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Indian Standard

METHODS OF TEST FOR DETERMINATION OF WATER SOLUBLE CHLORIDES IN CONCRETE ADMIXTURES

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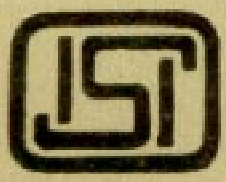

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METHODS OF TEST FOR DETERMINATION OF WATER SOLUBLE CHLORIDES IN CONCRETE ADMIXTURES

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Indian Standard

METHODS OF TEST FOR DETERMINATION OF WATER SOLUBLE CHLORIDES IN CONCRETE ADMIXTURES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 March 1973, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Various types of concrete admixtures are being used in this country, such as accelerators, retarders, water-proofers and air entraining agents. Some of these admixtures are likely to contain water soluble chlorides which are likely to cause corrosion of reinforcement in the reinforced concrete. In fact the use of such chlorides containing admixtures has been prohibited by IS : 456-1964*. However, the option of using such admixtures is left to the engineer-in-charge who has to use his discretion on the basis of relevant data in respect of the admixtures. As information on the percentage of water soluble chlorides in the admixtures is of vital importance it is considered necessary to bring out a standard dealing with the methods of test for determination of water soluble chloride content in concrete admixtures.

0.3 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 specifies the following methods of test for determination of water soluble chlorides in concrete admixtures:

a) Volumetric method,
b) Gravimetric method, and
c) Turbidimetric method.

*Code of practice for plain and reinforced concrete (*second revision*).
†Rules for rounding off numerical values (*revised*).

2. SELECTION OF METHOD

2.0 One of the three methods may be used appropriately depending on the concentration of the chlorides in the admixtures as per the declaration of the manufacturer.

2.1 The volumetric method may be used when the chloride concentration is nearly 1 percent or above.

2.2 The gravimetric method may be used when the chloride concentration is more than 2·5 percent.

2.3 The turbidimetric method may be used when the concentration of chloride is as low as 2 ppm and above.

2.4 Where a choice is open between volumetric and gravimetric methods volumetric method is preferable as it is quicker and less laborious. Turbidimetric method may be adopted when the chloride concentration is very low.

3. VOLUMETRIC METHOD

3.1 Reagents

3.1.0 *Quality of Reagents* — Unless otherwise specified, pure chemicals and distilled water (*see* IS:1070-1960*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3.1.1 *Nitric Acid* — 1:2·5 — 6 N.

3.1.2 *Sodium or Potassium Chloride Solution (Standard)* — 0·1 N.

3.1.3 *Potassium Chromate Indicator Solution*

3.1.4 *Silver Nitrate Solution* — 0·1 N.

3.1.4.1 *Preparation* — Weigh about 8·5 g of silver nitrate, dissolve in distilled water and make up to 500 ml in a volumetric flask.

3.1.4.2 *Standardization* — Standardize the solution against 0·1 N sodium chloride or potassium chloride solution using potassium chromate solution as indicator. Adjust the normality exactly to 0·1.

3.1.5 *Nitrobenzene*

3.1.6 *Ferric Alum Indicator Solution*

3.1.7 *Ammonium Thiocyanate Solution* — 0·1 N.

*Specification for water, distilled quality (*revised*).

3.1.7.1 *Preparation* — Weigh about 8·5 g of ammonium thiocyanate and dissolve it in 1 litre of water in a volumetric flask. Shake well, and standardize by titrating against 0·1 N silver nitrate solution using ferric alum solution as indicator. Adjust the normality exactly to 0·1.

3.2 Procedure

3.2.1 Weigh accurately sufficient quantity of the admixture such that about 0·1 g of chloride is present in the sample. Add enough hot water so as to make a volume of 150 ml, stir until dissolution is complete. If there is insoluble matter, filter and wash with water. Make up the clear solution thus obtained to a volume of 250 ml with water, shake well.

3.2.2 Pipette 50 ml of the solution into a 250-ml conical flask containing 5 ml of 6 N nitric acid. Add 10 to 15 ml of 0·1 N silver nitrate solution from the burette. Then add 2 to 3 ml of nitrobenzene and 1 ml ferric alum indicator and shake vigorously to coagulate the precipitate. Titrate the excess silver nitrate with 0·1 N ammonium thiocyanate until a permanent faint reddish brown colouration appears. Repeat the titration with another 50 ml portion.

3.2.3 From the volume of silver nitrate ($AgNO_3$) solution added subtract the volume of thiocyanate solution required. Take the average of the two determinations. Calculate the percentage of chloride (Cl) in the sample:

1 ml 0·1 N $AgNO_3 = 0{\cdot}003\,546$ g, Cl'

4. GRAVIMETRIC METHOD

4.1 Reagents

4.1.1 *Concentrated Nitric Acid*

4.1.2 *Dilute Nitric Acid* — 1 : 50.

4.1.3 *Silver Nitrate Solution* — approximately 0·1 N (*see* **3.1.4**).

4.1.4 *Dilute Hydrochloric Acid* — 1 : 100.

4.2 Procedure

4.2.1 Weigh out accurately sufficient quantity of the admixture such that about 0·05 g of chloride is present in the sample. Add enough hot water so as to make a volume of 150 ml, stir until the dissolution is complete. Filter and wash with water if there is insoluble matter. Add 1 to 2 ml of concentrated nitric acid. Then add the silver nitrate solution slowly and with constant stirring until the precipitation is complete. Add a slight excess (5 to 10 ml) of the silver nitrate solution. Heat the suspension nearly to boiling, while stirring constantly and maintain it at

this temperature until the precipitate coagulates and the supernatant liquid is clear. Set aside the beaker in the dark for one hour and filter through a previously weighed sintered glass or porcelain crucible. Transfer the last traces of silver chloride adhering to the beaker with a policeman. Wash the precipitate in the crucible with 1 : 50 nitric acid added in small portions until 3 to 5 ml of the washings collected in a test tube give no turbidity with 1 or 2 drops of dilute hydrochloric acid. Dry the crucible and contents in an air-oven at 130 to 150°C for one hour. Allow to cool in a desiccator and weigh. Repeat the process of drying and cooling until constant weight is attained.

4.2.2 Calculate the percentage of chloride in the sample:

0·1 g AgCl = 0·024 737 $Cl^{\prime}$

5. TURBIDIMETRIC METHOD

5.1 Apparatus

5.1.1 *Turbidimeter*

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 1 : 3.

5.2.2 *Silver Nitrate Solution* — *See* **4.1.3.**

5.2.3 *Standard Sodium Chloride Solution*

5.2.3.1 *Preparation* — Weigh accurately 0·164 9 g of sodium chloride (previously dried at 105 to 110°C for 2 h) and dissolve in 1 000 ml of distilled water in a volumetric flask. This solution contains 100 ppm chloride, that is, 100 mg/l.

5.3 Procedure

5.3.1 *Calibration of the Turbidimeter* — Take 5 ml of dilute nitric acid in a 100-ml volumetric flask, add 5 ml of silver nitrate solution and make up the volume with distilled water. Shake well and use the solution as 'blank' for adjusting the 'zero' of the galvanometer. Take 20 ml of the standard sodium chloride solution in a 100-ml volumetric flask, add 5 ml of dilute nitric acid and 50 to 60 ml distilled water. Shake well and add 5 ml of silver nitrate solution and make up the volume with distilled water. Shake well and use this turbid solution to adjust the galvanometer deflection to full scale.

5.3.1.1 Run in 1·0, 2·5, 5·0, 7·5, 10·0, 15·0, 17·5 and 20·0 ml standard chloride solution from a burette into separate 100-ml volumetric flasks. Take the first flask, add 5 ml of dilute nitric acid and 50 to 60 ml distilled water. Shake well, add 5 ml of silver nitrate solution and make up

the volume with distilled water. Shake well and measure the turbidity after checking the galvanometer 'zero' again. Repeat the above procedure with the remaining solutions.

5.3.1.2 Plot the galvanometer readings against chloride concentration in ppm.

5.3.2 *Determination of Chloride in the Test Sample* — Weigh accurately sufficient quantity of admixture such that it contains about 0·01 g of chloride and boil with 100 to 150 ml distilled water. Filter and wash with hot distilled water. Collect the filtrate and washings into a 500-ml volumetric flask and make up the volume. Take 50 ml (*see* Note) of this solution into a 100-ml volumetric flask, add 5 ml dilute nitric acid and 5 ml silver nitrate solution, and make up the volume with distilled water. Shake well and measure the turbidity after checking the galvanometer 'zero'. Read the chloride ion concentration in ppm from the calibration plot prepared earlier and then calculate the percentage of chloride in the sample.

$$\text{Percentage chloride} = \frac{\text{Weight of chloride in g}}{\text{Weight of the sample taken}} \times 100$$

NOTE — Suitable dilutions may have to be carried out such that the galvanometer reading falls within the range 2 to 15 ppm chloride whenever it is found necessary.

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2645-1964 Integral cement waterproofing compounds

3466-1967 Masonry cement (*first revision*)

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