

# Determining the Potential Alkali-Silica Reactivity of Combinations of Cementitious Materials, Lithium Nitrate Admixture and Aggregate (Accelerated Mortar-Bar Method)

## 1. Scope

1.1 This test method permits detection within 30 days of the potential for deleterious alkali-silica reaction of combinations of cementitious materials, lithium nitrate admixture and aggregate in mortar bars. The cementitious materials are composed of various proportions of hydraulic cement, pozzolans and ground granulated blastfurnace slag.

1.2 The test results are only valid for the specific combinations of pozzolan, slag, lithium nitrate admixture and reactive aggregates tested.

1.3 This test is not suitable for evaluating the potential for deleterious reaction of combinations of hydraulic cement and aggregate (that is, in the absence of pozzolans, ground granulated blast-furnace slag or lithium nitrate admixture).

1.4 The values stated in SI units are to be regarded as standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific precautionary statement is given in the section on Reagents.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C 125 Terminology Relating to Concrete and Concreting Aggregates

C 150 Specification for Portland Cement

C 151 Test Method for Autoclave Expansion of Portland Cement

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

C 490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete

C 494/C 494M Specification for Chemical Admixtures for Concrete

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

C 618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C 989 Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars

C 1240 Specification for Silica Fume for Use as a Mineral Admixture in Hydraulic-Cement Concrete, Mortar or Grout

C 1260 Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)

C 1293 Test Method for Concrete Aggregates by Determination of Length Change of Concrete Due to Alkali-Silica Reaction

C 1437 Test Method for Determining Flow of Hydraulic Cement Mortar

D 1193 Specification for Reagent Water

E 11 Specification for Wire-Cloth and Sieves for Testing Purposes

## 3. Definitions

3.1 For definitions of terms used in this test method, refer to Terminology ASTM C 125.

## 4. Significance and Use

4.1 This test method provides a means for evaluating the ability of pozzolans, ground granulated blast-furnace slag, lithium nitrate admixture, and combinations of these, to control deleterious internal expansion due to alkali-silica reaction when used with an aggregate intended for use in concrete. It is based on the Accelerated Test Method developed at the National Building Research Institute (NBRI) in the Republic of South Africa (1-4).

4.2 Results obtained using this test method may overestimate the reactivity of some types of aggregates if used in service with the same pozzolans, slag, lithium admixture and hydraulic cement of low alkali content.

4.3 Different levels of pozzolan, ground granulated blast-furnace slag and lithium nitrate admixture may require testing to determine the amount required to reduce expansion to an acceptable level. Pozzolans, ground granulated blast-furnace slag and lithium nitrate admixture may be tested separately or in combination.

4.4 It is recommended to test the same aggregate and hydraulic cement (without pozzolan, slag or lithium nitrate admixture) using Test Method C 1260.

4.5 This test method may underestimate the expansion of cementitious systems containing pozzolans with an alkali content > 4.0 % sodium oxide equivalent (5-8). It is recommended that such materials be tested using Test Method C 1293.

4.6 In the case of testing lithium nitrate admixture, this test method is only applicable for testing the admixture in the range from 50% to 150% dose.

## 5. Apparatus

5.1 The apparatus shall conform to Practice C 490, except as follows:

5.2 *Sieves*—Square hole, woven-wire cloth sieves, shall conform to Specification E 11.

5.3 *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  $5.1 \pm 0.3$  mm.

5.4 *Tamper and Trowel*—The tamper and trowel shall conform to Test Method C 109/C 109M.

5.5 *Containers*—The containers shall be of such a nature that the bars can be totally immersed in either the water or 1N sodium hydroxide (NaOH) solution. The containers shall be made of material that can withstand prolonged exposure to 80°C and must be resistant to a 1N NaOH solution (see Note 1). The containers must be so constructed that when used for storing specimens, the loss or gain of moisture is prevented by tight-fitting covers, by sealing, or both (see Note 2). The bars in the solution must be placed and supported so that the solution has access to the entire surface of each bar; therefore, ensure that the specimens do not touch the sides of the container or each other. The specimens, if stood

upright in the solution, shall not be supported by the metal gage stud.

**NOTE 1—The NaOH solution corrodes glass or metal containers.**

**NOTE 2—Some microwave-proof food storage containers made of polypropylene or high-density polyethylene have been found to be acceptable.**

5.6 *Oven, or Water Bath*—A convection oven or water bath with temperature control maintaining  $80.0 \pm 2.0^\circ\text{C}$ .

## 6. Reagents

6.1 *Sodium Hydroxide (NaOH)*—USP or technical grade may be used, provided the Na<sup>+</sup> and OH<sup>-</sup> concentrations are shown by chemical analysis to lie between 0.99N and 1.01N.

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

6.3 *Alkaline Soaking Solution*—Each liter of alkaline soaking solution shall contain 40.0 g of NaOH dissolved in 800 ml of water. If lithium nitrate admixture is used in the test, add 71 ml of the commercially available admixture multiplied by the decimal equivalent of the lithium nitrate admixture dose. (For example, to test a 75% lithium admixture dosage, each liter of alkaline soaking solution will contain 0.75 times 71 ml of commercially available lithium nitrate admixture.) This mixture shall be diluted with additional distilled or deionized water to obtain 1.0 liter of solution. The volume proportion of soaking solution to mortar bars in a storage container shall be  $4 \pm 0.5$  volumes of solution to 1 volume of mortar bars. The volume of a mortar bar may be taken as 184 ml. Include sufficient test solution to ensure complete immersion of the mortar bars.

6.3.1 **Warning**—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 Material 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 must always be used. These include full-face shields, rubber aprons, and gloves impervious to NaOH. Gloves must be checked periodically for pinholes.

## 7. Conditioning

7.1 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 2°C.

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

7.3 Maintain the storage oven or water bath in which the specimens are stored at a temperature of  $80.0 \pm 2.0^\circ\text{C}$ .

## 8. Sampling and Preparation of Test Specimens

8.1 *Selection of Aggregate*—Process materials proposed for use as fine aggregate in concrete as described in 8.2 with a minimum of crushing. Process materials proposed for use as coarse aggregate in concrete by crushing to produce as nearly as practical a graded product from which a sample can be obtained. Grade the sample as prescribed in Table 1. The sample shall represent the composition of the coarse aggregate as proposed for use.

8.1.1 When a given quarried material is proposed for use both as coarse and as fine aggregate, test only a selection of the fine aggregate, unless there is reason to expect that the coarse aggregate has a different composition than the fine aggregate. If such a difference is expected and if the differences might significantly affect expansion due to reaction with the alkalis in cement or from the environment of service, test the coarse aggregate in a manner similar to that employed in testing the fine aggregate.

Table 1 Grading Requirements		
Sieve Size		Mass, %
Passing	Retained on	
4.75 mm (No. 4)	2.36 mm (No. 8)	10
2.36 mm (No. 8)	1.18 mm (No. 16)	25
1.18 mm (No. 16)	600 $\mu\text{m}$ (No. 30)	25
600 $\mu\text{m}$ (No. 30)	300 $\mu\text{m}$ (No. 50)	25
300 $\mu\text{m}$ (No. 50)	150 $\mu\text{m}$ (No. 100)	15

8.2 *Preparation of Aggregate*—Grade aggregates to provide a sample meeting the requirements given 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 thereof 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 such portion individually in a clean container provided with a tight-fitting cover.

### 8.3 Selection and Preparation of Cement:

8.3.1 *Hydraulic Cement*—Use a hydraulic cement meeting the requirements of Specification C 150 (Note 3). In addition, the autoclave expansion in Test Method C 151 shall be less than 0.20 %.

**NOTE 3**—The alkali content of the cement has been found to have negligible (3) or minor (6) effects on expansion in this test.

8.3.2 *Preparation of Cement*—Pass cement for use in this test through an 850- $\mu\text{m}$  (No. 20) sieve to remove lumps before use.

8.4 *Selection of pozzolan, ground granulated blastfurnace Slag or lithium nitrate admixture*—Use one, or a combination, of the following:

8.4.1 Fly ash or natural pozzolan meeting the requirements of Specification C 618.

8.4.2 Silica fume meeting the requirements of Specification C 1240.

8.4.3 Ground granulated blast furnace slag meeting the requirements of Specification C 989.

8.4.4 Commercially available lithium nitrate admixture which nominally contains  $30 \pm 0.5$  wt% lithium nitrate admixture in water.

### 8.5 Preparation of Test Specimens:

8.5.1 *Number of Specimens*—Make at least three test specimens for each cementitious materials-aggregate combination.

8.5.2 *Preparation of Molds*—Prepare the specimen molds in accordance with the requirements of Practice C 490 except, the interior surfaces of the molds shall be covered with a release agent (see Note 4). A release agent is acceptable if it serves as a

parting agent without affecting the time of setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen.

**NOTE 4—TFE-fluorocarbon (Teflon) tape complies with the requirements for a mold release agent. (Teflon is a registered trademark.)**

**8.5.3 Proportioning of Mortar**—Proportion the dry materials for the test mortar using 1 part of cementitious materials (hydraulic cement plus pozzolan or ground granulated blastfurnace slag) 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 cementitious material and 990 g of aggregate made up by recombining the portions retained on the various sieves (see the section on Preparation of Aggregate) in the grading prescribed in Table 1. Use a water-cementitious material ratio equal to 0.47 by mass (see Note 5).

**NOTE 5—Ruggedness tests indicated that mortar bar expansions were less variable at a fixed water-cement ratio than when gauged to a constant flow (3).**

If silica fume or metakaolin are used, a high range water reducer (HRWR), meeting the requirements of Specification C 494/C 494M Type F, shall be used (if necessary) to provide adequate dispersion and workability of the mixture. The water-cementitious material ratio shall remain 0.47 by mass and the amount of HRWR shall be that to obtain a flow of  $\pm 7.5$  percentage points of a control mortar without silica fume or metakaolin as determined in accordance with Test Method C 1437 using 25 drops of the flow table. If liquid HRWR is used, the water present in the liquid must be included in the calculation of the water-cementitious material ratio. If lithium nitrate admixture is used, a 100% dose equals 0.0493 times the grams of portland cement in the mortar batch, on a mass basis. To maintain the 0.47 w/c, subtract 0.7 times the grams of lithium nitrate admixture used in the batch from the mixing water. For dosages higher or lower than 100%, simply multiply the 100% dose by the decimal equivalent of the percent of the 100% dose. (For example, for a 75% dose, multiply 0.75 times 0.0493 times the grams of portland cement in the mortar batch.) Or, for a volumetric basis, a 100% dose equals 0.0411 [ml/g] times the grams of portland cement in the mortar batch. If the lithium nitrate admixture dose is being calculated on a volumetric basis, then to maintain the 0.47 w/c, subtract 0.84 times the number of ml of lithium nitrate admixture used in the batch from the mixing water.

**8.5.4 Mixing of Mortar**—Mix the mortar in accordance with the requirements of Practice C 305.

**8.5.5 Molding of Test Specimens**—Mold test specimens within a total elapsed time of not more than 2 min and 15 s 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 gage 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.

## 9. Procedure

**9.1 Initial Storage and Reading**—Place each mold in the moist cabinet or room immediately after molds have been filled. The specimens shall remain in the molds for  $24 \pm 2$  h. Remove the specimens from the molds and, while they are being protected from loss of moisture, properly identify and make an initial comparator reading. Make and record the initial and all subsequent readings 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 water bath at  $80.0 \pm 2.0^\circ\text{C}$  for a period of  $24 \text{ h} \pm 2 \text{ h}$ .

**9.2 Zero Readings**—Remove the containers from the oven or water bath one at a time. Remove other containers only after the bars in the first container have been measured and returned to the oven or water bath. Remove the bars one at a time from the water and dry their surface with a towel paying particular attention to the two metal gage studs. Take the zero reading (see Note 6) of each bar immediately after drying, and read as soon as the bar is in position. Complete the process of drying and reading within  $15 \pm 5$  s of removing the specimen from the water. After taking comparator readings, leave the specimen on a towel until comparator readings have been taken on the remainder of the bars. Place all specimens made with each unique cementitious materials-aggregate combination in a separate container with sufficient alkaline soaking solution, at  $80.0 \pm 2.0^\circ\text{C}$  for the specimens to be totally immersed. Seal the container and return it to the oven or water bath.

**NOTE 6—The reference bar should be read prior to each set of specimens since the heat from the mortar bars may cause the length of the comparator to change.**

### 9.3 *Subsequent Storage and Measurement*—

Make subsequent comparator readings of the specimens periodically, at 2 to 3 day intervals, for 28 days after the zero reading, at approximately the same time each day. If readings are continued beyond the 28-day period, take at least one reading per week. The procedure is identical to that described in the 9.2, except that the specimens are returned to their own container after measurement.

## 10. Calculation

10.1 Calculate the difference between the zero comparator reading of the specimen and the reading at each period to the nearest 0.001 % of the effective gage length and record as the expansion of the specimen for that period. Report the average expansion of the three specimens of a given cementitious materials-aggregate combination to the nearest 0.01 % as the expansion for the combination for a given period.

## 11. Report

11.1 Report the following information:

11.1.1 Type and source of aggregate,

11.1.2 Type and source of hydraulic cement,

11.1.3 Type, source, and proportions of pozzolan

11.1.4 Grade, source, and proportions of ground granulated blast-furnace slag

11.1.5 Source and percent dosage of lithium nitrate admixture

11.1.6 Autoclave expansion and alkali content of cement as percent potassium oxide ( $K_2O$ ), sodium oxide ( $Na_2O$ ), and calculated sodium oxide ( $Na_2O$ ) equivalent ( $Na_2O_{eq} = \%Na_2O + 0.658 \times \%K_2O$ ),

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

11.1.8 Any relevant information concerning the preparation of aggregates, including the grading of the aggregate when it differs from that given in 8.2,

11.1.9 Any significant features revealed by examination of the specimens during and after test,

11.1.1 Amount of mixing water expressed as a mass fraction of the total cementitious material,

11.1.11 A graph of the length change data from the time of the zero reading to the end of the 30 day period.

## 12. Precision and Bias

12.1 The following precision statements are taken from Test Method C 1260, and are based on tests without pozzolans, ground slag or lithium nitrate.

12.2 *Within-Laboratory Precision*—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 % (9) (Note 7). 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 % (Note 7) of the mean expansion.

12.3 *Multi-Laboratory Precision*—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 % (9) (Note 7). Therefore, the results of two properly conducted tests in different laboratories on specimens of a sample of aggregate should not differ by more than 43 % (Note 7) of the mean expansion.

**NOTE 7—These numbers represent, respectively, the (1s %) and (d2s %) limits as described in Practice C 670.**

12.4 *Bias*—Since there is no accepted reference material for determining the bias of this test method, no statement on bias is being developed.

## 13. Keywords

13.1 aggregate; alkali-silica reactivity; length change; mortar; pozzolans, slag, sodium hydroxide, lithium nitrate

## REFERENCES

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