भारतीय मानक

मोर्टार व कांक्रीट में जल एवं अम्ल में घुलनशील क्लोराइड का निर्धारण — परीक्षण पद्धति

भाग 2 कठोर मोर्टार व कांक्रीट

Indian Standard

DETERMINATION OF WATER SOLUBLE AND ACID SOLUBLE CHLORIDES IN MORTAR AND CONCRETE — METHOD OF TEST

PART 2 HARDENED MORTAR AND CONCRETE

ICS 91.100.10;91.100.30

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

Chlorides in the concrete could be drawn from different sources like aggregates, mix water, admixtures and cement and could lead to durability problems namely, corrosion of reinforcing steel in concrete, if present in sufficient quantity. Chlorides could be present in different degrees of binding in the concrete matrix and could be determined as water soluble and acid soluble chlorides. In some cases of corrosion of carbonated concrete, the combined chlorides (water soluble and as acid soluble) will be let free in pore water and these chlorides are harmful to concrete. To minimize the chances of deterioration of concrete due to harmful chlorides, the level of these chlorides has been limited in various design codes. Therefore, this standard has been formulated to provide necessary guidance for determination of water soluble and acid soluble chlorides in concrete. This Part 2 of the standard covers volumetric method of test for determination of chlorides in hardened mortar and concrete and Part 1 of this standard covers the method of test for fresh mortar and concrete.

The composition of the committee responsible for the formulation of this standard is given in Annex A.

In reporting the results 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*)'.

Indian Standard

DETERMINATION OF WATER SOLUBLE AND ACID SOLUBLE CHLORIDES IN MORTAR AND CONCRETE — METHOD OF TEST

PART 2 HARDENED MORTAR AND CONCRETE

1 SCOPE

This standard (Part 2) covers volumetric method of test for determination of water soluble and acid soluble chlorides in hardened mortar and concrete.

NOTE — The source of samples for test in accordance with this standard may be either the stationary samples obtained from project sites or ready-mixed concrete plants.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
3025 (Part 32) : 1988	Methods of sampling and test (physical and chemical) for water and wastewater : Part 32 Chloride (*first revision*)

3 SAMPLING

The apparatus required for processing the sample shall be chosen for its suitability for the purposes of the investigation. A specimen to be tested for the determination of chlorides both acid and water soluble, shall not be removed from the structure until the concrete has become hard enough to permit removal without disturbing the bond between the mortar and the coarse aggregate. Normally concrete shall be 14 days old before the specimens are removed. Specimens that show abnormal defects or that have been damaged in removal shall not be used.

A core drill shall be used for securing cylindrical core specimens (at least 100 mm diameter). The diameter of the core should be at least 2.5 times the maximum size of the aggregates and the length of the core should be at least 95 percent of core diameter. For specimens taken perpendicular to the horizontal surface, a short drill is satisfactory. For inclined holes, a diamond drill is satisfactory. A saw having diamond or silicon carbide cutting edge shall be used for securing beam specimens from the structures or pavement.

Samples more than 25 mm in maximum dimension shall be reduced in size by use of jaw crusher or broken into smaller pieces by hammering carefully to avoid loss of smaller pieces. Crush the particles to less than 25 mm in maximum dimensions using a rotating puck grinding apparatus or by using a disk pulverizer, operated to restrict to negligible levels the loss of fine particles. Sieve the crushed samples through 850µm IS Sieve. Thoroughly blend the material by transferring it from one glazed paper to another at least 10 times.

4 METHOD OF TEST

4.1 Reagents

4.1.1 *Quality of Reagent*

Unless otherwise specified, pure chemicals of analytical reagent grade and distilled water (*see* IS 1070) shall be used in the test.

4.1.2 *Nitric Acid* (HNO_3) *Concentrated* (*Specific Gravity* 1.42)

Prepare the solution, 6N (approximately), by diluting 38 ml of concentrated nitric acid to 100 ml with distilled water.

4.1.3 *Ferric Alum* [$FeNH_4 (SO_4)_2 \, 12 \, H_2O$]

Dissolve 10 g of ferric alum in 100 ml of distilled water and add 1 ml of nitric acid.

4.1.4 *Potassium Chromate* (K_2CrO_4), 5 *Percent Solution*

Dissolve 5 g of potassium chromate in 100 ml of distilled water.

4.1.5 *Nitrobenzene* ()

4.1.6 *Silver Nitrate* ($AgNO_3$) *Solution,* 0.02 N

Weigh 1.7 g, of silver nitrate, dissolve in distilled water and dilute to 500 ml in a volumetric flask. Standardize the silver nitrate solution against 0.02 N sodium chloride solution using potassium chromate solution as indicator

(5 percent *w/v*) in accordance with the procedure given in IS 3025 (Part 32).

4.1.7 *Ammonium Thiocyanate* (NH_4SCN) *Solution*, 0.02 N

Weigh 1.7 g of ammonium thiocyanate and dissolve in one litre of distilled water in a volumetric flask. Shake well and standardize by titrating with 0.02 N silver nitrate solution using ferric alum solution as an indicator. Adjust the normality exactly to 0.02 N.

4.1.8 *Sodium Chloride* (NaCl), 0.02 N

Weigh 1.169 2 g of sodium chloride dried at 105 ± 2°C, dissolve in distilled water and make up to 1 000 ml in a volumetric flask.

4.2 Use of Filter Paper

In the methods prescribed in this standard, relative numbers of Whatman filter paper only have been prescribed since these are commonly used. However, any other suitable brand of filter papers with equivalent porosity may be used.

4.3 Procedure

4.3.1 *Water Soluble Chloride*

4.3.1.1 Weigh 1 000 ± 5 g of the pulverized mortar or concrete sample in a 2 litre capacity beaker and add 1 000 ml of distilled water (chloride free). Stir the mixture vigorously and warm gently for 15 min. After allowing the mixture to stand for 24 h for settling, decant about 200 ml of the supernatant solution into a clean dry 250 ml capacity beaker. Immediately, filter the solution through Whatman filter paper No. 1 and collect the filtrate.

4.3.1.2 Pipette 50 ml of filtrate in a 250 ml capacity conical flask. Add 5 ml of 6 N nitric acid. Add a known volume (X), preferably 25 ml of 0.2 N silver nitrate solution. Add 1 ml ferric alum and 5 ml of nitrobenzene. Shake vigorously to coagulate the precipitate. Titrate the excess silver nitrate with 0.2 N ammonium thiocyanate solution until a permanent faint reddish brown colour appears. Note down the volume (Y) of ammonium thiocyanate used.

4.3.2 *Acid Soluble Chloride*

4.3.2.1 Weigh about 1 000 ± 5 g of the pulverized mortar or concrete sample in a 2 litre capacity beaker and add 100 ml of 6N nitric acid and 900 ml of distilled water (chloride free), after stirring for few minutes. Stir the mixture vigorously and warm gently for 30 min. After allowing the mixture to stand for 10 to 15 min for settling, decant about 200 ml of the supernatant solution into a clean dry 250 ml capacity beaker. Immediately, filter the solution through Whatman filter paper No. 1 and collect the filtrate.

4.3.2.2 Pipette 50 ml of filtrate in a 250 ml capacity conical flask. Add 5 ml of 6 N nitric acid. Add a known volume (X), preferably 25 ml of 0.2 N silver nitrate solution. Add 1 ml ferric alum and 5 ml of nitrobenzene. Shake vigorously to coagulate the precipitate. Titrate the excess silver nitrate with 0.02 N ammonium thiocyanate solution until a permanent faint reddish brown colour appears. Note down the volume (Y) of ammonium thiocyanate used.

4.4 Calculation

Calculate the percentage of chloride (acid soluble/ water soluble) by mass of mortar or concrete as follows:

$$\text{Chloride, percent} = 0.00142\,(X - Y)$$

where

X = volume of silver nitrate added, in ml; and

Y = volume of 0.02 N ammonium thiocyanate consumed.

NOTE – Interference of silver chloride particles (which are generated *in-situ*) in titration by reacting with thiocyanate can be avoided by the addition of nitrobenzene which forms a film on silver chloride particles.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

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