

Recommended Methods of Soil Analyses for Canadian Prairie Agricultural Soils

S
593
R311
1988
c.1

Alberta
AGRICULTURE
300-1988



Library

Alberta
ENVIRONMENT

Alberta Department of the Environment
Library

RECOMMENDED

METHODS OF SOIL ANALYSES

FOR

CANADIAN PRAIRIE AGRICULTURAL SOILS

Prepared by
Alberta Agriculture
based on the methods used in
Alberta, Saskatchewan and Manitoba
Provincial Laboratories

JANUARY 1988



51551

MEMORANDUM

TO: THE MINISTER OF THE ENVIRONMENT

FROM:

THE ALBERTA ENVIRONMENTAL PROTECTION BOARD

Forwarded by
Alberta Environment
based on the findings set in
the Environmental Protection and
Control Act.

1989

PREFACE

This manual is intended to provide an outline of the analytical procedures in common use in Western Canada. These procedures provide data on which are based fertility and cultural recommendations for prairie agricultural soils. They have been reviewed and accepted by Soils Advisory Councils in Manitoba, Saskatchewan and Alberta.

The manual is not intended to provide a step by step description of procedures for the determination of soil nutrient elements. Such determinations can be carried out using a variety of analytical techniques. The technique used will be determined by the level of instrumentation available in a particular laboratory. Specific technique details can be obtained from Provincial Laboratory personnel using a particular method, or from qualified analytical chemists associated with instrument manufacturers.

Information pertinent to soil analysis methods in this manual may be obtained from one of the following provincial laboratories.

ALBERTA: The Director, Soils and Animal Nutrition Laboratory
Alberta Agriculture
6909 - 116 Street
Edmonton, Alberta, T6H 4P2
Telephone: (403) 436-9150, 427-8905

MANITOBA: Head, Manitoba Provincial Soil Testing Laboratory
Department of Soil Science
262 Ellis Building
University of Manitoba
Winnipeg, Manitoba, R3T 2N2
Telephone: (204) 474-8154

SASKATCHEWAN: The Director, Saskatchewan Soil Testing Laboratory
General Purpose Building
University of Saskatchewan
Saskatoon, Saskatchewan, S7N 0W0
Telephone: (306) 966-6890

The first edition of this manual was made possible through the contributions of many people including Mr. Jim Carson and Dr. Edward Redshaw, Alberta Agriculture Soils Branch, Mr. Dale Tomasiewicz, Director, Saskatchewan Soil Testing Laboratory, and Mr. Ken McGill, Head, Manitoba Provincial Soil Testing Laboratory. There will be continual update of this manual as new analytical techniques and procedures are developed by prairie laboratories.

D.H. Lavery, P.Ag.
A. Bollo-Kamara, Ph.D.
Alberta Agriculture
Soils and Animal Nutrition Laboratory
6909 - 116 Street
Edmonton, Alberta, T6H 4P2

SYNOPSIS

This manual is intended to provide an outline of the analytical procedures in use in the Western Canada Dairy Producers' Board which are based on the analytical and chemical procedures set out in the Analytical Manual, 1971. They are based on methods developed by the Dairy Producers' Board in Manitoba, Saskatchewan and Alberta.

The manual is not intended to provide a step by step description of procedures for the determination of any particular element and the results are to be used as a guide only. The analytical procedures are described in a general laboratory manual and the results are to be used as a guide only. The analytical procedures are described in a general laboratory manual and the results are to be used as a guide only.

The following list of laboratories is provided for information and should be obtained from one of the following provincial laboratories.

- ALBERTA: The Alberta Dairy and Animal Production Laboratory, Alberta Agriculture, 1000 - 11th Street, Edmonton, Alberta, T6H 4Z1. Telephone: (403) 427-2222, 27-2802.
- MANITOBA: Health, Animal Production and Food Testing Laboratory, Department of Soil Science, 150 St. James Street, Winnipeg, Manitoba, R3T 2X2. Telephone: (204) 478-2124.
- SASKATCHEWAN: The University of Saskatchewan, Animal Production Laboratory, University of Saskatchewan, Saskatoon, Saskatchewan, S7N 0W7. Telephone: (306) 974-8282.

The list of laboratories in this manual was made available through the cooperation of many people including Mr. Jim Taylor and Dr. Robert Anderson, Alberta Agriculture; Mr. Bill Johnson, Health, Animal Production and Food Testing Laboratory; and Mr. Ian Smith, Health, Animal Production and Food Testing Laboratory. There will be changes in the list of laboratories as new analytical techniques and procedures are developed by provincial laboratories.

D. H. Lawrence, Editor
D. J. Lawrence, Editor
Alberta Agriculture
Soil and Animal Production Laboratory
1000 - 11th Street
Edmonton, Alberta, T6H 4Z1

TABLE OF CONTENTS

<u>Method No:</u>	<u>Page</u>
1. SOIL REACTION (pH) AND ELECTRICAL CONDUCTIVITY (E.C.) . . .	7
2. HAND TEXTURE	10
3. ORGANIC MATTER	17
4. NITRATE-NITROGEN	19
5. AMMONIUM AND NITRATE-NITROGEN	22
6. PHOSPHORUS	23
7. POTASSIUM AND SODIUM	25
8. SPOT TESTS	27
9. SULFATE-SULFUR	28
10. TESTS FOR LIME REQUIREMENT	29
10(a). LIME REQUIREMENT BY INCUBATION	29
10(b). LIME REQUIREMENT BY DOUBLE BUFFER	32
11. ALUMINUM AND MANGANESE	34
12. COPPER, ZINC, MANGANESE AND IRON	35
13. BORON	36

TABLE OF CONTENTS

Page

Chapter No.

1	1	1
2	2	2
3	3	3
4	4	4
5	5	5
6	6	6
7	7	7
8	8	8
9	9	9
10	10	10
11	11	11
12	12	12
13	13	13
14	14	14
15	15	15
16	16	16
17	17	17

INTRODUCTION

A soil analysis, as defined for agricultural purposes, is "An operation or procedure which determines a property of the soil that is pertinent to the suitability of the soil to support plant growth". Soil analyses that provide reliable predictions of soil properties are the most practical basis known at the present time for soil fertility management. (Dahnke, Wm. C.). The process used to determine the reliability of a soil test in relation to plant growth response is known as "SOIL TEST CORRELATION".

For soil analysis information to be useful the levels determined must provide a basis for management recommendations. This process is known as soil test "CALIBRATION AND INTERPRETATION".

A complete soil testing program consists of four major parts:

- (1) Field research and method correlation and calibration
- (2) Field sampling and sample handling
- (3) Analytical procedures and laboratory operations
- (4) Data interpretation and recommendations

All four parts are essential for a soil testing program to function properly and provide reliable information for soil management. Without relevant field research from the region to correlate and calibrate soil tests the program will not be complete. Likewise when poor and improper sampling, analyses or interpretation procedures are used, the program will be damaged often resulting in misleading analyses and costly recommendations. This manual will concentrate on part 3. That is, the recommended "Analytical procedures and laboratory operations". The procedures presented in this manual have been extensively tested in Western Canadian laboratories. They are considered acceptable soil analysis procedures for use in providing data on which are based fertility and cultural recommendations for agricultural farm land. There may be more than one procedure that can be used for a particular determination. In this manual, for each determination, specific procedures and the Provincial Laboratory using them are identified. For details regarding a particular procedure the identified Provincial Laboratory should be contacted.

¹Dahnke, Wm. C. "Soil Test Correlation and Interpretation". Bull. 517, 1985, Agricultural Experiment Station, North Dakota State University, Fargo, North Dakota, 58105.

LABORATORY SAMPLING AND PREPARATION

SAMPLING

Procedures for field sampling are provided in Soil Sampling Guides available from provincial offices and laboratories. Since useable analytical results depend on sample quality, it is the responsibility of the submitter to ensure that soil samples are properly taken and handled prior to being submitted to the laboratory.

The sample submitted for analysis is a finite quantity that provides the laboratory with specific information not obtainable elsewhere. This includes composition, mineralogical and physical information. The most accurate analysis can only reveal what actually exists in the soil sample submitted. Therefore, it is first of all essential that the sample be representative of the producer's field. It now becomes the responsibility of the laboratory to ensure that no alteration or contamination of the sample occurs that might jeopardize the analytical results.

The soil sample received is unique. It cannot be readily replaced without considerable delay and effort. Hence, sample handling in the laboratory is very critical. The expense and complexity of analytical work and the importance of the conclusions that will be derived further emphasize the necessity for accurate laboratory sampling. The major steps in laboratory sampling are drying, grinding and taking a portion for analysis (subsampling).

DRYING

Sample drying is one of the major concerns in maintaining quality and producing reproducible analysis results. Air drying soil samples is recommended immediately after field sampling and prior to submission for analysis. Drying strongly inhibits biological activity in the sample that can cause marked changes in composition, particularly the available nitrogen content. Drying prior to submission will also enable reduced analytical turn around time in the laboratory.

Wet or moist samples will require drying upon receipt at the laboratory prior to grinding and analysis. This is achieved by spreading a thin layer of sample in drying pans and drying in a forced air cabinet (not an oven) at less than 30 degrees centigrade. Drying may also be carried out overnight at room temperature in a dust free area.

GRINDING

Soil samples for standard fertility analysis should be ground by stainless steel grinders or equivalent to pass through a 2 mm screen. Large, hard lumps occurring in samples should be broken into small fractions, using a jaw type crusher or wooden mallet, before small mill grinding is attempted.

Samples requiring total mineral analyses should be find ground by mortar and pestle, or by a noncontaminating mill, such as a SPEX MIXER MILL, fitted with either a methacrylate vial, a tungsten vial or polystyrene vial, depending on the nature of the sample and the type of analysis requested.

SUBSAMPLING

Laboratory subsampling of soil for analysis can be conducted by either weight or volume (scoop).

Whichever technique is used, it is important that the subsample truly represent the original sample submitted. Also, the subsampling method for any specific determination must be consistent with that used in the research calibration studies and must provide reproducible analysis results.

Provincial laboratories which are currently using either the weight or the scoop procedure have used the particular technique in all their method development and calibration studies.

Whether the weight or scoop method is used certain assumptions are made. For weight subsampling, assumptions are made concerning the average bulk density of the soil (which in most areas varies with soil texture and depth), since determining actual bulk density is a very lengthy procedure. In the Alberta Provincial Laboratory, the assumption is made the average bulk density is 1.47 grams per cubic centimeter. For scoop subsamples, the assumption is made that the actual sample weight in the levelled scoop will vary according to the bulk density of the soil in the field.

Differences will occur in soil test results for any given parameter (e.g. N, P or K) irrespective of the subsampling technique used. This analytical difference between one weighing and another, or one scooping and another, is due to the inherent heterogeneity of soil.

Analytical differences will exist between weight procedure subsamples and standard scoop subsamples because of actual weight differences between the subsamples. The degree of difference will vary with texture. Table 1 shows the weight variation that can occur among seven (7) soils of different textural classes.

SUBSAMPLING BY SCOOP (ALBERTA PROVINCIAL LABORATORY)

A critical factor in constructing scoops for subsampling soil is that the depth of the scoop bowl should be $\frac{2}{3}$ the inside diameter of the bowl. Specifications for several standard scoop sizes are listed in Table 11.

PROCEDURE FOR USING SCOOP

Stir the ground and screened sample with a spatula to loosen the soil.

TABLE I
WEIGHTS OF 5, 10, 15, AND 25 GRAM SCOOPS OF SOIL
FOR SEVEN SOIL CLASSES

Scoop Size	Heavy Clay	Clay	Clay Loam	Loam	Sandy Clay Loam	Sandy Loam	Sand
5g	5.76	5.20	5.25	6.13	4.98	5.31	6.93
10g	11.51	10.33	10.34	12.14	9.81	10.91	13.75
15g	17.17	15.48	15.55	18.27	14.76	16.35	20.64
25g	29.18	25.97	26.24	30.41	24.96	27.48	34.69

TABLE II
DIMENSIONS OF SCOOPS

Scoop Size	Calculated Volume (cc)	Diameter (mm)	Depth 2/3 Diameter (mm)
5g	4.55	20.56	13.71
10g	9.10	25.90	17.27
15g	13.65	29.66	19.77
25g	22.75	35.16	23.44

Take a heaping scoopful (use appropriate scoop size) of soil.

Holding the spatula firmly, tap the handle behind the scoop bowl 3 times to settle the soil.

Scrape excess soil off the scoop with the spatula to obtain a level scoopful.

Dispense into an appropriate container and tap the scoop again to ensure that no soil remains behind.

Calculate and report analytical results using the scoop size as the assumed weight of the soil.

SUBSAMPLING BY WEIGHT (MANITOBA PROVINCIAL LABORATORY)

Subsamples are weighed for all analyses. The results in ppm (parts per million) are multiplied by a factor to convert them to kg/ha (kilograms per hectare). This factor, which may be obtained from Table III, accounts for the differences in bulk densities associated with soils of different textures and at different depths of sampling.

In Manitoba organic soil subsamples are also weighed. Soils are classed as organic when the bulk densities are less than 0.9 grams per cubic centimeter (air-dried and ground through a 2 mm screen).

TABLE III

BULK DENSITY FACTORS FOR CONVERTING
PPM (PARTS PER MILLION) TO KG/HA (KILOGRAMS PER HECTARE)

Texture	0-15cm	15-30cm	30-60cm	15-60cm	30-75cm	60-75cm	60-90cm
Sand	2.5	2.5	5.2	7.7	7.9	2.6	5.2
LSand	2.1	2.3	5.1	7.4	7.7	2.6	5.1
SLoam	2.2	2.2	4.6	6.7	6.8	2.3	4.6
Loam	1.9	2.1	4.4	6.5	6.7	2.3	4.4
SiLoam	1.8	1.8	4.9	6.7	7.4	2.5	4.9
Silt	1.8	2.0	4.8	6.7	7.2	2.4	4.8
SCL	2.2	2.2	4.4	6.6	6.7	2.3	4.4
ClayL	2.1	2.1	4.2	6.3	6.4	2.2	4.2
SiCL	1.8	2.3	4.4	6.7	6.7	2.3	4.4
SC	2.6	2.6	4.4	7.1	6.7	2.3	4.4
SiC	1.8	1.9	3.9	5.8	5.8	1.9	3.9
Clay	1.8	1.8	4.4	6.3	6.7	2.3	4.4
Peat	0.6			1.8			
Muck	0.6			1.8			

STANDARD METHODS FOR SOILS ANALYSES

INTRODUCTION

The methods presented are the recommended standard procedures for analyses of soil samples. For each method a brief introduction is given with subsequent sections on reagents, equipment, procedures for extracting the soil nutrients and recommended analytical techniques.

The recommended quantities of reagents for preparing extracting solutions are for batch quantities. Smaller quantities of extracting solution can be prepared by making appropriate calculations for the desired concentration. Descriptions for the preparation of analytical reagents must be interpreted as dissolving the reagent in distilled water and adding enough distilled water to make the final volume to the desired quantity.

1. SOIL REACTION (pH) AND ELECTRICAL CONDUCTIVITY (E.C.)

ALBERTA PROVINCIAL LABORATORY (1:2 SOIL:WATER MIXTURE)

Introduction

Soil reaction (pH) and electrical conductivity (E.C.) can be determined simultaneously in a 1:2 soil:water mixture. The conductivity value determined is multiplied by a conversion factor (2.063), to give an approximate equivalent saturated paste value. The reason for this conversion is that most clients understand and have become accustomed to relating saturated paste values to crop growth conditions.

Reagents and Equipment

pH meter
pH indicating electrode
pH reference electrode
conductivity dip type electrode
conductivity meter

A single pH combination electrode may be substituted for the pH indicating and reference electrodes.

Procedure

With a 15 gram scoop dispense soil into vials or containers
Add 30 ml of distilled water and shake for 30 min.
Standardize the pH meter with standard buffers
Calibrate the conductivity meter

Analysis

Insert electrodes into the soil water mixture
Read the pH and E.C.
Multiply the E.C. value by 2.063
Results are reported in mS/cm

Comments

If the E.C. is equal to or greater than 1.0 (saturated paste equivalent), retain the water extract after the pH and E.C. measurements for the qualitative estimate of water soluble sulfate.

SASKATCHEWAN PROVINCIAL LABORATORY (1:1 SOIL:WATER MIXTURE)

Introduction

pH and E.C. are determined on a 1:1 soil:water mixture¹. The conductivity value determined is interpreted based on the criteria conventionally used with the saturation extract procedure and the observed relationship between the 1:1 mixture and saturation extract conductivity values.

¹Hogg, T.J. and J.L. Henry 1984, C.J.S.S. 64: 699-704.

Reagents and Equipment

pH meter
pH indicating electrode
pH reference electrode
conductivity dip type electrode
conductivity meter

Procedure

Weigh 50 grams of soil into small beakers
Add 50 ml of distilled water and stir intermittently for 30 min.
Standardize the pH meter with standard buffers
Calibrate the conductivity meter

Analysis

Insert electrodes into the soil water mixture
Read the pH and E.C. while stirring suspension
Correct the E.C. reading to 25 degrees centigrade
Record and report the 1:1 E.C. values as determined

MANITOBA PROVINCIAL LABORATORY (1:1 SOIL:WATER MIXTURE)

Introduction

Soil reaction (pH) and electrical conductivity (E.C.) are determined simultaneously by any one of two methods. One using a 1:1 soil:water mixture, the other as part of the salt analysis by the saturated paste method.

Reagents and Equipment

pH meter
pH indicating electrode
pH reference electrode
conductivity dip type electrode
conductivity meter

Procedure

Weigh 50 grams of soil into a container
Add 50 ml of distilled water
Shake for 30 min.
Allow mixture to settle
Determine the pH and E.C. in the supernatant
Report the pH and 1:1 E.C. value as determined

SATURATED PASTE METHOD

Place approximately 200 grams of soil in a 400 ml beaker. Add distilled water to the soil, while stirring with a spatula, until it becomes a paste. At saturation, the soil paste glistens and flows slightly when the container is tipped. Also, with the exception of clays, the soil slides freely off the spatula.

Let the mixture stand for 1 hour. If the appearance of the paste changes add soil or water to return it to the original state. Vacuum filter the paste with a large buchner funnel. Determine the pH of the paste and the E.C. of the extract collected using appropriate instrumentation.

The extract can be saved to determine water soluble cations and anions for salt analysis. (Calcium, potassium magnesium, sodium, chloride, sulfate, carbonate and bicarbonate).

2. HAND TEXTURE

ALBERTA PROVINCIAL LABORATORY

Introduction

Hand texture provides a semi-quantitative analysis of the textural classes of a soil. Further verification can be obtained by carrying out a quantitative textural mechanical analysis using the classical Bouyoucos or pipette methods.

The "Soil Texture Triangle" shown in Figure 1, contains the thirteen specific classes used to classify mineral soils on the basis of percent sand, silt and clay content. Some laboratories use five broader categories, superimposed on the texture triangle in Figure 1, to classify soils for fertility purposes. These five categories provide a more realistic classification when texture is determined by the rapid hand texture method. A sixth category is used to identify organic soils (those soils that do not contain discernible mineral fraction by hand texture).

The procedure for carrying out the semiquantitative hand texture analysis is explicitly described in Figure 2 with a detailed instructional diagram.

¹Obtained from Soils Survey: Canada Agriculture Research Branch, Edmonton, Alberta.

SOIL TEXTURE TRIANGLE - ALBERTA PROVINCIAL LABORATORY

ALBERTA PROVINCIAL LABORATORY

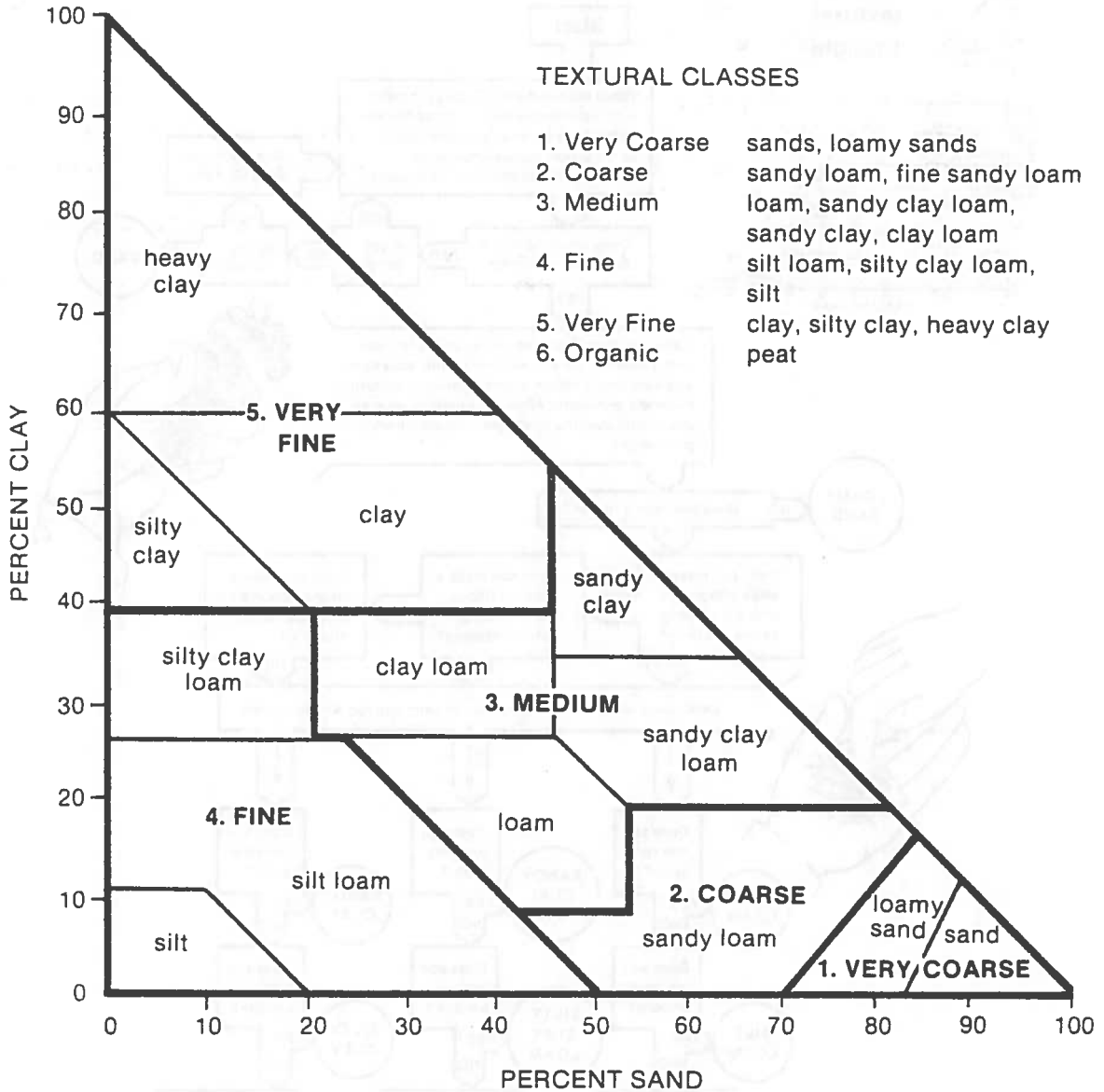


Figure 1: SOIL TEXTURE RATINGS

PROCEDURE FOR HAND TEXTURING BY FEEL - ALBERTA, SASKATCHEWAN AND MANITOBA LABORATORIES

ALBERTA, SASKATCHEWAN, & MANITOBA PROVINCIAL LABORATORIES

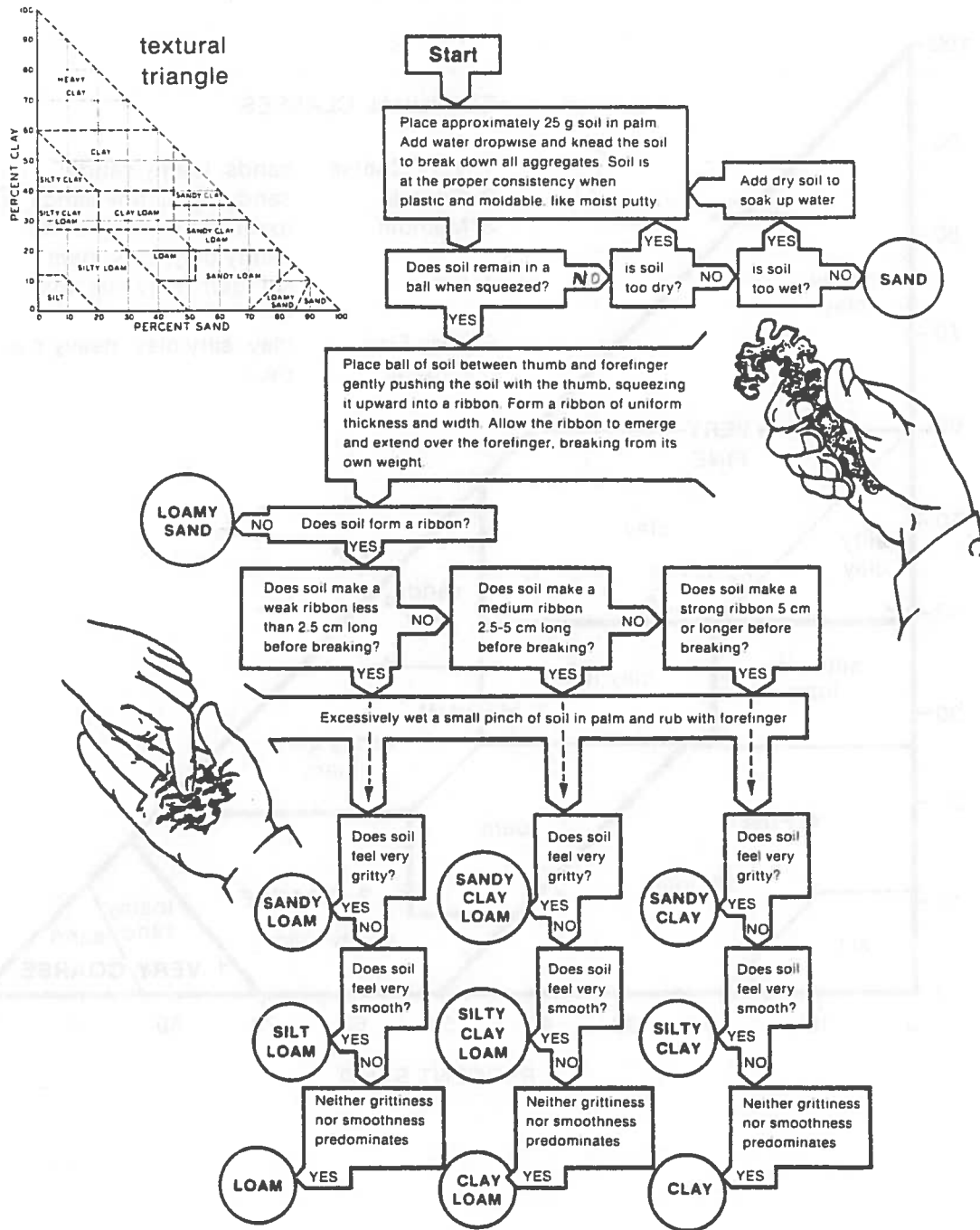


Figure 2. INSTRUCTIONAL DIAGRAM FOR TEXTURING BY FEEL

SASKATCHEWAN PROVINCIAL LABORATORY

Currently the hand texture analysis is used but seven categories are identified instead of six as described previously for Alberta. The soil textural diagram in Figure 3, is an attempt to rate texture on a linear scale of soil fineness.

The texture triangle is divided into six categories with the seventh for organic (peat) soil containing little or no discernible mineral fraction. The instructional diagram (Figure 2) for hand texturing by feel can still be used.



SOIL TEXTURE TRIANGLE - SASKATCHEWAN PROVINCIAL LABORATORY

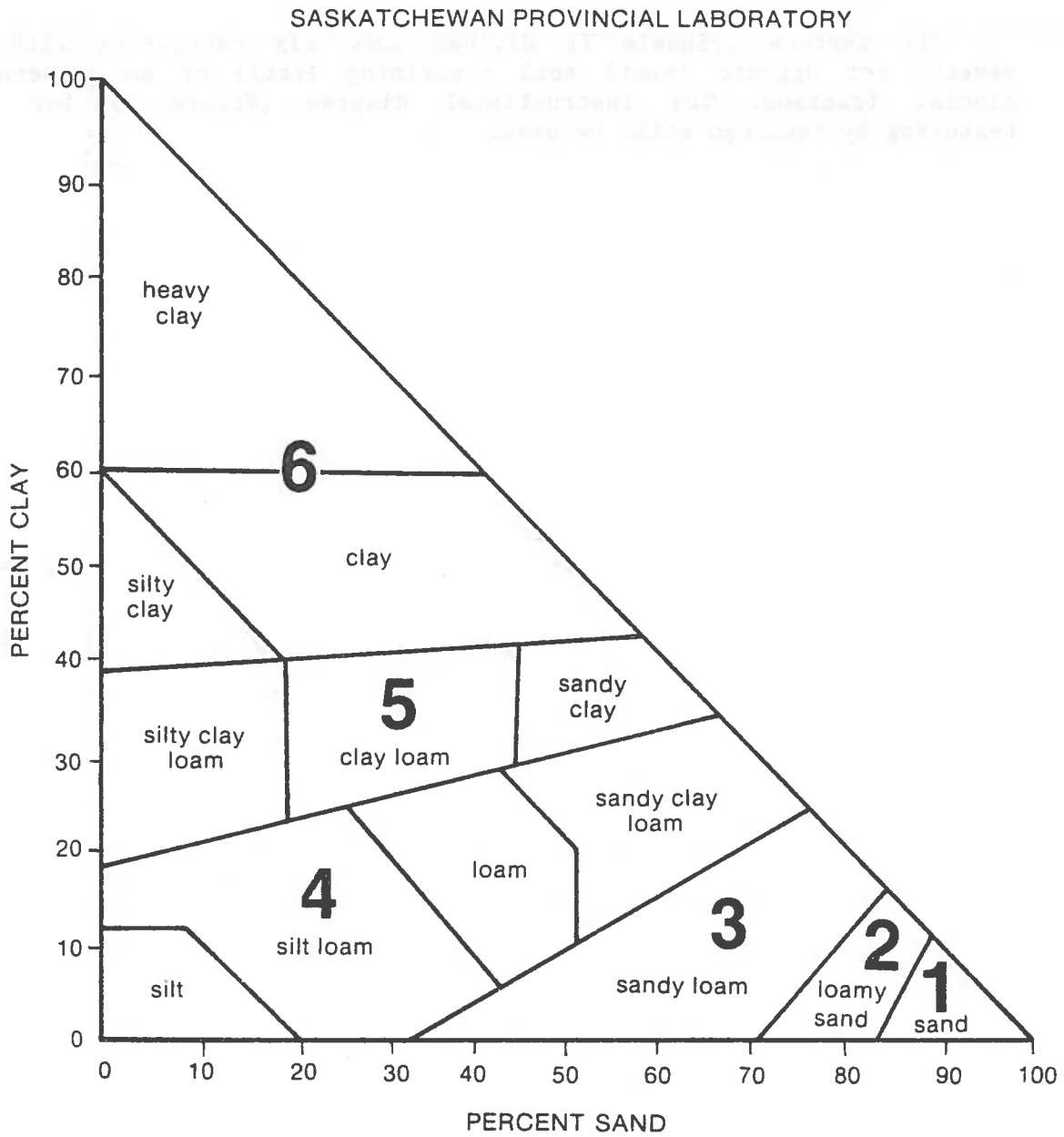
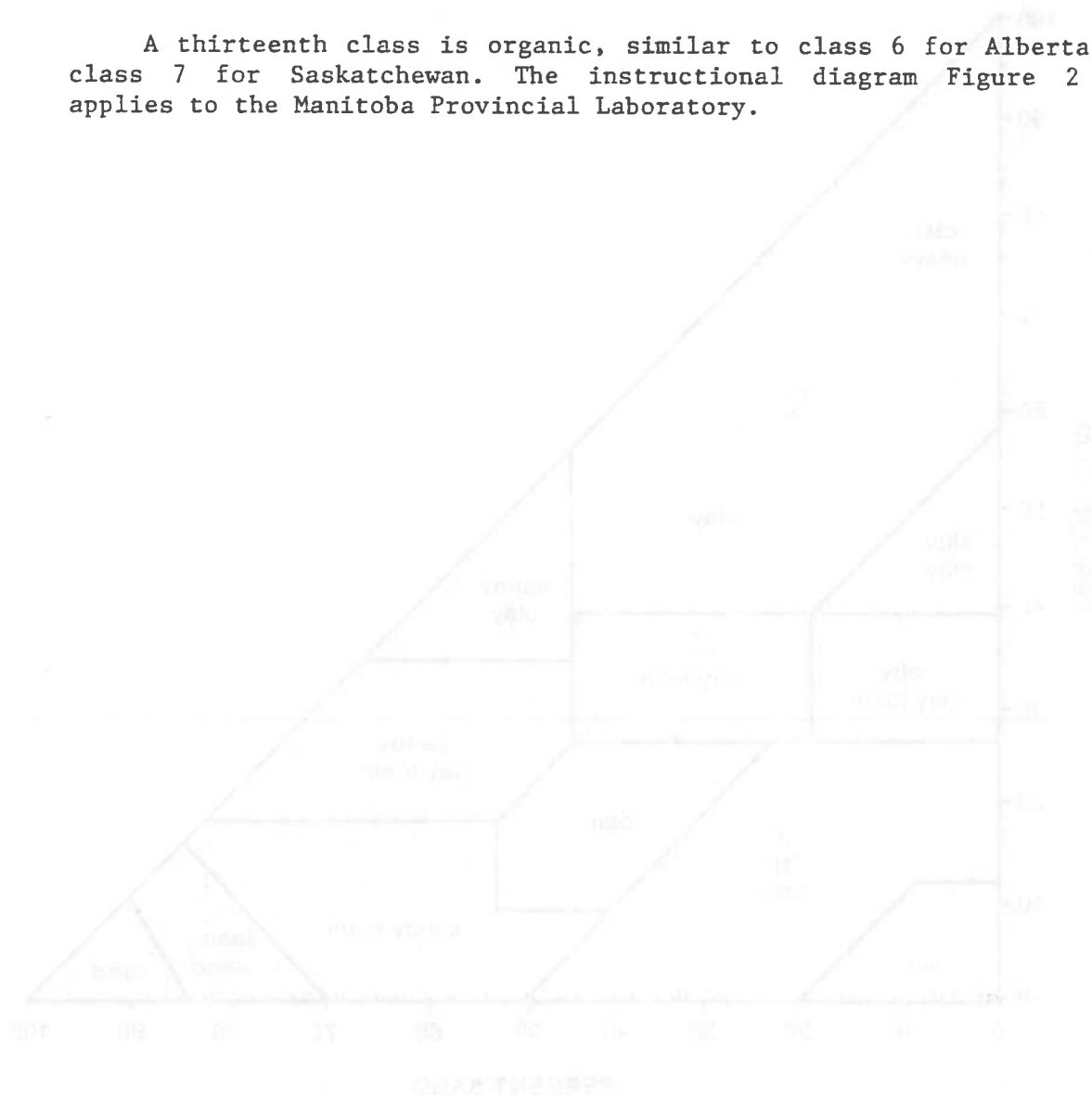


Figure 3. SOIL TEXTURE RATINGS

MANITOBA PROVINCIAL LABORATORY

Currently hand texture analysis is used and textural classes are identified using the 12 categories shown in Figure 4. Soils are labelled as sand, loamy sand, sandy loam, loam, sandy clay loam, etc. In addition modifiers are used extensively in the sandy loam class. For example a sandy loam may be further defined as fine sandy loam or very fine sandy loam.

A thirteenth class is organic, similar to class 6 for Alberta, and class 7 for Saskatchewan. The instructional diagram Figure 2 also applies to the Manitoba Provincial Laboratory.



SOIL TEXTURE TRIANGLE - MANITOBA PROVINCIAL LABORATORY

MANITOBA PROVINCIAL LABORATORY

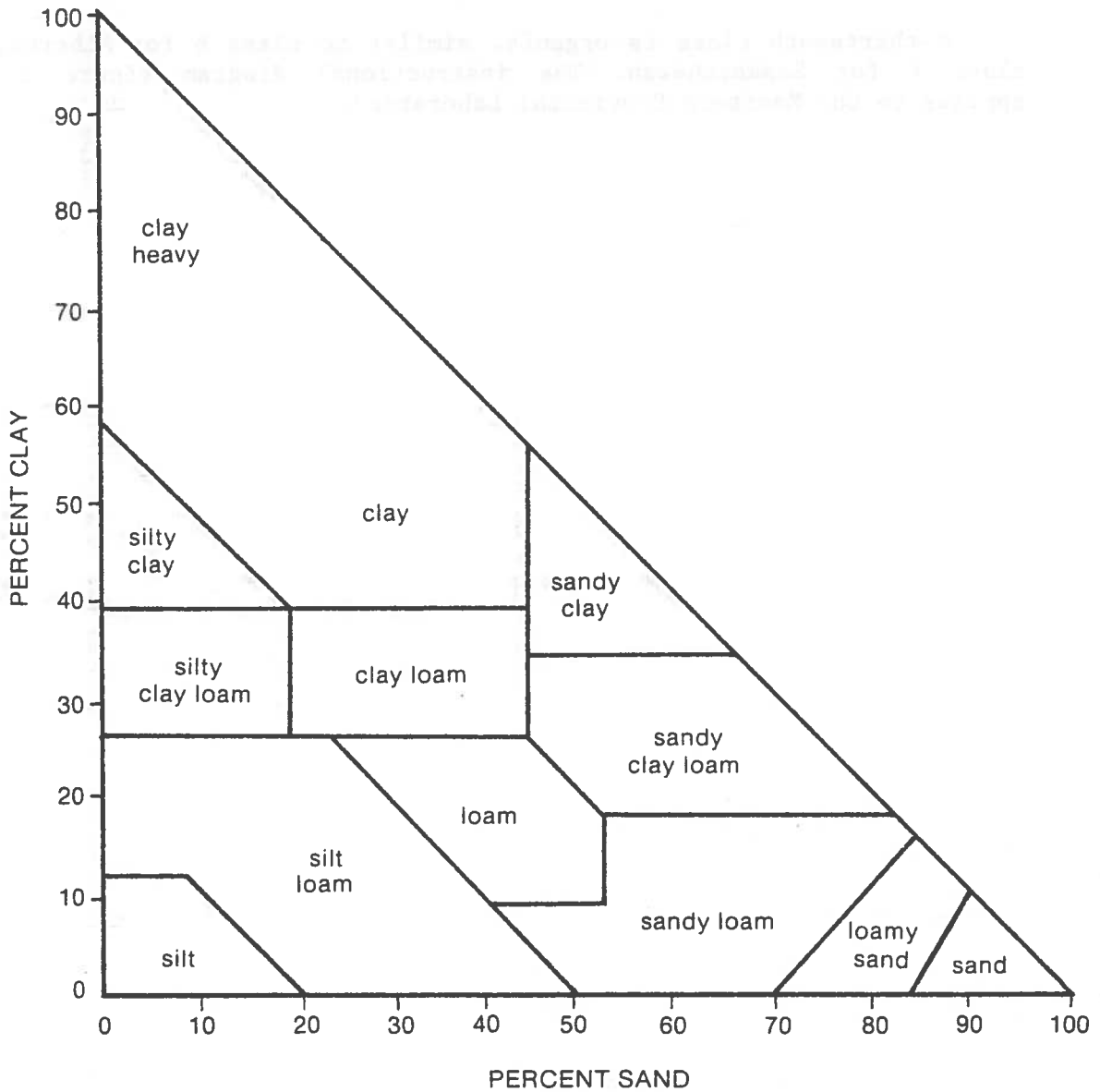


Figure 4. SOIL TEXTURE RATINGS

3. ORGANIC MATTER

ALBERTA PROVINCIAL LABORATORY - (LOSS ON IGNITION)

Introduction

The ignition method used in Alberta, was adapted from Ball¹. This analysis is not part of the routine analysis package but is offered on request for an additional fee. The method involves heating non-calcareous soils at 375 degrees centigrade in an oven or furnace and estimating the organic matter content by weight difference before and after ignition. This method is subject to errors caused by volatilization of substances other than organic material and incomplete oxidation of carbonaceous materials.

Reagents and Apparatus

No reagents required
Muffle furnace
Vycor or equivalent crucible

Procedure

Weigh out 20 grams of soil into a vycor or other suitable crucible of known weight
Dry the soil at 105 degrees centigrade for a minimum of 6 hours
Cool and reweigh (oven dry weight)
This weight is the value to use in calculating organic matter
Place the crucible and soil in a muffle furnace
Set the furnace temperature to 375 degrees centigrade
Ignite for 16 hours
Remove the crucible from the furnace.
Cool in a dessicator and weigh.

The percent ratio of weight loss during the ignition to the soil weight after drying at 105 degrees centigrade is recorded as the percent organic matter in the soil.

SASKATCHEWAN AND MANITOBA PROVINCIAL LABORATORY - (DICHROMATE METHOD-WET OXIDATION)

Introduction

This method was adopted from Walkley and Black². The method involves oxidation of the "easily oxidizable organic carbon" by chromic acid and back titration of the excess chromate. It is assumed that organic carbon is the only substance present that reduces dichromate, and that approximately 77% of the organic carbon is oxidized by this method. Soil organic matter is assumed to contain approximately 58%

¹Ball, D.F. Journal of Soil Science 15, No. 1, 1964.

²Walkley, A., and Black, I.A.; Soil Science 37: 29-37, 1934.

carbon. These numbers are obtained from averages but are satisfactory for comparison purposes. The method described herein is the spontaneous heating procedure where the heat of reaction is used in the oxidation process.

Reagents

Potassium dichromate - 1N

Dissolve 49.04 grams of reagent grade potassium dichromate in 1 litre of distilled water

Ferrous sulfate - 0.5N

Dissolve 139 grams of reagent grade ferrous sulfate in 900 ml of distilled water

Add 40 ml of concentrated sulfuric acid and dilute to 1 litre with distilled water. Standardize daily by titrating against potassium dichromate solution

Concentrated sulfuric acid - 96%

Procedure

Weigh 0.5 grams of soil into a 400 ml beaker

Add 10 ml of 1N potassium dichromate

Add 20 ml of concentrated sulfuric acid and swirl the solution immediately. Continue swirling vigorously for one minute

Allow to cool on a non heat conducting surface (asbestos pad) for 1 hour

Add 250 ml of distilled water

Titrate with 0.5N ferrous sulfate solution

Analysis

The titration can be carried out by automatic or manual procedures. Select the method for determining the endpoint (pH, millivolt or indicator). For more details contact the appropriate Provincial Laboratory.

Note: The procedure used in Saskatchewan Provincial Laboratory has been modified by using heating under reflux. The percent organic carbon assumed oxidized by this procedure is 87%.

4. NITRATE-NITROGEN

ALBERTA PROVINCIAL LABORATORY (AMMONIUM FLUORIDE - SULFURIC ACID EXTRACTION)

Introduction

Soil samples are extracted with a solution of 0.03N ammonium fluoride and 0.03N sulfuric acid.

Extracting Solution

Prepare a large batch of extracting solution, by dissolving 55.6 grams of ammonium fluoride and 77.0 grams (42 ml, specific gravity 1.84) of concentrated sulfuric acid in 50 litres of distilled water.

Standard Solution

Prepare a standard solution of 1000 ppm nitrogen by dissolving 7.22 grams of potassium nitrate in 1 litre of distilled water. Store in a refrigerator. Make appropriate standards from the stock solution by diluting with the extracting solution. A set of standard solutions with a range of nitrogen concentrations is required to calibrate the instrument.

Preparing and Extracting Soil Samples

Dispense one 5 gram scoop of soil into a 50 ml flask
Add 25 ml of extracting solution (1:5 extracting ratio)
Shake for 10 min.
Filter through #42 (or equivalent) filter paper
Collect the filtrate and analyze for nitrate-nitrogen

Analysis

Most laboratories use a colorimetric technique. However, other analytical techniques such as ion chromatography and electrochemistry can be used.

SASKATCHEWAN PROVINCIAL LABORATORY (CALCIUM CHLORIDE EXTRACTION)

Introduction

Soil samples are extracted with 0.001M calcium chloride. This same extract is used for the determination of sulfate-sulfur.

Extracting Solution

Prepare a large batch of 0.001 calcium chloride solution by dissolving 2.94 grams of calcium chloride (2 water of crystallization) in 20 litres of distilled water.

Standard Solution

Prepare appropriate standards using the extracting solution to calibrate the analytical instrument.

Preparing and Extracting Samples

Weigh 25 grams of soil into a 125 ml Erlenmeyer flask
Add 50 ml of extracting solution (1:2 extracting ratio)
Shake for 30 min.
Filter through Whatman #42 (or equivalent) filter paper
Collect the filtrate and analyze for nitrate-nitrogen

Analysis

Most laboratories use a colorimetric technique. However, other analytical techniques such as ion chromatography and electrochemistry can be used.

MANITOBA PROVINCIAL LABORATORY (SODIUM BICARBONATE EXTRACTION)

Introduction

Soil samples are extracted with 0.5M sodium bicarbonate at pH 8.5. The resulting extract is used for the simultaneous determination of nitrate-nitrogen and phosphorus.

Extracting Solution

Prepare a large batch of 0.5M sodium bicarbonate extracting solution as follows:

Dissolve 840 grams of sodium bicarbonate in 20 litres of distilled water
Adjust pH to 8.5 using sodium hydroxide
This solution is usually good for about one month
Prepare a new solution when in doubt
Always check the pH of solution before using

Standard Solution

Prepare appropriate standards using the extracting solution to calibrate the analytical instrument.

Preparing and Extracting Samples

Weigh 2.5 grams of 2 mm soil into a 125 ml Erlenmeyer flask
Add 50 ml of 0.5M extracting solution (sodium bicarbonate) (1:20 extracting ratio)
Make sure the extracting solution has a pH of 8.5
Add 1 gram of activated charcoal (free of phosphorus and nitrogen)
Shake at slow speed for 30 min.
Filter through Whatman #30 (or equivalent) filter paper
Collect the filtrate and analyze for nitrate-nitrogen

Analysis

Most laboratories use a colorimetric technique. However, other analytical techniques such as ion chromatography and electrochemistry can be used.

5. AMMONIUM AND NITRATE-NITROGEN

ALBERTA PROVINCIAL LABORATORY (POTASSIUM CHLORIDE EXTRACTION)

Introduction

Ammonium and nitrate¹ analyses can be performed simultaneously on the same extract. Bremner¹ provided the basis for the use of potassium chloride as an extractant for exchangeable ammonium, nitrate and nitrite. This extractant will quantitatively recover nitrate and nitrite added to soils. Also, ammonium added to soils that have low ammonium fixation capability is quantitatively recovered.

Extracting Solution

Prepare a 2 M solution of potassium chloride by dissolving 7456 grams of potassium chloride in 50 litres of distilled water.

Standard Solution

Prepare a 1000 ppm solution of ammonium and nitrate by dissolving 5.7153 grams of dried (100 degrees centigrade) ammonium nitrate in 1 litre of distilled water. Store in a refrigerator. Make appropriate standards from the 1 litre stock solution by diluting with the extracting solution. A set of standard solutions is required with a range of concentration to match the type of samples to be analyzed.

Preparing and Extracting Soil Samples

Dispense one 5 gram scoop of soil into a 100 ml vial
Add 25 ml of extracting solution (1:5 extracting ratio)
Shake for about 1 hour
Filter through Whatman #42 (or equivalent) filter paper
Collect the filtrate and store in a refrigerator until ready for analysis

Analysis

Conduct analysis determination on the filtrate by colorimetry or other equivalent technique for ammonium and nitrate.

Note: Saskatchewan and Manitoba Provincial Laboratories do not routinely determine ammonium-nitrogen.

¹Bremner, J.M. and D.R. Keeney. 1965. Steam distillation methods for determination of ammonium, nitrate and nitrite. Anal. Chem. Acta 32:485-495.

6. PHOSPHORUS

ALBERTA PROVINCIAL LABORATORY (AMMONIUM FLUORIDE-SULFURIC ACID EXTRACTION)

Introduction

Soil samples are extracted with a solution of 0.03N ammonium fluoride and 0.03N sulfuric acid.¹ Some laboratories use sodium bicarbonate as an extractant.

Extracting Solution

Prepare a large batch of extracting solution by dissolving 55.6 grams of ammonium fluoride and 77.0 grams of sulfuric acid in 50 litres of distilled water.

Standard Solution

Prepare a 2000 ppm solution of phosphorus by dissolving 8.7872 grams of potassium hydrogen phosphate in 1 litre of distilled water. Store in a refrigerator. Make appropriate standards from the stock solution by diluting with the extracting solution and prepare a set of standard solutions with a range of phosphorus concentrations to calibrate the instrument.

Preparing and Extracting Soil Samples

Dispense one 5 gram scoop of soil into a 50 ml Erlenmeyer flask
Add 25 ml of extracting solution (1:5 extracting ratio)
Shake for 10 minutes
Filter through Whatman #42 (or equivalent) filter paper
Collect the filtrate and analyze for phosphorus

Analysis

Most laboratories use a colorimetric procedure but other analytical techniques such as emission spectroscopy can be used.

ALBERTA, SASKATCHEWAN AND MANITOBA PROVINCIAL LABORATORIES (SODIUM BICARBONATE EXTRACTION)

Introduction

Sodium bicarbonate extraction² is used as an alternate method in the Alberta Provincial Laboratory. However, it is used on a routine basis in Saskatchewan and Manitoba Provincial Laboratories. Available potassium

¹Bray, R.H. and L.T. Kurtz. 1945. Determination of total organic and available forms of phosphorus in soils. Soil Sci. 59:39-45.

²Olsen, S.R., C.V. Cole, F.S. Watanabe, and L.A. Dean. 1954. Estimation U.S. Dept. of Agric. Circ. 939.

and nitrogen may be simultaneously determined with phosphorus in the same extract. The Manitoba Provincial Laboratory determines available nitrogen and the Saskatchewan Provincial Laboratory determines available potassium using this extract.

Reagents

Sodium bicarbonate
Sodium hydroxide

Extracting Solution

Prepare a large batch of 0.5M extracting solution by dissolving 840 grams of sodium bicarbonate in 20 litres of distilled water
Adjust the pH to 8.5 using 1M sodium hydroxide solution
Add mineral oil to avoid exposure to air
This solution is good for a month, however, check the pH daily

Preparing and Extracting Soil Samples

Weigh 5 grams of soil into a 250 ml flask
Add 100 ml of extracting solution (1:20 extracting ratio)
Saskatchewan and Manitoba weigh 2.5 grams of soil in 125 ml Erlenmeyer flasks and 50 ml of extracting solution. Saskatchewan adds carbon to the suspension before shaking.
Shake for 30 min.
Filter through Whatman #40 (or equivalent) filter paper.
Collect the filtrate and analyze for phosphorus.
Further pretreatment of the filtrate may be necessary before analysis.
Contact the appropriate laboratory for more details.

Analysis

The filtrate can be analyzed colorimetrically for phosphate and by flame photometer for potassium. However, both elements can be analyzed using an inductively coupled plasma.

7. POTASSIUM AND SODIUM

ALBERTA AND MANITOBA PROVINCIAL LABORATORIES (AMMONIUM ACETATE EXTRACTION)

Introduction

Soil samples are extracted with a neutral solution of 1N ammonium acetate for the determination of available potassium and sodium. Saskatchewan Provincial Laboratory determines potassium in the sodium bicarbonate extract described previously for phosphorus.

Extracting Solution

- 1N ammonium acetate
Dissolve 77.08 grams of ammonium acetate in 1 litre of distilled water
Adjust the pH to 7.00
- Use ammonium hydroxide if acidic to adjust the pH to 7
 - Use acetic acid if basic to adjust the pH to 7

Standard Solution

Prepare a 1000 ppm solution of sodium and potassium by dissolving 1.906 grams of potassium chloride and 2.543 grams of sodium chloride in 1 litre of distilled water. Make a series of standards with appropriate dilutions using the extracting solution. The series of standards should be appropriate to the analytical technique. If emission spectroscopy is used an internal standard may be necessary.

Internal Standard Solution

Prepare 1000 ppm solution of lithium by dissolving 9.86 grams lithium nitrate in 1 litre of distilled water. Dilute 5 ml of this stock to 1 litre using the extracting solution. Add a constant amount to all samples and standards.

Preparing and Extracting Soil Samples

ALBERTA PROVINCIAL LABORATORY

Dispense one scoop (5 grams) of soil into a 50 ml Erlenmeyer flask
Add 25 ml of ammonium acetate solution (1:5 extracting ratio) and shake for 5 min.
Filter through Whatman #40 (or equivalent) filter paper.
Collect the filtrate and analyze for potassium and sodium.

MANITOBA PROVINCIAL LABORATORY

Weigh 2.5 grams of air dry soil (2 mm) into a 125 ml flask
Add 25 ml of ammonium acetate solution (1:10 extracting ratio) and shake for 30 min.
Filter through Whatman #1 (or equivalent) filter paper.
Collect the filtrate and analyze for potassium and sodium.

Analysis

Sodium and potassium are alkali metals and should be analyzed with a low temperature flame if emission spectroscopy is chosen. Make sure a constant amount of internal standard is added to both standard and sample.

Sodium and potassium can also be analyzed using an inductively coupled plasma. In this case calcium and magnesium can also be determined in the same extract.

8. SPOT TESTS

ALBERTA PROVINCIAL LABORATORY

Introduction

These tests are used for rapid qualitative determinations of free sulphate and free lime.

Saskatchewan Provincial Laboratory does not use spot tests for free sulfate and free lime as part of the regular analysis.

Manitoba Provincial Laboratory determines quantitative free sulfate in the saturated paste extract. Free lime is determined as described below.

FREE SULFATE

This is a rapid qualitative test for free soluble sulfate in soils.

Reagents

Dissolve 7 grams of barium chloride in 100 ml of water.

Procedure

Place 1 ml of solution from the 1:2 soil water extract (refer pH and E.C. section) in a small test tube, add one drop barium chloride solution and mix. A permanent white precipitate indicates free sulfate.

FREE LIME

ALBERTA AND MANITOBA PROVINCIAL LABORATORIES

This is a rapid qualitative test for determining free lime in soils.

Reagent

Alberta uses 6M HCl, Manitoba uses 3M HCl.

Procedure

Add a few drops of 6M HCl to a small sample of soil in a spot plate. A rapid effervescence with the evolution of carbon dioxide confirms the presence of free lime. The degree of effervescence can be subjectively assessed as absent, very low, low, medium, high or very high.

9. SULFATE-SULFUR

ALBERTA, SASKATCHEWAN AND MANITOBA PROVINCIAL LABORATORIES (CALCIUM CHLORIDE EXTRACTION)

Introduction

In Alberta, extractable sulfate-sulfur is determined colorimetrically after extraction with 0.01M calcium chloride. Saskatchewan and Manitoba extract with 0.001M calcium chloride.

Extracting Solution

Prepare a large batch of 0.01M calcium chloride solution by dissolving 29.40 grams of calcium chloride in 20 litres distilled water. (2.94 grams for 0.001M solution)

Standard Solution

Prepare a 1000 ppm sulfur solution by dissolving 5.4299 grams anhydrous potassium sulfate in 1 litre 0.01M or 0.001M calcium chloride extracting solution. Prepare calibration standards from the stock solution diluting with 0.01M or 0.001M calcium chloride.

Preparing and Extracting Soil Samples

ALBERTA PROVINCIAL LABORATORY

Dispense one 10 gram scoop of soil into a 50 ml container
Add 20 ml 0.01M calcium chloride extracting solution (1:2 ratio)
and shake for 30 min.
Filter through Whatman #42 (or equivalent) filter paper
Collect the extract and analyze

SASKATCHEWAN AND MANITOBA PROVINCIAL LABORATORIES

Weigh 25 grams of dry soil into a 250 ml flask
Add 50 ml of 0.001M calcium chloride extracting solution (1:2 ratio)
and shake for 30 min.
Add 1 gram of activated charcoal and shake for 1 minute
Filter through Whatman #42 (or equivalent) filter paper
Collect the extract and analyze

Note: Saskatchewan Provincial Laboratory determines nitrate-nitrogen on this extract.

Analysis

The extract obtained can be analyzed by colorimetry, emission spectroscopy or ion chromatography.

¹Lazrus, A.L., K.C. Hill, and J.P. Lodge. A new colormetric microdetermination of sulfate ion. Paper presented at the Technicon Symposium, "Automation in Analytical Chemistry", New York, N.Y., Sept. 8, 1965. National Centre for Atmospheric Research, Boulder, Colorado.

10. TESTS FOR LIME REQUIREMENT

ALBERTA PROVINCIAL LABORATORY

Introduction

Two methods can be used, (1) the classical titration of the soil with calcium hydroxide involving intermittent shaking of the soil and calcium hydroxide mixture over a 72 hour period (Incubation Method) and (2) the more rapid Double Buffer Method.

Correlation studies, using acid soils from various regions, indicate that the double buffer and incubation methods gave results that were not statistically different for the soils studied. However, in situations where doubt may exist as to the validity of a prediction by the double buffer method, the lime requirement test should be repeated with the incubation method. This is most prevalent for soils with pH near neutral and pH greater than 6.00.

Note: Saskatchewan and Manitoba Provincial Laboratories do not determine lime requirement on farm soil samples.

10A. LIME REQUIREMENT BY INCUBATION

Equipment

pH meter accurate to 2 decimal places
Top loading balance accurate to 2 decimal places
Analytical balance accurate to 4 decimal places
Graduated cylinders (5, 10, 25, 50, 100 ml)
Eberback oscillating shaker
100 ml bottles or flasks
Racks to hold bottles or flasks
50 ml buret
20 litre plastic carboy
Soil grinder (particles to pass 2 mm sieve)

Reagents

Calcium hydroxide
pH 7 buffer
pH 4 buffer
Buffers are used for calibrating the pH meter.

Using Calcium Hydroxide

Calcium hydroxide powder can be weighed and mixed with the soil sample (calcium hydroxide powder method), or dissolved in water and added to the soil using a 50 ml buret (calcium hydroxide solution method). The latter procedure should only be used when a few samples are to be analyzed, since the calcium hydroxide solution must be prepared in small quantities to avoid contamination and deterioration due to carbon dioxide dissolution and precipitation of calcium carbonate. However, if

this method of titration is preferred it is advisable to prepare fresh solutions with each set of determinations. The weighed calcium hydroxide powder is the preferred method. It is the classical method and mimics the addition of lime to a soil.

Procedure For Solution Method

Prepare a 1.5 g per litre solution of calcium hydroxide using distilled water.

Weigh six 20 gram portions of a soil sample into 100 ml bottles Add calcium hydroxide and water to each portion as listed in

Table IV

Note: The equivalent tonnes of calcium carbonate for each mixture is also provided in Table IV

Place on a shaker and shake for 10 min.

Repeat shaking twice a day for 3 days

On the third day, shake and pour the slurry into containers

Measure the pH

TABLE IV
SUMMARY OF SOLUTION METHOD

Portion Number	Volume(ml) Calcium Hydroxide	Volume(ml) Water	Equivalent Tonnes of Calcium Carbonate
1	0	50	0
2	5	45	0.5
3	10	40	1.0
4	15	35	1.5
5	20	30	2.0
6	25	25	2.5

PROCEDURE FOR POWDER METHOD

TABLE V
SUMMARY OF POWDER METHOD

Portion Number	Powder(gm) Calcium Hydroxide	Equivalent Tonnes of Calcium Carbonate
1	0.0000	0
2	0.0075	0.5
3	0.0150	1.0
4	0.0225	1.5
5	0.0300	2.5
6	0.0375	2.5

Weigh six 20 gram portions of the soil sample into 100 ml bottles. Add powdered calcium hydroxide to these portions of sample in the amounts shown in Table V.

Add 50 ml of distilled water to each portion
Place on a shaker for 10 min.
Repeat shaking twice a day for three days
On the third day shake and pour the slurry into containers
Measure the pH

Data Reduction

Tabulate the six pH readings as shown in Table VI (the pH values are fictitious).

The amount of calcium carbonate (lime) required to reach a desired pH can be determined graphically or by mathematical computation.

TABLE VI
EXAMPLE OF DATA REDUCTION

Portion Number	pH	Tonnes/acre Calcium Carbonate
1	5.7	0
2	5.9	0.5
3	6.2	1.0
4	6.4	1.5
5	6.6	2.0
6	6.8	2.5

Graphical Determination

Plot pH as the ordinate (y axis) and tonnes of lime as the abscissa (x axis). Draw a line connecting the points. If the soil is to be limed to 6.5 then on the pH axis indicate 6.5. Draw a line parallel to the horizontal or x axis intersecting the curve at L2. From L2 draw another line parallel to the pH axis intersecting the x axis at L3. The reading of tonnes calcium carbonate at L3 is the desired lime requirement for the sample.

If a calculator or small computer is available with a least squares software package then more rapid determination is possible. The utilization of such programs is unique to a calculator or computer. If however you possess a mini computer with a Basic compiler (eg. TRS-80, IBM PC), a data reduction program can be obtained from the Alberta Provincial Laboratory.

10B. LIME REQUIREMENT BY DOUBLE BUFFER

ALBERTA PROVINCIAL LABORATORY

Equipment

Top loading balance accurate to 2 decimal places
Digital pH meter accurate to 2 decimal places
100 ml plastic vials
Eberback shaker

Reagents

Pyridine
Calcium chloride
Imidazole
Potassium chromate
TRIS (THAM) (tris hydroxymethyl aminomethane)
Hydrochloric acid
Ammonium hydroxide
pH 7 Buffer solution
pH 4 Buffer solution
Buffer solutions are used to calibrate the pH meter

Preparing Reagents

Weigh out two sets of the following:

4.28 g TRIS
1.54 g Imidazole
2.80 g Potassium dichromate
14.7 g Calcium chloride
Prepare a 10% pyridine solution in distilled water
Prepare a 0.5N HCl solution

The weights are those suggested by Yuan (1976). Dissolve each set of dry reagents in 0.9 litre distilled water and add 18.6 ml 10% pyridine solution to each solution. The original pH of this solution will be approximately 9.

Adjust one solution to pH 7. Label as Buffer 7
Adjust the other solution to pH 6. Label as Buffer 6.
Use the dilute acid for adjusting both buffers

This procedure requires patience and in most instances a trained technician. If excess acid is added so as to lower the pH below the required value add (1:1) ammonium hydroxide solution to raise the pH to the desired value. Bring the volume to 1 litre with distilled water.

Procedure

Weigh 5 grams of soil into each of two 100 ml vials
Add 50 ml of Buffer 7 to one sample
Add 50 ml of Buffer 6 to the other sample
Shake the samples for one hour
Read the pH of each soil suspension to 2 decimal places

Data Reduction

Tabulate pH readings for each sample

Let E7 = Equilibrium pH with Buffer 7

Let E6 = Equilibrium pH with Buffer 6

The buffering capacity L is expressed as follows:

$$L = d1-d2/E7-E6$$

Where: d1 = 7-E7; d2 = 6-E6

The Lime Requirement (LR) is then calculated as follows:

$$LR = ((6-E6) L(DpH-E6))10$$

Where: DpH is the desired pH (eg. 6.5)

LR is reported as the lime requirement in tonnes per acre

Useful References For Double Buffer Method

1. T.L. Yuan, Soil Sci. Soc., Am. Proc. 38, 437-440 (1974)
2. T.L. Yuan, Soil Sci. Soc., Am. J., 40, 800-802 (1976)
3. E.O. McLean, Commun. in Soil Science and Plant Analysis, 9(8), 699-715 (1978)
4. H. Ssali and J.K. Nuwamanya, ibid., 12(7), 643-659 (1981)

11. ALUMINUM AND MANGANESE

ALBERTA PROVINCIAL LABORATORY

Introduction

Acid soils often contain soluble forms of aluminum and manganese. As soil acidity increase (pH decreases) aluminum and manganese solubility increases and may reach toxic levels. Soluble aluminum and manganese are determined by extraction with a weak solution of calcium chloride.

Note: Saskatchewan and Manitoba Provincial Laboratories do not normally determine this form of aluminum and manganese on farm samples.

Extracting Solution

0.02M Calcium chloride (2 water of crystallization)
Dissolve 2.9404 grams calcium chloride in 1 litre of water

Procedure

Dispense one 10g scoop of soil into a 50 ml container
Add 20 ml of extracting solution (1:2 extracting ratio)
Shake for one hour
Centrifuge for 5 min. and filter through Whatman #42 (or
equivalent) filter paper
Collect the extract and analyze

Analysis

Atomic absorption or emission spectroscopy can be used to analyze the filtrate for aluminum and manganese. Pay particular attention to aluminum when analyzing by atomic absorption. Make sure a nitrous oxide acetylene flame is used.

12. COPPER, ZINC, MANGANESE AND IRON

ALBERTA, SASKATCHEWAN AND MANITOBA PROVINCIAL LABORATORY - (DIETHYLENE TRIAMINEPENTACETIC ACID (DTPA) EXTRACTION)

Introduction

Available levels of the essential micronutrients, copper, zinc, manganese and iron are determined by extracting with the strong complexing agent diethylene triaminepentacetic acid (DTPA).¹

Extracting Solution

0.005M DTPA, 0.01M calcium chloride and 0.01M triethamine (TEA) (pH 7.3). To make 1 litre of extracting solution weigh out the following:

1.9668 grams DTPA

1.4702 grams calcium chloride

14.9194 grams TEA

Put all three reagents in a one litre volumetric flask

Add about 900 mls distilled de-ionized water

Check the pH

If necessary adjust the pH to 7.3 with 6M HCl

Bring the volume to 1 litre

Procedure

Weigh 10 grams of air dried soil into a 50 ml flask

Add 20 ml of extracting solution (1:2 extracting ratio)

Shake for 2 hours

Filter through Whatman #42 (or equivalent) filter paper

Collect the extract and analyze for copper, zinc, manganese, and iron

Analysis

The extract can be analyzed by atomic absorption or atomic emission spectroscopy.

¹Lindsay, W.L., and W. A. Norwell. 1978. Development of DTPA soil test for iron, manganese, copper, and zinc. Soil Sci. Soc. Am. J. 42:421-428.

13. BORON

ALBERTA PROVINCIAL LABORATORY - (HOT WATER EXTRACTABLE BORON)

Introduction

Soluble forms of boron are extracted with hot water.

Extracting Solution

Distilled de-ionized water

Procedure

Weigh out 20 grams of soil into a 600 ml beaker

Add 40 ml of water (1:2 extracting ratio)

Attach to a reflux condenser and boil for 10 min.

An extraction unit can also be used in place of a reflux condenser (Labconco)

Centrifuge the contents and filter through Whatman #42 (or equivalent) filter paper

Collect the extract and analyze for boron

Analysis

The quantities of boron extracted by this method are very small, hence, a sensitive analytical technique must be used. The classical method for doing small amounts of boron in aqueous medium is the curcumin spectrophotometric determination. Emission spectroscopy with an ICP can be used to analyze small amounts of boron with better sensitivity than the classical colorimetric method. For further descriptions and applications of these techniques contact the appropriate laboratory.

SASKATCHEWAN PROVINCIAL LABORATORY - (AMMONIUM ACETATE EXTRACTABLE BORON)

Introduction

Ammonium acetate extractable boron has been found to correlate well with boron levels in plants.

Extracting Solution

1N Ammonium Acetate

Dissolve 77.08 grams of ammonium acetate in 1 litre of distilled water

Adjust pH to 4.8

Use ammonium hydroxide if acidic to adjust to pH 7.00.

Use acetic acid if basic to adjust pH to 7.00.

Procedure

Weigh 20 grams of soil into a 125 ml Erlenmeyer flask
Add 40 ml of ammonium acetate solution
Shake for 30 min.
Filter through Whatman #40 (or equivalent) filter paper
Collect the extract and analyze for boron

Analysis

The quantities of boron extracted by this method are very small, hence, a sensitive analytical technique must be used. The classical method for doing small amounts of boron in aqueous medium is the curcumin spectrophotometric determination. Emission spectroscopy with an ICP can be used to analyze small amounts of boron with better sensitivity than the classical colorimetric method. For further descriptions and applications of these techniques contact the appropriate laboratory.

MANITOBA PROVINCIAL LABORATORY - (CALCIUM CHLORIDE EXTRACTABLE BORON)

Introduction

The calcium chloride extraction procedure has provided more consistent results than the ammonium acetate extraction procedure.

Extracting Solution

0.02M Calcium chloride with 0.25g charcoal (washed in sodium chloride and air dried) and 0.5g/l polyacrylamide (M.W. over 5,000,000)

Reagents

Buffer Masking Agent

Add 250g ammonium acetate, 25g Na₄-EDTA and 10g Na₂-NTA to 400 ml water
Mix until dissolved and then add 125 ml of glacial acetic acid very slowly while mixing
Adjust the pH to 5.4 - 5.6 using either acetic acid or ammonium hydroxide

Azomethine-H

Dissolve 0.9g azomethine-H and 2g ascorbic acid in 30 - 40 ml distilled water
Bring up to 100 ml. (Prepare fresh each day)

Procedure

Weigh 10g of ground soil into a plastic pouch
Add 20 ml of 0.02M calcium chloride extract solution and seal the pouch
Place the pouch in boiling water and bring to a boil
After the pouch is boiling for 7 min. remove and filter the pouch contents through Whatman #42 (or equivalent) filter paper
Take a 4 ml aliquot of sample
Add 1 ml buffer agent and vortex
Add 1 ml azomethine - H solution and vortex
Let stand for 1 to 3 hours maximum then vortex and read with a colorimeter at 420 nm
Standard curve is 0.20 to 2.00 ppm with a cell pathlength of 1 cm.

