is determined by x-ray diffraction, which is based on Bragg's Law:

$$n\lambda = 2d \sin \theta,$$

where λ is the wavelength of the x radiation, d is the distance between crystal planes, θ is an angle at which the x-rays scattered from the crystal are in phase, and n is an integer. The thickness, d, is an identifying characteristic for clay minerals. Soils can have various layer-silicate clay minerals, such as chlorite, montmorillinite, vermiculite, and kaolinite that have various d spacings. By saturating the clays with Mg and K, further identification of clay mineral species, based on different d-spacing, is possible.

Materials and Equipment

1. For Removal of Organic Matter and Iron Oxides:
   a. Centrifuge tubes, 100 ml plastic, marked to indicate a 10-cm depth of liquid
   b. Watch glasses
   c. Water bath
   d. Centrifuge
   e. Vibrating stirrer

2. For Clay Separation, Preparation and X-ray Analysis:
   a. Centrifuge
   b. Beakers, 300 ml, tall, with watch glass covers
   c. Balance, capable of weighing to 0.0001 g
   d. Bottles, 200 ml
   e. Shaker
   f. Pipette, 5 ml and assorted sizes
   g. Beakers, 50 ml
   h. Oven
   i. Centrifuge tubes
   j. Hot water bath
   k. Glass mortar

l. Rubber policeman
m. Glass slides (for K) and glass slides or plastic disks (for Mg), cleaned with alcohol and a clean cloth; numbered with a felt pen for disks and a scriber for glass slides
n. Slide drying box with glass cover plate
o. X-ray diffractometer

Reagents

1. For Removal of Organic Matter and Iron Oxides:
   a. NaOAc buffer (pH 5.0), 1 N. 82 g of anhydrous NaOAc (136 g of $NaOAc \cdot H_2O$) and 27 ml glacial HOAC per liter, adjusted to pH 5.0. This reagent is only necessary if soil pH is 7.5 or greater.
   b. NaCl solution, 1 N. 58 g NaCl per liter.
   c. Sodium hypochlorite (NaOCl) 4 to 6% (available commercially as bleach) adjusted under a hood to pH 9.0 to 9.5 with 1 N HCl. Prepare fresh daily.
   d. Reagents for iron oxide extraction (1.a-e under Reagents, Chapter 15).
   e. Methanol.

2. For Clay Separation, Preparation, and Analysis:
   a. Sodium carbonate (Na_2CO_3), dilute solution. 2 g Na_2CO_3 per 18 liters of H_2O.
   b. Saturated NaCl solution. Approximately 357 g NaCl per liter of H_2O.
   c. Methanol.
   d. Magnesium acetate (MgOAc), 1 N. Dissolve 107 g $Mg(OAc)_2$ in H_2O, add a small crystal of thymol as a preservative, and dilute to 1 liter.
   e. Potassium acetate (KOAc), 1 N. Dissolve 98 g KOAc in H_2O, add a small crystal of thymol as a preservative, and dilute to 1 liter.

Overview of the Procedure

Step	Result	Approx. Time
For soils with pH $\geq$7.5, add pH 5.0 NaOAc	Dissolves carbonates, buffers pH	45 min.
Treat with NaOCl	Oxidizes, removes organic matter	45 min. for NaOCl
Extract Fe oxides with CBD procedure (Chap. 15)	Removes Fe oxide binding agents	2-4 hr
Rinse with methanol	Removes excess Na from soil	20-30 min
Add Na_2CO_3, centrifuge, decant clay	Separates clay fraction	20-30 min
Add NaCl	Flocculates clay	1 day
Add methanol, centrifuge, decant	Reduces volume of suspension	45-60 min
Transfer to bottles, shake	Disperses clay in suspension	15-30 min

Step	Result	Approx. Time
Dry, weigh 5-ml aliquot of clay suspension	Determines volume for 0.030 g clay sample	1 day
Add MgOAc and KOAc to separate samples, wash to remove excess K or Mg	Saturates clay with Mg and K	1 hour
Transfer saturated sample to slide, dry	Prepares sample for X-ray analysis	1-2 days
Run 30° chart, glycolate Mg sample, heat K sample, run 14° chart	Identifies, distinguishes clay minerals	1-2 days

A typical run (16 samples) requires approximately 1 week. X-ray analysis is performed at an outside laboratory.

Organic Matter Removal

1. Weigh 3.00 g of air-dry soil into a 100-cm centrifuge tube. Ideally the sample should yield 0.4 to 0.5 g of clay (see particle size analysis data for the clay content of the soil). If the soil has less than 5% clay, use a 6 g sample.

2. For soils with a pH $\geq$ 7.5, add 50 ml of NaOAc (pH 5.0) to the sample and heat it for 30 min at 90°C, stirring occasionally. Centrifuge at 1500-2000 rpm for 3 min (or until the sample is separated from the liquid) and discard the supernatant. If the soil is highly calcareous, two treatments may be necessary.

3. Add 15 ml of NaOCl solution to the samples (under a hood) and stir. Place the tubes in a hot water bath (90°C) and stir occasionally for 15 minutes. Centrifuge at 1500-2000 for 5 minutes and discard the supernatant. Repeat once for A horizons with 1-4% organic carbon, twice for samples with >4% organic carbon.

4. Add 50 ml of 1 N NaCl solution in 25-ml increments, stirring between increments. Centrifuge the samples at 1500-2000 rpm for 5 minutes and discard supernatant.

Iron Oxide Removal

1. Follow the procedure for CBD extraction (steps 2 through 6) in Chapter 15. Collect the supernatant if the iron oxide content is also being determined.

2. After the CBD extraction, wash the sides of the tube with 5-10 ml of distilled H_2O. Centrifuge at 1500-2000 rpm for 5 minutes (until the clay is settled) and discard the supernatant. Add 5-10 ml of H_2O, bring to the 10 cm line with methanol, stir, centrifuge and discard. Add water to keep sample wet for fractionation.

Clay Separation and Analysis

1. Fill the tubes to the 10 cm mark with dilute $NaCO_3$ solution. Stir with a glass rod and vibrating stirrer to bring the sample into suspension. Centrifuge at exactly 750 rpm for 3 minutes. Carefully decant the clay suspension into a 300-ml tall beaker.

2. Repeat Step 1 a total of 4 times, or until 200-250 ml of clay suspension has been collected. Discard the sand and silt remaining in the tubes.

3. Add 25-50 ml of saturated NaCl solution to the clay suspension in the beaker, stir, cover with a watch glass, and allow the clay to flocculate overnight.

4. Decant as much supernatant as possible from the flocculated clay. Transfer the remaining sample to centrifuge tubes (several tubes may be needed for each sample), centrifuge until the clay is separated, and discard the supernatant. Wash once with 10-15 ml of 1:1 H_2O:methanol and once with methanol, consolidating tubes of the same sample in the process. Transfer the washed clay to 200 ml bottles with water. Shake until the clay is well-dispersed.

   NOTE: If the samples have been stored for more than a day, place them on a shaker and shake until they are thoroughly dispersed.

5. Withdraw exactly 5 ml of dispersed clay from the bottle and pipette into a pre-weighed 50-ml beaker. Dry the samples overnight at 105°C.

6. Remove the dry samples, cool in a desiccator, and weigh them to the nearest 0.0001 g.

7. Calculate the volume of clay suspension needed to give 0.030 g of clay as follows:

   V (ml) = .150 / C (g), where

   C (g) = oven dried weight of clay in 5 ml of suspension
   V (ml) = suspension volume needed to give 0.030 g of clay

8. Shake the sample bottles until the clay is dispersed. Into each of two centrifuge tubes (one labelled for Mg saturation; the other for K saturation), pipette the calculated volume of suspension (V) needed to obtain 0.030 g of clay. Save the remaining clay in the bottle in case any slides have to be remade.

9. In one sample tube, add 50 ml of MgOAc; in the second tube, add 50 ml of KOAc. Add the reagents in 25-ml increments, stirring between increments. Place the tubes in a hot water bath for 15 minutes, stirring occasionally.

10. Remove the samples from the water bath and cool slightly. Centrifuge (approx. 1500 rpm for 3 min.) and discard the supernatant solution.

11. Remove the excess K and Mg by washing the samples twice with H_2O. Wash the sides of the tubes with H_2O, adding approximately 1/2 inch of water, and heat in the hot water bath to assist flocculation. Centrifuge and discard the supernatant. Use no more H_2O than necessary as too much may cause the clays to disperse.

12. Transfer the saturated clay sample to a small glass mortar, using a small amount (about 1.5 ml) of water and a rubber policeman to remove all the sample. Mix the clay well with the rubber policeman and transfer the sample to a labelled glass slide (for K-saturated clays) or glass slide or plastic disk (for Mg-saturated clays) with an eye dropper, taking care not to break the surface tension along the edge of the slide.

13. When all samples are transferred, or the slide drying box is filled, cover the box with the glass cover plate, taking care not to jar or disturb the slides until they are dry and the clay particles have oriented themselves (usually overnight - possibly longer).

    Note: Keep the slide drying box level for an even distribution of clay across the slide. In a sloping box, the surface tension on a slide may break, starting a reaction with other wet slides, or the clay film may dry thicker on the "downhill" end of the slide. Prevent contamination by keeping the slides covered at all times.

14. When slides are dry, place them in a marked slide box in proper numbered order. The slides are now ready to be analyzed on the x-ray diffractometer.

15. Run a 30° chart on the Mg-saturated samples. Next, place the slides or disks into a desiccator with ethylene glycol in the bottom, cover, and heat the desiccator in an oven (80°C) overnight. Cool and run 14° charts on the samples.

16. Run a 30° chart on the K-saturated samples. Place the slides in an oven, turn on the oven and heat the samples at 300°C for 2 hours (do not put slides in a hot oven as the clay may peel off the slide). Shut off the furnace and cool slowly to retard peeling of clay from the slide. Open the door a little to allow the temperature to drop to 100-150°C and the open the door a little more. When temperature reaches 50-70°C, transfer the samples to a desiccator, and run a 30° or 14° chart.

17. Place the slides analyzed in step 16 on an asbestos board in the oven and heat the samples to 550°C for 2 hours. Shut down the furnace and allow the samples to cool down overnight. When temperature reaches 50-70°C, transfer the samples to a desiccator, and run a 14° chart.

18. Assemble the charts into books, and make measurements made according to instructions. County chart books from previous years provide a form and arrangement consistent with previous years.

19. Estimate the amount of various clay minerals (to the nearest 5 to 10%) using the x-ray chart patterns and record the data.

References

Anderson, J. U. 1963. An improved pretreatment for mineralogical analysis of samples containing organic matter. Clays and Clay Minerals. 10:380-388.

Jackson, M. L. 1958. Soil chemical analysis. Prentice-Hall. Englewood Cliffs, NJ.

Jackson, M. L. 1969. Soil chemical analysis -- advanced course. 2nd ed., 8th printing, 1973. Publ. by the author, Dept. of Soil Sci., Univ. of Wisconsin, Madison, WI.

Mehra, O. P., and M. L. Jackson. 1960. Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate. Seventh Natl. Conf. on Clays and Clay Minerals. pp. 317-327.

APPENDIX A

LABORATORY DATA INPUT FORMS

The raw data generated by the soil characterization analyses are recorded on these Laboratory Data Input Forms and entered into the Soil Characterization Database (Ciolkosz and Thurman, 1992). The forms consist of seven separate pages. The data are recorded with the sample number (see Chapter 2) for ease in tracking. Variables and degree of accuracy are specified in the appropriate procedure.

The program shown in Appendix B converts the raw lab data into a final form for storage and/or output. This program is part of a larger database system that includes site and field descriptions by horizon as well as the laboratory data. For more information on the entire database system, see Ciolkosz and Thurman (1992).

References

Ciolkosz, E. J., and N. C. Thurman. 1992. Penn State soil characterization database system. Penn State University Agronomy Series (in preparation).

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LAB INPUT PAGE 1 OF 7

SAMPLE NUMBER			DEPTH (cm)		D I S	HORIZON		ROCK FRAGMENTS mm (VOLUME PERCENT; WEIGHT, GRAMS)						
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LAB INPUT PAGE 2 OF 7

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Pennsylvania State University
Soil Characterization Laboratory

SAMPLE NUMBER			CATION EXCHANGE CAPACITY								EXCHANGEABLE ACIDITY			
			CEC	CA CEC		MG CEC		NA CEC		K CEC		EXACID	EXACID	EXACID	EXACID
CNT	UNI	NUM	SOILWT	PPM	DF	PPM	DF	PPM	DF	PPM	DF	SOWT	BLML	SOML	NORM
---	---	---	4.00	--.-	---	-.--	---	-.--	---	--.--	---	2.00	--.--	--.--	-.---
---	---	---	4.00	--.-	---	-.--	---	-.--	---	--.--	---	2.00	--.--	--.--	-.---
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APPENDIX B

COMPUTER PROGRAM TO INPUT AND CALCULATE LABORATORY DATA

This appendix contains the laboratory data input and output structure and discription (S&D) files and the calculation program used by the Penn State Soil Characterization Laboratory. Procedure and chapter references are added to assist the reader. This program is part of the overall Soil Characterization Database system developed at Penn State (Ciolkosz and Thurman 1992).

Laboratory Data Input Structure and Description File (LAB.S&D) Corresponding to the Lab Data Input Forms (Appendix A)

VARIABLE	VARIABLE TYPE			LABINPUT.S&D DESCRIPTION
				<u>Rock Fragment Estimates (Chapter 2)</u>
RF1VP	N	2	0	Rock fragments (>250mm [10 in.] vol. pct. estimated in the field)
RF2VP	N	2	0	Rock fragments (250mm to 76mm [10 to 3 in.] vol. pct. estimated in the field)
RF3VP	N	2	0	Rock fragments (76 to 19mm [3.0 to 0.75 in.] vol. pct. estimated in the field)
RF3WT	N	4	0	Rock fragments (76 to 19mm [3.0 to 0.75 in.] weight in grams air dried)
RF4WT	N	4	0	Rock fragments (19 to 4.7mm [0.75 in. to 4.7mm] weight in grams air dry)
RF5WT	N	4	0	Rock fragments (4.7mm to 2.0mm weight in grams air dry)
LT2MMWT	N	4	0	Less than 2mm material (< 2.0mm weight in grams air dry)
				<u>Particle Size, Pipette Method (Chapter 5)</u>
PSLABNUM	C	3		Particle Size (PS) lab number for analysis container (fleaker and bottle)
PSFLKWT	N	6	2	PS fleaker weight(oven dry)
PSFLKPSOWT	N	6	2	PS fleaker weight plus soil weight (oven dry after H2O2 treatment and candling)
PSBWT20UM	N	6	3	PS bottle weight for 20 um pipette fraction (oven dry)
PSBWT5UM	N	6	3	PS bottle weight for 5 um pipette fraction (oven dry)
PSBWT2UM	N	6	3	PS bottle weight for 2 um pipette fraction (oven dry)
PSBP20UMWT	N	6	3	PS bottle plus 20 um pipette fraction weight (oven dry), incl. all <20 um material
PSBP5UMWT	N	6	3	PS bottle plus 5 um pipette fraction weight (oven dry), incl. all < 5 um material
PSBP2UMWT	N	6	3	PS bottle plus 2 um pipette fraction weight (oven dry), incl. all < 2 um material
PSBLANKWT	N	5	3	PS Na hexametaphosphate blank wt. (25ml pipette, 10 ml Na hex. per 1000 ml)
PSLABNUM2	C	3		PS lab number for analysis container (beaker)
VCSANDWT	N	5	2	Very coarse sand weight (2.0 to 1.0mm fraction) (oven dry)
CSANDWT	N	5	2	Coarse sand weight (1.0 to 0.5mm fraction) (oven dry)
MSANDWT	N	5	2	Medium sand weight (0.5 to 0.25mm fraction) (oven dry)
FSANDWT	N	5	2	Fine sand weight (0.25 to 0.10mm fraction) (oven dry)
VFSAND1WT	N	5	2	Very fine sand 1 weight (0.10 to 0.074mm) (oven dry)
VFSAND2WT	N	5	2	Very fine sand 2 weight (0.074 to 0.050mm) (oven dry)
TCLASSF	C	4		Textural classification estimated in the field
				<u>Bulk Density, 1/3 Atm. Water Content, COLE (Chapter 7)</u>
CLLETTERa	L	1		Clod identification letter (a,b,c etc.)
CLWTMAIRa	N	6	2	Clod weight moist (0.33 atmosphere moisture content) weighted in air
CLWTMWATa	N	6	2	Clod weight moist (0.33 atmosphere moisture content) weighted in water
CLWTODAIRa	N	6	2	Clod weight oven dry moisture content weighted in air

VARIABLE	VARIABLE TYPE			DESCRIPTION	LABINPUT.S&D
CLWTODWATa	N	6	2	Clod weight oven dry moisture content weighted in water	
CLRFWTAIRa	N	6	2	Clod rock fragment weight oven dry moisture content weighted in air	
CLRFWTWATa	N	6	2	Clod rock fragment weight oven dry moisture content weighted in water	

Repeat the sequence for clod identification letters b and c

15 Atm. Water Content (Chapter 7)

CAN15NUM	C	2		Can number for 15 atm. moisture content determination of < 2mm material
CAN15WT	N	6	3	Can weight for 15 atm. moisture content determination of < 2mm material
CAN15AMWT	N	6	3	Can plus soil weight of 15 atm. moisture content < 2mm material
CAN15AODWT	N	6	3	Can plus soil weight of 15 atm. oven dry moisture content < 2mm material

Reaction/pH (Chapter 8)

PHWL	N	3	1	pH (water, lab)
PHWF	N	3	1	pH (water, field)
PHKCLL	N	3	1	pH (KCl, lab)
PHKCLF	N	3	1	pH (KCl, field)
PHCACL2L	N	3	1	pH (CaCl2, lab)
PHCACL2F	N	3	1	pH (CaCl2, field)

Organic C (Chapters 10, 11)

OCMETH	C	1		Organic carbon method (W = Walkly-Black; C = combusion)
OCBLANKWT	N	5	3	Organic carbon blank weight (procedure without soil;grams)
OCBWT	N	8	4	Organic carbon bottle weight
OCBPCO2WT	N	8	4	Organic carbon bottle plus CO2 weight
OCSOILWT	N	5	3	Organic carbon soil sample weight

Moisture Correction Factor (Chapter 4)

MCCNUM	C	2		Moisture correction can number
MCCWT	N	6	3	Moisture correction can weight
MCCPADSOWT	N	6	3	Moisture correction can plus air dry soil weight
MCCPODSOWT	N	6	3	Moisture correction can plus oven dry soil weight
MC	N	5	3	Moisture correction factor (factor to convert air dry to oven dry weight basis)

Extractable Bases and CEC (Chapter 12)

CACECPPM	N	4	1	Exchangeable calcium (parts per million; 0, 2, 5, 8, 10 ppm standards)
CACECDF	N	3	0	Exchangeable calcium, dilution factor (e.g., 2X, 5X, 10X, etc.)
MGCECPPM	N	4	2	Exchangeable magnesium (parts per million; 0, 0.2, 0.5, 0.8, 1.0 ppm standards)
MGCECDF	N	3	0	Exchangeable magnesium, dilution factor (e.g., 2X, 5X, 10X, etc.)
NACECPPM	N	4	2	Exchangeable sodium (parts per million; 0, 0.4, 1.0, 1.6, 2.0 ppm standards)
NACECDF	N	3	0	Exchangeable sodium, dilution factor (e.g., 2X, 5X, 10X, etc.)
KCECPPM	N	5	2	Exchangeable potassium (parts per million; 0, 2, 5, 8, 10 ppm standards)
KCECDF	N	3	0	Exchangeable potassium, dilution factor (e.g., 2X, 5X, 10X, etc.)
CECSOILWT	N	4	2	Cation exchange capacity soil weigth (air dry, grams)

VARIABLE	VARIABLE TYPE			DESCRIPTION	LABINPUT.S&D
				BaCl2-TEA Extractable Acidity (Chapter 14)	
EXACIDBLML	N	5	2	Extractable acidity (ml of acid used in titration of blank)	
EXACIDSOML	N	5	2	Extractable acidity (ml of acid used in titration of soil sample)	
EXACIDNORM	N	5	3	Extractable acidity (normality of acid used in the titration)	
EXACIDSOWT	N	4	2	Extractable acidity soil weight (air dry, grams)	
				KCl-Extractable Al (Chapter 13)	
ALKCLSOWT	N	4	2	KCl-extractable Aluminum (KCl-Al) soil weight (air dry, grams)	
ALKCLPPM	N	4	2	KCl-extractable Al (ppm; 0, 2, 6, 10, 20 ppm standards)	
ALKCLDF	N	3	0	KCl-extractable Al, dilution factor (e.g., 2X, 5X, 10X, etc.)	
				CBD-Extractable Al, Mn, and Fe (Chapter 15)	
ALCBDPPM	N	4	1	CBD-extractable Al (ppm; 0, 2, 6, 10, 20 ppm standards)	
ALCBDDF	N	3	0	CBD-extractable Al, dilution factor (e.g., 2X, 5X, 10X, etc.)	
MNCBDPPM	N	4	2	CBD-extractable Mn (ppm; 0, 0.5, 1.5, 2.0, 3.0 ppm standards)	
MNCBDDF	N	3	0	CBD-extractable Mn, dilution factor (e.g., 2X, 5X, 10X, etc.)	
FECBDPPM	N	4	2	CBD-extractable Fe (ppm; 0, 1, 3, 4, 6 ppm standards)	
FECBDDF	N	3	0	CBD-extractable Fe, dilution factor (e.g., 2X, 5X, 10X, etc.)	
FECBDSOWT	N	4	2	CBD-extractable Fe, Al, Mn, soil weight (air dry, grams)	

Laboratory Data (Output) Structure and Description File (LAB.S&D)

VARIABLE	VARIABLE TYPE			DESCRIPTION	LABDATA.S&D
				Site Location and Horizon Information (Chapter 2)	
NUMBER	C	9		State, County (001 to 067), and pedon number (sequential within a county)	
SERIESNAME	C	18		Current soil series name	
HORIZONNUM	N	2	0	Horizon number (sequential from surface down)	
UDEPTH	N	3	0	Upper depth of horizon (centimeters)	
LDEPTH	N	3	0	Lower depth of horizon (centimeters)	
HORDISCONT	C	1		Horizon lithologic discontinunity	
MASTERLETT	C	3		Horizon master letter	
HORISUFFIX	C	4		Horizon suffix	
				Rock Fragments, Weight Percentages (Chapter 6)	
RF1WTP	N	4	1	Rock fragments (>250mm [10 in.] weight percent)	
RF2WTP	N	4	1	Rock fragments (250 to 76mm [10 to 3 in.] weight percent)	
RF3WTP	N	4	1	Rock fragments (76 to 19mm [3.0 to 0.75 in.] weight percent)	
RF4WTP	N	4	1	Rock fragments (19mm to 4.7mm [0.75 in. to 4.7mm] weight percent)	
RF5WTP	N	4	1	Rock fragments (4.7 mm to 2.0 mm fraction,weight percent)	
RFTWTP	N	5	1	Rock fragments total weight percent of <2mm + >2mm material	
LT2MMWTP	N	5	1	Less than 2mm weight percent of total soil material (<2mm + >2mm)	

VARIABLE	VARIABLE TYPE			DESCRIPTION
				Particle Size Distribution (Chapter 5)
VCSAND	N	4	1	Very coarse sand (2.0 to 1.0 mm fraction, pct. by weight of < 2.0 mm material)
CSAND	N	4	1	Coarse sand (1.0 to 0.5 mm fraction, pct. by weight of 2.0 mm material)
MSAND	N	4	1	Medium sand (0.5 to 0.25 mm fraction, pct. by weight of < 2.0 mm material)
FSAND	N	4	1	Fine sand (0.25 to 0.10 mm fraction, pct. by weight of < 2.0 mm material)
VFSAND	N	4	1	Very fine sand (0.1 to 0.05 mm fraction, pct. by weight of < 2.0 mm material)
VFSAND1	N	4	1	Very fine sand (0.10 to 0.07 mm fraction, < 2 mm material)
VFSAND2	N	4	1	Very fine sand (0.07 to 0.05 mm fraction, pct. by weight of < 2.0 mm material)
TSAND	N	5	1	Total sand (2.0 to 0.5 mm fraction, pct. by weight of < 2.0 mm material)
TSILT	N	5	1	Total silt (0.05 to 0.002 mm fraction, pct. by weight of < 2.0 mm material)
TCLAY	N	5	1	Total clay (< 0.002 mm fraction, pct. by weight of < 2.0 mm material)
CSILT	N	4	1	Coarse silt (0.05 to 0.02 mm fraction, pct. by weight of < 2.0 mm material)
CMSILT	N	4	1	Coarse + Medium silt (0.05 to 0.005 mm fract., pct. by wt. of < 2.0 mm mater.)
MSILT	N	4	1	Medium silt (0.02 to 0.005 fraction, pct. by weight of < 2.0 mm material)
MFSILT	N	4	1	Medium + Fine silt (0.02 to 0.002 fraction, pct. by weight of < 2.0 mm material)
FSILT	N	4	1	Fine silt (0.005 to 0.002 mm fraction, pct. by weight of < 2.0 mm material)
TCLASSL	C	4		Textural class (determined in the laboratory)
TCLASSF	C	4		Textural class (estimated in the field)
				Bulk Density, Porosity, COLE (Chapter 7)
BDMCLWRF	N	4	2	Bulk density moist clod with rock fragments(g/cc , 1/3 atmosphere moisture)
BDMLT2MM	N	4	2	Bulk density moist less than 2mm material (g/cc, 1/3 atmosphere moisture)
BDMLT2UC	N	4	2	Bulk density moist <2mm mat. (core uncorrected for rock frags.; g/cc, 1/3 atm.)
BDDLT2MM	N	4	2	Bulk density dry less than 2mm material(g/cc , oven dry)
BDGT2MM	N	4	2	Bulk density greater than 2mm material (rock fragments, g/cc, oven dry)
BDTSOIL	N	4	2	Bulk density total soil(g/cc ,<2mm + >2mm material, 1/3 atm. moisture)
BDMETH	C	1		Bulk density method (C = clod method, R = core method)
RFTVP	N	5	1	Rock fragments total volume percent (calculated from weight & BD data)
PORETSOIL	N	2	0	Porosity total soil (rock fragments + fine earth) vol. pct.
PORELT2MM	N	2	0	Porosity, <2mm (fine earth basis only, rock fragments not included) vol. pct.
COLELT2MM	N	5	3	Coefficient of linear extensibility of less than 2mm material
				Water Retention, Water Retention Difference (Chapter 7)
M03ACLWTP	N	5	1	Moisture content 1/3 atmosphere clod with rock fragments,weight percent
M03ALT2WTP	N	4	1	Moisture content 1/3 atmosphere less than 2mm weight percent
M03ALT2UC	N	5	1	Moisture content 1/3 atm. <2mm wt. pct. (core uncorrected for rock fragments)
M15ALT2WTP	N	4	1	Moisture content 15 atmosphere less than 2mm weight percent
M3LT2BUWTP	N	4	1	Moisture content 1/3 atm. <2mm bulk sample (not from clod or core) weight pct.
AWLT2MMWTP	N	5	1	Available water less than 2mm weight percent
AWLT2MMVP	N	5	3	Available water less than 2mm volume basis(cm/cm)
AWTSOILWTP	N	5	1	Available water total soil(rock fragments + less than 2mm, weigth percent)
AWTSOILVP	N	5	3	Available water total soil(rock fragments + less than 2mm, volume basis(cm/cm)
				Reaction/pH (Chapter 8)
PHWL	N	3	1	pH (1:1 water, lab)
PHWF	N	3	1	pH (1:1 water, field)

VARIABLE	VARIABLE TYPE	DESCRIPTION
PHKCLL	N 3 1	pH (1:1 KCl, lab)
PHKCLF	N 3 1	pH (1:1 KCl, field)
PHCACL2L	N 3 1	pH (1:1 CaCl2, lab)
PHCACL2F	N 3 1	pH (1:1 CaCl2, field)
		Organic C (Ch. 10, 11), CaCO3 Equivalent (Ch. 9), Total Sulfur (Ch. 10)
OCWTP	N 5 2	Organic carbon weigh percent(< 2 mm material)
OCMETH	C 1	Organic carbon method (W = Walkly-Black; C = combusion)
TN	N 5 2	Total nitrogen (Kjeldal method)
CN	N 6 2	Carbon/nitrogen ratio
CACO3EQ	N 5 1	Calcium carbonate equivalent (percent < 2 mm material)
TSULFWTP	N 5 3	Total sulfur weight percent (< 2 mm material)
		Extractable Bases, CEC (Ch. 12), Acidity (Ch. 14), KCl-Al (Ch. 13)
CA	N 4 1	Calcium exchangeable (meq/100g)
MG	N 4 1	Magnesium exchangeable (meq/100g)
NA	N 5 2	Sodium exchangeable (meq/100g)
K	N 5 2	Potassium exchangeable (meq/100g)
TB	N 5 1	Total bases exchangeable (meq/100g)
CAMG	N 5 1	Calcium/Magnesium Ratio
ALKCL	N 4 1	Aluminum exchangeable with 1N KCL (meq/100g)
EXACID	N 5 1	Extractable acidity (meq/100g)
CECSUM	N 5 1	Cation exchange capacity - sum of cation method(meq/100g)
CECNH4	N 5 1	Cation exchange capacity - ammonium acetate method(meq/100g)
BSSUM	N 5 1	Base saturation - sum of cations method(percent)
BSNH4	N 5 1	Base saturation - ammonium acetate method(percent)
ACIDSOLK	N 6 1	Acid soluble potassium (pounds/acre K; 1.0 N HNO3 method)
		CBD-Extractable Fe, Mn, Al (Chapter 15)
FE2O3	N 4 1	Iron oxides (percent < 2 mm material)
ALCBD	N 4 1	Aluminum extractable with CBD (percent)
MNCBD	N 4 1	Manganese extractable with CBD (percent)
		Clay Mineralogy (Chapter 16)
KAOL	C 2	Kaolinite (percent < 2 um clay)
ILL	C 2	Illite (percent < 2 um clay)
VERM	C 2	Vermiculite (percent < 2 um clay)
MONT	C 2	Montmorillonite (percent < 2 um clay)
CHLOR	C 2	Chlorite (percent < 2 um clay)
INTER	C 2	Intergrade (percent < 2 um clay)
QUARTZ	C 2	Quartz (percent < 2 um clay)

Laboratory Data Input and Calculation Program (LABINCAL.PRG)

The portion of the soil characterization database system computer program illustrated here converts the raw laboratory data into final lab results. For more information on the database system, see Ciolkosz and Thurman (1992). The calculations are explained in each chapter (chapter references have been added to each section to assist the reader). Programming comments are preceded by an asterisk (*).

```
*************************************************************************************
* PROGRAM      : LABINCAL.PRG                                                       *
* AUTHOR       : JERRY V. CIOLKOSZ                                                  *
* DATE         : MAY 23, 1992                                                       *
* DESCRIPTION  : CALCULATION ROUTINES LABINPUT.DBF FIELDS                           *
*              :                                                                    *
*************************************************************************************

PARAMETERS mCLODLETTER
*************************************************************
** BULK DENSITY (BD) **                                                (Chapter 7)
*************************************************************
CLWTMAIR=0
zCLWTMAIR='CLWTMAIR'+mCLODLETTER
CLWTMAIR=&zCLWTMAIR

CLWTMWAT=0
zCLWTMWAT='CLWTMWAT'+mCLODLETTER
CLWTMWAT=&zCLWTMWAT

CLWTODAIR=0
zCLWTODAIR='CLWTODAIR'+mCLODLETTER
CLWTODAIR=&zCLWTODAIR

CLWTODWAT=0
zCLWTODWAT='CLWTODWAT'+mCLODLETTER
CLWTODWAT=&zCLWTODWAT

CLRFWTAIR=0
zCLRFWTAIR='CLRFWTAIR'+mCLODLETTER
CLRFWTAIR=&zCLRFWTAIR

CLRFWTWAT=0
zCLRFWTWAT='CLRFWTWAT'+mCLODLETTER
CLRFWTWAT=&zCLRFWTWAT

* 1)
mCLVOLMOIST=CLWTMAIR-CLWTMWAT
* 2)
mCLVOLDRY = CLWTODAIR-CLWTODWAT
* 3)
mCLRFVOL = CLRFWTAIR-CLRFWTWAT
* 4)
mBDMCLWRF = IF(mCLVOLMOIST>0,CLWTODAIR/mCLVOLMOIST,0)
* 5)
mBDMLT2MM = IF(mCLVOLMOIST-mCLRFVOL>0,(CLWTODAIR-CLRFWTAIR)/(mCLVOLMOIST-mCLRFVOL),0)
```

```
* 6)
mBDDLT2MM = IF(mCLVOLDRY-mCLRFVOL>0,(CLWTODAIR-CLRFWTAIR)/(mCLVOLDRY-mCLRFVOL),0)
* 7)
mBDGT2MM = IF(mCLRFVOL>0,CLRFWTAIR/mCLRFVOL,0)
* 8)
**        cubed root  -1
mCOLELT2MM = IF(mBDMLT2MM>0,EXP(1/3*LOG(mBDDLT2MM/mBDMLT2MM)) -1,0)

** continued after rock fragments

*****************************************************************
** ROCK FRAGMENTS (RF) **                                                    (Chapter 6)
*****************************************************************
* 1)
mVOLLT76=RF4WT/IF(mBDGT2MM<>0,mBDGT2MM,2.45) + RF5WT/IF(mBDGT2MM<>0,mBDGT2MM,2.45) +
LT2MMWT/IF(mBDMLT2MM<>0,mBDMLT2MM,1.5)
* 2)
IF RF1VP+RF2VP+RF3VP <> 100
 mVOLRF1=mVOLLT76*RF1VP/(100-(RF1VP+RF2VP+RF3VP))
 mVOLRF2=mVOLLT76*RF2VP/(100-(RF1VP+RF2VP+RF3VP))
 mVOLRF3=mVOLLT76*RF3VP/(100-(RF1VP+RF2VP+RF3VP))
ELSE
 mVOLRF1=0
 mVOLRF2=0
 mVOLRF3=0
ENDIF
* 3)
mRF1WTV=mVOLRF1*IF(mBDGT2MM<>0,mBDGT2MM,2.45)
mRF2WTV=mVOLRF2*IF(mBDGT2MM<>0,mBDGT2MM,2.45)
mRF3WTV=mVOLRF3*IF(mBDGT2MM<>0,mBDGT2MM,2.45)
* 4)
mTSOILWT=mRF1WTV+mRF2WTV+RF3WT+RF4WT+RF5WT+LT2MMWT
* 5)
mRF1WTP=  IF(mTSOILWT>0,mRF1WTV/mTSOILWT*100,0)
mRF2WTP=  IF(mTSOILWT>0,mRF2WTV/mTSOILWT*100,0)
mRF3WTP=  IF(mTSOILWT>0,RF3WT/mTSOILWT*100  ,0)
mRF4WTP=  IF(mTSOILWT>0,RF4WT/mTSOILWT*100  ,0)
mRF5WTP=  IF(mTSOILWT>0,RF5WT/mTSOILWT*100  ,0)
mLT2MMWTP=IF(mTSOILWT>0,LT2MMWT/mTSOILWT*100,0)
* 6)
mRFTWTP=mRF1WTP+mRF2WTP+mRF3WTP+mRF4WTP+mRF5WTP

** bulk density continued                                                    (Chapter 7)
* 9)
IF mBDMLT2MM > 0 .OR. BDMLT2UC > 0
 mBDTSOIL =mLT2MMWTP/100*IF(mBDMLT2MM > 0,mBDMLT2MM,BDMLT2UC) + mRFTWTP/100*IF(mBDGT2MM
<> 0,mBDGT2MM,2.45)
ELSE
 mBDTSOIL=0
ENDIF
* 10)
mRFTVP = mBDTSOIL*mRFTWTP/IF(mBDGT2MM<>0,mBDGT2MM,2.45)
* 11)
*  CLOD AVERAGING ROUTINE
```

```
************************************************************
** PORE SPACE (PORE) **                                                    (Chapter 7)
************************************************************

mPORELT2MM= 100 - (mBDMLT2MM/2.60*100)

mPORETSOIL= mPORELT2MM*((100.0-mRFTVP)/100)

************************************************************
** PARTICLE SIZE (PS) **                                                   (Chapter 5)
************************************************************
* 1)
mSOILWT=PSFLKPSOWT-PSFLKWT
* 2)
m20PUMWT = (PSBP20UMWT-PSBWT20UM-PSBLANKWT)*40
m5PUMWT  = (PSBP5UMWT-PSBWT5UM-PSBLANKWT)*40
mTCLAYWT = (PSBP2UMWT-PSBWT2UM-PSBLANKWT)*40
* 3)
mMSILTWT = m20PUMWT - m5PUMWT
mFSILTWT = m5PUMWT  - mTCLAYWT
* 4)
mVCSAND  = ROUND(IF(mSOILWT>0,VCSANDWT  / mSOILWT *100,0)          ,1)
mCSAND   = ROUND(IF(mSOILWT>0,(CSANDWT-VCSANDWT) / mSOILWT *100,0)  ,1)
mMSAND   = ROUND(IF(mSOILWT>0,(MSANDWT-CSANDWT) / mSOILWT *100,0)   ,1)
mFSAND   = ROUND(IF(mSOILWT>0,(FSANDWT-MSANDWT) / mSOILWT *100,0)   ,1)
mVFSAND1 = ROUND(IF(mSOILWT>0,(VFSAND1WT-FSANDWT) / mSOILWT *100,0) ,1)
mVFSAND2 = ROUND(IF(mSOILWT>0,(VFSAND2WT-VFSAND1WT) / mSOILWT *100,0),1)
* 5)
mMSILT   = ROUND(IF(mSOILWT>0,mMSILTWT / mSOILWT *100,0),1)
mmmFSILT = ROUND(IF(mSOILWT>0,mFSILTWT / mSOILWT *100,0),1)
mTCLAY   = ROUND(IF(mSOILWT>0,mTCLAYWT / mSOILWT *100,0),1)
* 6)
mTSAND = mVCSAND+mCSAND+mMSAND+mFSAND+mVFSAND1+mVFSAND2
* 7)
mVFSAND = mVFSAND1+mVFSAND2
* 8)
mCSILT = IF(mTSAND+mMSILT+mmmFSILT+mTCLAY>0,100 - (mTSAND+mMSILT+mmmFSILT+mTCLAY),0)
* 9)
mTSILT = mCSILT+mMSILT+mmmFSILT
* 10)
mCMSILT = mCSILT+mMSILT
* 11)
mMFSILT = mMSILT+mmmFSILT
* 12) rounding correction
mTSAND=ROUND(mTSAND,1)
mTSILT=ROUND(mTSILT,1)
mTCLAY=ROUND(mTCLAY,1)
IF mTSAND+mTSILT+mTCLAY <> 100
  IF 100-(mTSAND+mTSILT+mTCLAY) <= 0.2 .AND. 100-(mTSAND+mTSILT+mTCLAY) >= -0.2
    mTCLAY=100-mTSAND-mTSILT
  ENDIF
ENDIF
```

```
* 13) ** TEXTURAL CLASSIFICATION **

** sands **
IF mTSAND > =85 .AND. 1.5*mTCLAY+mTSILT < = 15
 IF mCSAND+mVCSAND> =25.AND.mMSAND<50.AND.mFSAND<50.AND.mVFSAND<50
  mTCLASSL='COS '
 ELSEIF mVCSAND+mCSAND+mMSAND> =25.AND.mVCSAND+mCSAND<25.AND.mFSAND<50.AND.mVFSAND<50
  mTCLASSL='S  '
 ELSEIF mFSAND> =50 .OR. (mVCSAND+mCSAND+mMSAND<25 .AND. mVFSAND<50)
  mTCLASSL='FS '
 ELSEIF mVFSAND > = 50
  mTCLASSL='VFS '
 ELSE
  mTCLASSL='  '
 ENDIF

** loamy sands **
ELSEIF
(mTSAND>85.AND.mTSAND<90.AND.1.5*mTCLAY+mTSILT> =15).OR.(mTSAND> =70.AND.mTSAND< =85.AND.2*
mTCLAY+mTSILT< =30)
 IF mCSAND+mVCSAND> =25.AND.mMSAND<50.AND.mFSAND<50.AND.mVFSAND<50
  mTCLASSL='LCOS'
 ELSEIF mVCSAND+mCSAND+mMSAND> =25.AND.mVCSAND+mCSAND<25.AND.mFSAND<50.AND.mVFSAND<50
  mTCLASSL='LS '
 ELSEIF mFSAND> =50 .OR. (mVCSAND+mCSAND+mMSAND<25 .AND. mVFSAND<50)
  mTCLASSL='LFS '
 ELSEIF mVFSAND > = 50
  mTCLASSL='LVFS'
 ELSE
  mTCLASSL='  '
 ENDIF

** sandy loams **
ELSEIF
(mTCLAY<20.AND.mTSILT+2*mTCLAY>30.AND.mTSAND> =52).OR.(mTCLAY<7.AND.mTSILT<50.AND.mTSAND>4
3.AND.mTSAND<52)
 IF mCSAND+mVCSAND> =25.AND.mMSAND<50.AND.mFSAND<50.AND.mVFSAND<50
  mTCLASSL='COSL'
 ELSEIF mVCSAND+mCSAND+mMSAND> =30.AND.mVCSAND+mCSAND<25.AND.mFSAND<30.AND.mVFSAND<30
  mTCLASSL='SL '
 ELSEIF
(mFSAND> =30.AND.mVFSAND<30).OR.(mVCSAND+mCSAND+mMSAND>15.AND.mVCSAND+mCSAND+mMSAND
<30).OR.(mFSAND+mVFSAND>40.AND.mFSAND> =mVFSAND.AND.mVCSAND+mCSAND+mMSAND<15)
  mTCLASSL='FSL '
 ELSEIF mVFSAND > = 30 .OR.
(mVFSAND+mFSAND>40.AND.mVFSAND> =mFSAND.AND.mVCSAND+mCSAND+mMSAND<15)
  mTCLASSL='VFSL'
 ELSE
  mTCLASSL='  '
 ENDIF

** loam **
ELSEIF mTCLAY> =7.AND.mTCLAY<27.AND.mTSILT> =28.AND.mTSILT<50.AND.mTSAND<52
 mTCLASSL='L  '
```

```
** silt loam **
ELSEIF (mTSILT> =50.AND.mTCLAY> =12.AND.mTCLAY<27).OR.(mTSILT> =50.AND.mTSILT<80.AND.mTCLAY<12)
 mTCLASSL='SIL '

** silt **
ELSEIF mTSILT> =80.AND.mTCLAY<12
 mTCLASSL='SI  '

** sandy clay loam **
ELSEIF mTCLAY> =20.AND.mTCLAY<35.AND.mTSILT<28.AND.mTSAND> =45
 mTCLASSL='SCL '

** clay loam **
ELSEIF mTCLAY> =27.AND.mTCLAY<40.AND.mTSAND> =20.AND.mTSAND<45
 mTCLASSL='CL  '

** silty clay loam **
ELSEIF mTCLAY> =27.AND.mTCLAY<40.AND.mTSAND<20
 mTCLASSL='SICL'

** sandy clay **
ELSEIF mTCLAY> =35.AND.mTSAND> =45
 mTCLASSL='SC  '

** silty clay **
ELSEIF mTCLAY> =40.AND.mTSILT> =40
 mTCLASSL='SIC '

** clay **
ELSEIF mTCLAY> =40.AND.mTSAND<45.AND.mTSILT<40
 mTCLASSL='C   '

ELSE
 mTCLASSL='    '

ENDIF

*****************************************************************
** AVAILABLE WATER (AW) **                                    (Chapter 7)
*****************************************************************
* 1)
mM03ACLWTP = IF(CLWTODAIR>0,(CLWTMAIR-CLWTODAIR)/CLWTODAIR*100,0)
* 2)
mM03ALT2WTP = IF(CLWTODAIR-CLRFWTAIR>0,(CLWTMAIR-CLWTODAIR)/(CLWTODAIR-CLRFWTAIR)*100,0)
* 3)
mM15ALT2WTP = IF(CAN15AODWT-CAN15WT>0,(CAN15AMWT-CAN15AODWT)/(CAN15AODWT-CAN15WT)*100,0)
* 4)
IF (mM03ALT2WTP=0.AND.M03ALT2UC=0).OR.mM15ALT2WTP=0
 mAWLT2MMWTP  = 0
ELSE
 mAWLT2MMWTP  = IF(IF(mM03ALT2WTP>0,mM03ALT2WTP,M03ALT2UC)-
mM15ALT2WTP>0,IF(mM03ALT2WTP>0,mM03ALT2WTP,M03ALT2UC)-mM15ALT2WTP,0)
ENDIF
```

```
* 5)
mAWLT2MMVP  = mAWLT2MMWTP*IF(mBDMLT2MM>0,mBDMLT2MM,BDMLT2UC)/100
* 6)
mAWTSOILWTP = mAWLT2MMWTP*mLT2MMWTP/100
* 7)
mAWTSOILVP = mAWLT2MMVP*((100.0-mRFTVP)/100)

*****************************************************************
** MOISTURE CORRECTION (MC) **                                          (Chapter 4)
*****************************************************************
mMC=IF(MCCPODSOWT-MCCWT>0,1+((MCCPADSOWT-MCCPODSOWT)/(MCCPODSOWT-MCCWT)),1)
mMC=IF(mMC=0,1,mMC)

*****************************************************************
** ALUMINUM, MANGANESE AND IRON (AL, MN, FE) **                         (Chapter 15)
*****************************************************************

mFE2O3=FECBDPPM*IF(FECBDDF>0,FECBDDF,1)*3.574/(3.00/mMC)
* 3.00g = FeCBDSOWT

mMNCBD=MNCBDPPM*IF(MNCBDDF>0,MNCBDDF,1)*0.05/(3.00/mMC)
* 3.00g = FeCBDSOWT

mALCBD=ALCBDPPM*IF(ALCBDDF>0,ALCBDDF,1)*0.05/(3.00/mMC)
* 3.00g = FeCBDSOWT

mALKCL=(ALKCLPPM*IF(ALKCLDF>0,ALKCLDF,1)*50)/((2.00/mMC)*90)            (Chapter 13)
* for 2.00g (ALKCLSOWT) samples only

*****************************************************************
** ORGANIC CARBON (OC) **                                        (Chapters 10, 11)
*****************************************************************
* 1)
mCO2WT = OCBPCO2WT - OCBWT
* 2)
mOCWTP = IF (OCSOILWT*mMC>0,((mCO2WT-OCBLANKWT)*.2727)/(OCSOILWT/mMC)*100,0)

*****************************************************************
** EXCHANGE ACIDITY (EXACID) **                                         (Chapter 14)
*****************************************************************

mEXACID=((EXACIDBLML-EXACIDSOML)*EXACIDNORM)*100/(2.00/mMC)
* for 2.00g (EXACIDSOWT) samples only

*****************************************************************
** EXCHANGEABLE CATIONS (CA,Mg,Na,K),                                   (Chapter 12)
BASE SATURATION (BSSUM) AND CATION EXCHANGE CAPACITY (CECSUM) *
*****************************************************************
mNA=NACECPPM*IF(NACECDF>0,NACECDF,1)*0.217/(4.00/mMC)
* 4.00g = CECSOILWT

mK=KCECPPM*IF(KCECDF>0,KCECDF,1)*0.128/(4.00/mMC)
* 4.00g = CECSOILWT
```

```
mCA = CACECPPM * IF(CACECDF > 0,CACECDF,1) * 6.25/(4.00/mMC)
* 4.00g = CECSOILWT

mMG = MGCECPPM * IF(MGCECDF > 0,MGCECDF,1) * 10.25/(4.00/mMC)
* 4.00g = CECSOILWT

mTB = mNA + mK + mCA + mMG

mCAMG = IF(mMG < > 0,mCA/mMG,0)

mCECSUM = mNA + mK + mCA + mMG + mEXACID

mBSSUM = IF(mCECSUM < > 0,((mNA + mK + mCA + mMG)/mCECSUM) * 100 ,0)

*********************************************************************************
```

References

Ciolkosz, E. J., and and N. C. Thurman. 1992. Penn State soil characterization database system. Penn State Univ. Agron. Series (In preparation).

APPENDIX C

THE ATOMIC ABSORPTION SPECTROPHOTOMETER

Background and Theory

The principle behind atomic absorption spectrometry (AAS) is that atoms can be excited by the addition of light (photons) or, after excitation, can emit photons, as explained by the reaction:

$$\underset{\text{Absorption}}{M + hr \; ---> M^*} \; ---> \; \underset{\text{Emission}}{M + hr}$$

where M is the neutral atom, M* is the excited atom, h is Planck's constant, and r is the light frequency (Baker and Suhr, 1982).

In atomic absorption, light with a frequency characteristic of a particular atom is passed through a flame which contains that atom (an aspirated sample). The amount of light absorbed by the atoms in the flame is measured and is proportional to the concentration of the atom in the original solution. In flame emission, atoms are excited by the heat of the flame and emit photons of light of a wavelength characteristic of that atom as they fall back to ground state. The amount of emitted photons is detected and is proportional to the concentration of the atom in solution.

The AAS consists of five basic components that can be manipulated to optimize the AAS for analysis (Soil Survey Investigations Staff, 1991):

(1) Light source: A hollow cathode lamp (HCL) emits light specific to a particular element. HCL's specific to an element or group of elements are selected.

(2) Flame/ burner system: For many elements, air-acetylene is the preferred flame; for some, such as aluminum, nitrous oxide-acetylene is used to provide a hotter flame.

(3) Monochromator: The monochromator disperses light by wavelength. The slit width is adjusted to select the required wavelength.

(4) Detector: The detector is a multi-alkali cathode multiplier. The gain is adjusted for optimal detection.

(5) Display: The display can be set for absorbance, concentration, or emission intensity. Displayed readings are usually the average of several individual readings.

More information on the AAS can be found in Baker and Suhr (1982), Perkin-Elmer (1971), and Soil Survey Investigations Staff (1991).

The AAS is used to analyze extractable calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na) (Chapter 12); KCl-extractable aluminum (Al) (Chapter 13); and CBD-extractable iron (Fe), manganese (Mn), and Al (Chapter 15). Follow the extraction procedures described in the referenced chapters. Extractable Ca, Mg, Fe, and Mn are analyzed by atomic absorption using an air-acetylene flame. Extractable Al is analyzed by atomic absorption using a nitrous oxide-acetylene flame. Extractable K and Na are analyzed by flame emission using an air-acetylene flame.

This appendix describes the set-up and operation of the Perkin-Elmer Model 403 Atomic Absorption Spectrophotometer. Refer to Figures C.1 through C.5 for diagrams of the various parts of the Model 403.

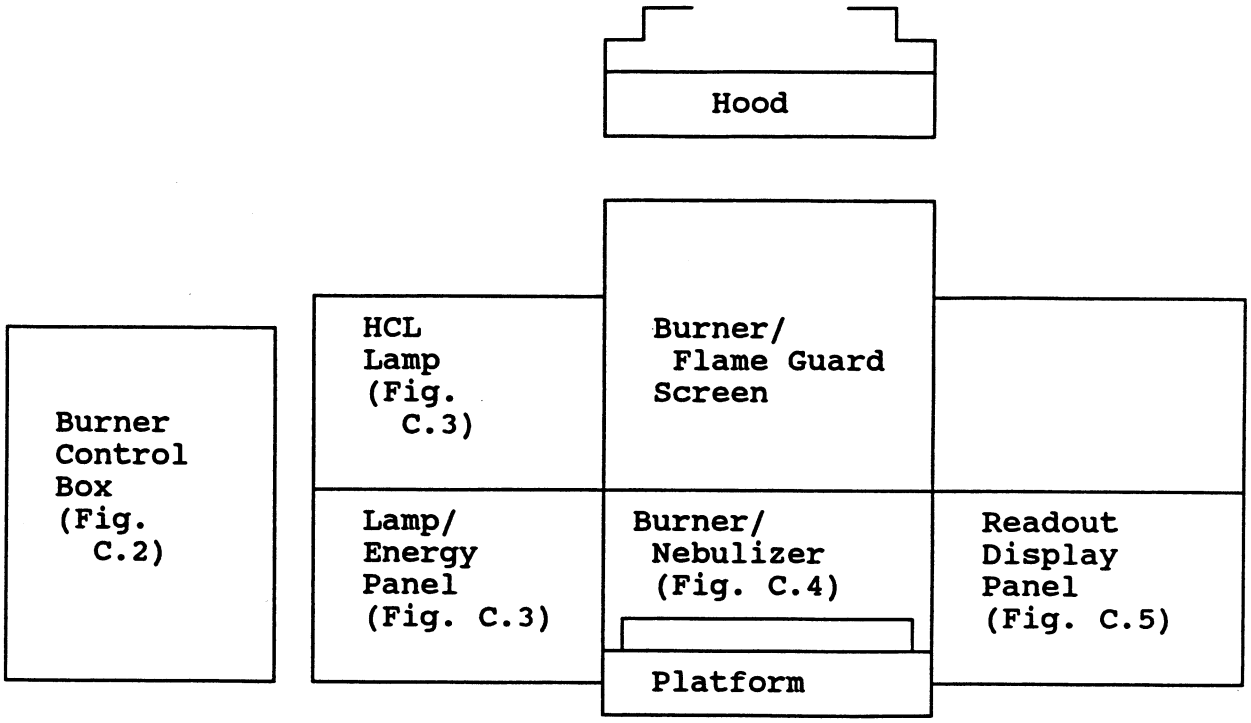

Figure C.1. General Schematic For the Perkin-Elmer Model 403 Atomic Absorption Spectrometer.

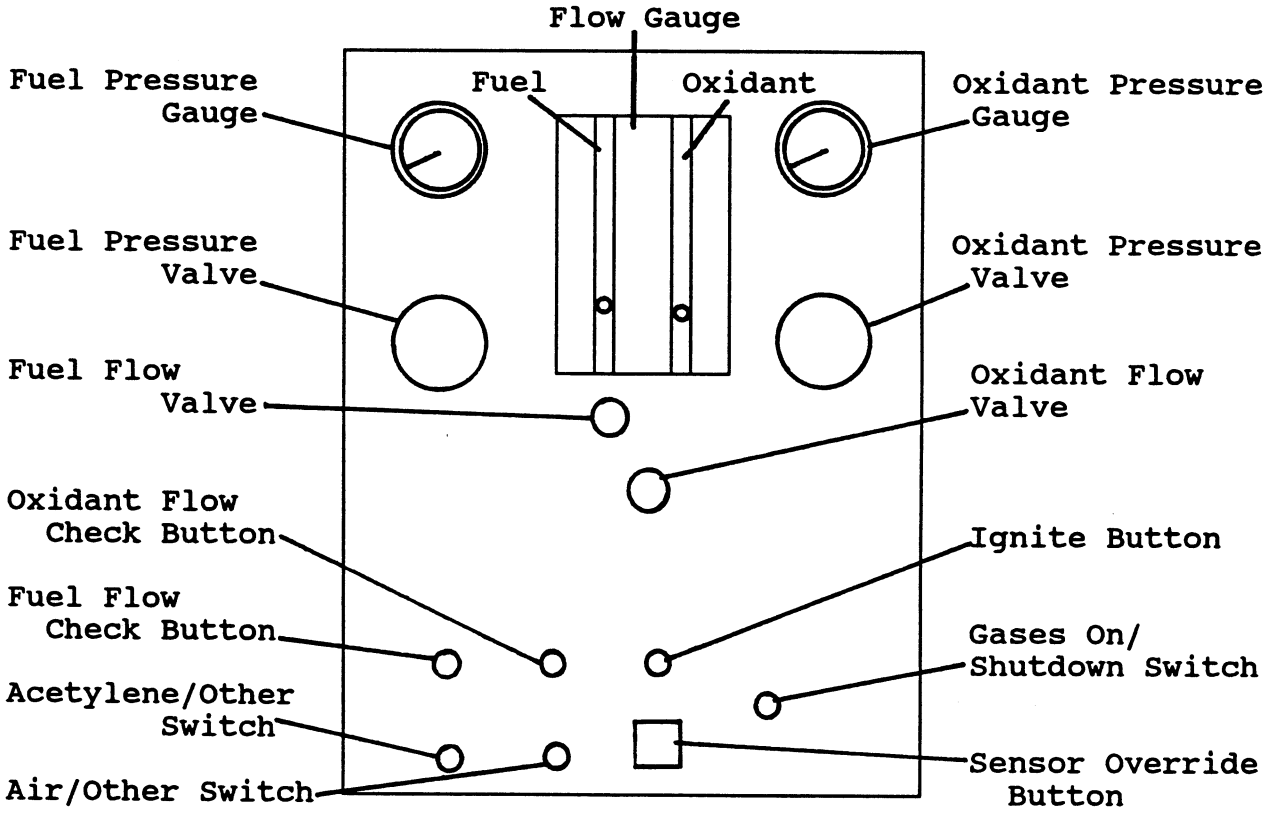

Figure C.2. Burner Control Box.

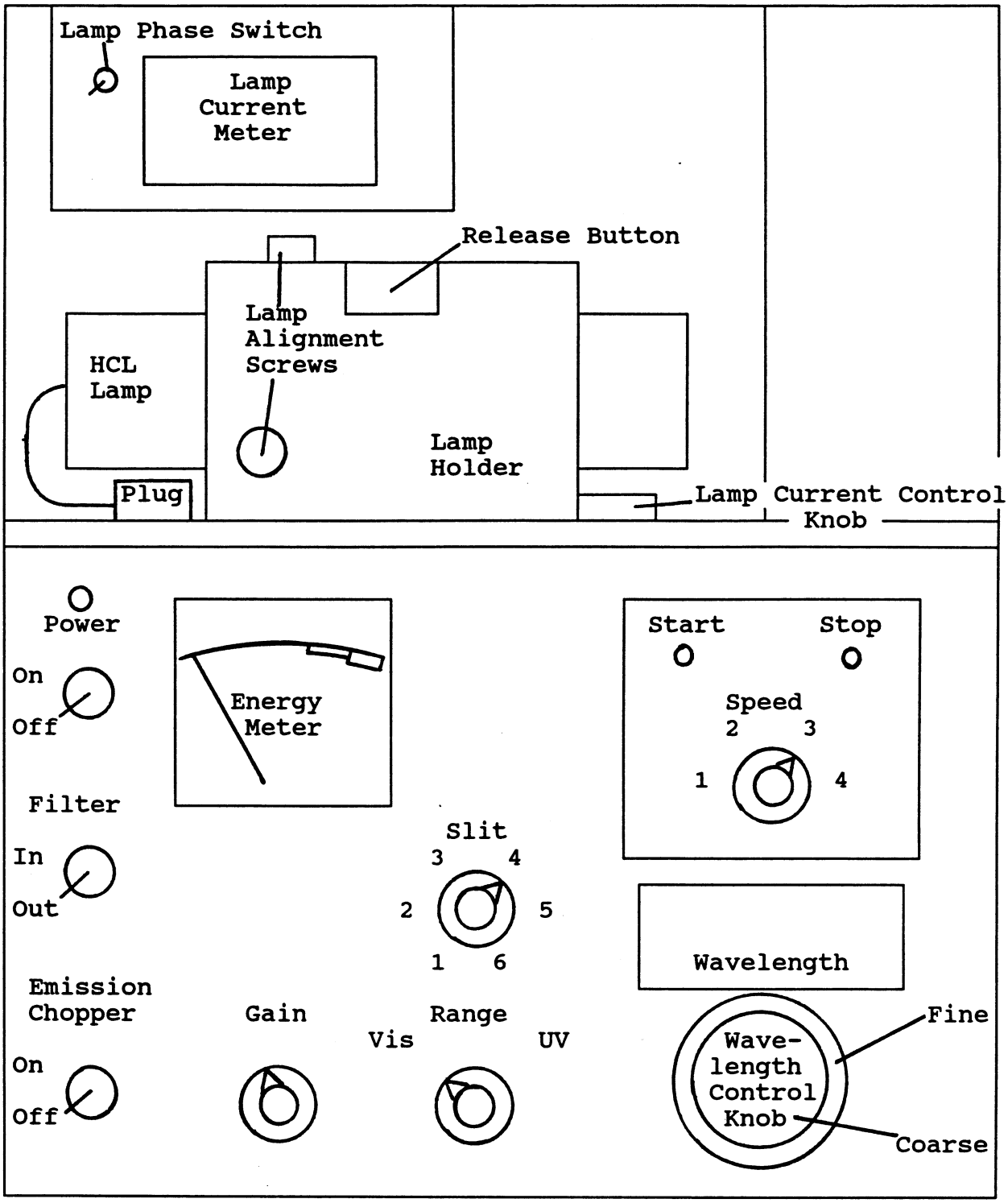

Figure C.3. HCL Lamp Compartment (Top) and Lamp/Energy Control Panel (Bottom).

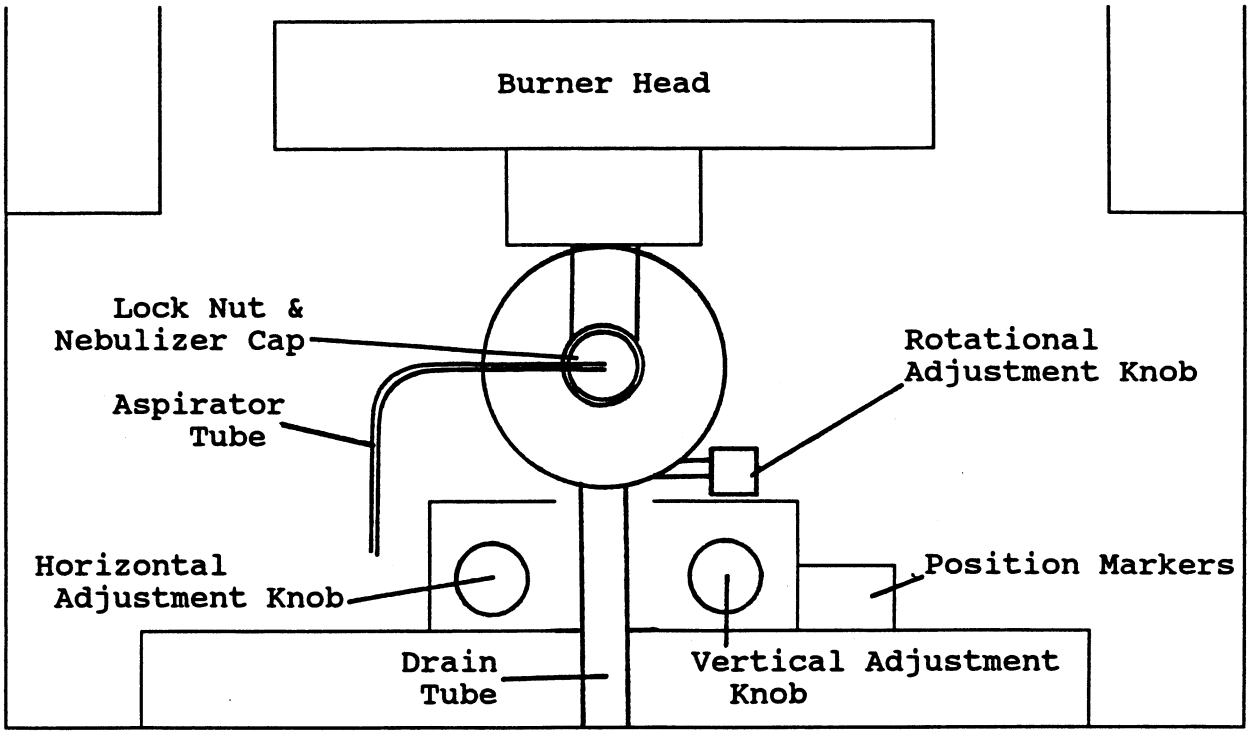

Figure C.4. Burner/Nebulizer.

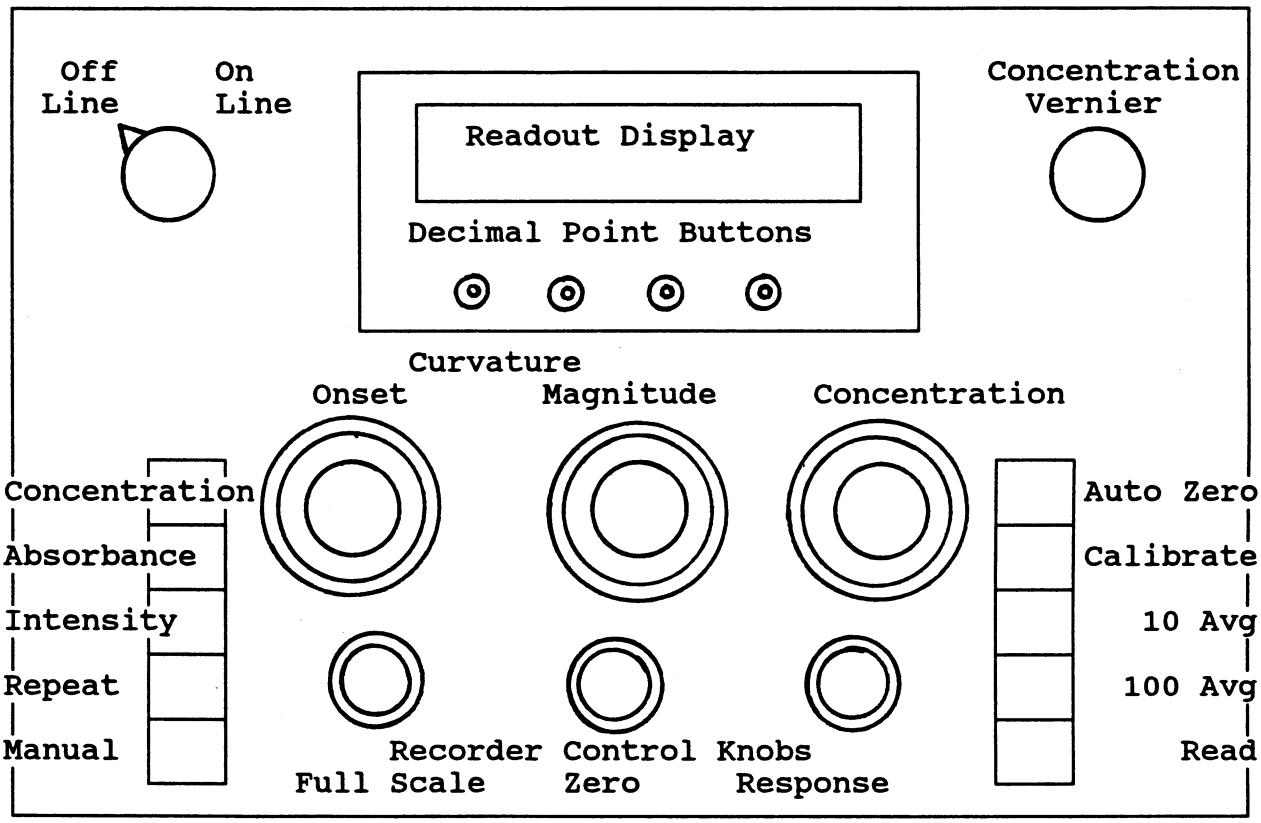

Figure C.5. Readout Display Panel.

Initializing the AAS

1. Before turning on the power source to the AAS, do the following:
   a. Set the gases switch on the burner control box (Fig. C.2) to shutdown.
   b. Unplug the HCL lamp (Fig. C.3), turn the lamp current control knob fully counterclockwise, and set the lamp phase switch to normal.
   c. On the lamp/energy control panel (Fig. C.3), turn the gain knob fully counterclockwise, switch the emission chopper off, and switch the filter out.
   d. In the burner/nebulizer compartment (Fig. C.4), place the aspirator tube in a beaker of distilled-deionized water and be sure that the drain tube has water in the loop and that the end is submerged in water. Install the proper burner head (see Table C.1), tightening the locking ring and connecting the safety wires. Use a 10-cm (length) head for air-acetylene flames and a 5-cm (length) head for nitrous oxide-acetylene flames.
   e. On the readout display panel (Fig. C.5), switch the display knob to off-line and depress the absorbance, repeat, and 10 average buttons. Set the curvature onset, magnitude and concentration knobs to zero. Switch the recorder full scale to 0.25A and turn the recorder zero and concentration knobs fully counterclockwise.
   f. Check the pressure in the gas tanks. Never run the AAS with less than 80 psi of pressure in the acetylene tank or 100 psi in the N_2O tank.
   g. Make sure that the AAS, burner control box, and deuterium arc power supply unit are all connected to a voltage regulator.
   h. Open the exhaust hood above the AAS (Fig. C.1).

2. Plug in the voltage regulator and turn on the power switch to the AAS. Press auto zero on the readout display panel.

3. Place the proper element lamp in the holder and plug in the lamp (Fig. C.3). Adjust the lamp current control knob to the operating current (mA) specified on the lamp.
   NOTE: K and Na are analyzed by flame emission and do not use lamps. For these elements, see the section on Flame Emission.

4. Select the slit, range, and wavelength on the control panel (Fig. C.3) according to Table C.1.

5. Slowly turn the gain knob clockwise and adjust the wavelength control knob above and below the initial wavelength setting to find a peak (the needle on the energy meter will deflect to the right). Locate the maximum peak with the wavelength control knobs and adjust the wavelength and the gain knobs so that the energy needle is located in the pink zone toward the lower limit of the red zone.
   NOTE: Monitor the energy level throughout the setup and adjust the gain as needed to keep the needle at the lower limit of the red zone.

6. Adjust the lamp alignment to maximize the energy reading by physically sliding the lamp forward and back in the holder and by turning the adjustment screws (Fig. 6.3) to move the lamp alignment up and down and from side to side.
   NOTE: The energy level may increase as the lamp warms up. Monitor the level and adjust the gain as needed.

7. Adjust the position of the burner head so that the light beam passes directly over, parallel to, and approximately 1 cm above the flame slit. Hold a piece of paper or card over the head to note the position of the light. Adjust the burner head with the three adjustment knobs on the head unit (Fig. C.4).

Table C.1. Set-up Specifications for the Perkin-Elmer 403 AAS.

Element	Wavelength (millimicrons)	Slit	Range	----- Burner Head ----- (oxidant-fuel)	Length (cm)	Lamp	Flow Rates Fuel	Oxidant
Ca	211 (209.4)	4	VIS	air-acetylene	10	Ca-Mg	32	55
Mg	285 (285.6)	4	UV	air-acetylene	10	Ca-Mg	32	55
Na	295 (292.2)	4	VIS	air-acetylene	10	None	32	55
K	383 (382.0)	4	VIS	air-acetylene	10	None	32	55
Fe	248 (249.5)	3	UV	air-acetylene	10	Fe	32	55
Al	309 (309.5)	5	UV	N_2O-acetylene	5	Al	55	35
Mn	279 (280.3)	3	UV	air-acetylene	10	Mn	32	55

NOTE: The wavelengths in parentheses provide the maximum energy peak for the Model 403 used in the Soil Characterization Lab.

Lighting the Burner For an Air-Acetylene Flame

NOTE: Do not look directly into the flame. Always keep the flame guard on the AAS closed or wear eye protection.

1. Switch the gas switches on the burner control box (Fig. C.2) to acetylene and air.

2. Open the valve on the air line and adjust the pressure on the pressure gauge to between 40 and 60 psi.

3. With the gas tank regulator valves closed (turned counterclockwise), open the acetylene gas tank. Turn the regulator valve until the outside line gauge reads between 12 and 15 psi.
   NOTE: If the acetylene tank pressure falls below 75-80 psi on the main tank (inner) gauge, acetone may pass through the gas control box and damage valves and tubing. For this reason, the tank is replaced when pressure falls below 80 psi.

4. Press the fuel (acetylene) flow check button on the burner control box (Fig. C.2) and adjust the fuel pressure valve to read 8 psi on the pressure gauge. Adjust the fuel flow valve to 32 on the flow gauge (read the middle of the ball).

5. Press the oxidant (air) flow check button on the burner control box (Fig. C.2) and adjust the oxidant pressure valve to read 30 psi on the pressure gauge. Adjust the oxidant flow valve to 55 on the flow gauge (read the middle of the ball).

6. Turn the gases switch from shutdown to on, and press the ignite button for 1 second and release it. If the flame does not ignite, wait 10 seconds and try again.

7. The flame should be even across the length of the slot. If the flame is uneven or interrupted in spots, shut down the gases, remove and clean the head after it cools.

Lighting the Burner For a Nitrous Oxide-Acetylene Flame

NOTE: UV light emitted by the flame can damage your eyes. Keep the flame guard on the AAS closed and wear eye protection when looking at the flame.

1. Install the smaller (5-cm) N_2O burner head, making sure the head is secured tightly and the safety wires are connected.

2. Switch the gas switches on the burner control box (Fig. C.2) to acetylene and air.
   NOTE: Flashbacks from the hot N_2O flame are most likely to occur during ignition and shutdown. For this reason, air is used as an oxidant when the flame is ignited, and the source is then switched to N_2O. The oxidant source is switched from N_2O back to air before the flame is shut down.

3. Adjust the air supply and the acetylene tank as described in steps 2 and 3 in the previous section. Plug in the pre-heater for the N_2O tank and open the N_2O tank. Adjust the gas regulator valve so that the outside line gauge reads 40 psi.
   NOTE: Replace the N_2O tank when the pressure drops below 100 psi.

4. Press the fuel (acetylene) flow check button on the burner control box (Fig. C.2) and adjust fuel pressure valve to read 8 psi on the pressure gauge. Adjust the fuel flow valve to 55 on the flow gauge (read the middle of the ball).

5. Press the oxidant (air - NO_2) flow check button on the burner control box (Fig. C.2) and adjust the oxidant pressure valve to read 30 psi on the pressure gauge. Adjust the oxidant flow valve to 35 on the flow gauge (read the middle of the ball).

6. Turn the gases switch from shutdown to on, and press the ignite button for 1 second and release it. If the flame does not ignite, wait 10 seconds and try again.

7. Allow the flame to burn for about 1 minute with acetylene and air and then switch the gas switch from air to other. The yellow flame should change to a short red flame. If not, immediately switch back to air and shut down the gases.

8. The flame should be even across the length of the slot. If the flame is uneven or interrupted in spots, shut down the gases, remove and clean the head after it cools.
   NOTE: The hot flame will produce deposits in and near the burner slot, causing a ragged flame. If the deposits clog the slot, a flashback may occur. Shut the flame off and use a razor blade to scrape the deposits from the surface and interior of the slot at frequent intervals.

Initializing For Flame Emission

1. Follow the procedures given in the section "Initializing the AAS", except for lamp adjustment.

2. Turn on the emission chopper (Fig. C.3) and select the intensity reading on the display panel (Fig. C.5). Switch the filter (Fig. C.3) in to analyze for K and leave the filter switch out to analyze for Na.

3. Light the burner flame as described in the section "Lighting the Burner For an Air-Acetylene Flame."

4. Adjust the wavelength, gain and concentration as follows:
   a. Turn the concentration dial (Fig. C.5) to 4.
   b. Aspirate the high standard and adjust the gain knob (Fig. C.3) so that the readout display (Fig. C.5) is 100.
   c. Aspirate a middle standard and adjust the wavelength control knob (Fig. C.3) to maximize the readout display.

Adjusting the Burner Head, Nebulizer, and Fuel Flow

NOTE: The burner head is adjusted to optimize sensitivity. Optimize the head with Mg standard for basic cations and Fe for Fe, Mn, and Al.

1. Adjust the burner head as follows (refer to Fig. C.4):
   a. Aspirate deionized H_2O from a beaker and press auto zero on the display panel (Fig. C.5).
   b. Raise the burner head (vertical adjustment knob) until it intercepts the light beam (a positive absorbance reading will appear on the readout display). Lower the head until the reading returns to zero.
   c. Aspirate the high standard and continue to lower the burner head until a maximum absorbance readout is obtained. Aspirate deionized H_2O and press auto zero.
   d. Aspirate the high standard again and adjust the horizontal and lateral adjustment knobs until a maximum absorbance readout is obtained. This aligns the flame with the light beam.

2. Adjust the nebulizer as follows:
   a. Loosen (turn clockwise) the thin locknut behind the knurled nebulizer cap (Fig. C.4).
   b. Aspirate the high standard and turn the nebulizer cap counterclockwise until air bubbles into the standard solution. Turn the nebulizer cap clockwise until a maximum reading is obtained. Tighten the locknut (snug, but not tight).

3. Aspirate the high standard and adjust the oxidant and fuel flow valves on the burner control box to get a maximum absorbance reading. Check the zero standard after each flow change and readjust as necessary.

Calibrating the AAS

NOTE: The AAS is calibrated with the standards prepared in conjunction with the specific extraction procedures. Table C.2 provides the linear range and sensitivity for each of the elements analyzed by the Soil Characterization Lab. This table is specific for the Model 403 used in this laboratory. The linear range provides a 1:1 correlation between the concentration of the standard solution and the concentration displayed on the readout. Beyond this range, the relationship becomes non-linear. The sensitivity, as used in this table, refers to the lowest concentration that can be distinguished from background. It approximates the working limit described by Baker and Suhr (1982) and has been determined on the Model 403 using standard solutions.

1. Depress the concentration button on the readout display panel (Fig. C.5)

2. Aspirate the 0 ppm standard and press auto zero.

3. Aspirate the low standard and use the concentration dial to adjust the readout to the concentration of the low standard.

Table C.2. The Linear Range and Sensitivity For Elements Analyzed by AAS.

Element	Extraction (Chapter)	Linear Range (ppm)	Sensitivity (ppm)
Ca	NH_4OAc (Ch. 12)	0 - 7.0	0.07
Mg	NH_4OAc (Ch. 12)	0 - 1.00	0.01
K	NH_4OAc (Ch. 12)	0 - 10.00	0.02
Na	NH_4OAc (Ch. 12)	0 - 2.00 0 - 10.0	0.01 0.1
Al	KCl (Ch. 13) CBD (Ch. 15)	0 - 20.0	0.5
Fe	CBD (Ch. 15)	0 - 6.0	0.1
Mn	CBD (Ch. 15)	0 - 3.00	0.05

4. Check the blank and re-zero if needed. Aspirate the remaining standards and record the readings. Within the linear range, the standard concentrations should be directly correlated with the readout display. If the readout departs from the actual concentration (this may occur at the high end of the standard range or when the high standard extends beyond the linear range), use the curvature correction described in step 5. Otherwise, proceed to the section "Analyzing the Samples."

5. If the readout display for the standards at the high end of the range deviate from the actual standard concentration, the curvature (deviation from the linear relationship) can be corrected with the following procedure. For example, suppose that the following readings have been obtained for Al standards:

Standard Concentration	Readout Display
2 ppm	2.0
6	6.0
12	12.0
20	18.5

The standard calibration is linear up to 12 ppm Al and becomes nonlinear somewhere between 12 and 20 ppm. Correct the curvature as follows:

a. Rotate the Curvature Magnitude dial (Fig. C.5) fully clockwise.
b. Aspirate a standard near the upper end of the linear range (12 ppm in the example) and turn the Curvature Onset dial until the readout display value (12.0) shifts away from the actual concentration. Turn the Onset dial back until the readout display returns to the value of the standard concentration.
c. Aspirate the standard that extends beyond the linear range (20 ppm in the example) and rotate the Magnitude dial until the readout display shows the actual standard concentration.
d. Once these steps are taken, do not change the Curvature dials until the samples have been analyzed.

Analyzing the Samples

1. Once the AAS is calibrated, aspirate the samples. The readout display is the equivalent of the sample concentration in ppm (no plotting or conversions are needed as long as the AAS is calibrated to provide a direct readout of concentration). Dilute and re-analyze any samples with concentrations higher than the high standard.

2. Check the 0 ppm and high standard readings occasionally (every 15 to 20 samples) to ensure proper standardization of machine.

Shutting Down the AAS

1. Aspirate deionized H_2O for 5 minutes after the last sample is analyzed.

2. Turn the gain and lamp current control knobs (Fig. C.3) fully counterclockwise and unplug the element lamp. Press auto zero on the display panel.

3. If N_2O was used, switch the oxidant to air and burn for 5 seconds.

4. Shut down the gases and close the main valves on the gas tanks. Press the fuel flow check button to drain the acetylene (until the pressure on acetylene line valve reads 0). Then press the oxidant flow check button to purge the system with air.
   NOTE: If N_2O was used, switch the oxidant switch to other and press the oxidant flow check button to drain N_2O from the lines. Then switch to air and purge the system. Unplug the N_2O pre-heater.

5. Turn off the power switch, unplug the voltage regulator, and shut the exhaust fan. After the AAS cools, replace the cover.

References

Baker, D. E., and N. H. Suhr. 1982. Atomic absorption and flame emission spectrometry. p. 13-37 in A. L. Page, R. H. Miller, and D. R. Keeney (Ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Perkin-Elmer. 1971. Instructions -- Model 403 Atomic Absorption Spectrophotometer. Perkin-Elmer. Norwalk, CT.

Soil Survey Investigations Staff. 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rpt. No. 42. National Soil Survey Center. Lincoln, NE.

APPENDIX D

MECHANICAL VACUUM EXTRACTOR

Background and Theory

The procedures for extractable bases (Chapter 12), KCl-extractable aluminum (Chapter 13), and exchangeable acidity (Chapter 14) rely on extraction by leaching. Because leaching rates vary with soil texture, methods that use Buchner funnels, filter flasks, and vacuum lines require constant attention to maintain uniform extraction rates among samples. This procedure is time-consuming and often results in variable extraction rates (Holmgren et al., 1977).

The mechanical vacuum extractor provides a constant, controlled leaching of soil samples of any texture. Syringes are mounted on a mechanical device that includes both stationary and moveable platforms (Figure D.1). The plunger in the lower syringe is held by the stationary platform and the syringes are raised away from the plunger by the moveable platforms. As the syringes are pulled away from the plunger, a vacuum develops, drawing extractant from the reservoir tube through the sample and into the lower syringe. The vacuum is constant for each sample and the extractor can be left running without constant monitoring.

Materials and Equipment

1. Mechanical vacuum extractor, 24 place
2. Syringes: upper reservoir, middle sample, and lower collection syringe with plunger
3. Filter pulp, ash-free
4. Silicone lubricant

Calibrating the Extraction Times

The control wheel below the stationary platform is used to adjust the rate of extraction. A graduated speed scale along one side of the wheel ranges from 0 (slow) to 45 (fast). The extraction times and speed settings in Table D.1 are specific for the mechanical vacuum extractor used in the Penn State Soil Characterization Lab. The settings are based on a 34-link drive chain on the extractor.

Table D.1: Extraction times and speed scale settings for the mechanical vacuum extractor.

Time (hr)	Speed Setting
0.5	37
0.75	29
1	23
2	15
3	11
4	9
6	7 1/2
8	6 2/3
10	6
12	5
16	4

Preparing for Extraction (refer to Fig. D.1)

1. Lower the upper moveable platforms so that the bottom platform is flush with the stationary platform. Adjust the hand crank so that the pin fits into the hole on the top of the extractor.
2. Inspect the syringes and plungers and discard any that are not in good physical condition. Lubricate the plungers with silicone lubricant to ensure good contact between the plunger and the syringe and to prevent air or extractant leaks.
3. Pack a 2.5-cm diameter (1g) ball of filter pulp into each sample syringe.
4. Moisten the filter pulp with a small amount (5-6 drops) of extractant.
5. Add the sample to the sample syringe and level the sample surface.
6. Hang the sample (middle) syringe in the slots on the top platform.
7. Install the lower syringe so that the plunger is hooked into the slot on the stationary platform and the syringe rests in the slot on top of the lower moveable platform.
8. Connect the noses of the middle and lower syringes with rubber tubing.
9. Add the extractant specified in the procedure to the sample syringe and stir with a glass rod or by tapping the syringe, being careful not to disturb the filter pulp.
10. Place reservoir syringes into the upper end of the sample syringes, add the specified amount of extractant, and place a small watch glass on top of the syringe.
11. Turn the control wheel to the setting time specified by the analysis.
12. Turn on the extractor and let it run until the procedure is completed. The extractor does not require monitoring and can be left to run overnight.

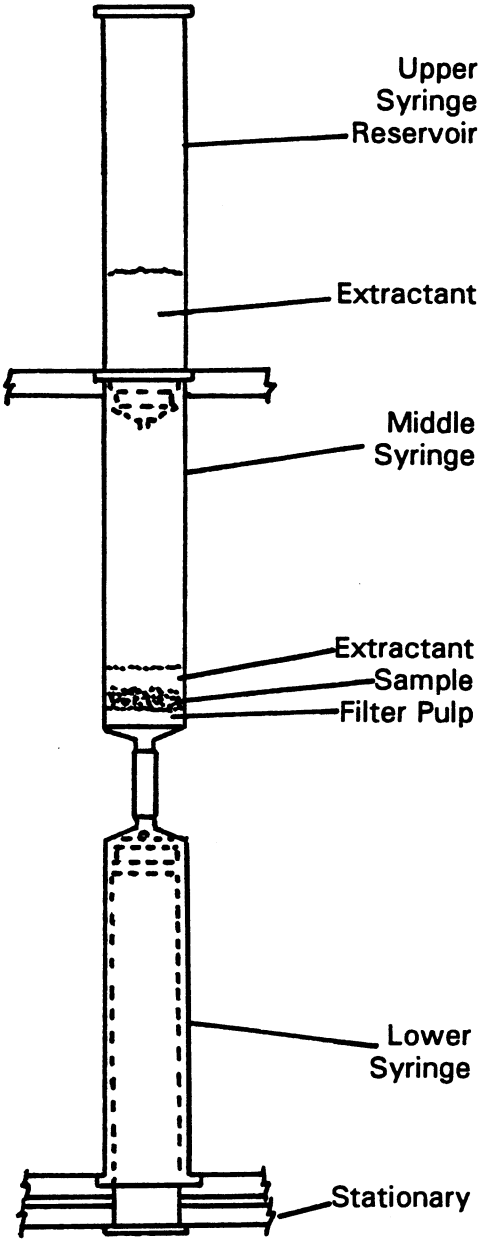

Figure D.1. Set-up for syringes.

References

Concept Engineering, Inc. 1978. Information manual for mechanical vacuum extractor. Lincoln, NE.

Holmgren, G. G. S., R. L. Juve, and R. C. Geschwender. 1977. A mechanically controlled variable rate leaching device. Soil Sci. Soc. Am. J. 41:1207-1208.

Soil Survey Investigations Staff. 1991. Soil survey laboratory methods manual. USDA SCS Soil Surv. Invest. Rpt. No. 42. Ver. 1.0. National Soil Survey Center. Lincoln, NE.

APPENDIX E

COLORIMETRIC METHODS FOR DETERMINING Al AND Fe

Prior to 1992 the Soil Characterization Laboratory used colorimetric methods to determine the concentration of KCl-extractable Al (Chapter 13) and CBD-extractable Fe (Chapter 15). KCl-extractable Al method was converted to an ionic state with acid and heat, and the color was developed with the reagent aluminon. Transmittance of the solution was determined at 530 nm on a spectrometer. The CBD-extractable Fe was complexed with o-phenanthroline and the solution transmittance was determined at 510 nm on a spectrometer. Transmittance readings were plotted with standard concentrations on log-normal graph paper and sample concentrations were determined from the standard curves.

The atomic absorption spectrometer (AAS) yields results similar to the colorimeter for KCl-Al and CBD-Fe (Thurman and Ciolkosz, 1992). The AAS is now used in the Soil Characterization Lab because it is quicker than the colorimeter, involves less glassware, and provides concentrations directly in ppm. The discontinued colorimetric procedures are described in this appendix for reference.

KCl-EXTRACTABLE Al -- COLORIMETRIC DETERMINATION

NOTE: Follow the extraction method described in Chapter 13. Instead of analyzing the samples on the AAS, use the colorimetric procedure described below.

Acid is added to sample aliquots and standards. These are heated to eliminate P and Si interferences. The aluminon reagent is added to develop a red color. Color transmittance of the standards and the samples are analyzed with a spectrometer. The transmittance readings for the standards are plotted on log-normal graph paper and sample concentrations are determined from the standard curve.

Materials and Equipment For Colorimetric Determination

1. Volumetric flasks, 50-ml
2. Hotplate
3. Pipettes, 2-ml for diluting samples, and assorted sizes for making standards
4. Spectrometer with cuvettes, $\pm$ 0.5% T in any position. Use only matched cuvettes (should give identical 100% transmittance readings for distilled H_2O without readjusting the spectrometer)

Reagents For Colorimetric Determination

1. HCl, 1 N. Dilute 86 ml of concentrated HCL to 1000 ml with H_2O.
2. Aluminon reagent. Add 120 ml of glacial HOAc to approximately 700 ml of H_2O in a 1-liter volumetric flask, and mix in 24.0 g of NaOH. Dissolve 0.35 g of aluminon and dilute to volume with H_2O. Adjust the pH to 4.2 $\pm$ 0.5. The reagent is stable for a week or more if properly stored. Replace with fresh reagent if the standard curves change significantly.
3. Al stock standard solution, 5 ppm. Pipette 10 ml of 1000 ppm of Al reference solution into a 100-ml volumetric flask and dilute to volume with H_2O. Pipette a 5-ml aliquot of this 100-ppm Al solution and dilute to 100 ml with H_2O. The resultant stock solution contains 5 ppm Al.

Al Determination by Colorimetry (Aluminon Reagent)

1. Transfer a 2-ml aliquot of the sample extract to a 50-ml volumetric flask. At the same time, pipette 0, 1, 2, 3, 4, 5, 6, 7, 8, and 10 ml of the 5-ppm Al stock standard in separate 50-ml volumetric flasks (these will be equivalent to 0. 0.1, 0.2, ..., 1.0 ppm Al). Do not bring the extracts or the samples to volume.

2. Add 3 ml of 1 N HCl to the samples and standards.
   NOTE: If the analysis cannot be completed in one day, stop at this point (the samples are stable).

3. Heat the samples and standards at 90-100°C on a steam hotplate for 30-60 min.

4. Cool and dilute to about 35 ml.

5. Add 10 ml of aluminon reagent, dilute to volume with H_2O, mix, and let stand for at least an hour.

6. Calibrate the spectrometer with the standard solutions, reading % transmittance (% T) at 530 nm. Use distilled H_2O to set the 100% T and read the transmittance for each standard. Plot the log of % T vs. the known ppm of Al for each standard.

7. Determine % T for the sample extracts, determine the concentration from the standard curve, and record the concentration (ALKCL PPM) on the input form. Check the standards periodically and recalibrate as needed.

8. Dilute any sample with a concentration greater than the high standard and reanalyze on the AAS. Any additional dilution factors will be incorporated into the calculations before entering the concentration on the input form.

Calculations

1. The general formula for calculating the concentration of extractable Al is:

$$\text{Al, meq/100 g} = \frac{\text{ppm sample} \times \text{ml extract} \times 1000 \times \text{d.f.} \times 100}{1{,}000{,}000 \times \text{g soil} \times \text{equiv. wt.}}$$

   where,

ppm sample	= ppm in sample extract
ml extract	= initial extract volume (50 ml)
g soil	= oven-dry soil weight (air-dry wt. / moisture correction)
d.f.	= dilution factor (an initial 25X plus additional dilutions)
equiv. wt.	= 9 for Al

2. This equation is simplified for the colorimetric procedure (incorporating constants, extract volume, soil weight, and equivalent weight of Al) as follows:

$$\text{Al, meq/100 g} = \frac{\text{ppm sample} \times 50 \times 25 \times \text{d.f.}}{10 \times (2.00 / \text{MC}) \times 9} = \frac{(\text{ppm sample} \times \text{d.f.}) \times 13.9}{(2.00 / \text{MC})}$$

   NOTE: The initial 25X dilution factor does not appear in the calculations for Al in the Database Computer Program (Appendix B). This 25X factor must be entered manually to use the program with the colorimetric method.

CBD-EXTRACTABLE Fe -- COLORIMETRIC DETERMINATION

NOTE: Follow the extraction method described in Chapter 15. Instead of analyzing the samples on the AAS, use the colorimetric procedure described below.

An o-phenanthroline indicator is added to standards and an aliquot of each extract to develop an orange color. The developed color will remain stable for at least 15 days (Olson and Ellis, 1982). Color transmittance of the standards and the samples are analyzed with a spectrometer. The transmittance readings for the standards are plotted on semi-log graph paper and sample concentrations are determined from the standard curve.

Materials and Equipment For Colorimetric Determination

1. Volumetric flasks, 100 ml
2. Pipettes, 2 ml for taking sample aliquots, and assorted sizes for standards
3. Spectrometer
4. Spectrometer cuvettes, $\pm$0.5% T in any position (should be oriented to give identical readings)

Reagents For Colorimetric Determination

1. Sodium acetate/acetic acid (NaOAc-HOAc) buffer. Dissolve 27.2 g of NaOAc (45.1 g of NaOAc·$3H_2O$) in 500 ml of H_2O. Add 123 ml of glacial HOAc (17.4N) and dilute to 1 liter with H_2O.
2. Hydroxylamine hydrochloride (NH_2OH·HCl), 5%. Dissolve 5 g of NH_2OH·HCl per 100 ml of H_2O. Store in a dark bottle out of the light.
3. o-Phenanthroline reagent. Dissolve 1.5 g of crystalline o-phenanthroline monohydrate in 100 ml of ethanol. Dilute to 1 liter with H_2O. Store in a dark bottle out of the light.
4. Reagent A. Mix 3 parts NaOAc - HOAc buffer, 1 part NH_2OH·HCl solution, and 1 part o-Phenanthroline reagent. Mix fresh each day.
5. Fe standard stock solution, 100 ppm. To 10-ml of 1000 ppm certified Fe reference solution, add 10 ml of the citrate-bicarbonate buffer used in the extraction and approximately 0.5 g of $Na_2S_2O_4$. Dilute to 100 ml with H_2O.

Procedure For Colorimetric Determination

1. Prepare the standards for calibration. Pipette 0, 1, 2, 3, 4, 5, and 7 ml aliquots of the 100 ppm Fe standard stock solution into separate 100-ml volumetric flasks. Add 25 ml of Reagent A, dilute to volume, and mix. These represent 0, 1, 2, 3, 4, 5, and 7 ppm Fe standards.

2. Pipette a 2-ml aliquot of the extract into a 100-ml volumetric flask, add 25 ml of Reagent A, dilute to volume, and mix thoroughly.

3. Let the standards and the samples stand for a minimum of one hour. The colors should be stable for at least 15 days (Olson and Ellis, 1982).

4. Calibrate the spectrometer with the standard solutions, reading % transmittance (% T) at 510 nm. The 0 standard is set at 100% T and a standard curve is plotted with the log of % T vs. ppm Fe in solution.

5. Determine % T for the sample extracts, calculate the concentration from the standard curve (using semi-log graph paper or a programmable calculator), and

record the concentration (FECBDPPM) on the input form. Check the standards periodically and recalibrate as needed.

Calculations

1. Percent Fe and Fe_2O_3 are calculated from ppm Fe as follows:

$$\%Fe = \frac{\text{extract vol (ml) x df(1) x df(2) x ppm Fe}}{\text{oven-dry sample wt. (g) x 10,000}}$$

where: extract vol. = 500 ml
df(1) = initial dilution factor of 50 (1 part extract to 50 parts)
df(2) = additional dilution factor (FECBDDF)
ppm Fe = ppm Fe from AAS analysis (FECBDPPM)
sample wt. = air-dry wt. (3.00 g) / moisture correction factor (MC)

$$\%Fe_2O_3 = \%Fe \times 1.43$$

2. The equations are simplified by incorporating the constant factors into the calculations as follows:

$$\%Fe_2O_3 = \frac{500 \times 50 \times df(2) \times ppm\ Fe \times 1.43}{(\text{air-dry wt.} / MC) \times 10{,}000}$$

$$\%Fe_2O_3 = (FECBDPPM \times FECBDDF \times 3.574) / (3.00 / MC)$$

References

Jackson, M. L. 1958. Soil chemical analysis. Prentice-Hall. Englewood Cliffs, NJ.

Jackson, M. L. 1969. Soil chemical analysis -- advanced course. 2nd edition, 8th printing, 1973. Publ. by the author, Dept. of Soil Science, University of Wisconsin, Madison, WI 53706.

Olson, R. V., and R. Ellis, Jr. 1982. Iron. p. 301-312 in A. L. Page, R. H. Miller, and D. R. Keeney (ed). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9. (2nd edition). Soil Sci. Soc. of Amer. Madison, WI.

Thurman, N. C., and E. J. Ciolkosz. 1992. A comparison of soil characterization laboratory methods: CBD-extractable Fe, Al, and Mn; KCl-extractable Al; and exchangeable acidity. Penn State University Agronomy Series (in preparation).

APPENDIX F

PROCESSING AND ANALYSIS OF RAMP SAMPLES

The Rural Abandoned Mine Program (RAMP) in Pennsylvania is administered by the Soil Conservation Service. In the past, the Soil Characterization Laboratory has supported this program by determining rock fragment content, total S, and H_2O extractable Al and Mn. Subsamples were sent to the Merkle Soil and Forage Testing Laboratory for analysis. The following sections outline the procedures used to process and analyze these samples.

SIEVING COARSE FRAGMENTS AND PREPARING THE ANALYTICAL SAMPLE

Materials and Equipment

1. Toledo counter balanced scale with counter weights
2. Paper gallon and pint container
3. Merkle soil test kit
4. 10 in. diameter nest of sieves - 3/4 in., 3/8 in., 0.187 in., and 2 mm sieves with cover and bottom pan
5. Brown paper, rolling pin and No. 12 rubber stopper
6. Top loading balance
7. Large ceramic mortar and pestle
8. plastic or paper bags, 1 pint

Procedure

1. Air-dry sample for processing.

2. Crush soil clods by spreading the sample on a piece of brown paper under the hood and rolling with a rolling pin. Be careful not to break down the coarse fragments.

3. Pour the sample into the nest of sieves. This can be done in increments if the sample is too large.

4. Use a rubber stopper to break clods until only clean fragments remain on the 3/4-in. sieve. Remove the top sieve and pour the fragments into an extra gallon container.

5. Repeat step 4 for each sieve size putting each size coarse fragment into a separate gallon container. Pour the less than 2 mm material from the bottom pan into a labeled gallon container.

6. Repeat steps 3 through 5 until the entire sample has been sieved and collected.

7. Weigh each gallon container on the Toledo countered balance. Use 500 g, 1 kg, or 2 kg counter weights on the rear balance pan until the scale registers within the 500 g range on the scale. Record the total sample weight (adding the amount of tare weight) on the data sheet (Table F.1).

8. Return a few of the 3/4-3 in., 3/8-3/4 in., and 0.187-3/8 in. size coarse fragments to the original sample bag. Place all of the 2 mm-0.187 in. size coarse fragments into a plastic bag. Put these two bags of material into the labeled gallon container with the less than 2 mm material.

9. Add the weights of the <2 mm and 2 mm-0.187 in. material. Divide the weight of the 2 mm-0.187 in. material by this sum, multiply by 250 and record on the data sheet. This is the wt. of the 2 mm-0.187 in. material to be recombined with the less than 2 mm material to get a 250 gm analytical sample.

10. Place a labeled pint container on the top loading balance and tare to zero. Weigh the amount of 2 mm-0.187 in. material calculated in step 9 into the container. Bring the weight to 250 gm with <2 mm material.

11. Pass the sample from the pint container through a 2 mm sieve. Pour the fragments retained on the sieve into the ceramic mortar and grind them to pass the 2 mm sieve. Pour the ground sample back in the pint carton and shake well to mix. This is the sample used for all subsequent analysis.

Table F.1. Example of Data Sheet for RAMP SAMPLES

	Weight (g)					
IDENT.	<2 mm	2 mm - 0.187 in	0.187 - 3.8 in	3/8 - 3/4 in	3/4 - 3 in	gm. 2 mm - .187 in. 250 gm anal. sample
R 85-1-1	1166	483	794	1166	2020	73.2
R 85-2-1	1958	341	306	346	1327	37.1
R 85-3-1	2040	345	410	492	1712	36.2

Al + Mn

	---- Mn ----		---- Al ----		---- Standards abs. ----		
IDENT.	abs.	ppm	abs.	ppm	ppm	Mn	Al
R 85-1-1	19		0		0	0	0
R 85-2-1	0		1		1	53	3
R 85-3-1	17		4		2	103	5
					3	153	8
					5	245	13
					8	375	19
					10	460	24

Total Sulfur

IDENT.	Sample Wt. (g)	Units Titrated	gm/l KIO3	% S	T/Ac CaCO3 Equiv.
R 85-1-1	0.272	18	2.0	0.030	
R 85-2-1	0.148	15	2.0	0.046	
R 85-3-1	0.128	35	2.0	0.123	

SUBMITTING A SUBSAMPLE TO THE ANALYTICAL (MERKLE) LAB FOR ANALYSIS

Procedure

1. Fill the plastic bag in the Merkle Lab kit about 1/3 full with material from the pint carton, secure the bag a the rubber band and return it to the Merkle Lab kit.

2. Complete the Merkle Lab sheet (pg. 3) for agronomic crops. Fill in the date, county location, and field no. or letter (RAMP I.D. No.) with the appropriate information. All other information on the form is exactly as shown in Table 16.1.

3. Place the completed sample kit in the campus mail.

WATER SOLUBLE ALUMINUM AND MANGANESE IN RAMP SAMPLES

This procedure is recommended by G. W. McKee, Professor of Agronomy, Pennsylvania State University.

Background and Theory

Most procedures for the exchangeable aluminum use a strong acid or salt, which removes all of the $A1^{+3}$ and Mn^{+2} into solution. This solution is then leached from the soil and an atomic absorbtion spectrophotometer is used to determine the ppm of $A1^{+3}$ and Mn^{+2} in the extracts.

The RAMP (Rural Abandoned Mine Project) samples are sent to the Soil Chracterization Laboratory by the Soil Conservation Service. The SCS is interested in the reclamation of these mine soils and are therefore interested in what nutrients are available or possibly toxic to the plants. By leaching the soil with distilled water, it is thought that the fraction of Al^{+3} and Mn^{+2} in the solution is more useful and it is then determined by using the atomic absorbtion spectrophotometer.

Materials, Equipment, and Reagents

1. Distilled water
2. Balance, sensitive to 0.01 g
3. Volumetric flasks, 250 ml
4. Reciprocating shaker
5. Vacuum flasks, 250 ml
6. Buchner funnels, 6-cm diameter
7. Filter paper, Whatman #42
8. Vacuum pump and apparatus
9. Storage bottles, plastic 50 ml, with caps
10. Atomic absorption spectrophotometer
11. 0.0, 1.0, 2.0, 3.0, 5.0, 8.0, and 10.0 ppm Al and Mn standards. Pipette 10 ml of 1000 ppm Al standard and 10 ml of 1000 ppm Mn standard into one 100 ml volumetric flask and dilute to volume with deionized-distilled H_2O. Pipette 0, 1.0, 2.0, 3.0, 5.0, 8.0, and 10.0 ml of this 100 ppm standard into separate 100-ml volumetric flasks and bring to volume with deionized-distilled H_2O.

Procedure

1. Weigh a 50.0 g sample into a 250 ml volumetric flask. Add 50 ml of deionized-distilled water, stopper the flasks, and shake them in an upright position for one hour in a reciprocating shaker at approximately 180 oscillations per minute.

2. Connect the vacuum flasks with Buchner funnels to the vacuum apparatus. Place #42 filter paper in the Buchner funnel. Wet the filter paper with water immediately before transferring the sample to insure that no soil particles will pass through the filter paper and contaminate the leached solution.

3. Transfer the suspension in the 250 ml volumetric flasks into the Buchner funnels and apply vacuum.

4. Transfer an undiluted aliquot of the extract into labeled 50-ml storage bottles.

5. Analyze for Al and Mn by atomic absorption (Appendix C).

References

McKee, G. W. 1979. Personal communication, Professor of Agronomy, The Pennsylvania State University.

McLean, E. O. 1965. Aluminum. p. 978-998 in C. A. Black (ed.). Methods of soil analysis. Part 2. Chemical and microbiological properties. Agron. No. 9, Amer. Soc. of Agron. Madison, WI.

Sobek, A. A., W. A. Schuller, J. R. Freeman, and R. M. Smith. 1978. Field and laboratory methods applicable to overburdens and minesoils. EPA-600/2-78-054. U.S. Environmental Protection Agency. Cincinnati, OH.

APPENDIX G

UNITS AND CONVERSIONS

Most technical journals and publications require that SI (International System) units be used in all submitted manuscripts. In some instances, the SI units differ from the common non-SI units, such as those used in this laboratory manual. Table G.1 provides a listing of the common non-SI unit, the preferred SI unit, and the conversion factor to change from common to SI units.

Table G.1. Conversion Factors For Common non-SI and SI Units.

Parameter	Common Unit	Conversion[a]	Preferred SI Unit
Clay, silt, sand, rock fragment content	percent (%)[b]	10	g kg^{-1}
Bulk density, particle density	g cm^{-3}	1.0	Mg m^{-3}
Pressure	atm	0.101	megapascal (MPa)
	bar	100	kilopascal (kPa)
	lb./in^2 (psi)	6.9 x 10^3	pascal (Pa)
Water content	percent (%)	0.01	kg kg^{-1}
Concentration	ppm	1.0	mg kg^{-1}
Concentration, org. C, ttl. S, free oxides (Fe, Mn, Al)	percent (%)	10	g kg^{-1}
Ion exchange conc. bases, Al, acidity, CEC	meq/100 g	1.0	cmol kg^{-1}
X-ray diffraction pattern	degree (°)[c]	1.75 x 10^{-2}	radian (rad)

[a] - Multiply the non-SI unit by the conversion to obtain the SI unit.

[b] - While g kg^{-1} is the preferred unit, percent is also acceptable in many cases.

[c] - While radian is the preferred unit, degree is also acceptable in many cases.

<u>References</u>

Soil Science Society of America. 1984. Glossary of soil science terms. SSSA. Madison, WI.

Soil Science Society of America. 1991. Conversion factors for SI and non-SI units. Soil Sci. Soc. Amer. J. 55 (no. 6): iv - v.

Agronomy Series Publications on the Pennsylvania State University Soil Characterization Laboratory

No. 25	Cunningham et al. 1972. Laboratory Characterization Data and Field Descriptions of Selected Pennsylvania Soils. (This publication gives all the Pennsylvania soil characterization data up to 1972. Following 1972, data was published in the PA Ag Expt. Station Progress report series Characteristics, Interpretations, and Uses of Pennsylvania Soils: Number 290, Dauphin Co.; 295, Northampton Co.; 300, Huntingdon Co.; 306, Warren Co.; 316, Armstrong Co.; 320, Bradford Co.; 323, Bedford Co.; 324 Bucks Co.; 326, Butler Co.; 341, Soils Developed from Cherty Limestone Material; 344, Soils Developed from Colluvium; 355, Soils Developed from Redbeds and Calcareous Material; 362, Soils Developed from Acid Shale; 381, Minesoils. All of the data listed above plus subsequent data obtained is now in the following computer database: Ciolkosz, E. J. and N. C. Thurman. 1993. Pennsylvania State University Soil Characterization Laboratory Database, Agronomy Dept., Pennsylvania State University, University Park, PA.)
No. 112	Ciolkosz, E. J. and R. R. Dobos. 1991. Pennsylvania State University Soil Characterization Laboratory Data Summary for Standard Samples.
No. 117	Thurman, N. C., E. J. Ciolkosz, and R. R. Dobos. 1992. Pennsylvania State University Soil Characterization Laboratory Methods Manual.
No. 118	Thurman, N. C. and E. J. Ciolkosz. 1992. A Comparison of Soil Characterization Laboratory Methods.
No. 124	Ciolkosz, E. J. and N. C. Thurman. 1992. Pennsylvania State University Soil Characterization Laboratory Database System.

Agronomy Series Publications on the Distribution and Genesis of Pennsylvania Soils

No. 21	Ciolkosz E. J., G. J. Latshaw, R. L. Cunningham, and W. D. Sevon. 1971. Parent Material, Topography, and Time as Soil Forming Factors in Eastcentral Pennsylvania.
No. 52	Marchand, D. E., E. J. Ciolkosz, M. F. Bucek, and G. H. Crowl. 1978. Quaternary Deposits and Soils of the Central Susquehanna Valley of Pennsylvania.
No. 64	Ciolkosz, E. J. et al. 1980. Soils and Geology of Nittany Valley.
No. 80	Ciolkosz, E. J., G. W. Petersen, R. L. Cunningham, and R. C. Cronce. 1983. Geomorphology and Soils of Nittany Valley.
No. 92	Ciolkosz, E. J., R. C. Cronce, and W. D. Sevon. 1986. Periglacial Features in Pennsylvania.
No. 95	Ciolkosz, E. J. and R. L. Cunningham. 1987. Location and Distribution of Soils of the World, United States, and Pennsylvania.
No. 100	Ciolkosz, E. J., T. W. Gardner, and R. R. Dobos. 1988. Paleosols in Pennsylvania.
No. 103	Ciolkosz, E. J. and R. R. Dobos. 1989. Distribution of Soils of the Northeastern United States.
No. 105	Ciolkosz, E. J., R. C. Cronce, and R. R. Dobos. 1989. Amorphous Material in Pennsylvania Soils.
No. 108	Ciolkosz, E. J. and R. R. Dobos. 1990. Color and Mottling in Pennsylvania Soils.
No. 116	Ciolkosz, E. J. and N. C. Thurman. 1992. Geomorphology and Soils of the Northeastern United States and Pennsylvania: A Series of Reprints.
No. 119	Ciolkosz, E. J., W. J. Waltman, and N. C. Thurman. 1992. Fragipans in Pennsylvania Soils.
No. 120	Clark, G. M. et al. 1992. Central Appalachian Periglacial Geomorphology: A Field Excursion Guidebook.
No. 125	Thorn, C. E., G. M. Clark, and E. J. Ciolkosz. 1993. Frost Action Environments.
No. 126	Ciolkosz, E. J., A. W. Rose, W. J. Waltman, and N. C. Thurman. 1993. Total Elemental Analysis of Pennsylvania Soils.
No. 127	Ciolkosz, E. J., W. J. Waltman, and N. C. Thurman. 1993. Iron and Aluminum in Pennsylvania Soils.
No. 128	Ciolkosz, E. J., M. K. Amistadi, and N. C. Thurman. 1993. Metals in Pennsylvania Soils.
In Press	Ciolkosz, E. J., N. C. Thurman, W. J. Waltman, D. L. Cremeens, and M. D. Svoboda. 1994. Argillic Horizons in Pennsylvania Soils.

(Continued on the inside of the back cover)