METHOD OF TEST FOR THE
DETERMINATION OF POTENTIAL ALKALI-CARBONATE REACTIVITY OF CARBONATE ROCKS BY CHEMICAL COMPOSITION

1. SCOPE

1.1 This method covers a procedure for testing quarried carbonate aggregates to determine whether or not they are potentially alkali- carbonate expansive.

1.2 Alkali-carbonate reactive aggregates are dolomitic limestones with a high clay content. Other carbonate rocks are not alkali- carbonate reactive. Dolomitic limestone may be recognized by determining CaO : MgO ratio. Clay content may be determined by measuring alumina (Al_2O_3) content.

1.3 Carbonate rocks which are not alkali-carbonate reactive may be alkali-silica reactive. This test procedure does not determine the potential for alkali-silica reactivity.

2. RELEVANT DOCUMENTS

2.1 ASTM C 702, Methods of Reducing Field Samples of Aggregate to Testing Size.

2.2 CSA-A23.2-1A, Sampling Aggregates for Use in Concrete.

2.3 CSA-A23.2-26A, Determination of Potential Alkali-Carbonate Reactivity of Quarried Carbonate Rocks by Chemical Composition.

3. APPARATUS

3.1 Crushing Equipment - a small jaw crusher or other suitable equipment capable of crushing approximately 2 kg of aggregate to pass a 2.00 mm sieve.

3.2 Grinding Equipment - a chatter box or other suitable equipment capable of grinding approximately 30 g of aggregate to pass a 150 µm sieve.

4. SELECTION AND PREPARATION OF TEST SAMPLES

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

4.2.1 The procedures outlined in ASTM C 702 should be followed to obtain representative test specimens of the aggregate sample. The mass of the test specimens is determined by the particle size of the aggregate as shown in Table 1.

Table 1

Pass	Retain	Approx. Mass, g
19.0 mm	13.2 mm	1000
13.9 mm	9.5 mm	500
9.5 mm	6.7 mm	200
6.7 mm	4.75 mm	75

4.2.2 No attempt should be made to obtain the required test specimen mass by adding or subtracting individual pieces. When preparing test specimens of aggregates larger in size than 20 mm, the mass of the sample should be such that a minimum of 200 particles will be obtained of each sieve size.

4.3 The various test specimens shall be combined and crushed, using a small bench crusher, or other suitable means, so that the entire sample passes a 2.00 mm sieve. Care should be taken that the jaw crusher is clean, and that no material is lost.

4.4 The crushed test specimen shall be mixed thoroughly, and further separated, using a sample splitter to obtain a specimen of about 30 g. This 30 g test specimen shall be pulverized, using suitable equipment so that it passes a 150 µm sieve. Any material retained on the 150 µm sieve shall be further treated to pass the sieve. Care should be taken that no material is lost.

4.5 The pass 150 µm sieve specimen shall be mixed thoroughly, and split to obtain specimens of a suitable size for chemical analysis.

5. TEST PROCEDURE

5.1 Where analytical data obtained in accordance with this method are required, any method may be used that meets the requirements of 5.2. A method is considered to consist of the specific procedures, reagents, supplies, equipment, instrument, etc., selected and used in a consistent manner by a specific laboratory.

Note 1: Examples of methods used successfully for analysis of carbonate rocks are given in references 1, 2, and 3.

5.1.1 If more than one instrument, even though substantially identical, is used in a specific laboratory for the same analyses, use of each instrument shall constitute a separate method, and each shall be qualified separately.

5.2 Qualification of a Method - Prior to use for analysis of aggregate, the method chosen must be qualified for such analysis.

5.2.1 Using the method chosen, make single determinations for each oxide on the Standard Reference Materials (see note). Complete two rounds of tests on non-consecutive days, repeating all steps of sample preparation. Calculate the differences between values and averages of the values for the two round of tests.

Note 2: The Standard Reference Materials are National Institute of Standards and Technology, Gaithersburg, Maryland, Reference Materials Nos. 1C and 88A.

5.2.2 The differences between duplicates shall not exceed the limits shown in column 3 of Table 2.

5.2.3 The average for each component shall not differ from the certified concentrations by more than the value shown in column 4 of Table 2.

6. REPORT

6.1 The report shall include the following:

6.1.1 Source of aggregate including a description of the elevation and location of the quarry face that was sampled.

6.1.2 Any relevant information concerning the preparation of the sample.

6.1.3 Maximum size of the aggregate.

6.1.3 The weight percentage of the various oxides to the nearest 0.1 percent.

6.1.4 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.

Table 2 Maximum Permissable Variation in Results

Column 1	Column 2	Column 3	Column 4
Standard reference material (S.R.M.)	Component	Maximum difference between duplicates	Maximum difference of the average of duplicates from the S.R.M. certificate values
N.I.S.T. 1C	CaO	0.6 %	± 0.5 %
	MgO	0.1 %	± 0.1 %
	Al_2O_3	0.1 %	± 0.2 %
N.I.S.T. 88A	CaO	0.5 %	± 0.5 %
	MgO	0.4 %	± 0.5 %
	Al_2O_3	0.1 %	± 0.1 %

7. INTERPRETATION OF RESULTS

7.1 A correlation between data obtained by this method, expansion of concrete prisms, rock cylinders, and performance of aggregates in concrete structures has been published (4). On the basis of this data, an area on the graph in Figure 1 has been established where potentially expansive alkali-carbonate reactive rocks are found. Aggregates that fall in this area should be considered to be potentially deleterious until the innocuous character of the aggregate is demonstrated by service records or by a supplementary test in accordance with CSA-A23.14A.

8. 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 alkali-carbonate reactive rock, obtained in a study with twelve 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 test, in different laboratories, expressed as a percent of their mean. These values are only applicable to carbonate rock with a chemical composition similar to that given in Table 3.

Table 3 Multi-Laboratory Variation

	CaO	MgO	CaO : MgO	Al_2O_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 C 670).

9. REFERENCES

9.1 SHAPIRO, L.; 1975, Rapid Analysis of Silicate, Carbonate and Phosphate Rocks - Revised Edition; United States, Geological Survey, Bulletin 1401, 76 p.

9.2 NORRISH, K., and CHAPPELL, B.W.; 1977, X-ray Fluorescence Spectrometry; in Physical Methods in Determinative Mineralogy, Ed by J. ZUSSMAN, Academic Press, London, Chapter 5, pp 201-272.

9.3 ASTM C25-83 Standard Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime; Annual Book of ASTM Standards, Vol. 04.01.

9.4 ROGERS, C.A.; 1985 Evaluation of the Potential for Expansion and Cracking Due to the Alkali-Carbonate Reaction; Ontario, Ministry of Transportation and Communications, Engineering Materials Office, Report EM-75, 38 p., Feb. Also published in Cement, Concrete and Aggregates, CCAGDP, Vol. 8, No. 1, Summer 1986, pp 13-23.

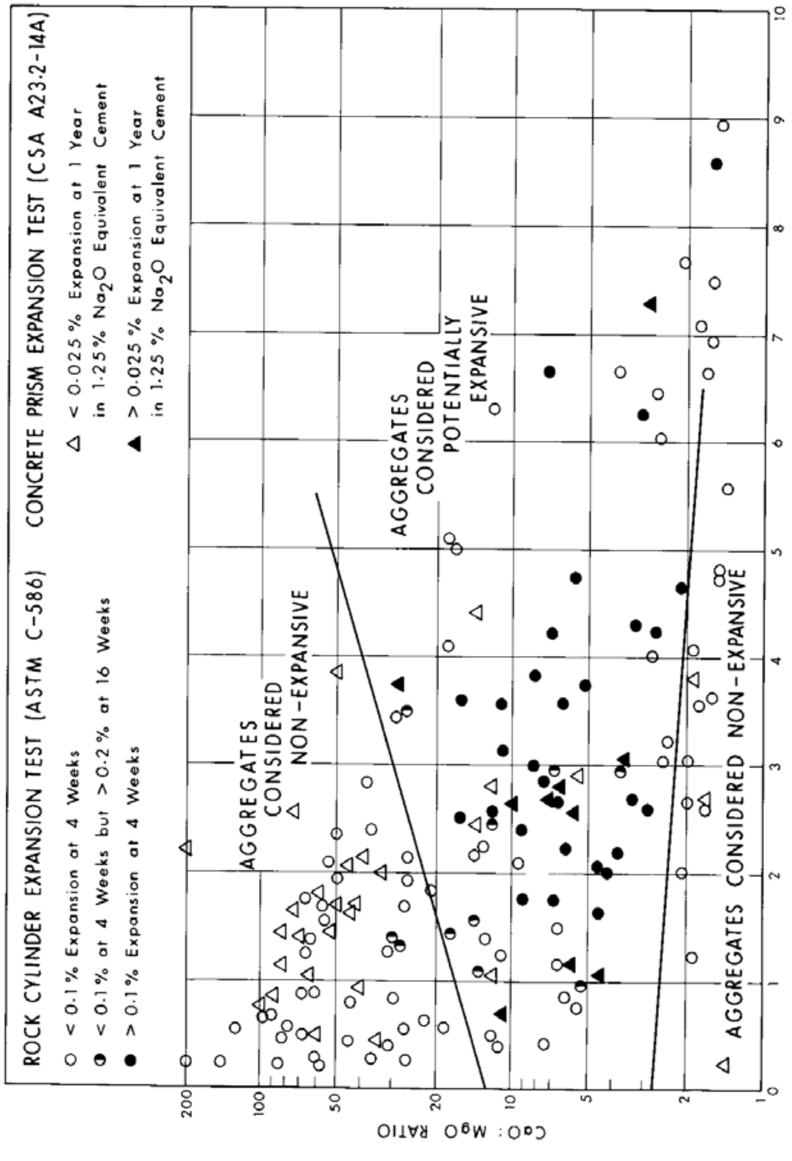

Figure 1: Illustration Of The Division Between Non-Expansive And Potentially Expansive Aggregates On Basis Of Chemical Composition