AS 1012.20–1992

Australian Standard®

Methods of testing concrete

Method 20: Determination of chloride and sulfate in hardened concrete and concrete aggregates

PREFACE

This Standard has been prepared by the Standards Australia Committee on Methods of Testing Concrete in response to the introduction of limits on chloride and sulfate in AS 3600, *Concrete structures.*

The method of solution provides aliquots (separate portions) which may be tested in an aggregate or concrete laboratory by a quantitative method to indicate whether chlorides or sulfides are present above a threshold detectable quantity which will require full quantitative determination in an analytical laboratory.

CONTENTS

METHOD

1 SCOPE This Standard sets out a method for the determination of chloride content and sulfate content (including easily oxidizable sulfide content) of concrete and concrete aggregates. The solutions obtained from the sample may also be used to determine the acid–soluble calcium oxide content of these materials.

The method of solution will react with and dissolve any easily oxidizable sulfides which are present and these will be reported as sulfate. Such sulfides have been shown to be deleterious to concrete and the method provides an approach to limiting their inclusion in concrete and concrete aggregates.

For compliance with the relevant requirements specified in AS 3600, 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. cementious materials, admixtures, 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.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS
1012 Methods of testing concrete
1012.14 Method 14: Method for securing and testing cores from hardened concrete for compressive strength
1152 Test sieves
3600 Concrete structures

BS
1881 Testing concrete
1881.124 Part 124: Methods for analysis of hardened concrete

3 PRINCIPLE The sample is dissolved in hot 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:

(a) *Distilled water* Distilled water shall be used for the preparation of all reagents and standard solutions.

(b) *Nitric acid (1:4)* Cautiously add 100 mL of nitric acid (15 mol/L or ϱ_{20} 1.42 g/mL) to 400 mL of water while stirring.

(c) *Silver nitrate solution* Dissolve 1 g of silver nitrate in 100 mL of water.

(d) *Methyl orange solution* Dissolve 0.1 g in 100 mL of water.

(e) *Barium chloride solution* Dissolve 5 g of barium chloride in 100 mL of water.

(f) *Ammonium hydroxide solution* Carefully add 100 mL of concentrated (28% (m/m) or ϱ_{20} 0.89 g/mL) ammonia solution to 400 mL of water with stirring.

(g) *Standard sodium chloride solution.*

(h) *Standard potassium sulfate solution.*

NOTE: Protective glasses should be worn when handling concentrated reagents.

5 APPARATUS The following apparatus is required:

(a) 6.7 mm, 1.18 mm and 150 μm sieves complying with AS 1152.

(b) A drying oven capable of operating in the temperature range 100°C to 110°C.

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

NOTE: A ring grinder is recommended.

6 SAMPLING AND SAMPLE PREPARATION

6.1 General Sampling requirements shall be fully detailed by the specifying authority.

NOTES:
1 It is recommended that samples from hardened concrete be taken in accordance with AS 1012.14.
2 Where the materials require special sampling techniques, i.e. dune sands, rainwater-leached and acid-etched surfaces, concrete subject to spray, estuarine and dredged aggregates, guidance is available in BS 1881: Part 124.

6.2 Sample preparation Ideally a minimum of 50 g passing the 150 μm sieve is required, but for investigations of sulfate or chloride penetration in concrete, a total sample of not less than 20 g shall be prepared. 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) Where necessary, grind any cements, fly ashes, slag or other powdered admixtures to pass a 150 μm sieve.

(e) 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 is as follows:

(a) Weigh out an accurate mass of the dried sample as given in Table 1 into a 250 mL beaker.

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