

Methods of Soil Analysis

as Used in the

OSC Soil Testing Laboratory

L. A. Alban
Mildred Kellogg



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INTRODUCTION

There are many methods currently in use for the testing of soils. Each section of the country appears to favor a particular method or group of methods and many times individual soil testing laboratories develop modifications of these methods. Then, too, procedures have changed greatly in the last 10 to 15 years. The development of new instruments, such as the flame photometer, and new titration procedures such as EDTA or Versene, have practically revolutionized soil testing. They not only have made the methods more accurate but have made them many times more rapid.

The methods for the analysis of soils as given here are those which are currently being used in the Oregon State College Soil Testing Laboratory. They have been selected on the basis of their reliability for predicting fertilizer and liming needs of Oregon soils and for their adaption to use for a large volume of soils. Changes will be made in these procedures from time to time as new methods and techniques are developed.

It is not the purpose of this bulletin to describe the various methods listed nor to compare them with any other methods in use. There are several books (1,2,6,8,10) and periodicals which devote many pages to the description of procedures involved in testing soils. Where applicable a reference is given to the originator of a particular method.

PREPARATION OF SAMPLE

Soil samples are air-dried and then passed through a mechanical crusher consisting of two rollers which turn toward each other. The soil then passes through a 10-mesh screen. The rollers are kept clean by large brushes held tightly against them. The soil samples are kept for at least 5 days before being tested, especially for potassium.

METHODS OF ANALYSIS

I. pH - 1:1 soil-water paste

A. Reagents

1. Standard buffer solutions - to standardize pH meter

B. Procedure

1. Weigh 10 grams of soil into a 50 ml beaker or No. 250 C.R. souffle cup. Add 10 ml of distilled water. Stir thoroughly.
2. Let stand for at least 30 minutes, stirring two or three times.
3. Read with a pH meter using a glass electrode.

II. LIME REQUIREMENT - Woodruff method (12)

A. Reagents

1. Buffer solution - Weigh out 800 grams calcium acetate, 160 grams p-nitrophenol, and 12 grams magnesium oxide. Dissolve in about 18 liters distilled water. Make up to 20 liters. Let settle for a week then adjust to pH 7.0 with HCl or NaOH.

2. Standard buffer solution for pH meter.

B. Procedure

1. Weigh 10 grams of soil into a 50 ml beaker or No. 250 C.R. soufflé cup. Add 10 ml of distilled water, stir and let stand for at least 15 minutes.
2. Add 20 ml of the buffered solution, mix well, and let stand for at least 20 minutes, stirring two or three times.
3. Read on glass electrode pH meter. East 0.1 drop from pH 7.0 is equivalent to 1000 lbs. CaCO_3 .

The buffer solution could be added after the soil pH is determined in Part I.

III. PHOSPHORUS - Sodium bicarbonate method (7)

A. Reagents

1. Sodium bicarbonate, 0.5 Molar - Dissolve 42.01 grams NaHCO_3 (Arm and Hammer, commercial grade can be used) in distilled water and make up to 1 liter. Adjust the pH to 8.5 with NaOH.
2. Ammonium molybdate - Dissolve 15.0 grams ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 300 ml of warm distilled water (60°C). Filter if necessary and add to the solution, after cooling, 342 ml of concentrated HCl gradually with mixing. Make up to 1 liter.
3. Stannous Chloride
 - a. Stock solution - Dissolve 10 grams $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 25 ml concentrated HCl. Prepare fresh every two months or less. Use large crystals rather than fine powder.
 - b. Dilute solution - Add 0.5 ml of the stock solution to 66 ml of distilled water. Prepare fresh for each set of determinations or at least once a day.
4. Standard phosphate solution - Dissolve 0.2195 gram pure KH_2PO_4 in distilled water. Dilute to 1 liter with bicarbonate extracting solution. This solution contains 50 ppm of phosphorus and serves as the base stock solution. Standard solutions of 0.5 to 5 ppm P can be made up when needed.

B. Procedure

1. Weigh out 2.5 grams of soil into a 50 ml shaking bottle and add 25 ml extractant. Shake for 30 minutes.
2. If resulting soil solution is colored, decolorize with Darco G-60. Use about $\frac{1}{4}$ teaspoon for each 25 ml extract, or more if necessary. Filter through Whatman No. 5 filter paper. The Darco G-60 may be added to the soil prior to shaking. (The Darco G-60 should be tested for phosphorus and if it contains enough to color the solution, it should be washed with sodium bicarbonate before using.)
3. Place 5 ml of the filtrate in a 25 ml volumetric flask. This can be done with an automatic pipette.
4. With an automatic pipette add 5 ml of ammonium molybdate solution to each flask and shake well. Remove all traces of the molybdate solution from the neck of the flask by washing down with distilled water until approximately 10 ml has been added.
5. Add 1.0 ml of the dilute stannous chloride solution, mix well immediately, make up to volume with distilled water and again shake thoroughly.

6. Read color intensity in the colorimeter using a 560 mu filter, ten minutes after addition of the stannous chloride solution. Determine amount of phosphorus from curve prepared by running a series of standards.

IV. POTASSIUM, CALCIUM, MAGNESIUM AND SODIUM - Flame Photometer method

A. Reagents

1. Ammonium acetate, 1 N - Add 700 ml of ammonium hydroxide to about 5 liters of distilled water. Then add 580 ml of acetic acid and make up to 10 liters. Shake vigorously. Adjust to pH 7.0 using a glass electrode pH meter.
2. Standard solutions for potassium, calcium and sodium - The following amounts of pure primary reagents, dissolved in a small amount of distilled water and made up to 1 liter with ammonium acetate, are used for standard solutions of 1000 ppm of the element:
 - a. Potassium - 1.9100 grams KCl
 - b. Calcium - 2.4973 grams CaCO_3
 - c. Sodium - 2.5418 grams NaCl
3. Standard solution for magnesium - Dissolve 0.5 grams pure Mg ribbon and 2.4973 gram CaCO_3 in 1:1 HCl and evaporate to dryness on hot plate. Take up residue in 500 ml NH_4OAc , add 125 ml of 1000 ppm solution, 50 ml of 1000 ppm Na solution, and make up to a liter with NH_4OAc . The magnesium standard solution should contain 1000 ppm Mg, 2000 ppm Ca, 125 ppm K, and 50 ppm Na.

B. Procedure

1. Weigh or measure 2 grams of soil into a 50 ml shaking bottle, add 20 ml of the ammonium acetate extractant, and shake for 30 minutes.
2. Filter through Whatman No. 5 filter paper.
3. Determine with the flame photometer using the following wave lengths:
 - a. Magnesium - 383 mu (285.2 mu if possible)
 - b. Calcium - 554 mu
 - c. Sodium - 580 mu
 - d. Potassium - 768 mu
4. Prepare a curve for each element by running a series of standards.

V. CATION EXCHANGE CAPACITY - Ammonium acetate method (9)

A. Reagents

1. Ammonium acetate, 1 N - Make up the same as in IV.
2. Ethyl alcohol, 95%.
3. Hydrochloric acid, 0.1 N
4. Boric acid solution, saturated
5. Standard sulfuric acid, approximately 0.1000N - Use 27.7 ml C.P. concentrated H_2SO_4 and make up to 10 liters. Mix well. Standardize against 25 ml of 0.1000 N Na_2CO_3 solution made by weighing out 2.6570 grams of oven dry C.P. Na_2CO_3 and making up to 500 ml with distilled water. Use mixed indicator.
6. Mixed indicator - 0.1 gram bromocresol green and 0.02 gram methyl red dissolved in 100 ml ethyl alcohol.

B. Procedure

1. Weigh out 10 grams of soil and place in a shaking bottle with 50 ml of ammonium acetate solution, shake for 30 minutes.

2. Transfer to a Buchner funnel and wash with an excess of 150 to 200 ml of ammonium acetate. Potassium, calcium, magnesium and sodium can be run on this extract by making filtrate up to a volume of 250 mls. Filtrate is run on the flame photometer using standards from IV.
3. Wash out the excess ammonium acetate with 150 to 200 ml ethyl alcohol and discard filtrate.
4. Change to a clean filter flask and wash the soil with 250 ml 0.1 N HCl to replace the ammonia.
5. Transfer the filtrate from steps 4 to an 800 ml Kjeldahl flask, add 10 grams NaCl, 10 mls concentrated NaOH, and an anti-bumping disc.
6. Attach immediately to the condenser and distill approximately one-third of the solution over into a previously placed 500 ml Erlenmeyer flask containing 50 ml saturated boric acid solution and 1 ml mixed indicator.
7. Lower the Erlenmeyer flask to prevent back-suction of the material and turn off burners.
8. Titrate the NH_3 with 0.1000N H_2SO_4 .

C. Calculation

1. $\text{ml H}_2\text{SO}_4 \times N \times \frac{100}{10} = \text{C.E.C. in m.e./100 grams of soil}$

VI. TOTAL SALTS (Conductivity) (10)

A. Procedure - 1:1 soil-water extract

1. Weigh 20 grams of soil into a 50 ml shaking bottle.
2. Add 20 ml distilled water and shake for 30 minutes. Filter with suction.
3. Determine the conductivity with a Solu-Bridge.

B. Procedure - saturation extract

1. Place 50 to 100 grams of soil in a 250 ml beaker, add distilled water and stir thoroughly until the soil is completely moist. Let the soil set for at least an hour. If free water collects on the surface the water content is too high. If the soil paste is stiff, the water content is too low. At saturation the soil surface should glisten and the soil flow just slightly when the beaker is tipped.
2. Transfer soil into a suction funnel and filter.
3. Determine conductivity with a Solu-Bridge.

This laboratory uses an RD26 Solu-Bridge which reads in millimhos/cm.

VII. ORGANIC MATTER - Walkley-Black method (11)

A. Reagents

1. Potassium dichromate, 1 N - Dissolve 40.04 grams $\text{K}_2\text{Cr}_2\text{O}_7$ in distilled water and make up to 1 liter. If this solution is carefully prepared, it will be exactly 1 N.
2. Ferrous-ammonium-sulfate, 0.4 N - Dissolve 159.6 grams $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ in distilled water containing 40 ml concentrated H_2SO_4 and make up to 1 liter. Determine normality periodically by titrating against the potassium dichromate solution.

3. O-phenanthroline ferrous sulfate complex - 0.025 M solution, "Ferroin".
4. Phosphoric acid - 14.6 M (85%)

B. Procedure

1. Pass soil through a $\frac{1}{2}$ mm sieve and weigh out 0.5 grams soil into a 500 ml Erlenmeyer flask.
2. Add 10 ml potassium dichromate and 20 ml concentrated H_2SO_4 . Mix rapidly and thoroughly for 1 minute. Let stand for at least 20 minutes or until cool.
3. Dilute to 150 ml with water and add 10 ml concentrated H_3PO_4 .
4. Titrate with 0.4 N ferrous-ammonium-sulfate. Use 6 drops O-phenanthroline indicator.
5. Run a blank simultaneously using same procedure.

C. Calculation

1. ml $Fe(NH_4)_2(SO_4)_2$ used (blank - sample titration) $\times \frac{N}{.4} \times .545 = \% \text{ O.M.}$

The factor .545 is derived as follows:

$$(.4 \text{ N}) \times \frac{12}{4000} \times \frac{1.72}{0.76} \times \frac{100}{0.5} = .545$$

in which 12/4000 is the meq. weight of carbon, 1.72 is the factor used based on the assumption that organic matter is 58% carbon, 0.76 is the percent recovery factor, and 0.5 is the weight of the sample.

VIII. TOTAL NITROGEN = Kjeldahl method (1)

A. Reagents

1. Concentrated sulfuric acid, low in N.
2. Concentrated sodium hydroxide (40%) - Add 10 kg sodium hydroxide flakes to 15 liters of water. Add the flakes slowly with constant stirring.
3. Catalyst - Na_2SO_4 , 100 grams; $CuSO_4$, 25 grams; selenium metal, powder 10 grams. Mix well.
4. Zinc, mossy.
5. Boric acid solution, saturated
6. See V. A. 5.
7. Mixed indicator - Dissolve 0.1 gram bromcresol green and 0.02 grams methyl red in ethyl alcohol and make to 100 ml.

B. Procedure

1. Weigh out 10 grams of soil and place in an 800 ml Kjeldahl flask.
2. Add 1 teaspoonful (10 grams) of catalyst and 30 ml concentrated H_2SO_4 .
3. Digest until clear (20 to 30 minutes) + one-half clearing time (10 to 15 minutes.) Cool.
4. Meanwhile place a 500 ml Erlenmeyer flask containing 50 ml boric acid solution and 4 drops of indicator under the condenser. Be sure the condenser tube is below the liquid surface.
5. Add 400 ml distilled water, one boiling stone and a small piece of mossy zinc to the Kjeldahl flask.

6. Pour in slowly and carefully down the side of the flask 60 ml of concentrated NaOH. Do not mix at this point.
7. Connect to condenser; then gently rotate solution until the material is thoroughly mixed.
8. Turn on distillation unit burners and distill over approximately 100 ml. Turn off burners and immediately lower the Erlenmeyer flasks.
9. Titrate NH_3 with 0.1000 N H_2SO_4 using the mixed indicator.
10. Run a blank whenever there is a change in reagents or at least once a day.

C. Calculation

1.
$$\frac{\text{ml H}_2\text{SO}_4 \text{ used (sample - blank titration)} \times N \times .014 \times 100}{\text{weight of material (grams)}} = \% \text{ nitrogen}$$

IX. BORON - Curcumin method (3)

A. Reagents

1. Curcumin - oxalic acid solution - Dissolve 0.04 gram finely ground curcumin in 100 ml 95% ethyl alcohol by warming slightly in warm water. When curcumin is dissolved, add 5 grams oxalic acid, dissolve, cool, and store in refrigerator for at least two days before using. When stored in a refrigerator the reagent should keep for a week or two.
2. Ethyl alcohol - 95%
3. Standard boron stock solution - Dissolve 0.5716 gram of C.P. boric acid in 1000 ml distilled water. This solution contains 0.1 mgm of boron per ml or 100 ppm and serves as the primary base stock solution. From this standard solution of 0.2 to 2.0 ppm B should be made up when necessary.

B. Procedure

1. Weigh 20 grams of soil into a 125 or 250 ml boron-free flask.
2. Add 40 ml distilled water and reflux for 5 minutes. Condensers should be of boron-free glass.
3. Place suspension in 50 ml centrifuge tube and centrifuge for 20 to 30 minutes at 2000 RPM. Help clarify by adding 0.02 gram calcium chloride before centrifuging.
4. If extract is colored add small amount of decolorizing charcoal and filter.
5. Place a 1-ml aliquot of the clear solution in a No.000 Coors evaporating dish.
6. Add 4 ml curcumin-oxalic acid solution and mix thoroughly by rotating the evaporating dish.
7. Evaporate to dryness on a water bath at $55^\circ \pm 3^\circ\text{C}$, and then continue to bake the residue at this temperature for a minimum of 15 minutes to insure complete dryness. Cool.
8. Treat the reaction products with 25 ml of 95% ethyl alcohol.
9. Filter through a Whatman No. 2 (or equivalent) filter paper. Filtering can be done directly into the comparison tubes, if desirable.
10. Read in colorimeter or spectrophotometer at 540 mu.
11. Determine amount of boron from curve prepared by running a series of standard solutions.

X. NITRATE-NITROGEN (4)

A. Reagents

1. Phenoldisulfonic acid - Dissolve 25 grams of pure (white) phenol in 150 ml concentrated H_2SO_4 . Add 75 ml of fuming H_2SO_4 . Mix the solution and heat for 2 hours at $100^\circ C$ in a boiling bath. Store solution in a brown bottle.
2. Ammonium hydroxide solution - Mix equal volumes of concentrated NH_4OH and distilled water.
3. Calcium hydroxide solution - Add excess $Ca(OH)_2$ to 1 liter of distilled water.
4. Standard nitrate solution - Dissolve 0.7221 gram of pure dry KNO_3 in distilled water and dilute to 1 liter. This solution contains 100 ppm N or nitrates and serves as the base stock solution. Standard solution of 0.5 to 10 ppm N should be made up when necessary.

B. Procedure

1. Shake 10 grams of soil with 50 ml of distilled water for 10 minutes. Add 0.2 gram $Ca(OH)_2$ powder and shake for an additional 5 minutes.
2. Filter and wash with an additional amount of water.
3. Transfer 5 ml of the filtrate to a 5-ml beaker. Add 1 ml $(Ca(OH)_2)$ solution and evaporate to dryness on a warm hot plate. Cool.
4. Add 1 ml of phenoldisulfonic acid and rotate the beaker so that the acid comes in contact with all of the residue.
5. Add 14 ml of distilled water and 5 ml of 1:1 ammonium hydroxide solution.
6. Transfer the solution to a colorimeter tube and read in a colorimeter at 420 mu.
7. Determine the amount of nitrate from curve prepared by running a series of standard solutions.

XI. AMMONIUM-NITROGEN (5)

A. Reagents

1. Potassium chloride extracting solution - Dissolve 149 grams KCl in 2 liters of distilled water.
2. Nessler reagent - In a 1000 ml volumetric flask dissolve 91 grams mercuric iodide and 70 grams potassium iodide, using as little water as possible. Add 224 grams KOH, dilute to 1 liter, mix well and cool. Let stand for a few days to allow any precipitate to settle. Decant into a brown glass bottle and store for use.
3. Sodium hydroxide - tartrate solution - Dissolve 80 grams sodium tartrate in about 500 ml of distilled water. Add 26 grams NaOH and after it is dissolved dilute to 1 liter.
4. Gum accacia solution - Dissolve 10 grams powdered gum accacia in 195 ml distilled water then add 5 ml Nessler's reagent. Gum accacia may be dissolved most readily by adding sufficient water to make a paste and then add the balance of water while stirring. Let settle a few days and decant into a brown glass bottle.
5. Copper sulfate solution - Dissolve 25 grams $CuSO_4$ in 1 liter of water.
6. Standard ammonium solution - Dissolve 0.4714 gram pure ammonium sulfate in 1 liter of distilled water. This gives a solution of 100 ppm N as ammonium. From this stock solution standards of 0.1 to 2.5 ppm N as ammonium can be made as needed.

B. Procedure

1. Place 10 to 20 grams of soil in a shaking bottle.
2. Add 100 ml extracting solution and 4 drops of copper sulfate solution.
3. Shake for 30 minutes and filter. Suction filtering can be used to speed up the operation.

Note: Beyond this point time is critical. Add all reagents to each sample before going onto next. Read color in 15 minutes.

4. Place 4 ml of sodium hydroxide-tartrate solution to a 50 ml volumetric flask. Add 20 ml of the filtered extract and mix. Add 1 ml Nessler's reagent and mix well. Then add 1 ml more of Nessler's reagent and again mix. This procedure keeps the solution from becoming turbid or flocculating. To develop the color fully, it may require 2 mls of Nessler's reagent at the last addition. Dilute immediately to the 50 ml mark. Mix the solution well.
5. After 15 minutes from the beginning of step 4, transfer solution to a colorimeter tube and read in a colorimeter at 420 mu.
6. Determine the amount of ammonium from curve prepared by running a series of standards.

XII. CALCIUM CARBONATE EQUIVALENT - for limestone sample (1)

A. Reagents

1. Hydrochloric acid, 1 N - Dilute 860 ml concentrated HCl to 10 liters with distilled water. Mix thoroughly. Standardize against primary Na_2CO_3 .
2. Sodium hydroxide, 1 N - Dissolve 420 grams sodium hydroxide pellets in about 5 liters of distilled water stirring constantly to prevent caking. Cool, then make up to 10 liters with distilled water. Mix thoroughly. Standardize against the standard 1 N HCl.
3. Phenolphthalein indicator - Dissolve 1 gram phenolphthalein in 50 ml ethyl alcohol and add 50 ml distilled water. Place in a dropper bottle.

B. Procedure

1. Weigh out 1 gram of finely ground material into 250 ml Erlenmeyer flask.
2. Add a known amount of 1 N HCl (usually 25 ml). Heat on hot plate until almost boiling. Remove, cool and wash down neck of flask with distilled water.
3. Titrate excess HCl with 1 N NaOH using phenolphthalein as indicator.

C. Calculations

1. 1 ml of 1 N HCl = 0.05 gram CaCO_3
2. $\text{grams CaCO}_3 \times 100 = \% \text{CaCO}_3 \text{ equivalent}$

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