# Methods of testing portland and blended cements

# Method 14: Length change of portland and blended cement mortars exposed to a sulfate solution

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# PREFACE

### Background

Since 1988, and during the development of AS 3972—1991, *Portland and blended cements*, as a performance-based Standard, the cement industry carried out work to develop a suitable test method for sulfate resistance based on the use of ISO/CEN mortar. At the time of publication of the 1991 edition of AS 3972 this work was still continuing.

The work was completed in early 1994 and Committee BD/10 on Cement agreed that AS 3972 be amended to define sulfate resisting cement Type SR in terms of its performance, based on the test method, and to include the test method for sulfate resistance as defined in this Standard.

A brief summary of the work undertaken by the cement industry to develop the test method and performance specification for Type SR cement is given below.

#### Test method development

A preliminary investigation of two methods, ASTM C452 and ASTM C1012, both modified to employ ISO/CEN mortar prisms, was carried out. Five cements were used in the preliminary investigation, including flyash and slag blended cements.

The principle of the ASTM C452 method is to increase the gypsum content of the cement to produce an  $SO_3$  content of 7%. Mortar bars made from this cement are then immersed in water and expansion is measured after 14 days. Results from this test method showed, contrary to documented field performance, that blended cements containing slag exhibited poor performance. This test method was considered not suitable for such cements.

The ASTM C1012 method is based on immersion of mortar bars in sodium sulfate solution for a period of time (up to one year) while measuring their expansion. Results from this test method using ISO/CEN prism  $(40 \times 40 \times 160 \text{ mm})$  showed very small expansions compared to the 25 mm square bars (American test) or the  $10 \times 40$  mm bars (German test). It was obvious then that measuring such small expansions was a source of large errors and poor repeatability of the test resulted.

Additional tests were carried out to develop a more rapid procedure. Initially, the use of thinner bars,  $10 \times 40 \times 160$  mm, produced encouraging results with the cements tested, showing substantially different expansions after relatively short exposure times. It was concluded then that a sulfate resistance test based on the ISO/CEN mortar and a modified prism is feasible.

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Further investigations examined the influences of various curing regimes: 20 MPa maturity, 3-day and 7-day curing before exposure to the sulfate solution, the type of sand and size of specimen. Five different cements were used, including flyash and slag blended cements.

It was concluded from these investigations that a modified ISO/CEN prism of  $15 \times 40 \times 160$  mm should be employed for the test. As the relative order of expansion of the five cements tested did not change with the three curing regimes, the 7-day curing period was recommended as being the most convenient and practical. Also, it was concluded that the determination of expansion after 16 weeks of exposure to the sodium sulfate solution was capable of distinguishing between the cement types and was recommended as the test duration.

Based on these conclusions and recommendations, work advanced to the second part of the investigation to develop the test method further and to determine its precision. It was considered desirable to get as many laboratories as possible to participate in this part of the investigation. An invitation was issued to over 40 organizations and laboratories around Australia to participate in the testing program. In the event, seven laboratories participated.

In the course of this work a number of cement samples were distributed to the seven participating laboratories to carry out ten repeat tests on each of the cements (under conditions of repeatability and reproducibility) to determine the precision of the test method.

#### Principal features of the method

The method uses the mortar and the mould used for the standard strength test in AS 2350.11 and the standard drying shrinkage test in AS 2350.13. The mould is modified to produce specimens with 15 mm thickness and to accept the stude required to measure the change in the length of the specimen.

The specimens are cured for 7 days, i.e. 2 days in the mould and 5 days in lime-saturated water before immersion in the standard sulfate solution. Measurement of the specimen's length is carried out initially after 7-day curing and before immersion in the sulfate solution, and subsequently every 2 weeks and up to 16 weeks of immersion in the sulfate solution.

#### **Expansion** limits

Once the test method had been developed and its precision established, it was possible to proceed with the necessary work to establish appropriate expansion limits for sulfate resisting cement, Type SR.

It was recognized from the outset that before expansion criteria could be suggested or recommended it was important to set principles for setting such limits.

The principles adopted were as follows:

- (a) The method of test should stand on its own merits and no attempt should be made to pass all cements as sulfate resisting cements.
- (b) The limit to be set should not be a world-wide absolute lower value but a value in the lower range for world cements which is technically defensible and internationally competitive.
- (c) The limit set should reflect the performance of cements which have been considered acceptable in sulfate resistance applications.

Based on these principles a database was established to provide the background for setting the limit. The database included various cement types produced in Australia and a number of cements obtained from overseas (South Africa, France, Germany and the USA) of known sulfate resistance performance. These included the so-called moderately sulfate resisting cements.

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Testing of overseas cements served to verify the test method and to assist in placing the expansion values of Australian cements in an international context. In general, moderately sulfate resisting cements are unlikely to meet the AS 3972 limit.

All cements were coded and laboratories had no knowledge of the expected performance, particularly of the overseas cements. The results reported by all laboratories were consistent with the performance reported by the suppliers of the cements. This has further increased confidence in the test method.

# METHOD

**1 SCOPE** This Standard sets out the method for the determination of length change of mortar bars of defined composition immersed in a sodium sulfate solution. The length change is taken as a measure of the sulfate resistance of portland and blended cement.

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

AS

2350	Methods of testing portland and blended cements
2350.11	Method 11: Compressive strength of portland and blended cements
2350.12	Method 12: Preparation of a standard mortar and moulding of specimens
ASTM	
C1012	Test method for length change of hydraulic-cement mortars exposed to a sulfate solution

**3 PRINCIPLE** The method determines length change of prismatic test specimens 40 mm  $\times$  15 mm  $\times$  150 mm in size after immersion in a sulfate solution.

The mortar is prepared and the specimens are moulded generally in accordance with AS 2350.12. The specimens are cured for 7 days before immersion in the standard sulfate solution. Measurement of the specimen length is carried out, initially after the 7-day curing and before immersion in the sulfate solution, and subsequently every 2 weeks and up to 16 weeks after immersion in the sulfate solution.

The standard sulfate solution to be used in this test method contains 0.352 mol/L of Na<sub>2</sub>SO<sub>4</sub> (50g/L). Some technical background on the composition of the test solution is given in ASTM C1012.

NOTE: This test method does not simulate the mechanism of sulfate attack by solutions of sulfate compositions other than that used. If evaluation of behaviour due to exposure to a given sulfate solution is desired, that solution should be used.

# 4 LABORATORY AND EQUIPMENT

**4.1 Laboratory** The conditions of the laboratory shall comply with the requirements of AS 2350.12.

**4.2** Mixer The mixer shall comply with the requirements of AS 2350.12.