

SAMPLING AND TESTING SOILS (CHEMICAL)

1. SCOPE: This test method is intended for soil that is to be used in preparation of seed beds for various plant growth needed in erosion control, landscaping and other uses.
2. APPARATUS AND MATERIALS: Apparatus and materials required are given in the Procedure.
3. SAMPLE: When set forth in Project Specifications, one (1) 2.3 kg (five pounds) sample per type of soil (as defined by Soil Science Society of America) shall be submitted to the Division of Materials for Chemical Analysis and Texture Classification (U. S. Department of Agriculture Textural Classification Chart). Approval or disapproval will be made by the Agronomy Section of the Division of Construction.
4. PROCEDURE:
 - 4.1. Sample Preparation:
 - 4.1.1. Sample should be air dry.
 - 4.1.2. Soil should be crushed with a mortar and pestle. Avoid crushing any rock or organic matter.
 - 4.1.3. Pass the soil through a No. 10 sieve.
 - 4.1.4. Mix the sieved soil thoroughly.
 - 4.1.5. Save a small jar (120 ml, or 4 ounces) of soil for the testing sample.
 - 4.2. Determination of Soil pH:
 - 4.2.1. Apparatus and Materials:
 - 4.2.1.1. pH Meter
 - 4.2.1.2. Buffer solution
 - 4.2.1.3. 150 ml beaker
 - 4.2.1.4. Distilled water
 - 4.2.2. Ph: Calibrate the glass electrode of the pH meter carefully with a buffer solution.

Weigh 5 grams of the soil sample (analytical balance) and place in a 150 ml beaker. Add 10 ml of distilled water from a graduated cylinder. Swirl the sample and water for 30 seconds, and allow to stand for 30 minutes. Swirl again, insert the electrodes, and read the meter. Record the reading. SAVE THE SAMPLE.

4.3. Determination of Exchangeable Hydrogen:

4.3.1. Apparatus and Materials:

4.3.1.1. The same equipment and reagents used in soil pH determinations (4.2.1.1 - 4) are also used in this determination.

4.3.1.2. In addition to the above, a nitrophenol buffer is used. 8 g paranitrophenol, 40 g $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ and 0.625 g MgO per liter of distilled water. This solution is adjusted to pH 7.0 by either HCl or MgO as required.

4.3.2. Exchangeable Hydrogen: Calibrate the pH meter with the prepared nitrophenol buffer to a pH of 7.0. Add 10 ml of the nitrophenol mixture with a pipette to the soil sample that was used in the pH determination. Swirl for 30 seconds, and allow to stand for 30 minutes. Swirl again, and read the meter. Each change of one tenth in pH on going from pH of 7.0 to pH of 6.0 is equivalent to 1 M.E. of hydrogen per 100 grams of soil. Do not try to run exchangeable hydrogen on an alkaline soil.

4.4. Determination of Phosphorous:

4.4.1. Apparatus and Materials:

4.4.1.1. High phosphate slide

4.4.1.2. Whatman No. 1 Filter Paper, 11 cm

4.4.1.3. 50 ml beaker

4.4.1.4. 50 ml Erlenmeyer Flask

4.4.1.5. 20 ml pipette

4.4.1.6. No. 1, or No. 2 Rubber Stopper

4.4.1.7. 0.5 ml rubber bulb pipette and vial

4.4.1.8. 5 ml comparator tube

4.4.1.9. Graduate pipette

- 4.4.1.10. Distilled water
- 4.4.1.11. Concentrated stannous chloride (preparation given at the end of procedure)
- 4.4.1.12. Molybdate solution (preparation given at the end of procedure)
- 4.4.1.13. 0.15 N H_2SO_4 Extraction solution

4.4.2. Phosphorus:

- 4.4.2.1. Put 5 g of soil sample into a 50 ml Erlenmeyer flask. Add 20 ml of extracting solution (0.15 N H_2SO_4) with a pipette. stopper, and shake for 2 minutes. Let it set for 15 seconds and then pour into a funnel containing Whatman No. 1 filter paper. The filtrate is collected in a 50 ml beaker. To another 50 ml beaker, add by pipette 10 ml of molybdate solution. To the 10 ml of molybdate, add 5 ml of the soil filtrate. Add 2.5 ml of diluted stannous chloride to the solution. Swirl the beaker and pour 5 ml into a comparator tube. Allow three minutes to elapse, and then make a reading on the High Phosphorus Slide. Available phosphorus is determined by multiplying the results of the color comparison (High Phosphate Slide) by 3. This will give you lbs. per acre.
- 4.4.2.2. Molybdate Solution for Phosphorus (1 liter): Weigh 9.0 grams (8.98 - 9.06) of ammonium molybdate (reagent grade), and add to 250 ml distilled water in a 600 ml beaker. It will help to heat the water, but do not exceed 50°C . Add 175 ml of exactly 10 N H_2SO_4 , transfer to a 1000 ml volumetric flask, and cool to at least 30°C before adjusting to final volume with distilled water. Mix thoroughly. Store in an amber bottle, or in the dark when not in use.
- 4.4.2.3. Stannous Chloride - Concentrated Solution: Dissolve 4 grams $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 25 ml concentrated HCl . Keep tightly stoppered (capped) in a small vial.
- 4.4.2.4. Dilute Stannous Chloride: Measure 0.5 ml of concentrated stannous chloride with a 0.5 ml rubber bulb pipette, and dilute to 20 ml with distilled water.

4.5. Determination of Potassium:

4.5.1. Apparatus and Materials

- 4.5.1.1. Potash Test Chart

- 4.5.1.2. 50 ml Erlenmeyer Flask
- 4.5.1.3. No. 1 Whatman filter paper, 9 cm
- 4.5.1.4. Funnel
- 4.5.1.5. Funnel vial marked at 5 ml
- 4.5.1.6. 10 ml pipette
- 4.5.1.7. Graduated pipette
- 4.5.1.8. No. 1 or No. 2 rubber stopper
- 4.5.1.9. Potash Reagent "A" (preparation given at end of procedure)
- 4.5.1.10. Potash Reagent "B" (preparation given at end of procedure)
- 4.5.2. Potassium:
 - 4.5.2.1. Place a 5 grams of soil sample into a 50 ml Erlenmeyer flask. Fold a filter paper (Whatman No. 1) and place in funnel. Add 10 ml of potash reagent "A" to the sample by pipette. Stopper and shake for 1 minute. Let the sample plus solution set for 30 seconds. Pour into the prepared filter. Obtain 5 ml of filtrate in the funnel vial. Add 2.5 ml of potash reagent "B" with a graduated pipette to the solution in the funnel vial. Shake the vial for 4 - 5 seconds. Record the exact 3-minute time interval, and then compare with the Potash Test Chart. Compare the visibility of the three black lines rather than the shades of yellow in the turbid mixture. The chart is read in pounds per acre.
 - 4.5.2.2. Preparation of Potash Reagent "A": Dissolve 5 grams of sodium cobaltinitrite, and 30 grams sodium nitrate, A. C. S. grade (Mallinckrodt's 7824 is good) in 70 ml distilled water in a 250 ml beaker, add 4 - 7 ml glacial acetic acid (A.C.S.), stir occasionally until solution turns dark (and reagents are apparently dissolved) and transfer all to a 100 ml volumetric flask with the aid of a small funnel and wash bottle. Make to 100 ml volume, mix somewhat by rolling the flask (do not invert) and let stand for 3 days with a small beaker inverted over the opening of the flask. Add the cobaltinitrite mixture to a container which will hold at least 2500 ml. Weigh out 300 grams of the sodium nitrate and by rinsing out the 100 ml volumetric flask and adding other distilled water, measure out a total of 2000 ml of distilled water with which to dissolve the sodium nitrate and add

the nitrate and water mixture to the 2500 ml container. After thorough mixing the pH should be checked and adjusted with a pH meter. Preferably, it should be pH $5.0 + 0.05$ but 5.0 ± 0.1 is acceptable. Keep this solution in good quality glass, or plastic, preferably in the dark with a minimum of air exposure.

- 4.5.2.3. Potash Reagent "B": This is isopropyl alcohol A. C. S. grade (Spec. Gravity at $25^{\circ}\text{C} = 7832$).

4.6. Determination of Calcium Magnesium:

- 4.6.1. Use the same procedure that is used for a complete limestone analysis. However, use a one gram sample in the soil analysis. Also, the conversion factors are different. They are given below.

4.6.2. Calculations:

- 4.6.2.1. pounds per acre of Calcium = $\text{Wt. of CaO} \times .71469 \times 2,000,000$
- 4.6.2.2. pounds per acre of Magnesium = $\text{Wt. of Mg}_2\text{P}_2\text{O}_7 \times .21847 \times 2,000,000$

4.7. Determination of Organic Matter:

4.7.1. Apparatus and Materials:

- 4.7.1.1. 425 μm (# 40) Sieve
- 4.7.1.2. 1 N Potassium dichromate
- 4.7.1.3. Concentrated sulfuric acid
- 4.7.1.4. Orthophenanthroline ferrous complex indicator
- 4.7.1.5. 0.35N Ferrous ammonium sulfate
- 4.7.1.6. Mortar and pestle

4.7.2. Percent Carbon:

- 4.7.2.1. This procedure consists of treating a soil with an oxident, and determining the oxident which was not consumed in oxidizing the soil. The procedure is the Walkley-Black, or Heat of Dilution variation of the Chromic Acid Oxidation Method.
- 4.7.2.2. The soil should be fine enough to pass a 425 μm (40) mesh sieve

(.420 mm). A mortar and pestle may be used to pulverize a sample.

- 4.7.2.3. Weigh to the nearest 0.01 gram, 1 - 3 grams of soil. Use approximately 1 gram of soils high in organic material and approximately 3 grams of soils low in organic material. Place the soil in a 250 - 500 ml Erlenmeyer flask. Add 15 ml of potassium dichromate from a burette, quickly followed by adding 30 ml of concentrated H_2SO_4 . Immediately swirl vigorously for 1 minute. Place on an asbestos sheet, or wire gauze, and let stand for 20 minutes. Slowly add 100 ml of distilled water, and swirl gently to mix. The solution will be warm. Cool the outside of the flask under a cold water tap until approximately room temperature is reached. Add 5 drops of orthophenanthroline ferrous complex indicator. Titrate the excess chromate with 0.35 N (actual normality may be different) ferrous ammonium sulfate. As the titration proceeds, the solution will turn green, then bluish-green just before the end point, and a murky red at the end point. Record all burette readings so the proper data for calculations are available.
- 4.7.2.4. Fifteen ml of potassium dichromate should be carried through the entire procedure described except that no soil shall be included. This is known as "blank" titration.
- 4.7.2.5. Calculations: The amount of ferrous ammonium sulfate required to reduce the excess chromate (which remains after the organic carbon in the soil sample is oxidized) subtracted from the amount of ferrous ammonium sulfate used in the blank titration will be chemically equivalent to the amount of chromate that reacted with (or oxidized) the organic carbon in the soil. This chromate is directly equivalent to the oxidized carbon.

$$\text{Percent of Carbon} = \frac{0.003 (X-Y) (Z) \times 100}{0.74 S}$$

Where:

X = mL blank titration

Y = mL sample titration

Z = N of ferrous ammonium sulfate

S = Weight of sample

Using the value, calculate the percent of organic matter.

Percent Organic Matter = $1.724 \times (\text{Percent of Carbon})$

5. CALCULATIONS: Calculations are covered in the Procedure.
6. PRECAUTIONS: Precautions are covered in the Procedure.

7. REPORT:

- 7.1. Soil Ph
- 7.2. Exchangeable Hydrogen
- 7.3. lbs./acre Phosphorus
- 7.4. lbs./acre Potassium
- 7.5. lbs./acre Calcium
- 7.6. lbs./acre Magnesium
- 7.7. % Organic Matter

APPROVED

DIRECTOR
DIVISION OF MATERIALS

DATE

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