Cement and Concrete Composites 19 (1997) 451–476
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0958-9465/97/$17.00

PII:S0958-9465(97)00024-3

Accelerated Tests for Assessing the Potential Exhibited by Concrete Aggregates for Alkali-aggregate Reaction

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Abstract

A number of tests are available for assessing the potential of aggregates for alkali-aggregate reaction. These are listed, for example, in ASTM C33-93. The majority of these test procedures have been widely used in researching aggregates, but there are serious reservations concerning the application of some of these tests to the selection of materials for structures. In recent years, many papers have been published detailing investigations of the tests. An examination of the literature shows that most of the tests are of little value for making engineering decisions. This paper reviews the literature relating to three commonly used tests, the ASTM C-289 Rapid Chemical Test, the ASTM C-227 Mortar Bar Test, and what has become ASTM C-1260-94 Accelerated Mortar Bar Expansion Test. Attention has been given to a detailed practical study of the accelerated mortar bar tests since these are becoming very widely used. The experimental work, in essence, represents a determination of the errors to be expected from application of the test to a single aggregate and the factors that influence these errors. The repeatability of the tests suggests an upper bound standard deviation of 0.0365% expansion. This figure is used to establish discriminant criteria for the recognition of potentially damaging materials. It is evident that diagnosis of potentially highly reactive materials can be made with confidence, but that a large number of tests are required to assess marginal types. Petrographic evaluation of the mortar following the test is essential, particularly where the results are borderline, though the presence of traces of gel has to be interpreted with caution. Much of the uncertainty in the test results derives from inherent aggregate variability. A test schedule can be designed that has small contributions to the error from the experimental steps, but the error due to aggregate variability remains. This can lead to the requirement for the manufacture of multiple batches of mortar for establishing acceptance criteria for even a single aggregate batch, and further consideration is needed as to the procedures necessary for the assessment of aggregate sources. © 1997 Elsevier Science Ltd. All rights reserved.

Keywords: ASR, accelerated tests, mortar bar test, repeatability, petrographic evaluation, acceptance criteria.

INTRODUCTION

From the engineering point of view, it is essential that tests are available for the detection of potentially reactive aggregates that are reliable and capable of giving a measure of the probability that the aggregate will be innocuous in service. Many of the tests currently in use are possibly suitable for research purposes but do not provide an adequate basis for discrimination in the selection of aggregate for construction. This paper reviews some of the more commonly used tests from the engineering

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standpoint and describes an investigation into the factors influencing expansion in one of the preferred accelerated mortar bar tests. The repeatability of the method is also assessed as is the definition of limits providing an acceptable probability that aggregates will be appraised correctly.

The test explored in detail is based on the original work of Oberholster and Davies,[1] which was designed to discriminate between innocuous and deleteriously expansive reactive aggregates when they are used in concrete. The procedure that we have used is similar to that now adopted as the recent designation ASTM C-1260-94[2] and the Canadian Standard A23.2-25A,[3] which was published in June 1994.

Since Stanton[4] characterised the expansive and damaging reaction between alkalies in the pore fluid of concrete and aggregate, numerous tests have been devised in an attempt to discriminate between supposedly potentially reactive and innocuous aggregates. This discrimination presumably ought to mean potentially reactive or potentially innocuous under the most severe conditions imaginable, though many tests specify particular compositional and environmental parameters that are not necessarily the most severe that could be applied. Some argue that all aggregates should be regarded as potentially reactive if the conditions are sufficiently aggressive.[5,6] There are good geological reasons for supposing that any aggregate source can contain, at some stage in its exploitation, unstable lithologies. The amount of material actually reactive is likely to be less than 1% by volume of the aggregate in even the most severe examples, and will be unevenly distributed.[55] It is also to be expected that some potentially harmful components when present in particular concentrations will, in practice, produce no damage in service.

Early, if not the first tests, are those introduced by ASTM. In 1950, the mortar bar test ASTM C-227[7] was introduced, and 2 years later, the chemical method ASTM C-289[8] was added. Both take advantage of ASTM C-295[9] for the petrographic examination of aggregates, which was also first issued in 1952. Except for minor modifications, these tests remain in the current book of standards. The limitations of these procedures are shown, however, by the publication of some 25 papers on testing that appeared in the proceedings of the 8th International Conference on Alkali-Aggregate Reaction in Kyoto in 1989,[10] and about 35 papers were published in the proceedings of the 9th International Conference held in London in 1992.[11] The introduction of the accelerated mortar bar test ASTM C-1260 in 1994[2] provides further evidence of the deficiencies in the previous standards and the need for improvement. There are also various tests based on concrete prisms that can test the aggregate in the size ranges to be employed in practice. Of these, perhaps that most widely employed is the Canadian standard.[12] However, concrete prism tests require at least a year for their completion, and there is evidence that a much longer period may be required, especially for some slowly reactive aggregates.

What is needed, therefore, is a practical test that allows defective materials to be identified under the most extreme conditions likely to be encountered in service and that can be carried out in a time scale that is appropriate to construction planning. Each such test also requires definition of criteria for the necessary discrimination to be made under the given environmental conditions. This implies a need for statistical knowledge of the errors attached to each method and the definition of the probability of avoiding damage at a level that the user will find acceptable. In general, the amount of information on the repeatability and reproducibility of the standard procedures is meagre, and the impact of poor reproducibility on the interpretation of test results is rarely considered. In some cases, including some standards, the repeatability of the tests is not defined or considered. A range may be stated without explanation as to its meaning. For the present work, the definitions used conform with current BSI and ISO practice and are as follows.

Standard deviation:	STDEV, as calculated using Excel 5 from $s = [(n\Sigma x^2 - (\Sigma x)^2)/n(n-1)]^{0.5}$, where n is the number of measurements of a value and x the values obtained.
Standard error:	$s/n^{0.5}$
Repeatability:	$r = 1.96\sqrt{2}s$, where r is a measure of the random error occurring once in 20 measurements and associated with one operator obtaining successive

results by the same method applied to the same material.

Reproducibility: $R = 1.96\sqrt{2}(s_1^2+s_2^2)^{0.5}$, where s_2 is the contribution to the variability due to test operators working in different laboratories, s_1 is within lab variation, and R is a measure of the random error occurring once in 20 measurements with the operators using the same method and materials. R is typically about one and a half to double the value of r.

ASTM C-227 mortar bar test

The ASTM C-227[7] Mortar Bar Test has been widely used, though it may require a year or more for completion. The mortar bars are made with cement that has more than 0.60% by weight of alkali as Na_2O equivalent, and the bars are stored over water at 38 ± 1.7°C, presumably meaning that the full range must be kept within these limits or the test result rejected. ASTM C-33-93[13] specifies that the alkalies should be substantially above 0.60% and preferably about 0.8% as Na_2O equivalent, but difficulties arise because this level of alkalies is not rigorously defined so that expansion could relate to the selected alkali level. The expected alkali levels in the eventual concrete are not related to the test method or result. This standard also states the following:

> While the line of demarcation between non-reactive and reactive combinations is not clearly defined, expansion is generally considered excessive if it exceeds 0.05% at three months and 0.10% at six months. The expansion is measured to the nearest 2 micrometres. All measurements should be within ±0.003% of the mean or the range of results should be ±15% of the average if the mean results are 0.02% or higher.

Presumably, the ranges given mean that the result is rejected if it falls outside the range. At 0.10%, the expansion is about 250 μm, and the acceptable range is therefore ±38 μm. The discriminant criterion is therefore 0.10% with a standard deviation of about 0.002% expansion.

Table 1. Data from an inter-laboratory study of the ASTM C227 procedure[12]

Mean expansion	*Standard deviation*	*Reproducibility (% expansion)*	*Number of laboratories*
0.013	0.005	0.015	5
0.015	0.003	0.008	5
0.024	0.015	0.041	5
0.026	0.025	0.073	5
0.049	0.055	0.154	5

This exacting repeatability is unlikely to be achievable in practice. The standard does not provide data on the repeatability of the test or its reproducibility. Sorrentino *et al.*[14] showed that the repeatability of the test can be as low as ±0.02% expansion at an expansion of 0.28%, i.e. more than ten times that required by the standard. Moreover, Oberholster[15] quotes various published researches and an inter-laboratory study that show that known reactive aggregates are not detected by the test. He also shows that the reproducibility of the method from the inter-laboratory study is highly variable (Table 1) with means and standard deviation depending on the alkali content of the cement.

The number of laboratories involved in this test was small, but these results clearly show a variation that indicates that the reproducibility worsens at a higher rate than the rate of increase in expansion, and the magnitude of the reproducibility suggests that the analytical measurements may not be normally distributed. To define a limit of 0.10% expansion means that the result might need to be recorded as, at best, 0.10 ± > 0.10 given the data of Table 1. The test will have to be repeated many times for a statistically valid discrimination to be made. This result alone shows that the test leaves much to be desired from a practical engineering point of view. The reason for the large standard deviation is discussed later, but one feature may be that the laboratories used slightly different methods.

Ranc *et al.*[16] discussed in detail earlier reservations with respect to the efficacy of this test and confirmed the reservations by showing that six aggregates known to be reactive in service were not detected at all as reactive by the test. These aggregates gave a mean expansion of 0.029 ± 0.009% after 6 months and 0.029 ± 0.017% with no aggregate reaching

0.10% after 12 months. These results echo the work of Shayan *et al.*[17] Rogers and Hooton[18] also found the procedure to be unsatisfactory and explained the lack of expansion as due to leaching of alkalies by condensation on the surface of the mortar bars. Sorrentino *et al.*[14] considered that the lack of expansion might, for some reactive aggregates, be due to the removal of aggregate below 0.15 mm as required by the standard. The procedure was improved by Ranc *et al.*[16] by increasing the alkalies to 1.25% as Na_2O equivalent by weight of cement and by preventing condensation. Nevertheless, the test again failed to detect the reactive aggregates, and Ranc *et al.* discuss various reasons for this behaviour.

This test therefore is clearly unacceptable for assessment of a range of aggregate types and in particular may not be suitable for discrimination of the aggregates that generate slow or delayed expansion. There is also a need for more careful definition of the experimental conditions. Doubt must be attached, for example, to the statements defining the required alkali concentrations in the test.[19] The results obtained may only be acceptable if it can be shown that the range of lithologies and microtextural features present in the rock are the same as those for which the test has proved to be satisfactory in the past. In the absence of suitable data for the reproducibility of the method, the reported failure to detect a wide range of potentially damaging aggregates, and the length of time required for the test, it can be difficult to interpret the observed magnitude of expansion in the test in terms that are useful in any way from the engineering point of view. The test is unfortunately still specified but should be abandoned.

ASTM C-289 chemical test

This test has been widely used, often in combination with the C-227 mortar bar test. It was designed specifically for testing rocks that are mainly silica, and is inappropriate for rocks containing carbonates, serpentine minerals, and probably for a wide range of rocks that are impure silicate lithologies. Grattan-Bellew[19] regarded this test as superseded, and Ranc *et al.*[16] have shown that the test is unreliable in that two of six aggregates known to be reactive in service were not identified as such. Figure 5 of Katawaki *et al.*[20] suggests that some 10% of aggregates may be identified incorrectly as reactive, while a smaller percentage is found to be innocuous by this test but reactive with the mortar bar test. Fournier and Bérubé[21] and Barisone and Restivo[22] have used acid treatment to remove carbonates in order to improve the discrimination effected by the procedure.

From an engineering point of view, the poor precision attached to the likely behaviour of the aggregate in this test must be regarded as unacceptable. In the authors' experience, there are two main problems associated with the test. The first of these is its widespread use on rocks for which it was not designed, and the second is the influence of minor or trace amounts of rock that are not in the specified size range for the test. In particular, it is sometimes necessary to wash the aggregate many times before traces of turbidity produced by suspended rock particles are removed. The presence of very finely divided siliceous material is considered likely to have a significant affect on the result of the test and can lead to rocks giving results that are indicative of potential for deleterious behaviour. Conversely, Sorrentino *et al.*[14] point out that removal of the finest fraction could mean removing the reactive fraction, and they have substantially modified the test to allow the fine fraction to be included. Their modifications mean that the test must be carried out, possibly repeatedly, over a few weeks and that both very high and very low results can result from aggregate considered to be innocuous.

No precision trials have been reported for this test, and since no controls are available, the bias of the method cannot be assessed. According to the standard,[13] replicate results are considered to be satisfactory if they fall in the range ±12 mmol/l when the test result is 100 mmol/l or less for the two variables measured, and 12% where the measured values exceed 100 mmol/l. Presumably, tests giving results outside this range are regarded as invalid. This means that there must be a broad band of uncertainty along the defined discriminant boundary. The standard also makes it plain that the result of the test does not predict the reactivity of slow–late expanding aggregates such as those ‘containing strained quartz, microgranulated quartz, or aggregates composed of meta-greywacke, metasiltstone, metaquartz and similar rocks’. There are, therefore, many conditions that must be met before the test result can be of value for practical purposes. Only results indicating that the aggregate is strongly reactive are meaningful because of the uncertainty of the discriminant boundary.

ACCELERATED MORTAR BAR TESTS

Alkali immersion methods

Numerous accelerated mortar bar tests have been developed, most of which centre on the procedure published by Oberholster and Davies,[1] which is known as the NBRI method. In this procedure, the mortar bar is immersed in 1 M NaOH solution at 80°C. In a comparison of several test procedures, Hooton and Rogers[23] found the NBRI method to be the most effective. The version of the NBRI test now adopted as ASTM C1260-94 has been tested as the ASTM provisional specification P-214. Bérubé *et al.*[24] has pointed out that this test may be too severe in that some innocuous aggregates have been found to react expansively. Conversely, it is reported by Hooton[25] that Stark found some reactive aggregates that are not detected. Possibly, these differences reflect slight differences in the way in which the test is carried out, as discussed below.

There is now a great deal of published information on tests of this type, which shows that the test detects reactive aggregates satisfactorily, particularly if the test bars are examined microscopically at the end of the test, and if the test is carried out for up to 56 days. Some data on the reproducibility of this type of test were given in Hooton.[26] The test used by Hooton was based on the NBRI specification but is quoted in ASTM C-1260 and was based on the work of six laboratories using three aggregates and three cements. The inter-laboratory precision was found to correspond with a coefficient of variation of 9.55% (one standard deviation) when the expansion was greater than 0.15%. More recently, more detailed studies of the repeatability have been published, including a very detailed interlaboratory study published by Rogers *et al.*[53] for one aggregate that shows that the reproducibility of the test is superficially rather poor. These studies demonstrate a need for further research and more careful definition of the experimental parameters and of the relationship between the test result and the cause of the expansion. Hooton[25] has published an account of the application of this test to a large number of aggregates with known service records. The test has also been adopted in Canada as CSA A23.2-25A,[3,27] and the Aggregate Committee of the Norwegian Concrete Society has also proposed a voluntary arrangement for the declaration and approval of aggregates used in concrete, which includes a slightly modified version of the NBRI test.[28] A proposal for a new Rilem standard currently under consideration is also based on this method.

It is this alkali immersion test that has therefore been chosen for the investigation reported here. No firm criteria have yet been established for the method, and slightly different procedures and criteria are recommended by the differing authorities. It is highly desirable that the minor differences between the various test specifications should be eliminated and that firm criteria should be established for the discrimination of innocuous and reactive materials. The aim of the present work is to examine the factors influencing the results obtained in the test and to discuss the precision and variation of the results obtained. Variables studied in the present work that can influence the results obtained are the size of the mortar bars, the type of studs embedded in the ends of the bars, the precision of the aggregate grading, the ratio of the volume of alkali to the surface area of the mortar, and the cement type employed. It is difficult to assess such factors unambiguously because of the apparently poor precision of the method. The tests were carried out independently by two separate laboratories, with one laboratory running all the tests at essentially the same time, while the other carried out the tests one at a time. Details of the test procedure used in the present study are summarized in Table 2. Some of these steps are more stringent than is necessary but were required for the evaluation of details of the procedure.

Enhanced alkali method

In these procedures, alkalies are added to the mortar during the mixing stage. A number of variants of the method have been developed in which the mortar is treated at an increased pressure in an autoclave. The use of increased pressure and temperature means that results can be obtained in a few days or even a few hours in some instances. Following the work of Tang,[29–31] Fournier *et al.*[32] have reviewed the procedures for rapid autoclave methods and

Table 2. Basic procedure used in the Mortar Bar tests

1. Grade the aggregate to produce the grading curve specified in ASTM C227-94.
2. Blend 800 g of cement with water to produce a water/cement ratio of 0.45 (crushed aggregate).
3. Blend into this mixture 2000 g of the aggregate to be tested and mix for 2 min.
4. Cast four mortar bars, filling in two or three stages and using vibration after each stage. (The bars usedmeasured 160 × 40 × 40 mm or 250 × 25 × 25 mm.)
5. Cover with polythene and cure in a sealed container containing water for 24 h with the mould mounted above the water and with the inner sides of the container being coated with cotton fabric that extends into the water.
6. Carefully remove the bars from the moulds and place in water in a sealed polypropylene box. The water must cover the bars by at least 3 mm. Thin plastic rods or a corrugated plastic sheet are placed beneath the bars.
7. Place the box in an oven and bring the temperature to 80 ± 1.5°C over about 4 h and maintain this temperature for 48 h.
8. Remove the bars one at a time and measure their length to 0.002 mm in not more than 20 s. Replace the bars in the water immediately after measuring.
9. Repeat this measurement a further three times changing the order in which the bars are measured and after returning the boxes with their charge to the oven for 2 h.
10. Decant the water and add the same volume of 1 M NaOH solution, which has been previously warmed to 80°C.
11. Return the box to the oven and repeat the length measurements as before at four convenient times over a period of 14 days. Further measurements were made after 28 days and longer periods in some cases.
12. Each length measurement is made with respect to a standard invar bar.

have proposed a detailed procedure after carrying out a wide range of comparative tests and a detailed review of the literature. They found that expansion is influenced by the pressure and temperature employed, the water/cement ratio, the alkali concentration, and the cement type. The autoclave curing time is also significant. They recommend the following procedure, which can be completed in 3 days.

Mortar bar type:	as ASTM C227
Water/cement ratio:	fixed at 0.50
Alkali concentration:	fixed at 3.5% Na_2O equivalent weight % of cement
Pre-autoclave curing:	2 days in water at 20°C
Steam curing:	5 h at 130°C and 0.17 MPa
Final measurement:	at room temperature (25°C) after cooling over an hour.

The repeatability of this test was measured for one aggregate giving a mean expansion of 0.297% with a repeatability of 0.026 for seven determinations. Aggregate is considered to be innocuous at an expansion of 0.15% or less. The reproducibility, if all laboratories use precisely the same method, is therefore likely to be about 0.06% at 0.3% expansion.

A version of the autoclave method has also been studied by Saloman and Gallais.[33] They used ultrasonic pulse velocity, dynamic elastic modulus, and length and weight changes to monitor the reaction. They recommended that the alkalies as Na_2O equivalent should be 4.0% by weight of cement, that the pressure should be 0.15 MPa, and that length measurement is more satisfactory than elastic modulus or USPV. They fixed the water/cement ratio at 0.5 and found that bars measuring 40 × 40 × 160 mm were more satisfactory than bars of a smaller cross-section. With these experimental conditions, discrimination between reactive and innocuous materials is made at 0.15% expansion. Criaud *et al.*[34] have also studied this method, keeping close to the specification of Tang *et al.*[29–31] The bars are immersed in 10% KOH solution and maintained in the autoclave at 150°C for 6 h. They found that the expansion was dependent among other things on the aggregate/cement ratio and have measured the repeatability and reproducibility of the method (see Table 3). An autoclave

Table 3. Example of relationship between mean and reproducibility for autoclave procedure[32]

Aggregate	*Mean (% expansion)*	*Standard deviation (s)*	*Reproducibility (% expansion)*	*Number of laboratories*
Limestone	0.026	0.008	0.022	4
Sand	0.053	0.011	0.030	2
Chert	0.146	0.008	0.022	4
Sand	0.227	0.079	0.219	5
Spratt	0.354	0.022	0.122	4

procedure has also been applied to concrete samples.[56]

In the medium term, it seems likely that the obvious advantages of this type of test will mean that it supersedes those in common use at the moment and that the method will be very valuable as an engineering, as well as a research, tool. However, the poor reproducibility and wide range of working practice make it unreliable for practical purposes at the moment. Despite these variations, the tests demonstrate very rapidly that reaction occurs even where concrete is tested, and some field reactions requiring decades can be produced in a few hours. The problem of deciding an appropriate level of alkalies is, however, important. Almost any siliceous aggregate can be made to react if the alkali level is high enough. Dense flint from South East England can cause expansion in concrete prisms if the alkalies exceed 2.5% by weight of cement and in the field if the alkalies exceed 2%. French[35] showed that concrete prism test results depend partly on the alkali level and partly on concrete quality. Hence, the manufacture of the bars requires strict definition of all parameters, including the alkali concentrations, and needs to be tested against a wide range of aggregate types of known field behaviour for discriminant criteria to be drawn reliably.

EXPERIMENTAL PROCEDURES

The two laboratories involved in the present testing used the same bulk sample of aggregate to make mortar bars of two sizes with three different cements and independent storage and measurement systems (Table 2). The sequence of testing and of making the bars was also left to the individual laboratories.

Aggregate

The aggregate for the tests was made up from innocuous quartz sands. These sands were carefully graded and blended to match that required by the ASTM C-227 specification. The coarsest fraction was then adjusted by replacing some of the original sand with fused silica so that the fused silica represented 10% of the total aggregate in the size range 2.36–4.75 mm and 5% of the total aggregate in the size fraction 1.18–2.36 mm so that the amount of fused silica in total made 15% of the aggregate. Sufficient material was prepared to make more than 100 mortar bars, and this carefully blended material was then divided by riffling and allocated to the two laboratories.

Cements

Most of the mortar bars were made using a single UK cement that was allocated to both laboratories. In addition, both laboratories used the same Norwegian cement, and one laboratory also made mortar bars using a rapid hardening Portland cement.

Mortar bar sizes and composition

Both laboratories made sets of four mortar bars with two different sizes: 25 × 25 × 250 mm and 40 × 40 × 160 mm. A total of five batches of four mortar bars were made of each size by both laboratories. These bars were all made with the same British ordinary Portland cement. Subsequently, three sets of four mortar bars in the two sizes were made with two other cements. For all mortar bars, a fixed aggregate/cement ratio of 2.25:1 and a fixed water/cement ratio of 0.44 were employed.

Moulding and storage

The mixture was placed in the moulds in two layers with compaction and vibration. The vibration at each stage was for 10 s. The surface was then trowelled smooth, and the mould was then cured for 24 h at 25°C in a closed polypropylene box containing water but not in contact with the water. The bars were then demoulded and cured in water at room temperature (25°C) for 48 h. They were then immersed in water in closed containers that were maintained at constant temperature in an oven at 80°C for 48 h. The lengths of the bars were then measured at a temperature of approximately 80°C. They were then placed in polypropylene containers with molar sodium hydroxide solution and, after sealing, were returned to the oven at 80°C. One laboratory used moulds in which ball-bearings were embedded at each end of each bar to act as the measuring points. The second laboratory used threaded bolts penetrating into the mortar for 15 mm at each end, and designed to take a ballbearing at each end for length measurement. Each set of mortar bars was enclosed in its own separate container. The moulds of two different sizes were filled from the same mixture, enough material having been prepared for both sets of bars. Also, both sets of bars were maintained under the same conditions and measured at various time intervals by both laboratories. One laboratory subsequently made a series of mortar bars of the two sizes that were maintained within various different volumes of alkaline solution.

Method of measurement

After 48 h, each set of mortar bars was removed from the oven in its container and placed in a water bath maintained in the range 80 ± 2°C. The initial length for each bar was measured using a digital gauge which could be read to 0.001 mm. Each bar was read once only within 20 s of being removed from the hot water. This measurement was repeated for the other bars in the set. The bars were then allowed to regain the temperature of 80°C in the oven, and the measurements were then repeated in reverse order. All measurements were made a total of four times, with the order being changed on each occasion, after allowing the temperature to recover to 80°C each time. The bars were measured at various ages up to 56 days in the alkaline solution.

RESULTS

The results of measurements made by laboratories A and B are given in Tables 4–7. The means and repeatabilities for the 250- and 160-mm-long bars for each of the two laboratories are given in Table 8, for each time of measurement. The repeatability as given in this table treats the mean results for each batch of four bars as the individual test result and is defined as the expected random error associated with a single test operator obtaining successive results on identical material. It is the magnitude of the difference between two single results that would occur in only one instance in 20 measurements. The observations are discussed in detail in a later section. If the individual results for the two laboratories are taken together, the repeatability at 14 days is 0.150% for 160-mm bars and 0.077% for 250-mm bars. These figures are likely to compare closely with the interlaboratory reproducibility for the procedure.

DISCUSSION

Expansion criteria

Various proposals have been made for the interpretation of the magnitude of expansion in tests of this type. These proposals vary according to the correlation with field experience and with rock type. They may be influenced also by individual features of the test. In 1983, Oberholster[36] defined divisions as follows: innocuous, <0.08%; slowly expansive, 0.08–0.20%; and rapidly expanding, >0.20%. These limits applied to various rock types and were based on a test of 10 days' duration and compared with the results of ASTM C-227 mortar bar and concrete prism tests. In 1986, Oberholster and Davies,[1] again using the correlation with ASTM C-227 to interpret the potential for reactivity, defined a division between innocuous and potentially reactive at an expansion of 0.11% in 12 days. Davies and Oberholster,[37] after an interlaboratory investigation, proposed boundaries after 12 days of testing as follows: innocuous, <0.10%; slowly expanding, 0.10–0.25%; and rapidly expanding, >0.25%. Again several aggregates were used in this test. Shayan *et al.*[38,39] proposed that aggregate should be regarded as innocuous if the expansion was less than 0.10% in 22 days, slowly expansive if the expansion exceeded 0.10% in 22 days, and rapidly expanding if the expansion exceeded 0.10% in 10 days. Hooton and Rogers[23] considered that an expansion of less than 0.10% at 14 days represented innocuous material, 0.10–0.25% expansion was ambiguous, and greater than 0.25% was considered to be reactive. Expansion of between 0.10 and 0.25% meant that further testing and evaluation were required. This test, according to these authors, appeared to be the most promising in making the distinction between reactive and non-reactive aggregates, and they were able to distinguish all 12 aggregates in their set that were known to be reactive. Grattan-Bellew,[19] in 1989, again selected the boundary of innocuous as being less than 0.10% after 14 days but found that different magnitudes of expansion could be defined for differing potentially reactive rock types. Hooton[25] investigated various test methods using aggregates with known petrographic characteristics and field performance in concrete. He found the alkali immersion procedure to be the most promising in terms of distinguishing between reactive, marginal and non-reactive aggregates. He proposed a boundary of 0.15% expansion between innocuous and reactive 16 days after casting, 14 days in alkali, but ambiguous results suggested that the test should be continued for 28 days and 56 days with the boundary between innocuous and reactive being drawn at 0.33% and 0.48%, respectively. Hooton and Rogers[40] used the same boundary but suggested a marginal zone below the discrimination boundary. This helped in the recognition of potential reactivity for some granites and gneisses containing microcrystalline quartz and strained quartz as the only reactive component.Grattan-Bellew[19] considered that two discriminant criteria were required according to the field conditions to be expected. A general limit of 0.10% expansion after 14 days needed to be modified to 0.2% for greywackes and argillites, 0.15% for other aggregate types, while for limestones the limit was less than 0.10%. Berube *et al.*[24] tested 142 aggregates. They detected all but one of the known reactive aggregates of Quebec but found the method too severe for many aggregates which have a good field record.

The recently introduced Canadian standard[3] states that the maximum expansion value for alkali-aggregate reactivity is 0.15% at 14 days. Several aggregates that expand greater than 0.15% after 14 days have not caused deleterious

Table 4. Replicate length changes for standard bars 25 × 25 × 250 mm; laboratory A % expansion

Days	*1*	*3*	*11*	*14*	*28*
Bar number					
1	0.034	0.261	0.420	0.455	0.523
2	0.023	0.193	0.364	0.386	0.477
3	0.034	0.239	0.409	0.432	0.534
4	0.034	0.239	0.409	0.432	0.557
Mean	0.031	0.233	0.401	0.426	0.523
Standard deviation	0.006	0.029	0.025	0.029	0.033
5	0.034	0.170	0.375	0.398	0.477
6	0.034	0.170	0.386	0.409	0.466
7	0.023	0.159	0.386	0.409	0.489
8	0.034	0.182	0.420	0.443	0.534
Mean	0.031	0.170	0.392	0.415	0.491
Standard deviation	0.006	0.009	0.020	0.020	0.030
9	0.045	0.159	0.375	0.443	0.511
10	0.034	0.148	0.398	0.432	0.523
11	0.023	0.170	0.398	0.398	0.545
12	0.023	0.159	0.398	0.420	0.477
Mean	0.031	0.159	0.392	0.423	0.514
Standard deviation	0.011	0.009	0.011	0.019	0.028
13	0.000	0.091	0.432	0.455	0.534
14	0.000	0.091	0.443	0.466	0.545
15	0.011	0.102	0.443	0.466	0.557
16	0.034	0.102	0.386	0.420	0.489
Mean	0.011	0.097	0.426	0.452	0.531
Standard deviation	0.016	0.007	0.027	0.022	0.030
17	0.000	0.182	0.375	0.409	0.511
18	0.000	0.205	0.409	0.432	0.557
19	0.023	0.193	0.386	0.409	0.523
20	0.011	0.170	0.420	0.398	0.545
Mean	0.009	0.188	0.398	0.412	0.534
Standard deviation	0.011	0.015	0.021	0.014	0.021
Overall mean	0.023	0.169	0.402	0.426	0.519
Mean standard deviation	0.010	0.014	0.021	0.021	0.028
Norwegian cement					
1	0.030	0.110	0.360	0.410	0.460
2	0.020	0.130	0.370	0.340	0.360
3	0.030	0.120	0.350	0.380	0.440
4	0.030	0.140	0.340	0.380	0.480
Mean	0.028	0.125	0.355	0.378	0.440
Standard deviation	0.005	0.013	0.013	0.029	0.043

expansion in field structures. Therefore, expansion in excess of the recommended limit calls for further testing of concrete specimens. A lower limit of 0.10% is recommended for quarried siliceous limestone. There are reports of field concrete made with aggregates containing granites, gneisses, and granodiorites, that exhibit less than 0.10% expansion at 14 days in the accelerated mortar bar test.

ASTM C1260[2] states that an expansion of less than 0.10% at 16 days after casting (14 days in the NaOH solution) is indicative of innocuous behaviour in most cases. Some granitic gneisses and metabasalts have been found to be expansively reactive in field performance even though their expansion was less than 0.10% at 16 days after casting. Expansion of more than 0.20% at 16 days after casting is indicative of potentially deleterious expansion. Expansion between 0.10% and 0.20% at 16 days after casting includes both aggregates that are known to be innocuous and deleterious in field performance. For these aggregates, it is particularly important to develop supplemental information. In such a situation, it may also be helpful to take comparator readings until 28 days.

In Norway, at SINTEF — Structures and Concrete — a modified version of the alkali immersion procedure has been practised to investigate natural aggregates, including crushed rock and blended aggregates.[41,42] The expansion is monitored by measuring length change of 40 × 40 × 160 mm bars after 4, 7, 12, 14, 28, and 56 days. The result at 14 days is used to evaluate the potential expansivity of the aggregate — less than or equal to 0.10% is considered innocuous, between 0.10% and 0.25% should be considered to be deleterious but slowly expanding, and 0.25% and above should be regarded as potentially deleteriously reactive and rapidly expanding. In Norway, further testing of the aggregate is recommended when petrographic examination indicates the presence of 20% or more of potentially reactive rock types in the aggregate, whereas aggregates with less than 20% are regarded as innocuous.[28]

Table 5. Replicate length changes for standard bars 25 × 25 × 250 mm; laboratory B % expansion

Days	*4*	*7*	*14*	*28*	*56*
Bar number					
1	0.245	0.306	0.388	0.480	0.531
2	0.214	0.265	0.347	0.429	0.480
3	0.194	0.245	0.327	0.408	0.449
4	0.245	0.316	0.418	0.510	0.582
Mean	0.224	0.283	0.370	0.457	0.510
Standard deviation	0.025	0.034	0.041	0.047	0.058
5	0.265	0.327	0.429		0.582
6	0.235	0.286	0.388		0.531
7	0.255	0.316	0.418		0.571
8	0.276	0.337	0.429		0.592
Mean	0.258	0.316	0.416		0.569
Standard deviation	0.017	0.022	0.019		0.027
9	0.235	0.286	0.367		0.571
10	0.214	0.265	0.337		0.531
11	0.235	0.296	0.367		0.571
12	0.224	0.286	0.357		0.510
Mean	0.227	0.283	0.357		0.546
Standard deviation	0.010	0.013	0.014		0.031
13	0.276	0.347	0.449		0.745
14	0.255	0.316	0.418		0.714
15	0.245	0.306	0.418		0.714
16	0.255	0.306	0.418		
Mean	0.258	0.319	0.426		0.724
Standard deviation	0.013	0.019	0.015		0.018
17	0.224	0.316	0.408		0.582
18	0.255	0.367	0.449		0.653
19	0.245	0.337	0.418		0.622
20	0.214	0.306	0.388		0.571
Mean	0.235	0.332	0.416		0.607
Standard deviation	0.019	0.027	0.026		0.038
Overall mean	0.240	0.307	0.397	0.457	0.591
Mean standard deviation	0.017	0.023	0.023	0.047	0.034

Many of the discriminant criteria therefore propose that aggregate should be considered innocuous if expansion is less than 0.10% after a period of about 14 days in 1 M NaOH solution at 80°C, though some propose more stringent conditions. This discriminant boundary is based on a comparison with field performance, where the aggregate has shown no reaction in service, and on a comparison with other test results, such as the ASTM C-227 mortar bar test. Neither of these comparisons can be regarded as wholly reliable. The conditions in the alkali immersion tests are much more severe than those found in the field, and if the alkali content of the cement paste is generally moderate in the field concrete or the conditions are not aggressive, then a reaction may not occur, even though in other circumstances, a deleterious reaction may take place. An expansion of 0.10% in the test is not necessarily comparable with the expansion of concrete containing the same aggregate, but if an expansion of 0.10% actually occurred in service then some cracking is a likely consequence. In addition, it is possible that some expansion may not be due to alkali-aggregate reaction and the value obtained must be interpreted also in terms of the reproducibility of the measurements. Thus, while significant expansion may be simple to interpret and require an aggregate to be rejected, or special conditions attached to its use, the diagnosis of innocuous material is less unambiguous. The cause of expansion therefore must be identified by microscopic examination of the test mortars. It may be possible for the expansion limit to the innocuous field to be

Table 6. Replicate length changes for standard bars 40 × 40 × 160 mm; laboratory A % expansion

Days	*1*	*3*	*11*	*14*	*28*
Bar number					
1	0.047	0.119	0.332	0.356	0.533
2	0.024	0.095	0.284	0.296	0.462
3	0.024	0.107	0.249	0.273	0.415
4	0.047	0.142	0.344	0.379	0.581
Mean	0.036	0.116	0.302	0.326	0.498
Standard deviation	0.014	0.020	0.044	0.050	0.074
5	0.047	0.095	0.284	0.308	0.462
6	0.083	0.178	0.308	0.320	0.486
7	0.059	0.130	0.320	0.344	0.521
8	0.071	0.142	0.344	0.367	0.533
Mean	0.065	0.136	0.314	0.335	0.501
Standard deviation	0.015	0.034	0.025	0.026	0.033
9	0.047	0.130	0.296	0.320	0.521
10	0.059	0.190	0.320	0.332	0.545
11	0.095	0.107	0.273	0.296	0.498
12	0.036	0.095	0.249	0.284	0.498
Mean	0.059	0.130	0.284	0.308	0.516
Standard deviation	0.026	0.042	0.031	0.022	0.023
13	0.083	0.095	0.261	0.356	0.403
14	0.047	0.083	0.237	0.261	0.367
15	0.071	0.107	0.284	0.296	0.427
16	0.059	0.095	0.273	0.296	0.415
Mean	0.065	0.095	0.264	0.302	0.403
Standard deviation	0.015	0.010	0.020	0.039	0.026
17	0.047	0.119	0.249	0.308	0.332
18	0.071	0.095	0.237	0.284	0.344
19	0.024	0.107	0.225	0.284	0.320
20	0.024	0.083	0.213	0.237	0.308
Mean	0.041	0.101	0.231	0.279	0.326
Standard deviation	0.023	0.015	0.015	0.030	0.015
Overall mean	0.053	0.116	0.279	0.318	0.449
Mean standard deviation	0.019	0.024	0.027	0.034	0.039
Norwegian cement					
1	0.09	0.16	0.3	0.38	0.48
2	0.08	0.16	0.29	0.34	0.46
3	0.08	0.14	0.33	0.36	0.46
4	0.08	0.15	0.3	0.34	0.45
Mean	0.083	0.153	0.305	0.355	0.463
Standard deviation	0.004	0.008	0.015	0.017	0.011

Table 7. Replicate length changes for standard bars 40 x 40 x 160 mm; laboratory B % expansion

Days	*4*	*7*	*14*	*56*
Bar number				
1				
2	0.206	0.279	0.392	0.650
3	0.186	0.258	0.361	0.619
4	0.186	0.258	0.382	0.640
Mean	0.193	0.265	0.378	0.637
Standard deviation	0.012	0.012	0.016	0.016
5	0.206	0.279	0.372	0.671
6	0.196	0.268	0.372	0.692
7				
8	0.155	0.196	0.299	0.537
Mean	0.186	0.248	0.348	0.633
Standard deviation	0.027	0.045	0.042	0.084
9	0.258	0.320	0.434	0.754
10				
11	0.268	0.330	0.423	0.723
12	0.310	0.392	0.495	0.805
Mean	0.279	0.348	0.451	0.760
Standard deviation	0.027	0.039	0.039	0.042
13	0.258	0.310	0.403	0.723
14				
15	0.258	0.310	0.403	0.723
16	0.237	0.289	0.372	0.681
Mean	0.251	0.303	0.392	0.709
Standard deviation	0.012	0.012	0.018	0.024
17				
18	0.235	0.299	0.413	
19	0.240	0.310	0.413	
20	0.236	0.289	0.403	
Mean	0.237	0.299	0.409	
Standard deviation	0.003	0.010	0.006	
Overall mean	0.229	0.292	0.396	0.685
Mean standard deviation	0.016	0.024	0.024	0.041

Table 8. Mean percentage expansions and repeatability

Days	*Lab A*		*Lab B*	
	Mean expansion	*Repeatability*	*Mean expansion*	*Repeatability*
250-mm bars				
1	0.023	0.023		
3	0.169	0.137		
4			0.240	0.046
7			0.307	0.062
11	0.402	0.039		
14	0.426	0.044	0.397	0.086
28	0.519	0.048		
56			0.591	0.226
160-mm bars				
1	0.053	0.039		
3	0.116	0.050		
4			0.229	0.108
7			0.292	0.108
11	0.279	0.091		
14	0.318	0.061	0.396	0.105
28	0.479	0.199		
56			0.685	0.169

defined from the data obtained from such examinations for the given aggregate type. For this purpose, qualitative recognition of the presence of alkali-silicate gel in the mortar bars coupled with damage to specific aggregate particles will be sufficient to demonstrate that an alkali-aggregate reaction has occurred.

It is evident from the foregoing paragraphs that there is little agreement or rigour concerning the values to be adopted for discriminating between reactive and innocuous materials. Consideration has also to be given to the repeatability of the test within the laboratory and the possible reproducibility in comparing laboratory results in order to interpret the discriminant criteria safely. The question to be answered should be either 'What is the probability of an aggregate being reactive in service, if it exhibits a particular level of expansion in the accelerated test?', or 'What is the probability of an aggregate being innocuous in service if it exhibits a particular level of expansion in the test?'.

Influence of alkali volume

Some experiments have been carried out using differing ratios of 1 M NaOH to the surface area of the aggregate. The results of this work lead to a conclusion that the amount of expansion increases progressively as the ratio of the volume of alkali to the surface area of the aggregate increases. This relationship is shown in Fig. 1. The result obtained from this work shows that the percentage expansion for the aggregate under test is given by $10.5(m/LD)^{0.25}$, where m is the mass of alkaline solution in the vessel in kilograms, and L and D are the length and breadth of the mortar bar in millimetres. It is evident from this that once the ratio exceeds 4 g/cm^2, the change in expansion is small but continuing. It is essential, therefore, that this ratio is fixed, and it is prudent to ensure that individual sets of bars containing specific aggregates under test are placed in their own containers with a specific aliquot of freshly prepared sodium hydroxide solution.

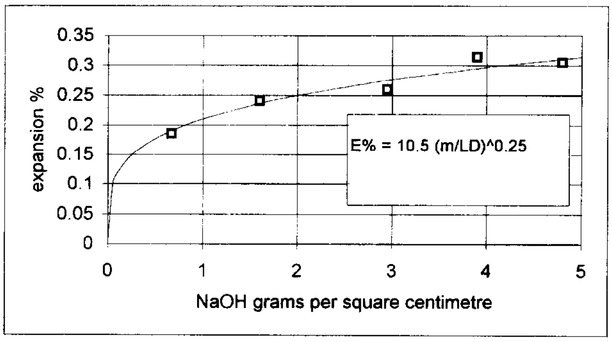


Fig. 1. Expansion vs. mass of alkali (grams/square centimetre of mortar bar surface) after 7 days in alkaline solution.

Cement type

The specification for accelerated mortar bar tests gives no requirement for the type of cement to be employed. Davies and Oberholster[43] investigated the effect of the cement composition upon expansion, using three different types of cement, all with different alkali contents and with differing fineness. They concluded that the different cement types had little influence upon the measured expansion. Grattan-Bellew[19] found that the expansion made with the cement having an alkali content of 1.08% was identical with that of bars made with a cement having an alkali content of 0.66%. Fournier and Bérubé[44] investigated the effect of cement composition on expansion by using ten different types of cement. They found no clear correlation between the chemical composition of the cement and the expansion obtained. However, cements with higher fineness values exhibited a higher degree of expansion, even if they did not have the highest alkali content. In the experiments reported here, no significant differences were found for the three cement types employed.

Practical considerations

The manufacture and measurement of the bars measuring 160 x 40 x 40 mm were found to be more convenient than the measurement of the longer bars. For the conventional ASTM C-227 mortar bar method, it has been found that the rate of expansion increases with increasing cross-sectional dimensions of the test specimens up to at least 100 mm.[45] In addition, Locker[46] reported a greater expansion for the 40 x 40 x 160 mm bars than with ASTM C-490 bars (25 x 25 x 285 mm) in tests that otherwise followed the ASTM C-227 mortar bar method. The results of the experiments carried out in the present work show that there is little or no difference in the percentage expansion of the thicker shorter bars than for the longer bars if other factors are constant. However, the repeatability of the measurements expressed as a percentage of the length of the bars is larger for 160-mm bars than it is for the 250-mm bars. Both systems of manufacture of the mortar bars proved satisfactory, but it was more difficult to ensure that the stainless-steel balls were attached to the mortar than it was for the inserted threaded measuring points. However, the threaded measurement points were found to be slightly more difficult to use with the loose ball-bearings than with the fixed bearings. Great care was taken in establishing the lengths by taking four readings at each reading interval. The time interval over which individual measurements were made was always only a few seconds following removal from the solution at 80°C. The effect of falling temperature was measured and is illustrated in Fig. 2. This shows the possible contribution of variation in time interval required for the measurement on the measurment error. The number of measurements carried out may have been excessive because the process takes a long time and hence gives a potential for continued expansion during the measurement period. This could influence the value of the repeatability at each time. Duplicate measurements for each bar might have sufficed.

Pessimum aggregate proportions

Shayan *et al.*[38] showed that a sandstone exhibited the pessimum effect in the accelerated test, and Shayan[47] has recorded a pessimum for the expansion of accelerated mortar bar tests using opal. The maximum expansion occurs where opal is 5% of the aggregate. This was the lowest opal level employed. The explanation for this is considered by Shayan to be due to the early generation of gel in the outer parts of the mortar bar. The larger amounts of reactive aggregate produce large amounts of gel that make the outer mortar an effective filter for NaOH. We have observed that the penetration of alkalies is in all cases slow and progressive and that bars of all aggregate types that we have tested react first in the outer zones (e.g. Wigum and French[48]). The bars made by Shayan were described as 'moisture-saturated' before being placed in the alkaline solution. In our experiments, the bars are also kept in water or alkaline solution. This may be a very important aspect of the test because if the bars are allowed to dry fully, they will exert a suction pressure on the alkali solution, and alkalies will penetrate rapidly. If the bars are maintained in a saturated condition, the transference of the alkalies will be by diffusion along a concentration gradient and will be retarded by chemical fixation in cement hydration products. The distribution coefficient of Na^+ and OH^- ions for equilibria involving cement hydrates, reaction products and pore fluid, and the diffusion coefficient for the Na^+ and $(OH)^-$ ions in the mortar system therefore will regulate the rate of reaction and the observed expansion. A large surface area for reaction will retard penetration of the diffusing species. It is therefore very important that

consideration be given to the amount of potentially reactive material in the mortar bar, which will influence the rate of ingression of alkalies, and

that the transference of the bars from the water to the alkalies be carried out under standard moisture conditions.

Further experimentation is required to confirm the magnitude of the difference in expansion created by allowing the bars to dry before placing them in the alkali solution relative to their remaining effectively saturated, but a variation in the rate of alkali penetration could have a very large effect on the reproducibility of the test.

With respect to the creation of a pessimum expansion, it is vital that the test be arranged to detect such possibilities. The reaction for most real aggregates involves only a small fraction of the rock with perhaps 1 or 2% of the rock actually taking part in reactions. Opal con-

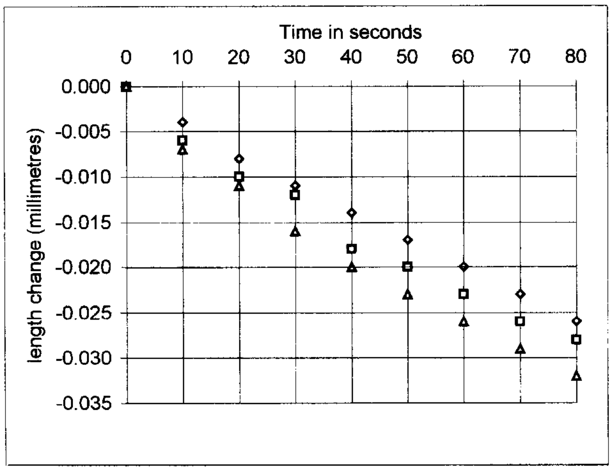


Fig. 2. Influence of falling temperature on length of mortar bars.

versely is likely to be wholly reactive, and the presence of 5% opal certainly should be detected before testing. Hence, for many aggregates, it may be prudent to test the material alone and with 50% replacement with an inert material. If the expansion is changed by a significant amount, in terms of the measured repeatability, on dilution, then either the aggregate might be rejected or further testing undertaken to define the pessimum relationship more precisely. The significant change in expansion must be defined in the terms of the repeatability of the measurement, the test being repeated if, say, the expansion difference exceeds the repeatability.

STATISTICAL APPRAISAL OF TEST DATA

Variation of standard deviation with mean expansion

A graphical display of the data for both 250-mm and 160-mm bars shows that the variance changes with the mean, i.e. the data are heteroscedastic, and hence log-transformation stabilises the variance, and the plot of $\log_{10}$ [standard deviation] is found to increase linearly with the mean. Also, the trends for lab A conform with those for lab B for both sets of bars with the exception of two sets of data for 160-mm bars for lab B (see Fig. 3). Visually, it appears that no distinction can be made between 250- and 160-mm bars, and this was checked by calculating the Kolmogorov–Smirnov two sample goodness-of-fit statistic. This showed that no difference can be found between the two sets of data for the two laboratories. Using a robust least-squares regression method trimmed to downweight outliers,[49] it was found that residuals were normally distributed, and a plot of residuals against estimated values showed no evidence of non-linearity. A linear model for $\log_{10}(s)$ variation with mean expansion is therefore satisfactory and gives

$$\log_{10}(s) = -1.8725 + 0.5867E,$$

where E is the percentage expansion.

The standard deviation (σ_0) for zero expansion is then a mean value of 0.013% expansion. The upper bounds for the standard deviation can be obtained from the distribution of the residuals of the regression equation. The 97.5th and 99.5th percentiles give multipliers of 2.600 and 2.745, respectively, which are upper bounds on the standard deviation for a given value of mean expansion based on the corresponding upper one-sided 95% and 99% prediction intervals. Hence

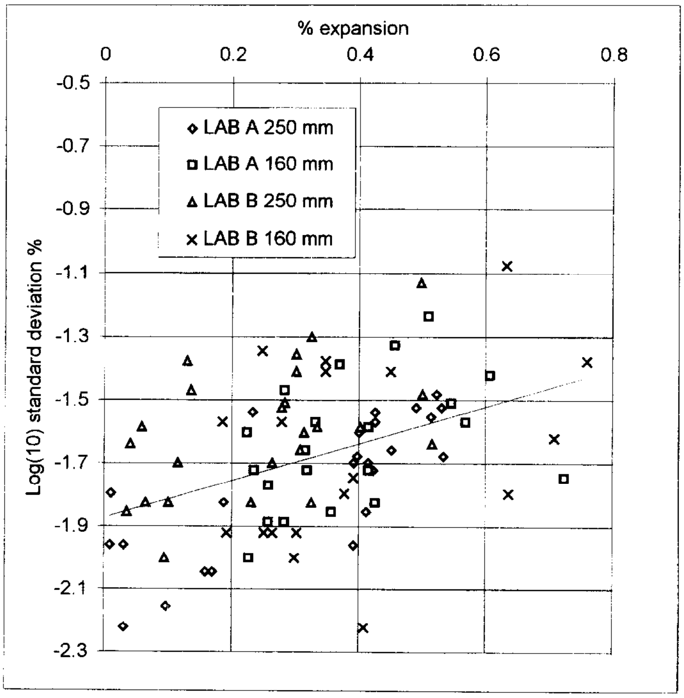


Fig. 3. Observed spreads of bar-expansion as a function of mean expansion for laboratories A and B and for 160- and 250-mm bars.

Table 9. Predicted variation in standard deviation with mean expansion

Mean expansion %	s_{mean} *expansion %*	s_{95} *expansion %*	s_{99} *expansion %*
0.00	0.013	0.035	0.037
0.10	0.015	0.040	0.042
0.15	0.016	0.043	0.045
0.20	0.018	0.046	0.048
0.25	0.019	0.048	0.052

$$\log_{10} s_{95} = -1.8725+0.5867E+0.4150, \text{ and}$$

$$\log_{10} s_{99} = -1.8725+0.5867E+0.4386$$

and the variations of the estimates of standard deviation with the mean expansion at various levels of confidence are as given in Table 9. Since it is common to consider that an expansion of $<0.10\%$ over a fixed period of time, as for example specified in ASTM C-1260, is not significant, this must represent the safe limit of measured expansion that could occur by chance or through some process of no significance for the durability of the concrete. Taking the safe estimate of s for zero expansion as 0.037% means that the limit of 0.10% is 2.70 standard deviations above the mean. As shown below, this figure (0.037%) is of general application, and the factor of 2.7 is close to the often-quoted safe limit of 2.75 standard deviations. The coefficient of variation given in ASTM C-1260 of 9.55% gives the variation with mean expansion summarized in Table 10.

The work of Oberholster[15] on repeatability gives an upper bound to the expected standard deviation of 0.05% at 0.250% expansion. This is of the same order as found in the present work. The expansion of 0.10% corresponds to about three times the estimated upper bound of the standard deviation if the distribution of data of tests is similar to that of the present work. The ratio N = mean expansion/0.037 therefore gives a measure of the potential for deleterious expansion. Expansion after the specified time giving a ratio of below $N = 3$ means that the test has failed to show up a potential for adverse alkali-aggregate reaction, whereas a ratio of, say, 8 might be taken to mean a potentially severe reaction. An expansion of, say, $N = 4$ may not always indicate a potential for deleterious alkali–aggregate reaction since it could represent a potential for expansion from some cause other than AAR combined with random error. Nevertheless, a value of $N = 4$ would make it unsafe to accept the aggregate. However, expansion cannot be used alone to identify AAR as the cause of expansion, and the cause of observed expansion must be found independently. Mortar bars therefore must be examined for evidence of adverse reaction using optical or electron microscopy of thin sections. Combining the test result with a petrographic examination leads to schemes for the interpretation of the likely behaviour of the aggregate such as that given in Table 11. This is particularly economical since the cost of the petrography can be less than half the cost of repeating the test and is also substantially quicker. It is also superior to the use of control tests because it provides different information and because the standard error in the measurement of the difference between a control and a test aggregate will be over 0.05%

Table 10. Comparison of mean predicted standard deviation based on ASTM C-1260 with present work

Mean expansion %	*ASTM C1260 precision (s) % expansion*	*Estimated mean s % expansion from present work*
0.00	0.006	0.013
0.10	0.010	0.015
0.15	0.014	0.016
0.20	0.019	0.018
0.25	0.024	0.019

How many tests?

In the present work, three and four mortar bars were used to obtain a single result. ASTM C-1260 provides for the manufacture of three bars. Since random errors can occur in the manufacture and measurement of the sets of bars, it seems desirable that at least two sets should be made for each aggregate. However, the number of tests required depends largely on the precision required for the measured expansion. ASTM C-1260 says that two laboratories carrying out the same test properly should not differ by more than 27% of the mean expansion. This very large difference presumably represents three standard deviations. It could obviously mean that while one laboratory would find an aggregate reactive, another might find it innocuous by the simple criteria currently defined. It is also intuitively obvious that where a result is close to the discriminant criterion, a

Table 11. Possible scheme for interpretation of test results

Expansion after 14 days/0.037	*Probability of system being reactive according to test*	*Evidence of AAR in thin section of mortar**	
		No	*Yes*
> 4	$> 99.9\%$	Reject	Reject
> 3–4	> 99–99.9%	Establish cause of expansion, consider repeating the test, consider use of PFA, GGBS	Reject
2–3	> 95–99%	Establish cause of expansion, consider repeating the test, continue test to 28 days, consider use of low alkali cement, PFA, GGBS, etc.	Reject
<2	$<95\%$	Accept aggregate	Consider repeating test or using low alkali cement, PFA, GGBS, etc.

*The qualitative recognition of gel in the thin section and of loss of aggregate or cracking within specific aggregate fragments is taken to be definitive. It may also be possible to assess the magnitude of reaction in terms of crack frequency/unit area and gel volume.

larger number of measurements are necessary than if a large expansion is measured.

The risks involved in decision making are α and β, which are defined as follows:
Decision based on experimental mean $\bar{x}$

Truth (which is unknown)	No expansion $> 0\%$ occurs	Expansion $> \delta\%$ occurs
No expansion $> 0\%$ occurs	$P_{\mathrm{correct}} = (1-\alpha)$	$P_{\mathrm{wrong}} = \alpha$
Expansion $> d\%$ occurs	$P_{\mathrm{wrong}} = \beta$	$P_{\mathrm{correct}} = (1-\beta)$

For the purposes of illustration, suppose that $\alpha = 0.01$ and $\beta = 0.001$, i.e. 1% and 0.10% risk, respectively. The corresponding quantiles of the cumulative normal distribution are then $Z_\alpha = 2.326$ and $Z_\beta = 3.090$. Since $\mu = 0.0$, $\sigma_0 = 0.037$ and if the change δ to be detected is $+0.10\%$, the classical normal theory estimate of the required sample size is

$$\hat{N} = \left[\frac{(Z_\alpha + Z_\beta)\hat{\sigma}_0}{\delta}\right]^2 = \left[\frac{5.416 \times 0.0365}{0.10}\right]^2$$

$$= 3.9 \rightarrow 4 \text{ replicates.}$$

However, Kupper and Haffner[50] showed that since this traditional and widely used formula is based on large-sample approximation theory, it gives estimated sample sizes that are too small; an effect that becomes worse as N becomes smaller. Their tables show that in order to have a 95% tolerance probability that μ is estimated with a confidence interval of $100\alpha\%$, the desired sample size is $N_{\mathrm{adj}} = 11$ rather than $N = 4$. A working approximation to their one-sample table for a 95% tolerance is given by

$$N_{adj} = 4.76 + 1.69\, N^{0.926}.$$

The corresponding critical value for the test criterion is given by

$$\bar{x}^* = \mu_0 + \sigma_0 Z_\alpha / \sqrt{\mathrm{N}_{\mathrm{adj}}} = (\mu_0 + \delta) - \sigma_0 Z_\beta / \sqrt{\mathrm{N}_{\mathrm{adj}}}.$$

Following the experiment, if $\bar{x}_{\mathrm{obs}} > \bar{x}^*$ then we accept the alternate hypothesis that $\mu_1 > \mu_0$ with at least a $100(1-\alpha)\%$ confidence that this decision is correct. If $\bar{x}_{\mathrm{obs}} < \bar{x}^*$, then we accept that $\mu_1 = \mu_0$ with at least a $100(1-\beta)\%$ confidence that the decision is correct, since the new population mean is $<(\mu_0 + \delta)$. These lead to the following estimates of sample sizes and corresponding critical values.

$\mu_0 = 0$	$\alpha = 0.01$	$\beta = 0.001$		
	$\delta = 0.10$	$\delta = 0.15$	$\delta = 0.20$	$\delta = 0.25$
$N_{\mathrm{adj}} =$	11	8	6	6
$\bar{x}^* =$	0.026	0.030	0.035	0.035.

If the limit of expansion is set lower than 0.10% then the number of measurements required becomes large because of the large standard deviation. For example, if the limit is set at 0.05%, the number of tests required is 27. If expansion at an early age is high then the number of replicates can be low, but avoidance of ambiguity at lower expansion requires consideration of the number of extra tests to be started and the length of time for which the tests are to be continued.

Assessment at an early age

Expansions measured at 1, 3, and 7 days provide useful data on the eventual expansion and on the number of tests required and the length of time required for the test. We make use of the fast initial response cumulative sum (FIR CUSUM) method[51] to detect drift of the mean expansion away from zero at as early a stage as possible. We are only interested here in positive drift, and the upper FIR CUSUM on the *i*th day is given by

$$S(U)_i = \max\{0, x_i - K + S(U)_{i-1}\},$$

where

$$S(U)_0 = K$$

and $\bar{x}_i$ is the observed mean expansion; H is the critical threshold, usually defined in terms of either a number of standard deviations above the mean or a given value. Here, H is set at 0.10% (corresponding with a drift of $2.74\sigma_0$); and $K = H/2 = 0.05\%$ is the headstart parameter, which ensures a rapid response of the monitoring process if the system has initially drifted away from the expected value of zero mean expansion. For example, consider the first batch of 250-mm bars of Table 4. These are listed in Table 12 along with the corresponding cusum values. This list shows that the critical expansion level is exceeded after only 3 days, and that four bars will suffice. As an example, the second value in the list for these mortar bars is found from $x_i = 0.034$, $K = 0.05$, $S(U)_{i-1} = 0.233$. The second part of the table shows a slow rate of expansion that might lead to an ambiguous condition after 14 days so that at least two and preferably three batches need to be tested. Here, there is evidence of real expansion but at a rate that is ambiguous during the early stages but might reach the critical level after 28 days.

Table 12. Illustration of calculation of FIR CUSUM

Day number	High expansion		Intermediate expansion	
	Mean expansion %	*FIR CUSUM*	*Mean expansion %*	*FIR CUSUM*
1	0.034	0.034	0.008	0.008
3	0.233	**0.217**	0.034	0.000
11	0.401	**0.568**	0.053	0.003
14	0.426	**0.944**	0.088	0.041
28	0.523	**1.417**	0.122	**0.113**

$N = 4$ bars, $H = 0.10$, and $K = 0.05$.

Contributions to variability

The contributions to the standard deviation of the expansion at any one time can derive from many sources. If the overall deviation increases with the mean then the deviation must include items that are related to the mortar itself rather than the system of measurement and storage. Consideration of the sets of 160-mm bars for laboratory A shows that the expansion is given by

$$E\% = kt^{0.5},$$

where t is the time in days. The slope of the straight line ($y = kt^{0.5} + c$) varies substantially (Figs 4 and 5). The range has been identified in this case as reflecting the ratio of the volume of alkali solution to the surface area of the prism. Other variables that would affect the slope include the proportion of reactive aggregate, the water/cement ratio, and the compaction of the mortar. Contributions to the measurement error include variations in the temperature of measurement, the limitations of the measurement system, wear and contamination of the end pieces and the precision with which the time of measurement is recorded. Consideration of the data for the five sets of 250-mm bars of laboratory A shows that for both measured results and the calculated expansion derived from a linear best-fit of expansion with root time, their standard deviation is close to 0.02%. However, the difference between measured and calculated expansion is large. This derives mainly from the measurements at 28 days where, for example, the mean expansion is 0.519%, whereas the calculated expansion is 0.568%. Examination of the data suggests that expansion is not linearly related to the square root of time after 14 days. If only expansions for up to 14 days are considered then the mean expansion and calculated expansions for 14 days are 0.427% and 0.430%, and the standard deviations are 0.016 and 0.011, respectively. The slope constants are very similar (0.1243, 0.1225, 0.1243, 0.1376, and 0.1246).

The best-fit straight lines drawn on Figs 4 and 5 intersect the expansion axis at negative values. This is interpreted as reflecting differences in the time of inception of the reaction due perhaps to differing diffusion rates for alka-

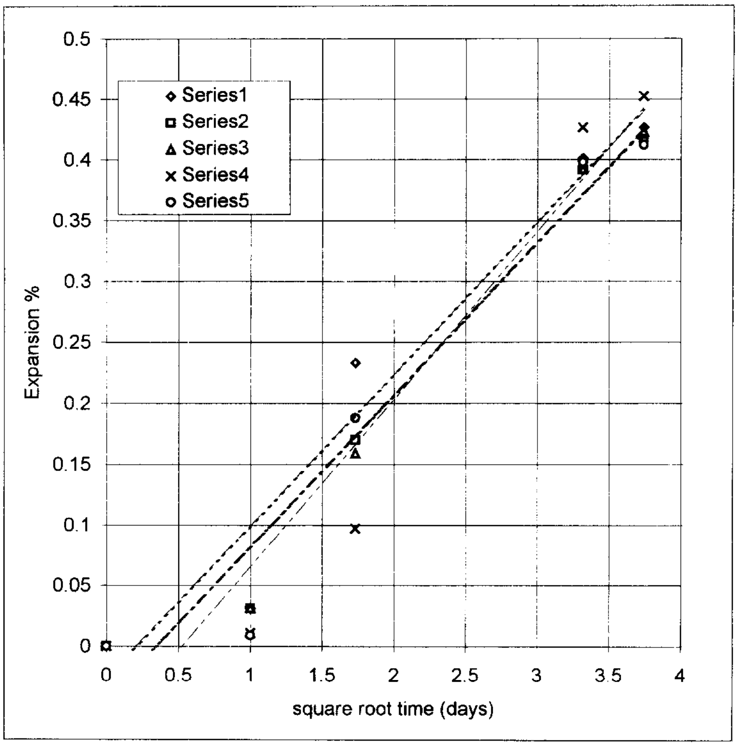


Fig. 4. Expansion vs. time for 250-mm long bars.

lies penetrating into the mortar. The 160-mm bars have a longer dormant period than the thinner 250-mm bars. The simple relationships also cannot be extrapolated to longer periods of time. However, two approaches have given excellent correspondence between modelled and actual expansion versus time. The first of these is

$$E\% = \beta_1(t-\beta_2)^{\beta_3} \qquad (1)$$

and is illustrated in Fig. 6 for the 250-mm bars of Table 4. This figure gives the three param-

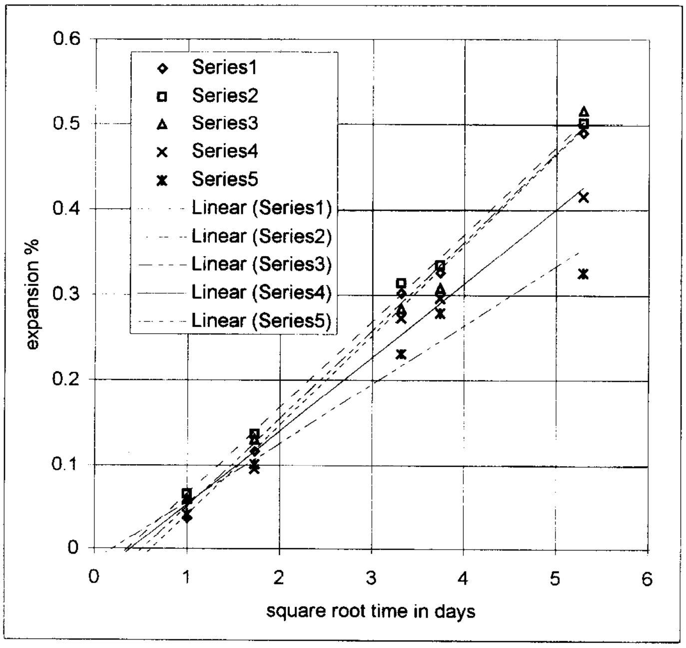
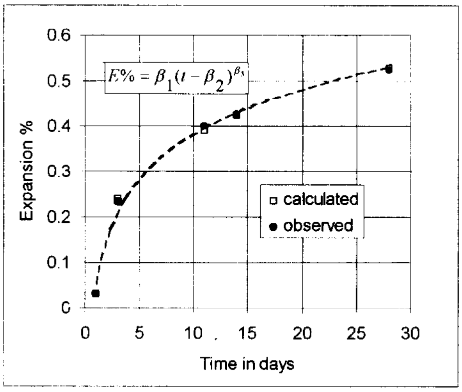


Fig. 5. Expansion vs. time in days for 160-mm long bars.

Series	β_1	β_2	β_3	R^2
1	0.19461	0.99767	0.3032	0.999
2	0.15733	0.98971	0.3598	0.980
3	0.14592	0.98201	0.3593	0.982
4	0.12610	0.99639	0.4608	0.938
5	0.15607	0.99947	0.3790	0.994



Data for series 1.

Fig. 6. Fitted expansions as function of time; model (1); plotted example is series 1. R^3 is coefficient of determination.

series	k	c	R^2	sd
1	0.1449	0.0484	0.993	0.014
2	0.1436	0.028	0.994	0.013
3	0.1504	0.0192	0.994	0.014
4	0.1716	-0.0215	0.963	0.040
5	0.1565	0.0116	0.998	0.008
mean	0.1534	0.0172	0.996	0.011

where % expansion = k*ln* t + c and
t is time in days. sd is the standard deviation of the residuals

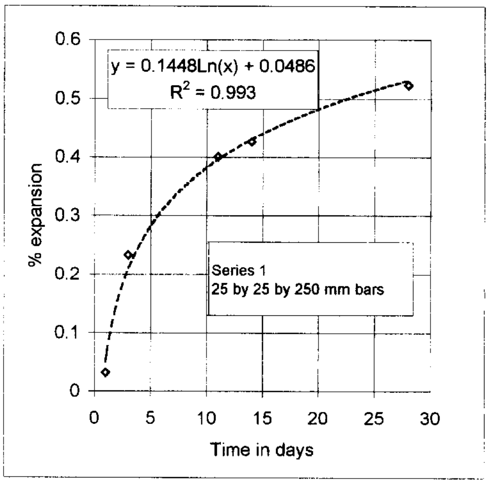


Fig. 7. Fitted expansions as function of time; model (2); plotted example is series 1.

eters of the formula and the coefficient of determination (R^2). Equally useful is the equivalent relationship

$$E\% = k\ln t + c, \qquad (2)$$

which is illustrated in Fig. 7. For both of these expressions, the residuals are very low with the main contributions to the residuals coming from expansion at early ages. The measurement system used to record the changes in length of the mortar bars allows each measurement to have a precision of $\pm 2\,\mu$m. If this is taken to be the standard deviation of each individual measurement, the standard deviation of a given length should be 4 μm. An increase in this standard deviation will derive from slight variations in the temperature at which the bars are measured. This gives a measurement standard deviation approaching 0.001% where the measured expansion is 0.4%. Hence, the range found in each set of four bars is not due to measurement error. This range can be compared with a standard deviation of 0.048% for 41 laboratories in the interlaboratory study made by Rogers *et al.*[53] for mortar with a mean expansion of 0.364% at 14 days. This is in keeping with the standard deviations obtained in the present work which are in the range of 0.02–0.03%.

All these figures evidently must include factors beyond those of the experimental measurements. The most likely cause of this large range is a variation in the composition of the mortar bars, and consideration must be given to the influence of the amount of reactive material in the mortar bars. We have carried out some experimental work on this. These influences will include a variation in the total amount of aggregate, a variation in the proportion of the reactive component in each given size range, and a variation in the grading curve of the aggregate. We have studied all three variables. The variation in the amount of reactive component in the individual size ranges has been found from Blyth's[54] improved binomial confidence limits based on the inverse Beta distribution. The variation in the amount of each given particle size in a single bar has been measured experimentally, as has the variation in total mass of aggregate in each bar. The standard deviation of the total amount of aggregate in each bar was found to be in the range $\pm$1.6–1.9% of the amount expected. The variation in amounts falling in each grading division for the sand used to make the specially graded aggregate was found to be as follows:

Size range (mm)	Mass %	Standard deviation mass percentage
2.36–5.0	7.1	0.62
1.18–2.36	8.4	0.60
0.60–1.18	16.9	0.49
0.30–0.60	51.3	1.27
0.15–0.30	16.3	0.97.

The amount and standard deviation for the reactive blend, obtained by considering volume proportions for the grading curve of the mortar bars, were found to be as follows:

	Amount mass %	Standard deviation (calculated mass %)
2.3–5.0 mm	10%	0.54
1.18–2.36	25%	0.76
0.60–1.18	25%	0.76
0.30–0.60	25%	0.76
0.15–0.30	15%	0.63.

The standard deviation of the glass in the two size ranges in our mortar bars was calculated to be 2.96 g in 37.5 g, and this, combined with the standard deviation for variation in the total amount of aggregate, gives a standard deviation of 1.2% to be attached to the 15% of glass.

These relationships were also modelled using a computer-based simulation. For the purposes of the simulation model, it is assumed that in any single trial, the total weight of the ith size fraction, w_i, is drawn from a normal distribution $N(\mu_i, \sigma_i)$, $i = 1,6$, where parameters are defined by the experimental results. An equivalent number of grains in the ith fraction is estimated from $238.732w_i/(r_i^3\rho_i)$, where r_i is the geometric mean radius (mm) of the size range, and ρ_i is the relative density. In the 1.18–2.36-mm fraction, which is the only one to have both glass and other sand, the number of glass grains expected to be present, n_g, is modelled in each trial by a random sample from a binomial distribution $B(n_g,\alpha)$, where α is the expected proportion of occurrences of n_g grains. The diameters of these glass grains are then assigned by sampling from a uniform distribution in the range 1.18–2.36 mm. Since the density of the glass is known, the equivalent individual grain weights can be calculated, summed, and the weight of sand other than glass obtained by difference from the overall weight of the size fraction. The total sample weight is then given by summing over all the simulated size grades and the overall percentage of glass is found. It was discovered that the simulated glass percentage had a small variable bias dependent on the magnitude of the difference between the glass and sand densities. This bias was corrected by setting α to 0.70. The results obtained from two independent sets of 10 000 trials were then as follows:

	Run 1	Run 2
% glass	15%	15%
Standard deviation of glass	0.80%	0.78%
Total sample weight	248.89g	248.89g
Standard deviation (g)	4.82g	4.75g

The result obtained therefore corresponds reasonably with the experimental values, indicating that the standard deviation to be attached to the 15% of glass is 0.8%.

If it is assumed that the expansion is related to the glass content and the error bar in glass content is related to the variation in expansion, then for an expansion of 0.40%, the standard deviation would be 0.02% or 0.03%, depending on the value used for the standard deviation — in keeping with the actual standard deviation found experimentally. It is of course evident that the relationship between the chosen aggregate proportion and the pessimum will affect the relationship between aggregate amount and expansion. Variation will be highest on the limbs of the pessimum curve and should be minimal close to the pessimum proportion or at the extreme limits of the pessimum. For any given rock combination therefore the range will be dependent on the rock that is reactive and its pessimum proportion and the particular aggregate composition with respect to the pessimum. A high range would mean that the mixture is on the steepest part of the pessimum curve. Further work is therefore required to evaluate the effect of the pessimum on the range of results to be expected for a given test procedure. It can, however, be concluded that the range of results obtained will give an indication of whether the aggregate mixture is close to the pessimum. It is also clear that the interlaboratory reproducibility will depend substantially on the pessimum composition and the statistics of the aggregate distribution between individual bars.

Towards a reliable test

Alkali immersion and autoclave methods offer excellent potential for development of a test but at present the likely interlaboratory reproducibility means that great care is required in the interpretation of the result. Tighter specification is required to minimise random errors and to evaluate possible influences of the pessimum on the results obtained. It may prove necessary to blend aggregates in order to improve diagnostic quality. It is evident that performance of an aggregate in service is not alone a sufficient indicator of the reliability of the test unless this is accompanied by other details of the field conditions, mix design, and alkali levels in the field concrete and the proportions of lithologies in the concrete.

It is easier to reject than to accept since if expansion exceeds the reproducibility of the test or three times the bounding standard deviation (in the present case, 0.037) and the mortar bar contains obvious alkali silicate gel, then the aggregate must be regarded as unreliable. It may be used with given suitable selections of mix design and cement type. If the expansion approaches or slightly exceeds the reproducibility without gel formation, the cause of the expansion must be found. If the measured expansion is less than the expected reproducibility, then ambiguity may remain, and the test needs to be continued and repeated. If a test result of 0.14% expansion is observed then, although this is less than the discriminant value given by Hooton,[25] the reproducibility would imply that the result is actually, say, 0.14 ± 0.08% and that there is about a 40% probability that the discriminant value would be exceeded in another test. Such a material could not be used with confidence. Clearly, the reproducibility of the test must be improved by an order of magnitude if it is to be used alone for engineering decisions involving the acceptance of aggregates that at present would be regarded as marginal. In view of the variation in aggregate proportions found for the present standard mortar bars, this does not mean an improvement in the system of mixing, measuring or storing, but an improvement in the representativeness of the aggregate in each bar. This could be achieved by the use of more bars or larger bars.

Procedural suggestions

(1) The improvement in test result by replication is evident from Tables 4–7. If all five results were taken to give a single test result, after 14 days the expansion would be as follows:

Laboratory A	160 mm bars	0.318% ± 0.027
	250 mm bars	0.426% ± 0.020
Laboratory B	160 mm bars	0.396% ± 0.047
	250 mm bars	0.397% ± 0.038

However, the number of bars required increases as the expansion declines, and a method such as the FIR CUSUM calculation should be used to study the progress of the expansion, and, if necessary, the number of bars should be increased to give three sets of at least four bars.

(2) The length of the bars is important, and marginally better results from the statistical point of view are to be obtained using 250-mm bars or longer than for the shorter bars. However, the thicker bars are easier to work with, have less tendency to cool during measurement and take the threaded end piece very well. Perhaps consideration should be given to the use of bars of, say, 300 × 40 × 40 mm, which would have substantially more aggregate and therefore reduce the effect of variation in aggregate amount and composition.

(3) The storage conditions greatly influence the result obtained and the early age variability appears largely to reflect the ratio of the volume of alkali solution to the surface area of the bar. This should be fixed for each bar or individual set of four bars at a volume ratio of four, and bars should not be stored in previously used alkali or collectively in large tanks. The small difference between the shorter and longer bars found for laboratory A appears to be largely attributed to variations in the volume ratio of alkali solution to mortar.

(4) The mortars should be kept wet at all stages since drying is likely to accelerate alkali ingression.

(5) In interpreting the result of the test, it is important that the aggregate tested matches that to be used in service. Separating individual rocks will be misleading. Opal obviously will give a strongly adverse reaction when present to a small extent but may appear innocuous at high concentrations. Tuffisite veins

may be reactive when tested alone but show no evidence of reaction when tested in an as-quarried mixture.

(6) It may be prudent to test certain aggregates in blends of 1:1 or other ratios with a known inert material with any significant difference in expansion between the pure aggregate and the blend being regarded as indicating a potential for reaction that requires further investigation.

(7) It is essential that a control aggregate and an inert aggregate should be available that can be tested along with the aggregate under investigation and with the same cement.

(8) Petrographic assessment of the cause of expansion is essential to the interpretation of the result obtained. Formation of recognisable amounts of gel coupled with damage to the aggregate in the test period should indicate rejection of the aggregate.

CONCLUSIONS

(1) Consistent results were obtained with a 14-day expansion of 0.4% which gave an upper bound standard deviation of 0.0365% expansion at zero expansion for both sizes of mortar bar. The figure for this upper bound of the standard deviation can be used as a unit of measurement of expansion with more than three times the standard deviation, indicating that significant expansion has occurred.

(2) Expansions less than 0.11% can arise from random errors, and expansion near this value requires the test to be conducted using at least three batches of four bars. The number of bars to be made and the duration of the test should be considered 3–4 days after the initiation of the first set of bars, but the current recommendations are inadequate for assessment of intermediate to low degrees of reaction. Consideration should then be given to making three sets of four bars as separate individual mixtures.

(3) In keeping with previous work, no significant differences were found with respect to the cement type or the details of the aggregate grading. The size of the mortar bar, however, is important, with large bars being easier to work with. The ratio of the volume of molar sodium hydroxide solution to the surface area of the mortar bar also critically affects the amount of expansion. This should be fixed at a ratio of 4:1, and a separate container should be used for each set of bars. It is possible that the rate of expansion is affected by the exchange of components with the ambient solution and hence will be influenced by the presence of bars of other mixtures in the solution or if the solutions employed have previously equilibrated with mortar.

(4) The accelerated mortar bar test such as the ASTM 1260-94 has considerable advantages over the slower and unreliable ASTM C-227 and over the unreliable ASTM C-289 for engineering purposes and, given appropriate quality control measures, should provide useable data. Experience is needed in interpreting the test result, particularly bearing in mind the differing styles of reaction that occur with different aggregates, and the differing rates at which expansion can develop. It is essential that the aggregate and the mortar bars should be examined petrographically in order to assess the nature of the material that is actually reactive and the style of reaction that is taking place, if any. Consideration of a scheme such as that given in Table 11 is required for the interpretation of the test result.

(5) The various forms of concrete prism test have the same problems of unknown repeatability and reproducibility as have affected the accelerated mortar bar tests. In addition, because the expansions are generally much smaller and require longer periods for their measurement, the concrete prism tests may be more difficult to interpret than the mortar bar tests. They have, of course, the advantage that the aggregate can be tested in a size closer to that in which it will be used in practice, and this may be particularly important where possibly slow, late expanding aggregates are involved. Proper correlation and close comparison with field experience and the observa-

tions of the bars themselves are the best guide to the validity of the results obtained in the accelerated mortar bar test, and, as was shown by Brown[52], the test can be carried out on aggregate removed from concrete damaged by the reaction if this is necessary. However, the use of field experience in interpreting both positive and negative test results may be unsatisfactory unless the full details of the field occurrence are available.

ACKNOWLEDGEMENTS

This study has been funded by the Norwegian Research Council, where it is part of the NORCON project. The paper is a part of B. J. Wigum's Dr.ing-study, which is supported financially by the Royal Norwegian Council for Scientific and Industrial Research.

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