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METHODS OF TEST FOR SOILS
PART XXVII DETERMINATION OF TOTAL SOLUBLE SULPHATES

(First Revision)

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Indian Standard

METHODS OF TEST FOR SOILS

PART XXVII DETERMINATION OF TOTAL SOLUBLE SULPHATES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part XXVII) (First Revision) was adopted by the Indian Standards Institution on 30 December 1977, 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. This part [IS : 2720 (Part XXVII)-1977] deals with the method for the determination of total soluble sulphates in soils. Sodium sulphate is present in some Indian soils. The salt is easily hydrated and dehydrated under the influence of climatic changes. There are enormous volume changes during this process of hydration and dehydration, which influence the engineering properties of soils. Both soluble sulphate content and moisture content of soil are subject to seasonal fluctuations and are mutually interdependent.

0.2.1 This standard was first published in 1968. In this revision a colorimetric or turbidimetric method, which is a rapid method has been added.

0.3 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. This has been met by basing the standard on the following publication:

INDIA. MINISTRY OF IRRIGATION & POWER. Central Board of Irrigation & Power, Publication No. 42. Standards for testing soils. 1963. Central Board of Irrigation & Power, New Delhi.

0.4 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*.

1. SCOPE

1.1 This standard (Part XXVII) lays down the procedure for determining the total soluble sulphate content of soils by: (a) precipitation method, (b) volumetric method, and (c) colorimetric or turbidimetric method.

2. PRECIPITATION METHOD (STANDARD METHOD)

2.1 Apparatus

2.1.1 *Analytical Balance* — sensitive to 0·001 g.

2.1.2 *Glass Beaker* — of 250 ml capacity.

2.1.3 *Glass Funnel* — 50 mm diameter.

2.1.4 *Glass Bottle* — of 250 ml capacity with a rubber cork.

2.1.5 *Crucible* — of 50 ml capacity.

2.1.6 *Heating Equipment*

2.1.7 *Pipette* — 25 ml.

2.1.8 *Burrette* — 50 ml with 0·1 ml graduation.

2.1.9 *A Mortar with Rubber Covered Pestle*

2.1.10 *Filter Paper* — Whatman No. 42 or equivalent.

2.1.11 *Muffle Furnace*

2.1.12 *Mechanical Shaker*

2.1.13 *Drying Oven*

2.2 Reagents

2.2.1 *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit and dilute with distilled water to 100 ml.

2.2.2 *Concentrated Hydrochloric Acid* — specific gravity 1·18 (conforming to IS : 265-1962†).

2.2.3 *Barium Chloride Solution* — 5 percent. Dissolve 5 g of barium chloride in 100 ml of distilled water.

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*Rules for rounding off numerical values (*revised*).

†Specification for hydrochloric acid (*revised*).

2.2.4 *Silver Nitrate Indicator Solution* — 0·5 percent. Dissolve 500 mg of silver nitrate in 100 ml of distilled water.

Note — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests. 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

2.3 Soil Sample — The soil sample shall be brought to a state in which it may be crumbled, if necessary, by drying it in an oven maintained at 105 to 110°C. The aggregations of particles shall be broken up in mortar with rubber covered pestle or the mechanical device. The sample shall be thoroughly mixed and then sub-divided by quartering.

2.4 Principle of the Method — The method depends upon preparing an aqueous extract of the soil and determining the sulphate content of this extract or an aliquot portion of it by the precipitation of sulphate as barium sulphate, filtering off the precipitate and weighing it.

2.5 Procedure — Take 10 g of soil from the sample prepared as given in **2.3**, in a 250-ml bottle with 100 ml of distilled water. Give occasional shaking for 2 h by means of the mechanical shaker. Allow the soil suspension to stand overnight (in case the soil is in a dispersed condition, add 0·5 to 1·0 g of pure potassium nitrate to flocculate the particles). Filter and take 25 ml of filtrate in a beaker and add concentrated hydrochloric acid to just neutralize the solution if it is found alkaline to phenolphthalein indicator. Add further 4 ml concentrated hydrochloric acid to make the solution acidic. Boil the solution. Remove the solution from the source of heat and add hot barium chloride solution in a fine stream with constant stirring, till there is no precipitation with a further addition. Place the beaker on a steam-bath for a minimum period of 4 h and allow the precipitate to settle. Filter the precipitate through ashless filter paper, wash free from chloride ions (*see* Note), dry and ignite. The filteration may also be done through a preweighed sintered glass crucible or a Gooch crucible. In the case of filter paper, after drying, ashing shall be done on a low flame and the precipitate then ignited over a burner or in a muffle furnace at 600 to 700°C for half an hour. Cool in a desiccator, weigh and note the weight of the residue. This is the weight of barium sulphate. A corresponding weight of sodium sulphate should be calculated and thus its percentage determined (*see* **2.6**).

Note — To check whether the residue is free of chloride ions, collect the washings in a separate test-tube at different time intervals and add a drop of 0·5 percent silver nitrate solution to it. The formation of white cloudy precipitate shows the presence of chloride ions in the precipitate. Continue washing until the white precipitate is not formed in the washings by the addition of silver nitrate solution. Hot water may be used for washing.

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*Specification for water for general laboratory use (*second revision*).

2.6 Calculations

2.6.1 Calculate as follows:

a) Sulphates (as SO_4), percent by mass $= 41{\cdot}15 \dfrac{W_1}{W_2}$

b) Sulphates (as $Na_2\ SO_4$), percent by mass $= 60{\cdot}85 \dfrac{W_1}{W_2}$

where

W_1 = mass in g of the precipitate, and

W_2 = mass in g of the soil contained in the solution taken for precipitation.

3. VOLUMETRIC METHOD (SUBSIDIARY METHOD)

3.1 Principle — The volumetric method depends upon insoluble barium sulphate forming and settling rapidly when barium chloride solution is added to the sulphate solution. The barium chloride reagent is added in excess and the excess is determined by the standard solution of barium chromate. With the formation of potassium chromate, the slight excess of chromate reagent becomes evident from the resultant yellow colour of the supernatent solution. The end point can be further tested (confirmed) by silver nitrate solution used as an external indicator. A brick red colouration is obtained when a drop of silver nitrate is added to a drop of the chromate solution.

3.2 Apparatus

3.2.1 *Analytical Balance* — sensitive to 0·001 g.

3.2.2 *Glass Beakers* — two, of 150 ml capacity.

3.2.3 *Glass Funnel* — 6 cm diameter.

3.2.4 *Measuring Flasks* — one of 100 ml and two of 500 ml capacity.

3.2.5 *Burette* — two, of 25 ml, 1/20 ml graduation.

3.2.6 *Conical Flasks* — six, of 150 ml capacity.

3.2.7 *Pipette* — 10 ml.

3.2.8 *Filter Papers*

3.2.9 *Heating Equipment*

3.2.10 *Drying Oven*

3.3 Reagents

3.3.1 *Barium Chloride Solution* — N/4. Dissolve 30·54 g of barium chloride in one litre of distilled water.

3.3.2 *Potassium Chromate Solution* — N/4. Dissolve 24·275 g of potassium chromate in a small amount of distilled water. Add a few drops of silver nitrate solution to it to remove any chloride, filter and dilute to 250 ml.

3.3.3 *Silver Nitrate Indicator Solution* — 0·5 percent. Dissolve 500 mg of silver nitrate in 100 ml of distilled water.

3.3.4 *Dilute Solution of Ammonium Hydroxide* — (sp gr 0·888). Mix ammonium hydroxide and distilled water in the ratio of 1 : 2 (one part of ammonia and two parts of distilled water).

3.3.5 *Concentrated Hydrochloric Acid* — sp gr 1·11 (conforming to IS : 265-1962*).

NOTE — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be used in tests. ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

3.4 Procedure — Weigh 10 g of the soil specimen obtained by the method specified in **2.3**, in a beaker and add about 50 ml water. Stir well, allow to decant, filter, wash the soil on filter paper with a small quantity of water and make the filtrate to 100 ml. Pipette out 10 ml of the water extract in a conical flask, make it slightly acidic by adding concentrated hydrochloric acid and heat to boiling. While boiling, add barium chloride solution (N/4) from the burette till the precipitation is complete and barium chloride solution is in slight excess.

3.4.1 Neutralize the solution with ammonium hydroxide and titrate the excess of barium chloride against potassium chromate solution (N/4). The end point may be confirmed if considered necessary, by using silver nitrate solution as an external indicator (*see* **3.1**).

3.5 Calculation — Calculate as follows:

Sulphates as sodium sulphate in soil,
percent by mass $= 0.0177 \times 100\,(x - y)$

where

x = volume of N/4 barium chloride added, ml;

y = volume of N/4 potassium chromate solution used in back titration; and

$x - y$ = N/4 barium chloride actually used for precipitating sulphate.

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*Specification for hydrochloric acid (*revised*).

†Specification for water for general laboratory use (*second revision*).

4. COLORIMETRIC OR TURBIDIMETRIC METHOD (SUBSIDIARY METHOD)

4.1 Apparatus

4.1.1 *Conical Flask* — 250 ml.

4.1.2 *Volumetric Flask* — 25 ml.

4.1.3 *Analytical Balance* — sensitivity 0·001 g.

4.1.4 *Photoelectric Colorimeter or Turbidimeter*

4.1.5 *Filter Paper* — Whatman No. 42 or equivalent.

4.2 Reagents

4.2.1 *Morgan's Extraction Solution* — 100 g of sodium acetate and 30 ml of 99·5 percent acetic acid dissolved and mixed in 500 ml of water and the volume made to 1 litre.

4.2.2 *Barium Chloride Crystals*

4.2.3 25 *Percent Gum Acatia*

4.3 Procedure — Weigh 20 g air-dry soil specimen in a 250-ml conical flask. Add 100 ml of Morgan's extraction solution. Shake the suspension for one-half hour and filter through Whatman's No. 42 filter paper or equivalent. Take 10 or 20 ml aliquot and transfer to a 25-ml volumetric flask. Add 1 g of barium chloride crystals (ground to pass 500-micron IS sieve and to be retained on 250-micron IS sieve) to the aliquot in the flask and shake for 1 minute. Add 1 or 2 ml of 25 percent gum acatia. Pour distilled water up to the mark of volumetric flask and shake for a minute. Precipitate the suspension and take the reading between 5 to 30 minutes after precipitation either by photoelectric colorimeter using blue filter or by turbidimeter. Sulphate is then determined by the standard sulphate curve.

4.4 Preparation of Standard Sulphate Curve

4.4.1 *Stock Solution* — Dissolve 0·888 g anhydrous sodium sulphate $Na_2 SO_4$/l alcoholic (N) ammonium chloride (NH_4Cl). This gives a concentration of 0·60 mg of SO_4/ml.

NOTE — Absolute alcohol should be used for the preparation of the solution.

4.4.2 *Working Standard Solution* — Dilute 0·60 mg SO_4/ml stock solution with alcoholic (N) ammonium chloride to give 0·06 mg SO_4/ml. Take 2, 4, 6, 8, 10 ml of this to give a range of 0·12 — 0·60 mg of SO_4.

4.4.3 The standard curve should be prepared by taking readings with photoelectric colorimeter using blue filter or by turbidimeter using the working standard solution.

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