METHOD OF TEST FOR ACCELERATED DETECTION OF POTENTIALLY DELETERIOUS ALKALI-SILICA REACTIVE AGGREGATE BY EXPANSION OF MORTAR BARS

1. SCOPE

1.1 This test method allows detecting within 16 days the potential for deleterious expansion of mortar bars due to the alkali-silica reaction.

1.2 This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific precautionary statement is given in Note 5.

2. RELEVANT DOCUMENTS

2.1 ASTM C 109, Test Method for Compressive Strength of Hydraulic Cement Mortars (using 2in or 50-mm Cube Specimens)

2.2 ASTM C 295, Practice for Petrographic Examination of Aggregates for Concrete

2.3 ASTM C 305, Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

2.4 ASTM C 490, Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar, and Concrete

2.5 ASTM C 511, Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

2.6 ASTM C 856, Practice for Petrographic Examination of Hardened Concrete

2.7 ASTM D 1193, Specification for Reagent Water

2.8 ASTM E 11, Specification for Wire-Cloth Sieves for Testing Purposes

3. SIGNIFICANCE AND USE

3.1 This test method provides a means of detecting the potential of an aggregate used in concrete for undergoing alkali-silica reaction and resulting potentially-deleterious internal expansion. It is based on the NBRI Accelerated Test Method [1, 2, 3]. It may be especially useful for aggregates that react slowly or produce expansion late in the reaction.

Note 1: This procedure is similar in all important matters to the CSA version of this test. It differs from CSA in that a standard supply of cement is specified and the use of a laboratory control aggregate is also specified.

3.2 When excessive expansions are developed, it is recommended that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity. Sources of such supplementary information include: (1) petrographic examination of the aggregate (Practice C 295) to determine if known reactive constituents are present; (2) examination of the specimens after tests (Practice C 856) to identify the products of alkali reactivity.

3.3 When it has been concluded from the results of tests performed using this test method and supplementary information that a given aggregate should be considered potentially-deleteriously reactive, additional studies, possibly using alternative methods, may be appropriate to develop further information on the potential reactivity.

4. APPARATUS

4.1 The apparatus shall conform to Specification C 490, except as follows:

4.1.1 Sieves: Square hole, woven-wire cloth sieves shall conform to ASTM E 11.

4.1.2 Mixer, Paddle, and Mixing Bowl: Mixer, paddle, and mixing bowl shall conform to the requirements of Practice C 305, except that the clearance between the lower end of the paddle and the bottom of the bowl shall be 5.1 ± 0.3 mm.

4.1.3 Tamper and Trowel: The tamper and trowel shall conform to Test Method C 109.

4.1.4 Containers: The containers must be of such a nature that the bars can be totally immersed in either the water or 1N NaOH solution. The containers must be made of material that can withstand prolonged exposure to 80°C and must be inert to a 1N NaOH solution (Note 2). The containers must be so constructed that, when used for storing specimens, the loss of moisture is prevented by tight-fitting covers, by sealing, or both (Note 3). The bars in the solution must be placed and supported so that the solution has access to the whole of the bar, therefore, the specimens must not be allowed to touch the sides of the container or each other. The specimens, if placed on end in the solution, shall not be supported by the metal gauge stud.

Note 2: The NaOH solution will corrode glass or metal containers.

Note 3: A covered container that has been found to be acceptable for this purpose is sold by Rubbermaid as a microwave-proof food storage container.

4.1.5 Oven: A convection oven with temperature control maintaining $80.0 \pm 2.0^{\circ}$ C.

5. REAGENTS AND MATERIALS

5.1 Sodium Hydroxide (NaOH): USP or technical grade may be used, provided the Na⁺ and OH⁻ concentrations are shown by chemical analysis to lie between 0.99N and 1.01N.

5.2 Purity of Water: Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

5.3 Sodium Hydroxide Solution: Each litre of solution shall contain 40.0 g of NaOH dissolved in 900 mL of water, and shall be diluted with additional distilled or deionized water to obtain 1.0 L of solution. The volume proportion of sodium hydroxide solution to mortar bars in a storage container shall be 4.0 ± 0.5 volumes of solution to 1 volume of mortar bars (Note 4).

Note 4: The volume of a mortar bar may be taken as 184 mL.

Note 5: **Precaution**: Before using NaOH, review: (1) the safety precautions for using NaOH; (2) first aid for burns; and (3) the emergency response to spills, as described in the manufacturer's Materials Safety Data Sheet or other reliable safety literature. NaOH can cause very severe burns and injury to unprotected skin and eyes. Suitable personal protective equipment should always be used. These should include full-face shields, rubber aprons, and gloves impervious to NaOH. Gloves should be checked periodically for pin holes.

5.4 Control Aggregate: A supply of 'Spratt' alkali-silica reactive aggregate prepared as described in 7.2. This aggregate is available from: The Soils and Aggregates Section, Ministry of Transportation, 1201 Wilson Avenue, Downsview, Ontario, M3M 1J8.

6. CONDITIONING

6.1 Maintain the temperature of the molding room and dry materials at not less than 20°C and not more than 27.5°C. The temperature of the mixing water, and of the moist closet or moist room, shall not vary from 23°C by more than 1.7°C.

6.2 Maintain the relative humidity of the molding room at not less than 50 % . The moist closet or room shall conform to Specification C 511.

6.3 Maintain the storage oven in which the specimens are stored in the containers at a temperature that shall not vary from 80°C by more than 2.0°C.

7. SAMPLING AND PREPARATION OF TEST SPECIMENS

7.1 Process materials proposed for use as fine aggregate in concrete as described in 7.2 with a minimum of crushing. Process materials proposed for use as coarse aggregates in concrete by crushing to produce as nearly as practical a graded product from which a sample can be obtained. The sample shall have the grading as prescribed in 7.2, and be representative of the composition of the coarse aggregate as proposed for use.

7.1.1 When a given quarried material is proposed for use both as coarse and as fine aggregate, test it only by selection of an appropriate sample crushed to the fine aggregate sizes, unless there is reason to expect that the coarser size fractions have a different composition than the finer sizes and that these differences might significantly affect expansion due to reaction with the alkalies in cement. In this case, test the coarser size fractions in a manner similar to that employed in testing the fine aggregate sizes.

7.2 Preparation of Aggregate: Grade all aggregates to which this test method is applied in accordance with the requirements prescribed in Table 1. Crush aggregates in which sufficient

quantities of the sizes specified in Table 1 do not exist until the required material has been produced. In the case of aggregates containing insufficient amounts of one or more of the larger sizes listed in Table 1 and, if no larger material is available for crushing, the first size in which sufficient material is available shall contain the cumulative percentage of material down to that size as determined from the grading specified in Table 1. When such procedures are required, make a special note in the test report. After the aggregate has been separated into the various sieve sizes, wash each size with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. Dry the portions retained on the various sieves and, unless used immediately, store each portion individually in a clean container provided with a tight-fitting cover.

Sieve Size		Mass, %
Passing	Retained	
4.75 mm	2.36 mm	10
2.36 mm	1.18 mm	25
1.18 mm	600 µm	25
600 µm	300 µm	25
300 µm	150 µm	15

Table 1 Grading Requirements

7.3 Preparation of Cement: Pass cement for use in this test through a 850 µm sieve to remove lumps before use.

7.4 Preparation of Test Specimens:

7.4.1 Number of Specimens: Make at least three test specimens for each cement-aggregate combination.

7.4.2 Preparation of Molds: Prepare the specimen molds in accordance with the requirements of Specification C 490, except that the interior surfaces of the mold shall be covered with a release agent (Note 6). A release agent will be acceptable if it serves as a parting agent without affecting the setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen.

Note 6: TFE-Fluorocarbon tape complies with the requirements for a mold release agent..

7.4.3 Proportioning of Mortar: Proportion the dry materials for the test mortar using 1 part of cement to 2.25 parts of graded aggregate by mass. The quantities of dry materials to be mixed at one time in the batch of mortar for making three specimens shall be 440 g of cement and 990 g of aggregate made up by re-combining the portions retained on the various sieves (see 7.2) in the grading prescribed in Table 1. For natural fine aggregates, use a water to cement ratio equal to 0.44

by mass. For coarse aggregates or manufactured sands, use a water to cement ratio equal to 0.50 by mass (Note 7).

Note 7: Ruggedness tests indicated that mortar bar expansions were less variable at a fixed water to cement ratio than when gauged to a constant flow [3]. The water to cement ratios selected should give acceptable workability in most cases [4].

7.4.4 Mixing of Mortar: Mix the mortar in accordance with the requirements of Practice C 305.

7.4.5 Molding of Test Specimens: Mold test specimens with a total elapsed time of not more than 2 min and 15 sec after completion of the original mixing of the mortar batch. Fill the molds with two approximately equal layers, each layer being compacted with the tamper. Work the mortar into the corners, around the gauge studs, and along the surfaces of the mold with the tamper until a homogeneous specimen is obtained. After the top layer has been compacted, cut off the mortar flush with the top of the mold and smooth the surface with a few strokes of the trowel.

8. PROCEDURE

8.1 Initial Storage and Measurement: Place each mold in the moist cabinet or room immediately after molds have been filled. The specimens shall remain in the molds for 24 ± 2 h. Remove the specimens from the molds and, while they are being protected from loss of moisture, properly identify and measure for initial length. Make and record the initial and all subsequent measurements to the nearest 0.002 mm. Place the specimens made with each aggregate sample in a storage container with sufficient tap water to totally immerse them. Seal and place the containers in an oven or room at $80 \pm 2.0^{\circ}$ C for a period of 24 h.

8.2 Zero Measurements: Remove the containers from the oven one at a time. Remove other containers only after the bars in the first container have been measured and returned to the oven. Remove the bars one at a time from the water and dry their surfaces with a towel paying particular attention to the two metal gauge measuring studs. Take the zero measurement (Note 8) of each bar immediately after drying, and read as soon as the bar is in position. Complete the process of drying and measuring within 15 ± 5 s of removing the specimen from the water. After measurement, leave the specimen on a towel until the remainder of the bars have been measured. Place all three specimens in a container with the 1N NaOH, at 80 ± 2.0 °C. The samples must be totally immersed. Seal the container and return it to the oven.

Note 8: The comparator bar should be measured prior to each set of specimens since the heat from the mortar bars may cause the length of the comparator to change.

8.3 Subsequent Storage and Measurement: Undertake subsequent measurement of the specimens periodically, with at least three intermediate readings for 14 days after the zero reading, at approximately the same time each day. The measuring procedure is identical to that described in 8.2, except that the specimens are returned to their own container after measurement. If the mean

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expansion of the mortar bars exceeds 0.14 % at 14 days, the bars shall be returned to the container and stored for a total of 28 days. At least one reading per week shall be taken between 14 and 28 days. If the expansion after 28 days exceeds 0.30 % the bars shall again be returned to the container and stored for a total of 56 days. At least one reading per week shall be taken between 28 and 56 days.

9. CALCULATION

9.1 Calculate the difference between the zero length of the specimen and the length at each period of measurement to the nearest 0.001 % of the effective gauge length and record as the expansion of the specimen for that period. Report the average expansion of the three specimens of a given cement-aggregate combination to the nearest 0.001 % as the expansion for the combination for a given period.

10. **USE OF CONTROL AGGREGATE**

Every 10 samples, but at least once every six months in which a sample is tested, a sample 10.1 of the control aggregate described in 5.4 shall be tested. The mean multi-laboratory expansion of this aggregate with high alkali cement has been found to be 0.42 % with a standard deviation of 0.062 % at 14 days in solution [5]. Therefore results obtained with this material should fall in the range 0.30 - 0.54 %, nineteen times in twenty.

11. REPORT

11.1 The report shall include the following:

11.1.1 Type and source of aggregate,

11.1.2 Alkali content of cement as percent potassium oxide (K₂O), sodium oxide (Na₂O), and calculated sodium oxide (Na₂O) equivalent, if the standard cement was not used,

11.1.3 Average length change in percent at each reading of the specimens,

11.1.4 Any relevant information concerning the preparation of aggregates, including the grading of the aggregate when it differs from that given in 7.2,

11.1.5 Any significant features revealed by examination of the specimens during and after test.

11.1.6 Amount of mixing water expressed as mass percent to cement,

11.1.7 Type, source, proportions, and chemical analyses, including Na₂O and K₂O, of any pozzolans employed in the tests,

11.1.8 A graph of the length change data from the time of the zero reading to the end of the period of testing.

11.1.9 A graph of the length change data from the time of the zero reading to the end of the 14-day period of the aggregate used as a control.

12. PRECISION

12.1 Within-Laboratory Test Variation: It has been found that the average within-laboratory coefficient of variation for materials with an average expansion greater than 0.1% at 14 days is 2.94%*. Therefore, the results of two properly conducted tests within the same laboratory on specimens of a sample of aggregate should not differ by more than 8.3%* of the mean expansion, 19 times out of 20.

*These numbers represent, respectively, the 1s% and d2s% limits as described in ASTM Standard C 670

12.2 Multi-Laboratory Variation: It has been found that the average multi-laboratory coefficient of variation for materials with an average expansion greater than 0.1% at 14 days is 15.2%*. Therefore, the results of two properly conducted tests within the same laboratory on specimens of a sample of aggregate should not differ by more than 43%* of the mean expansion, 19 times out of 20.

*These numbers represent, respectively, the 1s% and d2s% limits as described in ASTM Standard C 670

13. **REFERENCES**

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