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Indian Standard

METHODS OF TEST FOR SOILS

PART XXIV DETERMINATION OF CATION EXCHANGE CAPACITY

(First Revision)

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Indian Standard

METHODS OF TEST FOR SOILS

PART XXIV DETERMINATION OF CATION EXCHANGE CAPACITY

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part XXIV) (First Revision) was adopted by the Indian Standards Institution on 16 February 1976, 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 establishing uniform procedures for the determination of different characteristics of soils and also for facilitating a comparative study of the results, the Indian Standards Institution has brought out the 'Indian Standard methods of test for soils (IS : 2720)' which is being published in parts. So far 38 parts have been published. This part (Part XXIV) which was originally published in 1967 covers the method for the determination of cation exchange capacity of soil. Cation exchange capacity gives an indication of the affinity of soil for water and its capacity for swelling.

0.2.1 In this revision, a method to determine the exchangeable metallic cations and a method to determine the exchangeable hydrogen ions separately have been given. The sum of the exchangeable ions determined by these methods gives the total exchangeable ions in the soil; however, because of fixation of potassium and ammonium by illite and vermiculite type of minerals, the cation exchange capacity values as determined by these ions are generally lower than the true values. A single method which determines the cation exchange capacity (metallic and hydrogen ions together) has also been included. In keeping with the latest trend the term 'base exchange capacity' has been replaced by the term 'cation exchange capacity'.

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.

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*.

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

1. SCOPE

1.1 This standard (Part XXIV) lays down the methods for the determination of exchangeable metallic cation and exchangeable hydrogen ion, sum of which will be the cation exchange capacity of soils.

1.1.1 It also lays down a single method which determines the cation exchange capacity (metallic and hydrogen ions together).

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition shall apply.

2.1 Cation Exchange Capacity — Cation exchange is the physio-chemical process whereby one type of ions (cation) adsorbed on soil particles is replaced by another type. The cation exchange capacity signifies the capacity of soil to retain cations up to its highest limit; or it can also be defined as the power of the soil to combine with cation in such a manner that they cannot be easily removed by leaching with water, but can be exchanged by an equivalent amount of other cations.

3. DETERMINATION OF EXCHANGEABLE METALLIC CATIONS

3.1 Standard Method

3.1.1 *Reagents*

3.1.1.1 *Ammonium acetate solution* — 1 N. Prepared by adding 576 ml of glacial acetic acid (relative density 1·052) diluted to 5 litres, to 540 ml (of relative density 0·88) or 750 ml (of relative density 0·91) of ammonium hydroxide solution diluted to 5 litres.

The above two solutions should be mixed in equal volumes. The *p*H of the mixed solution should be determined in a comparator using bromothymol blue as indicator. If *p*H is close to 7·0, a little more of acetic acid solution or ammonium hydroxide solution should be added to have the *p*H exactly 7·0.

3.1.1.2 *Hydrochloric acid* — 0·05 N. Dilute 50 ml of N hydrochloric acid solution to one litre in a volumetric flask.

3.1.1.3 *Sodium hydroxide* — 0·05 N.

NOTE — The solution should be standardized afresh while using.

3.1.1.4 *Methyl red indicator*

3.1.2 *Apparatus*

3.1.2.1 *Buchner funnel* — Designation 110 (*see* IS : 5009-1968*).

3.1.2.2 *Pipettes* — 25 and 50 ml (*see* IS : 4162-1967† and IS : 1117-1958‡).

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*Specification for Buchner funnels.

†Specification for graduated pipettes.

‡Specification for one-mark pipettes.

3.1.2.3 *Cylinders graduated* — 100 and 250 ml capacity (*see* IS : 878-1956*).

3.1.2.4 *Filter paper* — Whatman No. 44 or equivalent, 15 or 18·5 cm diameter.

3.1.2.5 *Silica basins* — 500 and 100 ml capacity.

3.1.2.6 *Water-bath*

3.1.2.7 *Hot-plate* — (*See* IS : 365-1965†).

3.1.2.8 *Muffle furnace*

3.1.2.9 *Burette* — 50 ml with stand (*see* IS : 1997-1967‡).

3.1.2.10 *Chemical balance* — of 0·1 or 0·2 mg sensitivity.

3.1.2.11 *Beaker* — 400 ml capacity (*see* IS : 2619-1971§).

3.1.2.12 *Flask* — 1 litre capacity.

3.1.3 *Procedure*

3.1.3.1 Transfer 50 to 100 g (*see* Note 1) of soil to 400-ml beaker, add 250 ml of 1 N ammonium acetate solution, stir well and allow to stand overnight at room temperature. Then decant through a 15- or 18·5-cm filter paper and leach with further portions of the ammonium acetate solution in small quantities, allowing the filter to drain completely between each addition, in a 1-litre flask. Continue leaching until one litre of the filtrate has been collected (*see* Note 2).

NOTE 1 — Take 50 g of soil when more than 20 to 25 milli-equivalent percent of exchangeable cation are present and 100 g when less than 10 milli-equivalent percent are present, as determined by the rapid method with 0·05 N hydrochloric acid.

NOTE 2 — Where possible the extraction may be done by centrifuge.

3.1.3.2 Transfer a suitable aliquot of the leachate corresponding to 10 g of soil, to 500-ml silica basin and evaporate to small volume on the water-bath. Transfer the solution at this stage to a 100-ml silica basin and continue the evaporation to dryness, finishing the operation over a hot-plate. Ignite in a muffle furnace, gently at first and then for 20 minutes at a medium to full red heat, so converting the acetates of the exchangeable metal ions to carbonates or oxides. Support the silica basin on a triangle to prevent the bottom being overheated.

3.1.3.3 When cold, add 20 to 50 ml of 0·05 N hydrochloric acid, or sufficient quantity to ensure an excess of acid at this stage; warm gently and leave to stand until solution of the alkalis is complete. Then titrate the excess of acid with 0·05 N sodium hydroxide using methyl red as indicator. If the methyl red is absorbed as the titration proceeds add further a drop or two just before the end-point.

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*Specification for graduated measuring cylinders.

†Specification for electric hot-plates (*revised*).

‡Specification for burettes (*first revision*).

§Specification for glass beakers (*first revision*).

3.1.4 *Calculations* — Calculate the amount of total exchangeable metallic ions originally present in the soil, in milli-equivalents percent from the following expression:

$$meq = (V_a - V_b) \times N \times \frac{1\,000}{V_e} \times \frac{100}{W}$$

where

meq = total exchangeable metallic ions, in milli-equivalents percent;
V_a = volume of standard hydrochloric acid taken;
V_b = volume of standard sodium hydroxide used in back titration;
N = normality factor;
V_e = volume of extract taken; and
W = weight of soil extracted.

3.2 Rapid Method — (*See* Note).

NOTE — The rapid method is not suitable for highly calcareous soils (calcium carbonate in excess of 15 percent).

3.2.1 *Reagents*

3.2.1.1 *Hydrochloric acid solution* — 0·05 N.

3.2.1.2 *Lime water* — freshly prepared and of known normality.

3.2.1.3 *Bromothymol blue indicator*

3.2.2 *Apparatus*

3.2.2.1 *Pipettes* — 100 and 25 ml (*see* IS : 1117-1958* and IS : 4162-1967†).

3.2.2.2 *Test tube* — 175 × 32 mm with stopper (*see* IS : 2618-1963‡).

3.2.2.3 *Filter paper* — Whatman No. 30 or equivalent.

3.2.2.4 *Erlenmeyer flask* — 125 ml capacity.

3.2.2.5 *Beaker* — 250 ml capacity (*see* IS : 2619-1971§).

3.2.2.6 *Chemical balance*

3.2.2.7 *Burette* — 100 ml with stand (*see* IS : 1997-1967||).

3.2.2.8 *Glass rod* — (*See* IS : 7374-1974¶).

3.2.3 *Procedure*

3.2.3.1 Pipette 100 ml of 0·05 N hydrochloric acid into a test-tube (175 × 32 mm), add a weighed amount of soil (1 to 5 g), stopper, stir well and allow to stand overnight. The amount of soil taken should be sufficient to neutralize not more than about 20 percent of the standard acid. In general, the larger amount may be taken for sandy loams or soils slightly

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*Specification for one-mark pipettes.

†Specification for graduated pipettes.

‡Specification for test-tubes.

§Specification for glass beakers (*first revision*).

||Specification for burettes (*first revision*).

¶Specification for glass rods and tubing for laboratory glassware.

podolized. For heavy clays and base saturated soils, only 1 to 2 g should be used. The values obtained by this method are equilibrium values and therefore depend on the proportion of soil to acid.

3.2.3.2 Filter through a dry 11-cm Whatman No. 30 or equivalent filter paper, collecting the filtrate in a dry 125-ml Erlenmeyer flask, rejecting the first portion. Titrate a 25-ml aliquot against standard lime water using bromothymol blue as indicator. As the end-point is approached add 2 or 3 more drops of indicator to overcome absorption by any sesquioxide precipitate. Titrate a blank similarly.

3.2.4 *Calculation*

3.2.4.1 Then the approximate value for total exchangeable metallic ions in milli-equivalents percent is given by the expression:

$$meq = (B - T) \times N \times \frac{100}{25} \times \frac{100}{W}$$

where

meq = total exchangeable metallic ions,
B = blank titration in ml of lime water of known normality,
T = actual titration in ml of lime water of known normality,
N = normality of the lime water, and
W = mass of soil taken.

If any calcium carbonate is present (say up to 15 percent), express its amount in milligram equivalents percent, and deduct it from the value calculated for total exchangeable metallic ions.

4. DETERMINATION OF EXCHANGEABLE HYDROGEN IONS

4.1 Reagents

4.1.1 *Barium Acetate Solution* — 1 N of *p*H 8·1, adjusted with barium hydroxide.

4.1.2 *Phenolphthalein Indicator* — 1 percent.

4.1.3 *Sodium Hydroxide* — 0·05 N.

4.2 Apparatus

4.2.1 *Carbon Filter Funnel* — (*See* Note under **4.3.1**).

4.2.2 *Conical Flask* — 500 ml capacity.

4.2.3 *Beaker* — 600 ml capacity (*see* IS : 2619-1971*).

4.3 Procedure

4.3.1 Transfer 25 g of soil to the funnel with care to prevent soil from sticking to the sides of the carbon filter funnels (*see* Note). Level off the soil and place silica sand on the top to a depth of about 5 mm. Pour 350 ml of the barium acetate solution to the soil. Leaching rate from the funnel should not exceed 10 to 20 drops per minute.

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*Specification for glass beakers (*first revision*).

When all the extraction solution is passed through, measure the volume in a graduated cylinder and transfer it to a 600-ml beaker. Add approximately 10 drops of 1 percent phenolphthalein solution and back titrate the extraction solution with 0·05 N sodium hydroxide solution to a faint pink colour. Make a blank titration on 100 ml of barium acetate solution extracted through the carbon filter funnel but without the soil sample.

NOTE — It is a special type carbon funnel having arrangement for flow rate regulation by screw clamp, fine glass wool and acid washed silica sand (coarse). This funnel is wetted with a little of barium acetate solution and the glass wool compressed. A small quantity of pure silica is then spread over the glass wool.

4.4 Calculation — Express the results obtained in terms of *meq* of H^+ per 100 g of soil

$$meq \text{ exchangeable } H^+ \text{ per 100 g soil} = (T-B) \times N \times \frac{100}{\text{mass of soil in g}}$$

where

T = volume of sodium hydroxide solution used in titration of soil extraction, ml;

B = volume of sodium hydroxide solution used for the blank titration, ml; and

N = normality of standard sodium hydroxide solution.

5. METHOD FOR DETERMINATION OF CATION EXCHANGE CAPACITY (METALLIC AND HYDROGEN IONS TOGETHER)

5.1 Reagents

5.1.1 *Sodium Acetate Solution* — 1 N. Adjusted to *p*H 5·0 containing 82 g of salt and about 28 ml of glacial acetic acid per litre.

5.1.2 *Sodium Acetate Solution* — Neutral, 1 N. Containing 82 g of salt per litre, with *p*H adjusted to 7·0 with acetic acid to neutralize sodium hydroxide normally formed by hydrolysis of this salt.

5.1.3 *Calcium Chloride* — Neutral, 1 N. Containing approximately 109 g of calcium chloride, hexahydrate or 73 g of calcium chloride, per litre of carbondioxide-free distilled water, with *p*H adjusted to 7·0 with calcium hydroxide.

5.1.4 *Acetone* — 80 percent.

5.1.5 *Special Solutions* — The following special solutions are required for calcium versene procedure.

5.1.5.1 *Standard calcium solution* — 0·500 5 g of pure dried calcium carbonate is dissolved in a minimum of 0·2 N hydrochloric acid. The solution is boiled to expel carbondioxide and is then diluted to 1 litre. The solution is 0·010 0 N with respect to calcium.

5.1.5.2 *Ammonium chloride-ammonium hydroxide buffer of pH 10* — This buffer is made up of 100 ml of 1 N ammonium chloride and 500 ml of 1 N ammonium hydroxide.

5.1.5.3 *Eriochrome black T indicator* — This solution is prepared by dissolution of 0·5 g of the indicator with 4·5 g of hydroxylamine hydrochloride in 100 ml of methanol.

5.1.5.4 *Sodium cyanide solution* — 2 percent.

5.1.5.5 *Standard versene solution* — A 2-g portion of disodium versenate (disodium dihydrogen ethylenediamine tetra acetic acid) is dissolved in 900 ml of water. Then approximately 50 mg of magnesium chloride crystals ($MgCl_2.6H_2O$) are added to the solution. The normality of versene is then determined by titration of 25 ml portion of the standard calcium solution.

5.3 Apparatus

5.3.1 *Centrifuge Tubes* — 100 ml.

5.3.2 *Centrifuge* — (*See* Note under **5.4.1**).

5.3.3 *Beaker* — 250 ml (*see* IS : 2619-1971*).

5.3.4 *Conical Flask* — 500 ml.

5.3.5 *Pipette* — 25 ml (*see* IS : 4162-1967†).

5.3.6 *Burette* — with stand (*see* IS : 1997-1967‡).

5.4 Procedure

5.4.1 Take 5 g of soil in a 100-ml centrifuge tube and stir in 50 ml of 1 N sodium acetate of *p*H 5·0 with a policeman-tipped rod. Digest the soil suspension in a near boiling water-bath for 30 minutes with intermittant stirring. Remove the salts by centrifugation of the suspension and decantation of the clear supernatant liquid (*see* Note). Give two additional washings with 1 N sodium acetate of *p*H 5·0, the 30 minutes boiling water-bath treatment being repeated if the sample is known to be calcareous. If still the presence of salts is doubted, give two more washings with 1 N sodium acetate of *p*H 5·0. Then give the sample 5 washings with 1 N calcium chloride solution. Remove the excess salts by washings (usually 5) with 80 percent acetone, until the excess calcium chloride is removed as indicated by a negative $AgNO_3$ test for chloride in the last of the washings. Finally replace calcium by means of 5 washings with a neutral 1 N sodium acetate solution. Place in a 500-ml conical flask, the calcium solution (about 250 ml) resulting from displacement in the determination of cation exchange capacity. Then, add 10 ml of the ammonium chloride-ammonium hydroxide buffer solution to bring the solution to *p*H 10; and then add 10 drops of erichrome black T indicator solution and 1 ml of 2 percent sodium cyanide solution. Similarly prepare a blank of sodium acetate and titrate it to a

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*Specification for glass beakers (*first revision*).

†Specification for graduated pipettes.

‡Specification for burettes (*first revision*).

bright blue end-point with standardized (about 0·01 N) versene solution. Titrate the test sample to the same colour.

NOTE — In lieu of centrifuge washing, the sample may be washed in an ordinary funnel with filter paper.

5.5 Calculation

5.5.1 Calculate the milli-equivalent cation exchange capacity per 100 g of soil as follows:

$$\textit{meq}\text{ exchange capacity per 100 g} = \text{ml of versene solution} \times N \times \frac{100}{\text{mass of soil in g}}$$

where N is the normality of the versene solution.

(*Continued from page* 2)

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AMENDMENT NO. 1 NOVEMBER 1987

TO

IS : 2720 (Part 24) - 1976 METHODS OF TEST FOR SOILS

PART 24 DETERMINATION OF CATION EXCHANGE CAPACITY

(*First Revision*)

(*Page* 5, *clause* **3.1.2.3**) — Substitute ' IS : 878-1975* ' *for* ' IS . 878-1956* '.

(*Page* 5, *clause* **3.1.2.7**) — Substitute ' IS : 365-1983† ' *for* ' IS : 365-1965† '.

(*Page* 5, *clause* **3.1.2.9**) — Substitute ' IS : 1997-1982‡ ' *for* ' IS : 1997-1967‡ '.

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

'*Specification for graduated measuring cylinders (*first revision*).

†Specification for electric hot plates (*second revision*).

‡Specification for burettes (*second revision*).'

(*Page* 6, *clause* **3.2.2.1**) — Substitute ' IS : 1117-1975* ' *for* ' IS : 1117-1958* '.

(*Page* 6, *clause* **3.2.2.7**) — Substitute ' IS : 1997-1982|| ' *for* IS : 1997-1967|| '.

(*Page* 6, *foot-notes with* ' * ' *and* ' || ' *marks*) — Substitute the following for the existing foot-notes:

'*Specification for one-mark pipettes (*first revision*).

||Specification for burettes (*second revision*).'

(*Page* 9, *clause* **5.3.6**) — Substitute ' IS : 1997-1982‡ ' *for* ' IS : 1997-1967‡ '.

(*Page* 9, *foot-note with* ' ‡ ' *mark*) — Substitute the following for the existing foot-note:

'‡Specification for burettes (*second revision*).'

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Reprography Unit, BIS, New Delhi, India