8 Conditioning

8.1

The temperature of the moulding room, apparatus, and dry materials shall be determined at not less than 20 °C and not more than 26 °C. The temperature of the mixing water and of the moist closet or moist room shall be 23 ± 2.0 °C.

8.2

The relative humidity of the moulding room shall be maintained at not less than 50%. The moist closet or room, if used, shall conform to ASTM C511.

8.3

The storage oven in which the specimens are stored in the containers shall be maintained at a temperature of 80 ± 2.0 °C.

9 Sampling and preparation of test specimens

9.1 General

9.1.1

A sample of the aggregate to be tested shall be obtained in accordance with CSA A23.2-1A. Samples for alkali-aggregate reaction testing shall, if possible, be obtained from a stockpile of aggregate produced in a manner identical to that which will be used in subsequent production.

9.1.2

Bulk samples shall be reduced to test specimen size in accordance with the procedures of CSA A23.2-2A, omitting where relevant the requirement about not preparing samples of an exact predetermined mass.

9.1.3

Materials proposed for use as fine aggregate in concrete shall be processed as described in Clause 9.2 with a minimum of crushing. Materials proposed for use as coarse aggregates in concrete shall be processed by crushing to produce, as nearly as practicable, a graded product from which a sample can be obtained. The sample shall have the grading specified in Table 1 and be representative of the composition of the coarse aggregate as proposed for use.

9.1.4

When a given quarried material is proposed for use both as coarse and as fine aggregate, each aggregate shall be tested.

9.2 Grading

All aggregates to which this Test Method is applied shall be graded in accordance with the requirements in Table 1. Aggregates in which sufficient quantities of the sizes specified in Table 1 do not exist shall be crushed until the required material has been produced. If aggregates contain 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, a special note shall be made to that effect in the test report. After the aggregate has been

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separated into the various sieve sizes, each size shall be washed with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. The portions retained on the various sieves shall be dried and, unless used immediately, stored individually in a clean container provided with a tight-fitting cover.

Note: In order to produce a graded aggregate sample that is representative of the original coarse aggregate material, the following procedure may be used:

- a) Start with a representative 6 kg (including 2 kg of each fraction 5 to 20 mm) subsample prepared by quartering or other suitable means to ensure a representative portion of the original sample collected following CSA A23.2-1A.
- b) Using a small jaw crusher (or other appropriate equipment), crush the coarse aggregate particles by multiple passes.
- c) Sieve the material over a 5 mm sieve between each pass until all material passes the sieve. Care should be exercised not to close the opening between the jaws or the disks too rapidly because this can produce significant amounts of fine dust.
- d) Separate the material into the various size fractions required for the test.
- e) If insufficient quantities of some of the fractions are produced, ground the particles using a disk pulverizer by progressive passes. The material can be sieved over a suitable sieve between each pass until all material passes the sieve. The material is then separated into the smaller size fractions and blended with the previously produced material.

Table 1Grading requirements(See Clauses 9.1.1, 9.2, and 9.4.3.)

Sieve size					
Passing	Retained	Mass, %			
5 mm	2.5 mm	10			
2.5 mm	1.25 mm	25			
1.25 mm	630 µm	25			
630 μm	315 µm	25			
315 μm	160 µm	15			

9.3 Cement

Portland cement meeting the requirements of Clause 7.4 shall be used. Cement for use in this test shall be passed through a 710 μ m sieve to remove lumps before use.

9.4 Preparation of test specimens

9.4.1

At least three test specimens shall be made for each aggregate.

9.4.2

The specimen moulds shall be prepared in accordance with the requirements of ASTM C490/C490M, except that the interior surfaces of the mould shall be covered with a release agent.* A release agent shall be suitable 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.

* TFE-Fluorocarbon (Teflon) tape complies with the requirements for a mould release agent.

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9.4.3

The dry materials for the test mortar shall be proportioned using 1 part cement to 2.25 parts 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 recombining the portions retained on the various sieves (see Clause 9.2) in the grading prescribed in Table 1. For natural fine aggregates, a water-to-cement ratio equal to 0.44 by mass shall be used. For crushed coarse aggregates or manufactured sands, a water-to-cement ratio equal to 0.50 by mass shall be used.

Notes:

- 1) Ruggedness tests indicate that mortar bar expansions were less variable at a fixed water-to-cement ratio than when gauged to a constant flow.
- 2) The water-to-cement ratios selected should give acceptable workability in most cases.

9.4.4

The mortar shall be mixed in accordance with the requirements of ASTM C305.

9.4.5

Test specimens shall be moulded with a total elapsed time of not more than 2 min and 15 s after completion of the original mixing of the mortar batch. The moulds shall be filled with two approximately equal layers, each layer being compacted with the tamper. The mortar shall be worked into the corners, around the gauge studs, and along the surfaces of the mould with the tamper until a homogeneous specimen is obtained. After the top layer has been compacted, the mortar shall be cut off flush with the top of the mould and shall be smoothed with a few strokes of the trowel.

10 Procedure

10.1

The procedure shall be performed as specified in Clauses 10.2 and 10.3.

10.2

Place each mould in the moist cabinet or room immediately after moulds have been filled. Alternatively, the mortar bar moulds may be covered with wet burlap or placed over water in a sealed container, and maintained at 23 ± 2.0 °C. When burlap is used, it shall be saturated but not dripping and shall cover the top and sides of the samples without being in direct contact with the mortar surface. The burlap shall be completely covered with a polyethylene sheet in such a manner as to prevent drying of the burlap.

Leave the specimens in the moulds for 24 ± 2 h. Remove the specimens from the moulds 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, at room temperature, to totally immerse them. Seal and place the containers in an oven at 80 ± 2.0 °C for a period of 24 h.

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 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. Ensure that the elapsed time between removal of the container from the oven and completion of the measurements is no more than 5 min. After measurement, leave the specimen on a

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towel until the remainder of the bars have been measured. Place all three specimens in a container with the 1 N NaOH, preheated at 80 \pm 2.0 °C. Totally immerse the samples. Seal the container and return it to the oven.

* The comparator bar should be measured prior to each set of specimens since the heat from the mortar bars might cause the length of the comparator to change. The lower measuring stud of the comparator should be wiped dry after each measurement to prevent corrosion.

10.3

Undertake subsequent measurements of the specimens periodically, with at least three intermediate readings for 14 d after the zero reading, at approximately the same time each day. In some cases, if measurements are continued beyond the 14 d period, take at least one reading per week. Follow the measuring procedure described in Clause 10.2, but return the specimens to their containers after measurement.

11 Calculation

The difference between the zero length of the specimen and the length at each period of measurement shall be calculated to the nearest 0.001% of the effective length and recorded as the expansion of the specimen for that period. The average expansion of the three specimens shall be reported to the nearest 0.01% as the expansion for a given period.

12 Use of a control material

12.1

When testing is conducted, the laboratory shall demonstrate its ability to conduct the test. At the time of testing or at least every six months, testing with a known reactive aggregate shall be conducted.

12.2

As a means of validating the testing laboratory and validating the testing process, Spratt aggregate shall be tested. After 14 d in solution, the expansion of mortar bars made with Spratt aggregate shall be between 0.30% and 0.55%. After 28 d of testing, the expansion shall be between 0.47% and 0.98%. When expansion data are obtained that fall outside these limits, mortar cast with aggregates from the beginning of the first test of the Spratt aggregate until the beginning of the next test with Spratt aggregate shall be retested.

Note: Spratt coarse aggregate is available in 25 kg bags from the Soils and Aggregates Section, Materials Engineering and Research Office, Ontario Ministry of Transportation (Soils-Aggregates@ontario.ca).

13 Reporting

13.1 Required information

The following information shall be included in the test report, as applicable:

- a) sample number or identification marks;
- b) type and source of aggregate, location of aggregate within the source (e.g., bench level, area within a pit, etc.);
- c) type and source of Portland cement;
- d) alkali content of cement as percentage potassium oxide (K₂O), sodium oxide (Na₂O), and calculated sodium oxide (Na₂O) equivalent;
- e) average length change percentage at each reading of the specimens;

- f) any relevant information concerning the preparation of aggregates, including the grading of the aggregate when it differs from that given in Clause 8.2;
- g) any significant features revealed by examination of the specimens and the sodium hydroxide solution during and after the test;*
- h) amount of mixing water expressed as water-to-cement ratio;
- i) a graph of the length change data from the time of the zero reading to the end of the period of testing;
- j) a graph of the length change data from the time of the zero reading to the end of the 14 d period of the control aggregate;
- k) expansion of mortar bars made with the Spratt aggregate tested closest in time to that of the results of the sample being reported;
- I) name of the certified technician who performed the test;
- m) identification of the certified laboratory that performed the test (i.e., name and address);
- n) name and signature of the person responsible for the review and approval of the test report; and
- o) any deviations from the test procedure.

* In some cases, the solution might become cloudy due to the presence of alkali-silica gel.

13.2 Optional information

The following information may be included in the test report:

- a) name of the person or organization who sampled the aggregate; and
- b) date the sample was taken or received by testing laboratory.

14 Precision

14.1 Single-laboratory precision

It has been found that the average single-laboratory coefficient of variation for materials with an average expansion greater than 0.1% at 14 d 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 C670.

14.2 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 d is 15.2%.* 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%* of the mean expansion, 19 times out of 20.

* These numbers represent, respectively, the 1S% and D2S% limits as described in ASTM C670.

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A23.2-26A Determination of potential alkali-carbonate reactivity of quarried carbonate rocks by chemical composition

1 Scope

This Test Method specifies the laboratory procedure for testing quarried carbonate aggregates by chemical analysis to determine whether they are potentially alkali-carbonate expansive.

2 Reference publications

This Test Method refers to the following publications, and where such reference is made, it shall be to the editions listed below, including all amendments published thereto:

CSA Group

A23.2-1A:19 Sampling aggregates for use in concrete

A23.2-2A:19 Sieve analysis of fine and coarse aggregate

A23.2-14A:19

Potential expansivity of aggregates (procedure for length change due to alkali-aggregate reaction in concrete prisms at 38 °C)

ASTM International

C25-17 Standard Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime

C670-15 Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction

C702/C702M-18

Standard Practice for Reducing Samples of Aggregate to Testing Size

Other publications

Norrish, K., and Chappell, B.W. 1977. X-ray Fluorescence Spectrometry. *Physical Methods in Determinative Mineralogy*, 201–272. Ed. J. Zussman. London: Academic Press.

Rogers, C.A. 1986. Evaluation of the Potential for Expansion and Cracking of Concrete Caused by the Alkali-Carbonate Reaction. *Cement, Concrete and Aggregates*, ASTM, Summer: 13–23.

Shapiro, L. 1975. *Rapid Analysis of Silicate, Carbonate and Phosphate Rocks (Revised Edition)*. United States Geological Survey Bulletin 1401.

Shehata, M. H., Jagdat, S., Rogers, C., and Lachemi, M. 2017. Long-term effects of different cementing blends on alkali-carbonate reaction. *ACI Materials Journal*, 114(4): 661–672.

3 Significance and use

3.1

Deleteriously expansive alkali-carbonate reactive (ACR) aggregates in Ontario are dolomitic limestones with a high clay or acid insoluble residue content. Dolomitic limestone can be recognized by determining the CaO:MgO ratio. Clay content can be determined by measuring alumina (Al₂O₃) content (Rogers, 1986). This Test Method has been found to be a useful screening procedure for expansive carbonate rock in Ontario; it might not have similar application elsewhere. Users of this Test Method in other geographic locations should establish the usefulness of this Test Method with suspect alkalicarbonate reactive rocks in their area.

3.2

Carbonate rocks that are not alkali-carbonate reactive might be alkali-silica reactive. This Test Method does not determine the potential for alkali-silica reactivity.

3.3

Recent work using laboratory blends of known Ontario ACR highly expansive dolomitic limestone with known non-expansive dolostone have shown that the test did not predict the expansivity in all cases. Aggregate blends containing expansive aggregate contents of 15 and 20% fell in the lower "non-expansive" field of Figure 1, but showed expansions in excess of 0.040% (0.047 and 0.076%) at one year in the concrete prism test (Shehata *et al.*, 2017).

4 Apparatus

The apparatus shall consist of a small jaw crusher or other suitable equipment capable of crushing aggregate to pass a 2.5 mm sieve and a shatter box or other suitable equipment capable of grinding approximately 30 g of aggregate finer than 2.5 mm to pass a 160 μ m sieve.

5 Sample selection

5.1

The procedures specified in CSA A23.2-1A shall be followed to obtain a representative sample of the aggregate to be tested. The sample, if possible, shall be obtained from a stockpile of aggregate produced in a manner identical to that which will be used in subsequent production.

5.2

Bulk samples shall be reduced to test specimen size in accordance with the procedures of CSA A23.2-2A, omitting where relevant the requirement about not preparing samples of an exact predetermined mass.

6 Test specimen preparation

6.1

The procedures specified in ASTM C702/C702M shall be followed to obtain representative test specimens of the aggregate sample. The mass of test specimens shall be determined by the particle size of the aggregate as given in Table 1. No attempt shall be made to obtain the required test specimen mass by adding or subtracting individual pieces.

Nominal maximum aggregate size, mm	Minimum mass of sample, kg
14 and less	2
20	3
28	4
40	5
56	10
80	18

Table 1 Test specimen size (See Clause 6.1.)

6.2

The test specimen shall be crushed using a small jaw crusher or other suitable means so that the entire sample passes a 2.5 mm sieve. The jaw crusher shall be clean and no material shall be lost.

6.3

The crushed test specimen shall be mixed thoroughly and a representative specimen of 30 ± 5 g shall be prepared using a sample splitter or other suitable means. The test specimen shall be pulverized using suitable equipment so that it passes a 160 μ m sieve. Any material retained on the 160 μ m sieve shall be further treated until it is able to pass the sieve. Care shall be taken so that no material is lost.

6.4

The material passing the 160 μ m sieve shall be mixed thoroughly. Specimens of a suitable size shall be obtained for chemical analysis.

7 Hazards

This Test Method does not purport to address the safety problems associated with its use. The user of this Test Method shall establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

8 Test procedures

8.1 General

8.1.1

Where analytical data obtained in accordance with this Test Method are required, any method may be used that meets the requirements of Table 2. A method shall be considered to consist of the specific procedures, reagents, supplies, equipment, instruments, etc., selected and used in a consistent manner by a specific laboratory.

Note: Examples of methods used successfully for analysis of carbonate rocks are given in Shapiro (1975), Norrish and Chappell (1977), and ASTM C25.

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8.1.2

If two or more instruments, even if they are substantially identical, are used in a specific laboratory for the sample analyses, use of each instrument shall constitute a separate method and each shall be qualified separately.

8.2 Qualification of a method

8.2.1

Prior to use for analysis of aggregate, the method chosen shall be qualified for such analysis.

8.2.2

Using the method chosen, duplicate determinations shall be made for each oxide on the standard reference materials.

Note: The standard reference materials are available from the National Institute of Standards and Technology (NIST), Gaithersburg, Maryland, Reference Materials ID, 88B.

8.2.3

The differences between duplicates shall not exceed the limits specified in Column 3 of Table 2.

8.2.4

The average for each component shall not differ from the qualified concentrations by more than the value specified in Column 4 of Table 2.

Table 2 Maximum permissible variation in results

(See Clauses 8.1.1, 8.2.3, 8.2.4, and 9.)						
Standard reference material (SRM)	Component	Maximum difference between duplicates, %	Maximum difference of the average of duplicates from the SRM certificate values, %			
NIST 1D	CaO	0.6	±0.5			
	MgO	0.1	±0.1			
	Al ₂ O ₃	0.1	±0.2			
NIST 88B	CaO	0.7	±0.6			
	MgO	0.4	±0.5			
	Al ₂ O ₃	0.1	±0.1			

8.3

The aggregate specimen shall be tested using the chosen analytical method.

9 Reporting

9.1 Required information

The following information shall be included in the test report, if applicable:

- a) sample number or identification marks;
- b) type and source of aggregate;

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- c) a description of the elevations and the location of the quarry face that was sampled, or if the sample was taken from a stockpile, this shall be reported;
- d) maximum nominal size of the aggregate;
- e) percentage mass of the various oxides in the sample to the nearest 0.1%;
- f) a description of the type of analytical method used, together with data to show that the method used meets the precision and accuracy limits shown in Table 2;
- g) name of the technician who performed the test;
- h) identification of the certified laboratory that performed the test (i.e., name and address);
- i) name and signature of the person responsible for the review and approval of the test report; and
- j) any deviation from the test procedure.

9.2 Optional information

The following information may be included in the test report:

- a) name of the technician who sampled the aggregate; and
- b) date the sample was taken.

10 Interpretation of results

Correlations among data obtained by this Test Method, expansion of concrete prisms and rock cylinders, and performance of aggregates in concrete structures have been published.* On the basis of these data, an area on the graph in Figure 1 has been established where potentially expansive alkalicarbonate reactive rocks are found. Aggregates whose results of analysis fall in this area shall be considered to be potentially deleterious until the innocuous character of the aggregate is demonstrated by service records or by a supplementary tests in accordance with CSA A23.2-14A.

* For further information see Rogers (1986).

11 Precision

Preliminary data indicate that the multi-laboratory coefficient of variation (for experienced laboratories) is dependent upon the element being analyzed. Multi-laboratory coefficient of variation for an alkalicarbonate reactive rock, obtained in a study with 12 participants, is given in Table 3. The D2S values are the limits that should not be exceeded by the difference between the results of two properly conducted tests, in different laboratories, expressed as a percentage of their mean. These values are only applicable to a carbonate rock with a chemical composition similar to that specified in Table 3.

Tabla 2

Multi-laboratory variation (See Clause 11.)					
	CaO	MgO	CaO:MgO	Al ₂ O ₃	
Value	43	6	7	2	
C of V*	1.5%	6.9%	7.6%	13%	
D2S [†]	4.3%	20%	22%	37%	

* Coefficient of variation.

+ D2S = acceptable maximum difference between two results; see ASTM C670.