भारतीय मानक

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

भाग 1 ताजा मोर्टार व कंक्रीट

Indian Standard

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

PART 1 FRESH MORTAR AND CONCRETE

ICS 91.100.10; 91.100.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Price Group 3

FOREWORD

This Indian Standard (Part 1) 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 as acid soluble chlorides. In some cases of corrosion of carbonated concrete, the combined chlorides (water soluble and 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 1 of the standard covers volumetric method of test for determination of chlorides in fresh mortar and concrete, and Part 2 of this standard covers the method of test for hardened 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 1 FRESH MORTAR AND CONCRETE

1 SCOPE

This standard (Part 1) covers volumetric method of test for determination of water soluble and acid soluble chlorides in fresh 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

A sample of fresh concrete or mortar shall be collected within a period of two hours from the time of addition of water to the ingredients that is, cement, coarse and fine aggregates and admixtures, etc. However, every effort shall be made to collect the samples immediately after mixing. Samples shall be obtained by taking uniformly distributed increments (preferably without stopping the mixing operations, provided sampling can be safely carried out), and mixed thoroughly to form a combined bulk sample. The number of increments and size of bulk sample necessarily depends on the quantity of the material, its variability and the accuracy required of the test results.

At least three approximately equal sample increments totalling 0.02 m^3 shall be taken in a clean and dry receptacle across the stream of mortar or concrete. This receptacle shall be constructed of non-absorbent material, preferably of metal and shall be such that the sample retained is not segregated. A flat surface without retaining sides will not fulfill this purpose. Where three sample increments are taken they shall be taken at about the time when one quarter, one half and three quarters of the concrete have been discharged from the mixer, and if more than three are taken they shall be at correspondingly shorter, but of equal intervals.

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)

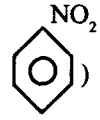
Prepare the solution, 6N (approximately), by diluting 38 ml of concentrated nitric acid to 100 ml with distilled water.

4.1.3 *Ferric Alum* [$FeNH_4 (SO_4)_2$ 12 H_2 O]

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

4.1.4 *Potassium Chromate* ($K_2 CrO_4$), 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 *m/v*) in accordance with the procedure given in IS 3025 (Part 32).

4.1.7 *Ammonium Thiocyanate* (NH_4 SCN) *Solution,* 0.02N

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 fresh mortar or concrete sample in a 2 litre capacity beaker and add 500 ml of distilled water (chloride free). Stir the mixture vigorously for 15 minutes. After allowing the mixture to stand for 10 to 15 minutes 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 0.02 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 fresh mortar or concrete sample in a 2 litre capacity beaker and add 50 ml of 6 N nitric acid and 450 ml of distilled water (chloride free) after stirring for few minutes. Stir the mixture vigorously for 15 minutes. After allowing the mixture to stand for 10 to15 minutes 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 standard 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:

$$\text{Chloride, percent} = 0.0007\,1\,(X - Y),$$

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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BUREAU OF INDIAN STANDARDS

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Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
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Regional Offices :

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