

Australian Standard®

AS 1012.20.2:2016

Methods of testing concrete

Method 20.2: Determination of water-soluble chloride in aggregates and hardened concrete

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PREFACE

This Standard has been prepared by the Standards Australia Committee CE-012, Aggregates and Rock for Engineering Purposes, in consultation with the Committee BD-042, Methods of Testing Concrete, to supersede, in part, AS 1012.20—1992, *Methods of testing concrete*, Method 20: *Determination of chloride and sulfate in hardened concrete and concrete aggregates*.

It is widely accepted that water-soluble chloride poses a threat to the corrosion of steel reinforcement in concrete. Therefore, a test method which accurately determines the water-soluble chloride in concrete and aggregate is considered an important tool. With limited crushing of aggregate to pass an 850 µm sieve, a more accurate measurement of water-soluble chloride ions in concrete can be made.

The Volhard titration method is the only non-instrumental titration permitted. Alternatively, a potentiometric titration by ion selective electrode can be used. One such method, Method 4B described in the Canadian Standard CSA A23.1-09/A23.2-09 (R2014) (see copyright information below), is described in this Standard.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

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METHOD

1 SCOPE

This Standard sets out a method for the determination of water-soluble chloride content of concrete aggregates and hardened concrete. A boiling water extraction procedure is used and the material to be tested is ground coarser to pass an 850 μm sieve. The method assumes that personnel conducting the procedure are trained and competent in conducting chemical tests and in the preparation of standard solutions.

2 NORMATIVE REFERENCE

The following document is required for the implementation of this Standard:

NOTE: Documents referenced for informative purposes are listed in the Bibliography.

AS

1152 Specification for test sieves

3 PRINCIPLE

The sample is stirred in hot water to provide a solution from which an aliquot is tested for water-soluble chloride content.

4 SAFETY PRECAUTIONS

Procedures using concentrated mineral acids, including the preparation of dilute solutions of these acids, shall be carried out in a fume cupboard.

WARNING: CONCENTRATED ACIDS POSE HAZARDS OF ACID BURNS RESULTING IN OFTEN IRREVERSIBLE DAMAGE TO THE SKIN OR EYES. INGESTION OF THE CONCENTRATED ACID, INHALATION OF THE VAPOUR, OR SKIN CONTACT CONSTITUTES A SERIOUS HEALTH HAZARD.

Full length rubber gauntlets and a face mask should be worn when handling the acid.

Appropriate documentation should be consulted as to safety precautions.

5 REAGENTS AND STANDARD SOLUTIONS

Analytical quality reagents shall be used. The following are required:

- (a) *Distilled water* Distilled water shall be used for the preparation of all reagents and standard solutions.
- (b) *Nitric acid* Prepare a 1:4 diluted solution of concentrated nitric acid. Cautiously add 100 mL of nitric acid (15 mol/L or 1.42 g/mL) to 400 mL of water while stirring.
- (c) *Methyl orange indicator* Dissolve 2 g of methyl orange per litre of 95% ethyl alcohol.
- (d) *Silver nitrate standard solution* Prepare a solution of 0.05 mol/L silver nitrate and standardize against 5.00 mL of standard 0.05 mol/L sodium chloride solution, diluted with water to 150 mL.
- (e) *Sodium chloride solution* Prepare exactly 1 L of a 0.05 mol/L sodium chloride solution. Dry sodium chloride at $105 \pm 5^\circ\text{C}$ to constant mass. Measure out 2922.2 mg of dried reagent, and dissolve in water and dilute to exactly 1 L in a volumetric flask and mix thoroughly. This solution is the standard and requires no further standardization.

NOTE: Methods for the preparation of standard solutions are available in many reference chemical texts. Reference can also be made to CSA A23.2-4B.

6 APPARATUS

The following apparatus is required:

- (a) *Balance* An analytical balance of adequate capacity and with a limit of performance of ± 0.0005 g.
- (b) *Burette* 10 mL capacity with 0.05 mL graduations.
- (c) *Fume cupboard*.
NOTE: Advice on suitable fume cupboards can be found in AS/NZS 2243.8.
- (d) *Grinder* Capable of grinding hardened concrete and aggregates to pass an 850 μm sieve.
- (e) *Ion selective electrode* Either a silver, chloride/sulphide ion selective electrode or a silver billet electrode coated with silver chloride with a reference electrode.
- (f) *Oven* Capable of operating in the temperature range 100°C to 110°C .
- (g) *Potentiometer* With millivolt scale graduated to 1 mV.
- (h) *Sieves* Punched plate and woven wire test sieves of aperture 6.7 mm, 1.18 mm, and 850 μm complying with AS 1152.
- (i) *Stirrer* Magnetic stirrer and coated stirring bars.

7 SAMPLING AND SAMPLE PREPARATION

7.1 General

Sampling requirements should be fully detailed by the specifying authority.

Sampling shall be as follows, appropriate to the source of the sample:

- (a) For sampling of an aggregate, or a hardened concrete, mortar or grout, take a sample of at least 1 kg, to be reduced by crushing and grinding as detailed in Clause 7.2, Items (a) to (d).
- (b) For sampling from a core taken from a concrete structure to determine the chloride profile, a sample of at least 30 g is required for each depth increment obtained by sawing.

cutting. Reduce the sample by crushing and grinding as detailed in Clause 7.2, Items (c) and (d). Use the larger core diameter to minimize the effect of lubricating water on the water-soluble chloride determination.

- (c) For sampling by direct dry drilling or profile grinding from a concrete structure to determine the chloride profile, a sample of at least 10 g is required for each depth increment. Dry the sample to constant mass as detailed in Clause 7.2, Item (d).

NOTES:

- 1 It is recommended that aggregate samples be taken in accordance with AS 1141.3.1.
- 2 It is recommended that samples from hardened concrete be taken from concrete cylinders prepared in accordance with AS 1012.8.1, except that the concrete specimens for assessment should not be subject to standard moist curing, but sealed in the plastic sample bags after stripping and until delivered to the laboratory for testing of water-soluble chloride.

7.2 Sample preparation

A minimum of 30 g sub-sample passing the 850 μm sieve is required for samples obtained according to Clause 7.1, Items (a) and (b), from which a 3 g test portion is used for each test.

The procedure shall be as follows:

- (a) Progressively crush large particles until the reduced sample is able to pass a 6.7 mm sieve and split the sample to not less than 500 g.
- (b) Further crush the reduced sample until it will pass a 1.18 mm sieve and split to no less than 200 g.
- (c) Crush or grind the reduced sample to pass an 850 μm sieve. Avoid exceptionally fine grinding and avoid loss of sample. Divide the sample to a not less than 30 g sub-sample.
- (d) Dry the sub-sample to a constant mass at a temperature of $105 \pm 5^\circ\text{C}$.

8 PROCEDURE

8.1 Preparation of the test solution

The procedure is as follows:

- (a) Select a representative test portion of 3 g. Weigh and record the test portion to the nearest 0.001 g (M) and place it into a 250 mL beaker.
- (b) Add 75 ± 1 mL of water, cover with a watch glass, bring to a boil and boil for 5 min. Allow to stand for 24 hours in an atmosphere free of hydrochloric acid (HCl) fumes.
NOTE: It is important to keep the beaker covered during heating and digestion to prevent the loss of chloride by volatilization.
- (c) Fit a 250 mL or 500 mL Büchner funnel and filtration flask with a 90 mm double filter paper consisting of a fast, ashless (less than 0.01% ash) paper over a medium, ashless paper.
- (d) Wash the papers with the diluted nitric acid before the water washing to prevent chlorine contamination of the test solution.
- (e) Wash the filter papers with four 25 mL increments of water using suction filtering.
- (f) Discard the washings and rinse the flask once with a small portion of water.
- (g) Reassemble the suction apparatus and filter the mixture prepared in Item (b).
- (h) Rinse the beaker and the filter paper twice with small portions of water.
- (i) Transfer the filtrate from the flask to a clean 250 mL beaker.