

Australian Standard®

AS 1012.20.1:2016

Methods of testing concrete

Method 20.1: Determination of chloride and sulfate in hardened concrete and aggregates— Nitric acid extraction method

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METHOD

1 SCOPE

This Standard sets out the method for determining the presence of chlorides and sulfates (including easily oxidizable sulfides) in hardened concrete, mortar, grout and concrete aggregates.

This method differs from AS 1012.20.2 in that a nitric acid method of extraction is used instead of boiling water extraction and that a finer fraction of material, passing the 150 µm sieve is used.

The nitric acid extraction method will dissolve any easily oxidizable sulfides which are present and these will be reported as sulfate.

For compliance with the relevant requirements specified in AS 3600 and AS 5100, additional tests and calculations may be required.

NOTES:

- 1 Separate test methods are available for the analysis of the other concrete-making materials, e.g. cementitious materials, admixtures and water.
- 2 Gross variations in the chloride and sulfate contents of concrete and concrete-making materials can occur over distances of even a few millimetres. This can be caused, for example, by leaching with rainwater, surface evaporation and crusting and penetration of salt and ground waters. Sampling technique is of prime importance. As the effect of the use of water during coring, especially small diameter cores, can be significant, the amount of water used should be restricted.
- 3 The solutions obtained from this method may also be used to determine the acid-soluble calcium oxide content of these materials.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1012	Methods of testing concrete
1012.8.1	Method 8.1: Method of making and curing concrete—Compression and indirect tensile test specimens
1012.8.2	Method 8.2: Method of making and curing concrete—Flexure test specimens
1012.8.3	Method 8.3: Methods of making and curing concrete—Mortar and grout specimens
1012.8.4	Method 8.4: Method for making and curing concrete—Drying shrinkage specimens prepared in the field or in the laboratory
1012.14	Method 14: Method for securing and testing cores from hardened concrete for compressive strength
1012.20.2	Method 20.2: Determination of water-soluble chlorides in hardened concrete and concrete aggregates—Water extraction method
1143	Methods for sampling and testing aggregates
1143.3.1	Method 3.1: Sampling—Aggregates
1152	Specification for test sieves
3600	Concrete structures
5100	Bridge design

3 PRINCIPLE

The sample is heated to boiling in 20% (V/V) nitric acid to provide a solution from which aliquots may be tested for chloride and sulfate content.

4 REAGENTS

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

- Distilled water (water)* Distilled water shall be used for the preparation of all reagents and standard solutions.
- Nitric acid (20%)* Slowly add 100 mL of nitric acid (15 mol/L or ρ_{20} 1.42 g/mL) to 400 mL of water while stirring.
- Silver nitrate solution* Dissolve 1 g of silver nitrate in 100 mL of water.
- Methyl orange solution* Dissolve 0.1 g of methyl orange in 100 mL of water.
- Barium chloride solution* Dissolve 5 g of barium chloride in 100 mL of water.
- Ammonium hydroxide solution* Slowly add 100 mL of concentrated (28% (m/m) or ρ_{20} 0.89 g/mL) ammonia solution to 400 mL of water with stirring.
- Standard sodium chloride solution.*
- Standard potassium sulfate solution.*

5 APPARATUS

The following apparatus and equipment are required:

- Balance* An analytical balance of adequate capacity and with a limit of performance of ± 0.0005 g.
- Crusher* Capable of reducing the concrete sample to a size suitable for grinding.

- (c) *Grinder* A grinder capable of grinding hardened concrete and concrete-making materials to pass a 150 µm sieve.

NOTES:

- 1 A ring grinder is recommended.
 - 2 Alternative methods of size reduction may be used.
- (d) *Oven* A drying oven capable of operating in the temperature range 100°C to 110°C.
- (e) *Sieves* 6.7 mm, 1.18 mm and 150 µm sieves complying with AS 1152.
- (f) *Graduated flask* 250 mL reading to 1 mL.
- (g) *Pipette* 50 mL capacity.
- (h) *Burette* Readable to 0.5 mL (optional).
- (i) *Sample divider* For fine material.

6 SAMPLING AND SAMPLE PREPARATION

6.1 General

A representative sample shall be obtained from either concrete or aggregates.

NOTES:

- 1 It is recommended that samples from hardened concrete be taken in accordance with the AS 1012.8 series or AS 1012.14 as detailed by the specifying authority.
- 2 It is recommended that samples from aggregates be taken in accordance with AS 1141.3.1.

6.2 Sample preparation

The procedure is as follows:

- (a) Progressively crush large samples until the 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 the sample to not less than 50 g.
- (c) Grind the reduced sample to pass a 150 µm sieve. Avoid exceptionally fine grinding and avoid loss of sample.
- (d) Dry the sample to a constant mass at a temperature of 100°C to 110°C.

7 PROCEDURE

7.1 Preparation of the sample solution

The procedure shall be as follows:

- (a) Weigh out an accurate mass of the dried sample as given in Table 1 into a 250 mL beaker.
- (b) Disperse in 75 mL of distilled water [see Clause 4(a)] and add the relevant amount of nitric acid [see Clause 4(b)] as shown in Table 1.
- (c) Bring the solution to the boil in a covered beaker and boil gently for 2 min.
NOTE: It is important that no significant evaporation occurs, as chloride loss will be significant if the nitric acid is concentrated to more than 50%.
- (d) After cooling, allow the coarse material to settle and decant the solution into a 250 mL graduated flask. Wash the beaker and coarse residue several times with distilled water by decantation.