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1 Appendix 1: METHOD BASED ON ORGANIC CARBON CONTENT OF SOIL

1.1. SCOPE

The method for determining the percentage by weight of the organic matter present in soils based on the determination of the organic carbon content of the soil.

1.2. APPARATUS

Oven - thermostatically controlled to maintain the temperature between 105° and 110°C, with the interior of non-corroding material.

Chemical Balance - sensitive to 0'001 g.

Volumetric Flask - two, of one-liter capacity.

Burettes - two, of 25 ml, graduated in 0.1 ml,

Pipettes - 10-ml pipette and 1-ml pipette fitted with a rubber teat.

Conical Flasks - two, of 500-ml capacity.

Graduated Measuring Cylinders - 200-ml and 25-ml.

Desiccator -- with any desiccating agent other than sulphuric acid.

Glass Weighing Bottle - approximately 25 mm in diameter and of 50 mm height fitted with a ground glass stopper.

Sieves - 10-mm IS Sieve and 425-micron IS Sieve and receivers.

Wash Bottle

1.3. REAGENTS

The reagents used shall be of analytical quality.

Potassium Dichromate Normal Solution - Dissolve 49.035 g of potassium dichromate in distilled water to make one liter of solution.

Ferrous Sulphate, Approximately 0'5 N Solution - Dissolve approximately 140 g of ferrous sulfate in 0.5 N sulphuric acid to make one liter of solution (add 14 ml of concentrated sulphuric acid to distilled water to make one liter of solution for 0.5 N sulphuric acid).

Sulphuric Acid, Concentrated - of specific gravity 1.836 .

Orthophosphoric Acid, 85 Percent - of specific gravity 1.70 to 1.75.

Indicator Solution – 0.25g of sodium diphenylamine-sulphonate dissolved in 100 ml of distilled water.

1.4. STANDARDIZATION OF FERROUS SULPHATE SOLUTION

Ten milliliters of the standard potassium dichromate solution shall be run from a burette into a 500 ml conical flask. 20 ml of concentrated sulphuric acid shall then be added carefully, and the mixture swirled and allowed to cool for some minutes. 200 ml of distilled water shall then be added to the mixture followed by 10 ml of phosphoric acid and 1 ml of the indicator, and the mixture shall be shaken thoroughly. Ferrous sulfate solution shall then be added from the second burette in 0.5 ml increments, the contents of the flash being swirled until the color of the solution changes from blue to green. A further 0.5 ml of potassium dichromate shall then be added changing the color back to blue. Ferrous sulfate solution shall then be added drop by drop with continued swirling until the color of the solution changes from blue to green after the addition of a single drop. The total volume of ferrous sulfate solution used (X) shall be noted to the nearest 0.05 ml (1 ml ferrous sulfate solution is equivalent to 10.5/X ml potassium dichromate).

1.5. SOIL SAMPLE FOR TEST

The soil sample as received from the field shall be prepared. The portion of the air-dried sample selected for the purpose of this test shall be weighed, its moisture content determined using a separate sample for the purpose, and the equivalent oven-dried weight (W_1) recorded. It shall then be sieved on a 10-mm IS Sieve and all particles other than stones crushed to pass through the sieve. The equivalent weight on the oven-dry basis of the material passing 10-mm IS Sieve (W_2) shall be calculated and recorded to the nearest 0 percent of its total weight. A sample weighing approximately 100 g shall be obtained from the material passing the 10 mm IS Sieve by quartering. This sample shall then be pulverized so that it passes the 425-micron IS Sieve.

NOTE 1 - This -method gives high results of organic content in soils containing sulfide, The sulfide can be destroyed at this stage by the addition of dilute (2 N) Sulphuric acid. Acid shall be added until no further evolution of hydrogen sulfide occurs.

NOTE 2 - This method gives high results of organic content in soils containing chlorides. The chlorides may be removed at this stage by washing the soil with distilled water until no turbidity is obtained when a drop of the washing water is tested with silver nitrate solution. Alternatively, the effect 0 chlorides on the determination can be partly eliminated by using concentrated sulphuric acid in which silver sulfate has been dissolved in place of the concentrated Sulphuric acid. If the ratio of carbon to chloride does not exceed unity, 25 g of silver sulfate per liter of sulphuric acid will be sufficient to precipitate the chloride.

A 5-gram soil sample shall be taken from the thoroughly mixed portion of the material passing the 425 microns IS Sieve and used for the test.

1.6. PROCEDURE

The sample shall be placed in a glass weighing bottle and weighing to 0•001 g. A small quantity, from 5 g to 0.2 g depending on the organic content (see« Note) shall be transferred to a dry 500-ml conical flask, the weighing bottle reweighed and the equivalent weight on oven-dry basis of soil specimen removed (W_s) calculated by difference and allowing for the moisture content of the soil.

NOTE - The size of the specimen for chemical analysis will vary with the amount of organic matter present in the soil. As much as 5 gm. may be required for soil low in organic matter and as little as 0.2 g with very peaty soil. After a number of determinations have been made, the experience will indicate the most suitable size of the specimen to be taken. In unfamiliar types of soil, it is suggested that a series of specimens of varying sizes should be weighed out and tested. The determination giving a total of 5 to 8 ml dichromate solution reduced should be taken al the correct result.

Ten milliliters of N potassium dichromate solution shall be run into the conical flask from a burette, and add 20 ml of concentrated sulphuric acid very carefully from a measuring cylinder. The mixture shall be thoroughly swirled for about one minute and allowed to stand on a heat insulating surface, such as asbestos, or wood, for 30 min to allow oxidation of the organic matter to proceed. During this period the flask shall be protected from draughts. Distilled water, 200 ml, shall then be added along with 10 ml of orthophosphoric acid and one ml of the indicator (see Note). The mixture shall be shaken vigorously. If the the indicator is absorbed by the soil, a further 1 ml of the solution shall be added. Ferrous sulfate solution shall then be added from the second burette in 0•5 ml increments, the contents of the flask being swirled until the color of the solution changes from blue to green. A further 0•5 ml of• potassium dichromate shall then be added, changing the color of the solution back to blue. Ferrous sulfate solution shall then be added drop by drop with continued swirling until the color of the solution changes from blue to green after the addition of a single drop. The total volume of the ferrous sulfate solution used (γ) shall be noted to the nearest 0.05 ml.

Note - If complex ferric ions which interfere with the endpoint are present in the soil, after the addition of 10 ml of orthophosphoric acid. 0.2 g of sodium fluoride may be added before the addition of the indicator.

1.7. CALCULATION

The total volume (V ml) of potassium dichromate used to oxidize the organic matter in the soil is given by the following formula:

$$V = 10.5\,(1 - r/X)$$

where

r = total volume of ferrous sulfate used in this test, and

X = total volume of ferrous sulfate used in the standardization test

The percentage of organic matter (OM) present in the oven-dried the sample shall be calculated from the following formula:

$$OM, percent\ by\ weight\ = \frac{0.67w2V}{WI\,W3},$$

where

W2= weight on the oven-dry basis of the soil sample passing 10 mm IS Sieve,

V= total volume of potassium dichromate used to oxidize the organic matter,

WI= weight on the oven-dry basis of the total soil sample taken for the test before sieving, and

W3 = weight on the oven-dry basis of the soil specimen used in the test.

NOTE- The method is based on the determination of the organic carbon content of the soil, and it assumes that soil organic matter contains an average of 50 percent of carbon by weight. With the technique employed approximately 77 percent of the carbon in the organic material is oxidized. These factors are included in the formula.