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METHODS OF TEST FOR SOILS

PART XXII DETERMINATION OF ORGANIC MATTER

(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHODS OF TEST FOR SOILS

PART XXII DETERMINATION OF ORGANIC MATTER

(First Revision)

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Indian Standard

METHODS OF TEST FOR SOILS

PART XXII DETERMINATION OF ORGANIC MATTER

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part XXII) (First Revision) was adopted by the Indian Standards Institution on 25 February 1972, after the draft finalized by the Soil Engineering Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 With a view to establish uniform procedures for the determination of different characteristics of soils and also for facilitating comparative studies of the results, the Indian Standards Institution is bringing out this ' Indian Standard Methods of Test for Soils ' (IS : 2720) which is being published in parts. Thirtythree parts of this standard have been published so far. This part deals with the method of test for the determination of organic matter content in the soil. Organic matter is an undesirable constituent of the soil from the engineering point of view since it causes swelling or shrinkage of the soil when the moisture content or the applied load changes. The estimation of organic matter, therefore, forms an important part of soil examination.

0.3 A wide variety of both dry and wet combustion methods are in use for the determination of the organic matter of soils. Two methods which are widely in use are given in this revision of the standard. These methods give reproducible results and the results are sufficiently accurate for common engineering purposes. The first method is given as the standard method and the other as an alternative method.

0.4 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (revised).

SECTION I METHOD BASED ON ORGANIC CARBON CONTENT OF SOIL

1. SCOPE

1.1 Section 1 of this standard (Part XXII) lays down 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.

2. APPARATUS

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

2.2 Chemical Balance — sensitive to 0.001 g.

2.3 Volumetric Flask — two, of one-litre capacity.

2.4 Burettes — two, of 25 ml, graduated in 0.1 ml.

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

2.6 Conical Flasks — two, of 500-ml capacity.

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

2.8 Desiccator — with any desiccating agent other than sulphuric acid.

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

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

2.11 Wash Bottle

3. REAGENTS

3.1 The reagents used shall be of analytical quality.

3.1.1 Potassium Dichromate Normal Solution — Dissolve 49.035 g of potassium dichromate in distilled water to make one litre of solution.

3.1.2 Ferrous Sulphate, Approximately 0.5 N Solution — Dissolve approximately 140 g of ferrous sulphate in 0.5 N sulphuric acid to make one litre of solution (add 14 ml of concentrated sulphuric acid to distilled water to make one litre of solution for 0.5 N sulphuric acid).

NOTE — This solution is unstable in air and should be kept tightly stoppered. It should be standardized against the potassium dichromate solution at least once in a week.

3.1.3 Sulphuric Acid, Concentrated — of specific gravity 1.836 (see IS : 266-1961*).

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

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

4. STANDARDIZATION OF FERROUS SULPHATE SOLUTION

4.1 Ten millilitres of the normal potassium dichromate solution shall be run from a burette into a 500-ml conical flask. Twenty millilitres of concentrated sulphuric acid shall then be added carefully and the mixture swirled and allowed to cool for some minutes. Two hundred millilitres 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 sulphate solution shall then be added from the second burette in 0.5 ml increments, the contents of the flask being swirled, until the colour of the solution changes from blue to green. A further 0.5 ml of potassium dichromate shall then be added changing the colour back to blue. Ferrous sulphate solution shall then be added drop by drop with continued swirling until the colour of the solution changes from blue to green after the addition of a single drop. The total volume of ferrous sulphate solution used (X) shall be noted to the nearest 0.05 ml (1 ml ferrous sulphate solution is equivalent to $10.5/X$ ml potassium dichromate).

5. SOIL SAMPLE FOR TEST

5.1 The soil sample as received from the field shall be prepared in accordance with IS : 2720 (Part I)-1972†. The portion of the air dried sample selected for the purpose of this test shall be weighed, its moisture content determined using 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 oven-dry basis of the material passing 10-mm IS Sieve (W_2) shall be calculated and recorded to the nearest 0.1 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 (see Notes 1 and 2). 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 sulphides. The sulphides can be destroyed at this stage by the addition of dilute (2 N)

*Specification for sulphuric acid.

†Methods of test for soils: Part I Preparation of dry soil specimen for various tests (first revision).

sulphuric acid. Acid shall be added until no further evolution of hydrogen sulphide 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 of chlorides on the determination can be partly eliminated by using concentrated sulphuric acid in which silver sulphate has been dissolved in place of the concentrated sulphuric acid specified in 3.1.3. If the ratio of carbon to chloride does not exceed unity, 25 g of silver sulphate per litre of sulphuric acid will be sufficient to precipitate the chloride.

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

6. PROCEDURE

6.1 The sample shall be placed in a glass weighing bottle and weighed 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_3) 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 g may be required for soil low in organic matter and as little as 0.2 g with a very peaty soil. After a number of determinations have been made, experience will indicate the most suitable size of 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 as the correct result.

6.2 Ten millilitres 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 indicator is absorbed by the soil, a further 1 ml of the solution shall be added. Ferrous sulphate solution shall then be added from the second burette in 0.5 ml increments, the contents of the flask being swirled, until the colour of the solution changes from blue to green. A further 0.5 ml of potassium dichromate shall then be added, changing the colour of the solution back to blue. Ferrous sulphate solution shall then be added drop by drop with continued swirling until the colour of the solution changes from blue

to green after the addition of a single drop. The total volume of the ferrous sulphate solution used (Y) shall be noted to the nearest 0.05 ml.

NOTE — If complex ferric ions which interfere with the end point 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.

7. CALCULATIONS

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

$$V = 10.5 (1 - Y/X)$$

where

Y = total volume of ferrous sulphate used in this test, and
 X = total volume of ferrous sulphate used in the standardization test (see 4.1).

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

$$\text{OM, percent by weight} = \frac{0.67 W_2 V}{W_1 W_3} \text{ (see Note)}$$

where

W_2 = weight on oven-dry basis of the soil sample passing 10-mm IS Sieve,
 V = total volume of potassium dichromate used to oxidize the organic matter (as in 7.1),
 W_1 = weight on oven-dry basis of the total soil sample taken for the test before sieving, and
 W_3 = weight on 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 given in 7.2.

8. REPORT

8.1 The test results should be recorded suitably. A recommended *pro forma* for recording the results is given in Appendix A.

8.2 The organic matter content present shall be reported to the nearest 0.1 percent of the original oven-dry soil.

**SECTION 2 DETERMINATION OF ORGANIC MATTER
(ALTERNATIVE METHOD) BY THE CHROMIC
OXIDATION EQUIVALENT OF SOIL ORGANIC MATTER**

9. SCOPE

9.1 Section 2 of this standard (Part XXII) lays down the method for determining the percentage by weight of the organic matter present in soils, by the chromic oxidation equivalent of soil organic matter.

9.2 Determination of organic matter of soil by chromic acid method is the most rapid and popular type of analysis and has the advantage of moderately satisfactory discrimination of humus from highly condensed forms including graphite and charcoal.

10. APPARATUS

10.1 Electric Hot Plate — with adjustable temperature.

10.2 Beakers — 250-ml and 400-ml.

10.3 Heat Resistant Glass Test Tubes — 30 × 200 mm.

10.4 Heat Resistant Glass Beaker (Bath) — of 2-litre capacity.

10.5 Thermometer — one, 0 to 250°C range.

10.6 A One-Litre Shallow Heat Resistant Glass Tray (Bath)

10.7 Torsion Balance — sensitive to 0.05 g.

10.8 Capillary Tube, Stirrer and Compressed Air Supply

10.9 Stirring Rod — with ends flattened.

10.10 Volumetric Pipettes

10.11 Burette — 50-ml.

11. REAGENTS

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

11.2 Chromic Acid, 0.4 N Solution — Exactly 19.61 g of potassium dichromate (oven dry) is dissolved in about 50 ml of water and then the solution is diluted to one litre with concentrated sulphuric acid.

11.3 Ferrous Ammonium Sulphate 0.2 N Solution — Exactly 78.44 g of ferrous ammonium sulphate is dissolved in 300 ml of water containing 20 ml of concentrated sulphuric acid, and the solution is diluted to 1 litre with water. This solution is made freshly, or titrated against the standard chromic acid each day.

11.4 Orthophenanthroline Indicator 0.025 M Solution

12. SOIL SAMPLE FOR TEST

12.1 The soil sample shall be ground to pass a 212-micron IS Sieve and 0.25 g of soil (0.05 g if peat, 1.00 g if soil has less than one percent organic

matter) shall be taken in a 250-ml beaker or 30 × 200 mm test tube (see also 5.1).

13. PROCEDURE

13.1 Oxidation of Organic Matter — From a pipette, 20-ml of 0.4 N chromic acid solution shall be added to the soil sample in the 250-ml beaker or test tube, and similar quantity shall be taken for the standardization blank. The vessel with mixture shall be placed in the orthophosphoric acid bath and heated on the electric hot plate at such a rate that a temperature of 155°C is reached in 20 to 25 min. The contents of tube or beaker shall be mixed every 5 min during the heating period. The temperature is held at 155 to 160°C for an additional 5 min. The thermometer shall be kept in the blank, which is simultaneously heated, to follow the solution temperature.

13.2 The vessels with samples and blank shall be then removed from the bath, allowed to drain in the air for 30 sec, and then placed in a water bath at room temperature for 2 min. The thermometer shall be removed with care not to break it by thermal shock.

13.3 Back Titration — The chromic acid solution, now cooled to room temperature, shall be diluted with water to 75 to 200 ml, either in the tube or 250-ml beaker. Then 5 ml of 85 percent orthophosphoric acid and 4 drops of orthophenanthroline indicator shall be added. The solution shall be back titrated with the 0.2 N ferrous ammonium sulphate until solution colour turns from green to red at the end point. An air jet stirrer should be used with the tube. The colour at start is dark brownish, and then shifts sharply from blue to red at the end point. The blank shall be similarly titrated. More chromic acid shall be added to fresh samples if the amount added proves to be inadequate; not over one-half of the chromic acid should be consumed by oxidation of organic matter.

14. CALCULATION OF RESULTS

14.1 The percentage of organic matter in a soil shall be estimated as follows:

$$\text{Organic matter, percent (in soil)} = 20 \left(1 - \frac{T}{S} \right) \times \frac{0.23}{x} \quad (\text{see Note})$$

where

T = sample titration, ml of approximately 0.2 N ferrous solution;

S = standardization blank titration, ml of approximately 0.2 N ferrous solution; and

x = weight of soil sample taken, in g.

NOTE — In deriving the constant 0.23, it is assumed a 4 valance change of carbon occurs, 58 percent carbon occurs in soil organic matter, only carbon is oxidized, and only 90 percent of total soil organic matter is oxidized.

14.2 The organic matter content present shall be reported to the nearest 0.1 percent of the original oven-dry soil.

APPENDIX A

(Clause 8.1)

PRO FORMA FOR RECORDING TEST RESULTS

Determination of Organic Matter Content of Soil

Project..... Details of soil samples.....

Sample number				
Total weight of original sample (W_1), in g				
Weight of soil passing 10-mm IS Sieve (W_2), in g				
Weighing bottle number				
Weight of weighing bottle and dry soil before taking specimen for test, in g				
Weight of weighing bottle and dry soil after taking specimen for test, in g				
Weight of dry soil specimen used (W_3), in g				
Volume of ferrous sulphate solution added to standardize potassium dichromate solution (X), in ml				
Volume of ferrous sulphate solution used to oxidize excess potassium dichromate solution (T), in ml				
Volume of potassium dichromate solution used to oxidize or- ganic matter in soil $V = 10.5$ ($1 - T/X$), in ml				
Percentage of organic matter in soil = $\frac{.6 W_2 V}{W_1 W_3}$				
Remarks				

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 331 01 31, 331 13 75

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TO

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PART XXII DETERMINATION OF ORGANIC MATTER

(First Revision)

Alterations

(Page 5, clause 3.1.3, line 2) - Substitute
'IS:266-1977*' for 'IS:266-1961*'.

(Page 5, clause 5.1, line 2) - Substitute
'IS:2720(Part I)-1983†' for 'IS:2720(Part I)-1972†'.

(Page 5, foot-notes with '' and '+' marks)* -
Substitute the following for the existing foot-notes:

'*Specification for sulphuric acid (*first revision*).

'†Methods of test for soils: Part I Preparation of
dry soil specimen for various tests (*second revision*).'

(BDC 23)