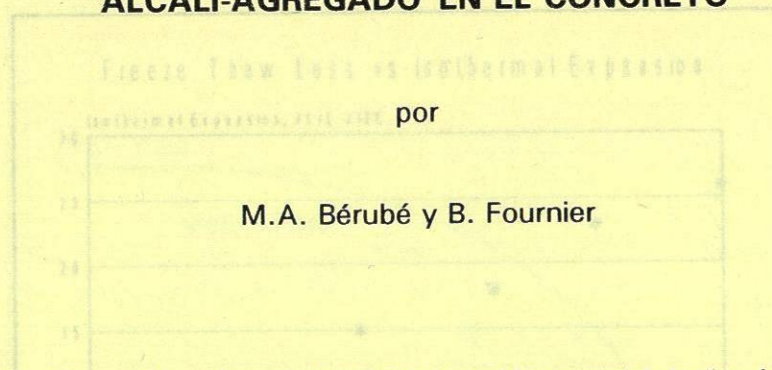


## ENSAYES PARA DETERMINAR LA REACCION ALCALI-AGREGADO EN EL CONCRETO

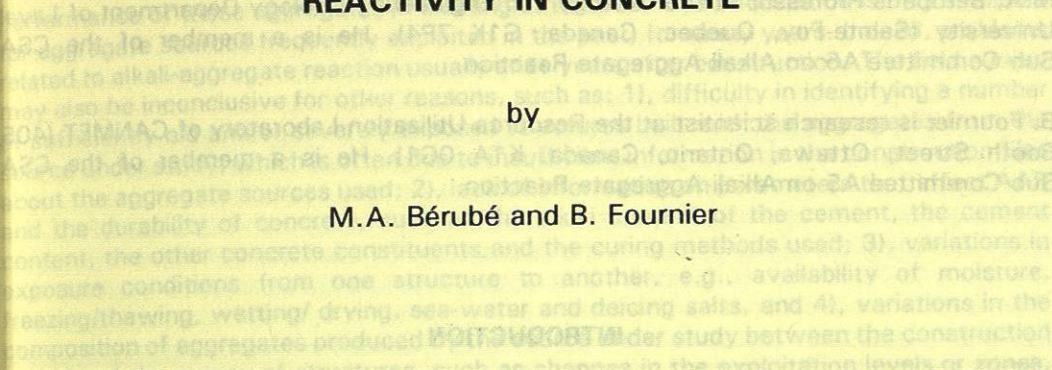


M.A. Bérubé y B. Fournier

**Sinopsis:** La información más realista sobre la reacción potencial alcalina de los agregados para concreto es aportada por su comportamiento en las estructuras existentes. Sin embargo, si dicha información no está disponible o se juzga poco confiable por diversas razones, los agregados deben ser ensayados en el laboratorio. Este trabajo revisa y discute los principales métodos de ensaye de la reacción álcali-agregado usados en Norteamérica. En el actual estado del conocimiento, sólo algunos métodos son considerados estadísticamente confiables para ser utilizados en la mayor parte de los tipos de agregados para concreto. El examen petrográfico ASTM C-259 es siempre el primer paso a seguir. El otro método rápido de ensaye recomendado, es el Método Acelerado para Barras y Mortero (ASTM C 9 - P 214 ó la propuesta CSA). Este ensaye no puede ser usado para rechazar materiales, ya que es severo para un gran número de agregados inocuos, pero representa una poderosa herramienta de selección, ya que a sólo algunos agregados deletéreos no pueden ser detectados. El método de ensaye más realista es el Método de Prisma de Concreto CAN/CSA A23.2-14A. Por consiguiente, se propone un diagrama de decisión para la evaluación de agregados, que está, sin embargo, basado principalmente en la experiencia Canadiense del AAR (reacción álcali-agregado) y puede no ser necesariamente aplicable a todos los agregados encontrados en otros países.

**Palabras Clave:** Métodos de ensaye, método acelerado de ensaye, reacción álcali-agregado, alcalinos, concreto, durabilidad, expansión, mortero (material).

## TESTING FOR ALKALI-AGGREGATE REACTIVITY IN CONCRETE



M.A. Bérubé and B. Fournier

**Synopsis:** The most realistic information on the potential alkali-reactivity of concrete aggregates is provided by their field performance in existing structures. However, if such information is not available or judged not reliable for a number of reasons, the aggregates have to be tested in the laboratory. This paper reviews and discusses the principal testing methods for alkali-aggregate reactivity that are used in North America. At the present state of knowledge, only a few methods are considered statistically dependable enough to be applicable to most types of concrete aggregates. The Petrographic Examination ASTM C 295 is always the first step to do. The only other rapid test method that is recommended is the Accelerated Mortar Bar Method (ASTM C 9 - P 214 or CSA proposal). This test cannot be used for rejecting materials, because it is severe for numerous innocuous aggregates, but remains a powerful screening tool since only a few deleterious aggregates cannot be detected. The most realistic testing method is the Concrete Prism Method CAN/CSA A23.2-14A. Accordingly, a decision chart, which is, however, mainly based on Canadian experience of AAR and could not necessarily apply to all aggregates found in other countries, is proposed for aggregate evaluation.

**Keywords:** Testing methods; accelerated testing methods; alkali-aggregate reaction; alkalies; cements; concrete; durability; expansion; mortar (material)



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## INTRODUCTION

**Basic concepts** -- For many years, aggregates were believed to be essentially inert and chemically inactive in concrete mixtures. It is now well established that the properties of aggregates greatly affect the strength, the durability and the structural performance of concrete. In concrete, the aggregates are subjected to a highly basic and alkali environment where some mineral phases, generally stable in normal environmental conditions, can produce significant deteriorations as a result of deleterious chemical reactions commonly called alkali-aggregate reactions.

Two types of alkali-aggregate reactions (AAR) are described in the CAN/CSA A23.1-M90 (Appendix B) which differ fundamentally in the type of mineral phases and the mechanisms involved (1) (see Table 1): A), alkali-carbonate reaction (ACR), and B), alkali-silica reaction (ASR). The former involves fine-grained argillaceous dolomitic limestones that are mainly found in Ontario (Canada), and in a few states in the USA. The alkali-silica reaction may be subdivided into two categories according to the type of reactive silica involved: A1), ASR which occurs with poorly crystalline or metastable siliceous minerals, and volcanic or artificial glasses, and A2), ASR which occurs with quartz-bearing rocks. The latter is presented separately because of the delayed onset of expansion and cracking that can be observed either on concrete prisms tested in the laboratory or on concrete structures (which can be up to 20 years).

Three conditions must prevail to initiate and maintain alkali-aggregate reactions in concrete: 1), the aggregates must be reactive; 2), the alkalis must remain abundant in the concrete pore solution; usually, these are mostly supplied by the cement, but some may also be provided by chemical or mineral admixtures, by some mineral phases present within the aggregate particles such as altered feldspar, micas and zeolites, or by secondary sources such as sea-water and deicing salt, and 3), the concrete must be exposed to high humidity, over 80 to 85% R.H. according to many authors.

**Predicting the potential alkali-reactivity of concrete aggregates** -- A critical question arises when planning the construction of a concrete structure to be subjected to conditions that might promote the development of AAR: *Are the proposed aggregates alkali-reactive in concrete?* To answer this question, the most realistic information is provided by the field

performance of these aggregates in existing structures. This information is only available for aggregate sources frequently exploited in the past, for many years. Indeed, problems related to alkali-aggregate reaction usually arise years after construction. The field survey may also be inconclusive for other reasons, such as: 1), difficulty in identifying a number of sufficiently old and/or severely exposed structures built with the aggregates from the source under study, which is often due to insufficient information in the construction files about the aggregate sources used; 2), lack of information on parameters that affect AAR and the durability of concrete, such as the alkali content of the cement, the cement content, the other concrete constituents and the curing methods used; 3), variations in exposure conditions from one structure to another, e.g., availability of moisture, freezing/thawing, wetting/ drying, sea-water and deicing salts, and 4), variations in the composition of aggregates produced by the source under study between the construction period and the survey of structures, such as changes in the exploitation levels or zones, and modifications in the methods of exploitation or preparation. For instance, a structure that contains very reactive aggregates might not have suffered from AAR if a low alkali cement or effective supplementary cementing materials in sufficient amounts had been used in concrete.

Because of the reasons mentioned above, the actual determination of the potential alkali-reactivity of aggregates is often only possible through laboratory testing programs. An engineering judgement is then necessary to predict the risk for deleterious expansion with the aggregate investigated, based on the laboratory results, the known limitations of the testing methods used, the past field performance of concrete aggregates that are quite similar to the one under study, and the conditions to which the particular structure to be built will be subjected.

The daily reality in the construction industry is also that, on many occasions, the aggregates must be evaluated within a very short period of time; this calls for testing methods that are rapid, reliable, simple, and reproducible. This paper reviews the principal testing methods for AAR, standardized or not, that are commonly being used in North America. These methods are listed in Table 2 with the types of AAR for which they can be used (ACR and/or ASR). Many other methods used in other countries are reviewed and discussed in (2).

First, it must be pointed that all these methods are accelerated tests, even the CSA Concrete Prism Method, which requires one year, and that they cannot exactly reproduce field exposure conditions. Indeed, they all try to predict in less than one year what may happen in the field after five, ten, twenty years or even more. To achieve this goal, one or many of the following test conditions are generally used: 1), increase the alkali concentration to which the aggregate is subjected using high-alkali mixtures or immersing the samples in alkaline solutions; 2), store the test samples at high temperature, for example 38°C, 80°C, and even more in autoclave treatments; 3), subject the samples to high pressure as in the autoclave; 4), subject the samples to high humidity environments such as 100% R.H. or immersing them in aqueous solutions, and/or 5), increase the specific area in reducing the aggregate samples to powder or sand size.



### PETROGRAPHIC EXAMINATION ASTM C 295

Potentially reactive mineral phases and corresponding host rock types are listed in Table 1. The petrographic examination of aggregates in thin sections under the optical microscope generally allows recognition of these potentially mineral phases or rock types. Techniques such as X-Ray diffraction, measurement of the undulatory extinction angle of quartz grains, and scanning electron microscopy might be also useful (ASTM C 295). It must be mentioned that the effectiveness of the undulatory extinction angle method is presently questioned (3). When conducted by a petrographer with experience of AAR, the petrographic examination can sometimes be sufficient for accepting or rejecting aggregates for use in concrete, in accordance with the past field performance of petrographically similar aggregates. If there is doubt, the petrographic examination will help selecting additional tests to be performed, considering the nature of the aggregates under study, thus preventing poor choices and reducing the amount of work. Indeed, some testing methods are not capable of detecting some deleterious aggregates, while being too severe for innocuous ones. In fact, the petrographic examination must always be performed prior to or in parallel to any other quality control test.

### CHEMICAL METHODS

#### Chemical Method CSA Proposal A23.2-26A

This method, which is in the process of being adopted in the Canadian standards for AAR (1), covers the evaluation of the potential alkali-carbonate reactivity of quarried carbonate rocks. In this test method, a representative sample of the aggregate to be tested is reduced using a small jaw crusher and a disk pulverizer such as to pass a 160  $\mu\text{m}$  sieve. The material is then carefully homogenized and sent for chemical analysis for  $\text{CaO}$ ,  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$ . The results thus obtained are plotted on a graph showing zones associated with "aggregates considered non-expansive" and "aggregates considered potentially expansive" (Fig. 1). Aggregates for which the results in this test are falling in the "considered potentially expansive" zone should be considered as such until their innocuous character is demonstrated by either satisfactory service record or concrete prism test results.

#### Chemical Method ASTM C 289

The Chemical Method ASTM C 289 is certainly one of the most widely used tests for evaluating the potential reactivity of silica-bearing aggregates. Its popularity is mainly because it requires only small quantities of material and results can be obtained within a few days. In this test, 25 g sub-samples of crushed aggregate particles, 150-300  $\mu\text{m}$  in size, are immersed in 25 mL 1N NaOH solution at 80°C for 24 hours. The solution is then filtered and analyzed for dissolved silica (Sc) and reduction in alkalinity (Rc). The results are plotted on a standard chart showing three fields corresponding to innocuous, deleterious and potentially deleterious aggregates (Fig. 2).

This test is applicable to alkali-silica reactivity only. However, a significant number of known alkali-silica reactive aggregates from all over the world pass the test, while other aggregates with good field performance fail (2). There are several reasons for this:

**Poor representativity of the aggregates used to design the test** -- The original ASTM decision chart was based on expansion of mortar bars, field performance or petrographic examination, in some cases, of 71 rocks from USA containing very reactive siliceous mineral phases such as opal, calcedony and volcanic glass. However, the increased application of the chemical test to aggregates from various countries led to the conclusion that the original chart is not universally applicable (4).

**Mineral interference** -- Mineral phases such as calcium, magnesium and iron carbonates, hydrated magnesium silicates, gypsum, zeolites, clay minerals, organic matter and iron oxides have proved to create interferences which result in: 1), underestimated Sc values due to precipitation of silica or interference during chemical analysis, or 2), overestimated Rc values due to reactions with  $\text{Na}^+$  and  $\text{OH}^-$  ions (5). In particular, calcium carbonate leads to precipitation of some of the silica dissolved as CSH, thus causing the acceptance of several reactive aggregates (6) (Fig. 2). A modified version of the Chemical Method ASTM C 289, which consists in performing the test procedure on the dried insoluble residue, 0 to 300  $\mu\text{m}$  in size, of the aggregate to be investigated, has recently been proposed to overcome carbonate interference (6,7). This method, despite producing interesting information on the basic chemical stability of the insoluble residue of the investigated aggregates in an alkaline solution, showed only limited success in differentiating potentially reactive from non-reactive aggregates.

**Crushing and sieving effects** -- During the processes of crushing and sieving of the aggregate sample to obtain the material required for the test, 150 - 300  $\mu\text{m}$  in size, much of the reactive phases may be taken out from the test specimens when sieving to discard the < 150  $\mu\text{m}$  fraction, for instance the reactive quartzitic cement around the non-reactive quartz grains in some Potsdam sandstones (2).

**Conclusion** -- The past experience has shown that the Chemical Method ASTM C 289 is severe for a number of innocuous aggregates, while being not severe enough for many deleterious ones. Moreover, modified procedures or limit criteria based on regional geological considerations were assessed with either limited or good success. In Canada, this test is not used anymore by many agencies and has been discarded of the new proposed version of the CSA Standards for AAR (1).

### MORTAR BAR METHODS

#### Mortar Bar Method ASTM C 227

Mortar bars are made with the aggregate meeting specific grading requirements and a cement with the highest alkali content representative of the general use intended, or



available in the laboratory making the tests. The ratio of cement to graded aggregate is 2.25. The amount of mixing water is adjusted such as to get a specified flow. The bars, 25 x 25 x 285 mm in size, are kept in their moulds for 24 hours at 23°C and then stored at 38°C and 100% R.H., in sealed specified containers. Length change measurements are made at frequent intervals. The expansion limits are 0.10% at 6 months, or 0.05% at 12 months. According to Grattan-Bellew (8), these limits should be reported to 12 and 6 months, respectively. Such limits are already being used by many agencies.

The test does not apply to alkali-carbonate reactive aggregates, and also proved to be not capable of detecting many slow-late expanding alkali-silica/silicate reactive aggregates in particular greywackes and argillites (9), as well as all the reactive aggregates in UK (10). A number of parameters have shown to greatly affect the results:

**Effect of container and wicking** -- The test is largely affected by the presence or absence of wicks inside the storage container (11) (Fig. 3). With containers with wicks, for instance the proposed reference ASTM container, a large number of reactive aggregates satisfy the test requirements due to excessive leaching of alkalis from the mortar bars, thus leading to lower expansions (11). As a result, numerous tests performed in the past and from which it was concluded that the aggregates were innocuous are doubtful if the bars have been stored in containers with wicks inside.

**Effect of alkali content** -- The variations permitted in the alkali content of the cement for the test may also explain some of the experimental variations observed in the past (14). A current practice in many Canadian laboratories consists in adding NaOH to the mixture water so as to increase the alkali content to 1.25% (Na<sub>2</sub>O eq.) by mass of cement.

**Effect of water/cement** -- No water/cement is specified in the mortar bar method of ASTM C 227 as water is added to reach a specified flow. However, it has been observed that variations in the w/c may significantly affect the expansion results (Fig. 5). The behaviour might be attributed to the smaller quantity of "free" pore water in lower w/c mixtures, which results in higher alkali concentrations in the pore solution. The low porosity of low w/c mixtures also offers less room for stocking the deleterious reaction products, while such mixtures are possibly less susceptible to alkali leaching because of a relatively lower permeability.

**Conclusion** -- In Canada, the CSA Concrete Prism Method, which does not take moisture into longer, while considered much more reliable, is preferred to the Mortar Bar Method of ASTM C 227. However, when this mortar test has to be performed, it is highly recommended: 1), to use a container without wicking; 2), to increase the alkali content to 1.25% (Na<sub>2</sub>O eq.) of the cement mass by adding NaOH to the mixture water; 3), to control the water/cement to 0.50 (0.44 for uncrushed natural sands), and 4), to test in parallel with a well-known (or reference) reactive aggregate. The above recommendations concerning storage conditions and the alkali content have been taken into account in the recent development of the French equivalent Mortar Bar Method AFNOR P 18-585.

### Accelerated Mortar Bar Method (ASTM C 9 - P 214 & CSA Proposals)

Mortar bars for this test are prepared in accordance with ASTM C 227. After 24 hours of initial moist curing in the molds, the bars are placed in a sealed plastic container filled with water at 23°C and the containers immediately placed in an oven stove at 80°C. The next day, the zero reading is taken and the bars transferred to a 1N NaOH solution at 80°C for two weeks (12 days in the original NBRI proposal (12)), and measured hot each working day. In this test, expansion of mortar bars generally increases when increasing the w/c (2,8,13). The ASTM C 9 - P 214 (14) and CSA (1) proposals specify using a fixed w/c of 0.50 for coarse aggregates and manufactured sands, and 0.44 for natural sands.

**Expansion limit criteria and test performance** -- There is a general agreement that aggregates which expand less than 0.10% after 14 (or 12) days be considered innocuous (Table 3). Indeed, up to now, only a few deleteriously reactive aggregates were found to satisfy this quite severe criterion, including alkali-carbonate reactive rocks from the Kingston area (Ontario, Canada) (15), some Potsdam sandstones from the Montreal area (Quebec, Canada) (2,16) (Fig. 6), some granite and gneisses of Grenville age from Maryland and Virginia (USA) (17), and a particular phyllite from Australia (18). As shown in Table 3, less or more severe criteria are also used for aggregate acceptance by other workers or agencies. In some cases, different limit criteria are proposed according to the type of aggregate tested.

On the other hand, there is also a general agreement that all above criteria are severe for numerous aggregates with good field performance (2), and that materials that exceed the proposed limits should require further testing. This is clearly evidenced by the results obtained at Laval University on a number of quarried silicate and carbonate aggregates from Quebec (2,16) (see Figs. 6 and 7).

**Conclusion** -- The Accelerated Mortar Bar Method should be used with care for rejecting aggregates. Indeed, many innocuous aggregates that perform well in the field and/or in other laboratory tests on mortar or concrete specimens, have been reported to have failed this accelerated test when using limits of 0.10% or 0.15% expansion at 14 days. Then further testing is required for aggregates exceeding the proposed limits. Nevertheless, this test remains a useful screening tool as it is capable of recognizing within two weeks most deleterious aggregates, while correctly recognizing a high proportion of innocuous aggregates. This test method has been applied to a large number of aggregates in several countries. In addition, its precision has also been assessed by several workers (8,13,19,20), with very satisfactory results.



### CONCRETE PRISM METHODS

#### Concrete Prism Method CAN/CSA-A23.2-14A (Current Procedure)

Three concrete prisms, not less than 75 x 75 x 300 mm and not more than 120 x 75 x 450 mm in size, are made with the coarse aggregate under study, a non-reactive sand and a normal Portland cement containing between 0.8% and 1.2% alkalis ( $\text{Na}_2\text{O}$  eq.). The specified cement content is 310 kg/m<sup>3</sup> and the amount of mixture water is adjusted so as to give a slump of  $80 \pm 10$  mm. Since 1986, the test method requires the total alkali content being raised to 1.25% ( $\text{Na}_2\text{O}$  eq.) by mass of cement, by adding NaOH to the mixture water. This corresponds to a total alkali content of 3.88 kg/m<sup>3</sup> of concrete. The prisms are stored either in a moist curing room at 23°C or above water in sealed containers at 38°C. Length change measurements are made periodically. The prisms stored at 38°C shall be taken out of the high temperature storage condition  $16 \pm 4$  h before measurement. The test prism are then immersed for 30 minutes in a water bath at 23°C before measurement. The 23°C storage condition was first proposed for detecting the alkali-carbonate reactivity, while the so-called "accelerated" version at 38°C is currently being used for detecting the alkali-silica reactivity. The expansion at one year should not exceed 0.025% at 23°C or 0.04% at 38°C.

**Influence of mixture proportionings (w/c, coarse aggregate/sand)** -- According to the standard, water is added to the concrete mixture to give the specified flow, whatever the water/cement. However, similarly to that observed in the Mortar Bar Method ASTM C29 (Fig. 5), variations in the w/c may have a significant effect on the expansion process. For instance, a lower ratio normally leads to a higher strength, a lower permeability, a lower porosity, but in turn to a higher alkali concentration in the "free" pore water, and to less space for stocking the expansive reaction products. A number of parameters may affect the water demand in plain concrete mixtures are not sufficiently well controlled by the current procedure, such as the particle shape, which may be greatly influenced by the type of crusher used to prepare the aggregates, and the ratio between coarse and fine aggregates, which can vary between 50 : 50 and 65 : 35, thus causing also variations in the amount of reactive aggregate particles. In practice, most laboratories which perform the CSA concrete prism test on a routine basis use to fix the coarse/fine aggregate and w/c ratio.

**Influence of storage conditions** -- According to Rogers & Hooton (11,21), significant amounts of alkali are progressively leached from the test concrete prisms, with the amount of expansion obtained in the long term being related to the amount of alkalis remaining in the prisms. For instance, after 130 weeks of testing, specimens made with alkali-carbonate reactive aggregate from Ontario (Canada) suffered 63% alkali leaching at 23°C (in moist curing room), and 42% at 38°C (in plastic pails with wicks). A number of experiments have shown that the one-year expansion of concrete prisms stored above water at 38°C, in sealed containers, was similar or even lower when the wicks were removed from the containers (8,22), while lowered by about 15 to 20% when the concrete specimens are immersed in water 30 minutes before each measurement

specified in the current CSA standard), compared with no immersion (22), as a result of alkali dilution in water. Leaching of alkalis from the concrete prisms has been mentioned as one of the possible factors to explain why the expansion curves are flattening out after a few months of testing.

**Effect of cement content** -- The current CSA concrete prism method can detect a wide variety of reactive aggregates, with the exception of a number of slowly-reactive quartz-bearing aggregates such as some greywackes, argillites, quartzwackes, quartzites, phyllites, arkoses, sandstones, and granites that are found in gravel deposits or exploited in a number of quarries in Eastern Canada (2). According to Rogers (21,23), in order to recognize these slowly-reactive aggregates, the test should be modified to prevent excessive alkali-leaching, for instance by storing the prisms in sealed plastic bags, or to compensate for this phenomenon by adding more alkalis and/or using more cement in the concrete mixture.

**Experimental variations (reproducibility)** -- A multilaboratory study using an alkali-carbonate reactive aggregate from Ontario and which involved twenty different laboratories suggested that the coefficient of variation for the CSA Concrete Prism Test conducted at 38°C is about 23% (21), which is quite high.

**Conclusion on the current test method** -- Despite of all the above limitations, the current CSA concrete prism test is still considered in Canada as the most realistic method used for evaluating the alkali-reactivity potential of concrete aggregates in the laboratory. Indeed, the test is capable of recognizing most reactive aggregates except a number of slow-late expanding ones for which a higher cement content around 410 kg/m<sup>3</sup> content seems to be required (15).

However, in our opinion, testing together the coarse aggregate with the sand used in AAR affected concrete structures incorporating such slowly reactive aggregates might have allowed detection of many of them, since the sands used often presented a composition and potential reactivity that are quite similar to those of the corresponding coarse aggregates. For instance, the 1-year expansion obtained with the current CSA procedure for a coarse greywacke that is very similar to the one used in the AAR affected Mactaquac dam, in New Brunswick (Canada), was 0.034% when using a well-known non reactive sand, but increased to 0.066% in presence of a sand that was very similar to the one used in the dam (24).

#### New Proposed Concrete Prism Method CAN/CSA-A23.2-14A

In response to the various problems that have been progressively identified with the current CSA Concrete Prism Test, a CSA Task Group recently proposed a number of modifications to the current test method (1). The procedure proposed, which is in the final steps of the process for being introduced in the Canadian Standards, includes the following modifications: