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Accelerated Laboratory Testing for High Early Strength Concrete for Alkali Aggregate Reaction

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EXECUTIVE SUMMARY

Aggregate makes up a significant portion of concrete. However, unlike cement, there are limited choices as to the composition and source of aggregate to be used. It has been shown in the laboratory and in the field that certain types of aggregate can cause deleterious expansion through a reaction known as alkali-silica reaction (ASR). In order to ensure the durability of new pavements and projects, the susceptibility of aggregates and cements need to be investigated. An accelerated test for ASR susceptibility was used in which mortar bar length changes were measured and used to indicate the potential of being reactive.

Aggregate consisting of granite, mylonite, phyllonite, and ultramylonite were tested.

These aggregates have the same chemical and mineralogical composition, however they all have differences in grain size. For the samples studies, smaller grain size indicated a higher amount of deformation.

Two aggregate types were chosen for which the different types of cements would be tested. The choices were a mildly reactive granite and a highly reactive phyllonite.

Five cements from four different manufactures were tested. Each cement was used in two sets of experiments: one containing granitic aggregate and the other containing phyllonitic aggregate. The four cements were Type I/II portland cement (I/II), Type III portland cement (III), two calcium sulfoaluminate cements (CSA I, and CSA II), and a calcium aluminate cement (CA).

After 16 days casting, the order of expansion of cements containing granitic (mildly reactive) aggregate is:

$$CA \ll CSA I \ll CSA II \ll I/II \ll III$$

while at 32 days the order became:

$$CA \ll CSA I \ll I/II \ll CSA II \ll IIII$$

For cements containing phyllonitic (highly reactive) aggregate, the order of expansion at 16 days is:

$$CA \ll CSA I \ll CSA II \ll I/II \ll III$$

while at 32 days the order became:

$$CA \ll I/II \ll III \ll CSAI \ll CSAII$$

Based on the data from the tests, one cement (CA) was clearly highly resistant to ASR. The other four cements failed the test when using highly reactive aggregate based on the upper expansion limit of 0.20 percent 16 days after casting according to ASTM C 1260. Even when using mildly reactive aggregate, both I/II and III cements fall into the ambiguous range, while CSA bars expanded less than 0.10 percent. However, due to large uncertainties inherent to the accelerated test, it is very difficult to rank the order of performance for these three cements. This test only shows that these four cements did not perform as well as CA cement.

1.0 INTRODUCTION

Since the reaction was first discovered in California by Stanton in 1940,(1) much has been reported in literature about the damage to concrete structures caused by this deterioration process. The alkali silica reaction (ASR) is a reaction in which certain aggregates react with the highly alkaline pore solution of concrete. As the reaction name implies, the reactive aggregate contains silica. However, not all siliceous aggregates are reactive. In general, the aggregates that cause harmful reactions in concrete are those containing amorphous silica (glasses and opal), unstable crystalline polymorphs of silica (cristobalite and tridymite), poorly crystalline forms of silica (andesite and rhyolite), and microcrystalline quartz-bearing rocks (quartzite and greywacke). The highly alkaline pore fluids of concrete are able to depolymerize the reactive silica present in the aggregates, forming products of different compositions in the concrete pores.

In the presence of moisture, the reaction products (gel products) change in volume and may expand to such a degree that the concrete tensile strength is reached and the material cracks. As soon as the material's integrity is affected, other processes of deterioration may take place.

Silica exists in a crystalline or in a non-crystalline state. The non-crystalline forms of silica, often called amorphous silica, exhibit a featureless x-ray pattern. The expansion behavior of a concrete element depends on the type, size, and amount of aggregates containing reactive silica present in the concrete mixture. Amorphous silica is also composed of silicon tetrahedra, but it is arranged in such a way as to produce a random three-dimensional network without regular lattice structures. As a consequence of the absence of an ordered arrangement, the structure of amorphous silica is open with "holes" in the network where electrical neutrality is not satisfied (as on the surfaces of crystalline silica) and the specific surface is large. The reactivity with aqueous solutions increases as a consequence of the large area available for reactions to take place.

The structure of crystalline silica is built by repetition of a basic unit—the silicon tetrahedron—in an oriented three-dimensional framework. The silicon tetrahedron consists of a silicon ion, Si⁴⁺, in the center of the tetrahedron, surrounded by four oxygen atoms in the corners. Depending on the combination of the tetrahedra, different silicon to oxygen ratios develop in the structure. The following silicate structures are possible: island or independent, double, rings, chains, bands, sheets, and frameworks.

The structure of quartz (SiO₂), for example, is of the framework type. The silicon tetrahedra are joined through the vertices by oxygen, each of which is linked to two silicon atoms; the oxygen to silicon ratio equals to 2:1, thus electrical neutrality is attained. On the surfaces, however, a complete tetrahedron does not evolve, and unsatisfied charges develop on

the silica surfaces because each silicon is linked to only three oxygen atoms instead of four, and each surface oxygen to only one silicon instead of two. This means that the surface oxygen bears a single negative charge, and the surface silicon bears a single positive charge.

In the presence of moisture or water, the unsatisfied charges of the amorphous silica surfaces and holes are neutralized by the ions OH⁻ and H⁺. The hydroxyl ions link to the positively charged silicon ions, and the hydrogen cations to the negatively charged oxygen ions, thereby hydrating the silica surfaces. The hydroxyl-oxygen ions are bonded to the silicon ions in the same way as the other oxygen ions present in the lattice, and the strongest bonds in the structure are the ones between silica and oxygen. The depolymerization of silica by hydration can be expressed as

$$-Si - O - Si - H2O \rightarrow -Si - OH + OH - Si -$$
 (1)

In addition, the silica surfaces are left with a weakly acidic character when some hydrogen ions are freed because the interatomic bonding in silica, Si - O - Si, is stronger than the O-H bond.(2) In the process of depolymerization, all four bonds of a tetrahedron may react, and the silica tetrahedron ends up having a silicon in the center and hydroxyls in the corners, forming the monomer Si(OH)₄, which is also called orthosilicic acid, monosilicic acid, or silicic acid – H₄SiO₄. With time, the Si(OH)₄ molecules will condense to form particles of colloidal dimensions.

Carman suggested that in an alkaline medium where NaOH is present, for example, the hydrogen cations, H⁺ in expression (1) are replaced by Na⁺ cations, which, unlike the H⁺ proton, cannot enter into the electronic structure of the oxygen ion and are much more easily hydrated.(2) As a result, Na⁺ ions are readily ionized and a sol (colloidal solution) is formed of silica particles having a strong negative charge and the cations forming a characteristic diffuse

layer of counter-ions. The diffuse layer is constituted of the cations present in the fluid medium.

In the case of an alkaline medium containing only sodium ions, the reactions may be expressed as

$$-Si - OH + Na^{+} + OH^{-} = -Si - O^{-}Na^{+} + H_{2}O$$
 (2)

After the acidic hydroxyl groups at the surface of the silica have been neutralized, if there still is an excess of NaOH, then the internal -Si - O - Si- links can be disrupted as indicated by the following reaction

$$-Si - O - Si - + 2 NaOH = -Si - O Na^{+} + Na^{+}O - Si - + H_{2}O$$
(3)

This reaction product, known as alkali silica gel, can be expansive which can lead to loss of concrete durability.

As mentioned earlier, not all siliceous aggregates are reactive. Table 1 summarizes the types of rocks that are reactive. The main difference between these two groups of rocks is the crystalline structure, or lack thereof, of their mineral constituents.

The first set of rocks lack minerals with a crystalline structure. This leads to high reactivity because of the high energy of the structure due to the disarray. This provides favorable thermodynamic conditions for the reaction. Since the structure is in disarray, this creates channels and holes in the structure providing pathways for reactive ion migration, and also a greater surface area on which the reaction can occur.

The second category contains rocks that have minerals with a crystalline structure. The most common form of crystalline silica is quartz. Whereas all rocks of the first category are extremely reactive, the reactivity of this second category ranges from mildly reactive to very reactive. It is still speculation as to why some quartz and crystalline forms of silica are reactive while others are not.

Table 1 Alkali-reactive Silica Minerals and Rocks(3)

Category (1)	Alkali-reactive poorly crystalline or metastable silica minerals, and volcanic or artificial glasses (Classical alkali-silica reaction)		
Reactants:	Opal, tridymite, cristobalite; volcanic glasses; artificial glasses, beekite.		
Rocks:	Rock-types containing opal such as shales, sandstones, silicified carbonate rocks, some cherts, flints, and diatomite.		
	Vitrophyric volcanic rocks: rhyolites, dacites, latites, andesites, and their tuffs, perlites, obsidians; all varieties with a glassy groundmass; some basalts.		
Category (2)	Alkali-reactive quartz-bearing rocks.		
Reactants:	Chalcedony; cryptocrystalline to microcrystalline quartz; quartz with deformed crystal lattice, rich in inclusions, intensively fractured or granulated; poorly crystalline quartz at grain boundaries; quartz cement overgrowths (in sandstones).		
Rocks:	Cherts, flints, quartz veins, quartzites, quartz-arenites, quartzitic sandstones which contain microcrystalline to cryptocrystalline quartz and/or chalcedony.		
	Volcanic rocks such as those in category (1) but with devitrified, cryptocrystalline to microcrystalline groundmass.		
	Micro-granular to macro-granular silicate rocks of various origins which contain microcrystalline to cryptocrystalline quartz: a) Metamorphic rocks: gneisses, quartz-mica schists, quartzites, hornfelses,		
	phyllites, argillites, slates;b) Igneous rocks: granites, granodiorites, charnockites;c) Sedimentary rocks: sandstones, greywackes, siltstones, shales, siliceous		
	limestones, arenites, arkoses; Sedimentary rocks (sandstones) with epitaxic quartz cement overgrowths.		

When this occurs in concrete, the force caused by the expansion of the alkali-silica gel causes cracking to occur. The crack pattern is irregular, and is known as *map cracking*. It is possible to have light colored carbonated reaction product around the crack area. Petrographic examination of the concrete, chemical analysis of the reaction product, or an ultra violet spray that detects alkali-silica gel can all be used to detect the presence of an alkali-silica reaction.

This report presents the results from an investigation into accelerated ASR testing. The first part discusses the aggregates and cements investigated as well as the test method used. The

investigation demonstrates that even aggregates once thought to be innocuous can cause expansion. The second part presents the results of accelerated tests using five proprietary cements from four different manufacturers. The third part is a discussion on the validity of these tests, and explores the possibility of using other tests. Following a Summary section, a proposed test procedure.

2.0 MATERIALS AND TEST DESCRIPTION

The following sections describe the aggregates and cements examined in this study and test procedure used to evaluate them.

2.1 Description of the Rocks Used for Testing

The rocks used for testing were obtained from the Santa Rosa mylonite zone in Southern California. Mylonitization is a process in which mineral components of a rock undergo large and dominantly ductile deformation. (4) Except for mechanical abrasion at the boundaries, quartz and mica are highly strained and flattened, and recrystallize by subgrain formation. This results in grain size reduction and development of strong preferred orientation in quartz and mica. However, during mylonitization, harder components such as feldspars remain largely coherent as clasts. In the extreme case of phyllonite and ultramylonite (highly deformed rocks), even the harder mineral components succumb, and the grain size becomes extremely small.

All samples used in this study were collected in upper Palm Canyon area of the Santa Rosa mylonite zone. The mylonite zone lies on the eastern edge of the Peninsular Ranges batholith and was pervasively deformed in a ductile regime shortly after the emplacement of the batholith in the late Cretaceous. The mylonite zone is 200 m to 1.5 km wide and strains are very large as evaluated from a study of enclaves by Wenk.(5) Granite deformed to mylonite at the

outer margin of the batholith. Locally within the mylonite, extremely deformed areas of phyllonite and ultramylonite were developed that are best exposed in upper Palm Canyon.(4) Previous studies quantified the processes that occurred during the transformation of granite to mylonite and phyllonite, particularly the development of preferred orientation,(4, 6) and the evolution of microstructure. It is found that the deformation process does not produce any bulk chemical or mineralogical change.

2.1.1 Granodiorite

The granodiorite sample (PC 89) is a white and gray rock with black specks consisting of approximately 27 percent quartz, 52 percent plagioclase, 6 percent orthoclase, and 8 percent biotite. The composition indicates that the rock is a granodiorite. The rock is medium to fine grained and is holocrystalline. Abundant anhedral sections of quartz and plagioclase with scattered flakes of brown biotite are present. Orthoclase is not observed in appreciable quantities. No foliation or preferred orientation can be observed.

2.1.2 Mylonite

The mylonite sample (PC284) is a gray and white rock. Unlike PC89, the gray and white areas of the rock are arranged in layers. These layers represent compositional layering formed through the reduction of grain size and recrystallization due to ductile deformation. Feldspar augen are common in the matrix of quartz and biotite. This metamorphic rock would be classified as a mylonite. Quartz grains have a grain size ranging from 0.5 to 1.5 mm, biotite from 0.25 to 1.5 mm, and plagioclase from 0.5 to 3.5 mm. Biotite and quartz exhibit strong preferred orientation. Foliation and lineation can be observed in hand samples.

2.1.3 Phyllonite

The phyllonite sample (PC 92) is a gray, metamorphic rock. No crystals can be seen with the unaided eye. With the use of a microscope, a few remaining feldspar augen are seen. Grain size range from 0.05 to 0.15 mm. Quartz and biotite grains are no larger than about 3 µm. The foliation and lineation are visible in the hand specimen. Biotite is observed to have a stronger preferred orientation than in mylonite while quartz grains have lost some degree of preferred orientation.

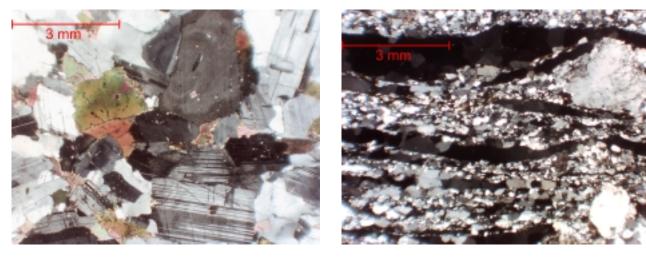
2.1.4 Ultramylonite

The ultramylonite sample (PC 86) is a massive black rock. The only difference from phyllonite (PC 92) appears to be the color. No microscopic evidence can be found for the difference in outward appearance. Because of the darker color, this rock is classified as an ultramylonite.

Photomicrographs of these rocks are shown in Figure 1.

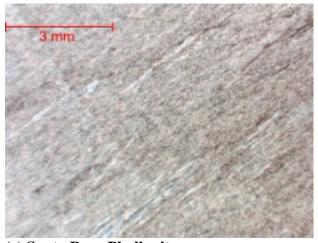
2.2 Texture Analysis of Rock Samples

In many rocks, minerals exhibit a preferred orientation pattern; this is expressed in anisotropy of physical properties. This "directionality," also known as "texture," develops, for example, in sediments during preferred settling of platy clay particles parallel to the bedding planes. More importantly, it develops in metamorphic rocks during ductile deformation.(7) Rotations of crystals occur due to intracrystalline slip, caused by non-equiaxed shape, and patterns may be modified during dynamic recrystallization.



(a) Peninsular Ranges Granodiorite





(c) Santa Rosa Phyllonite

Figure 1. Photomicrographs of rocks used for testing.

The orientation distribution of crystals is most commonly represented in pole figures. The pole figure is constructed by placing a polycrystalline sample inside a sphere and intersecting a crystal direction (e.g., the c-axis or [0001] of quartz) with the surface of the sphere producing "poles." Pole densities can then be contoured and expressed as a smooth distribution usually normalized such that contours express pole densities in multiples of a uniform distribution.

Crystal orientations can be measured with a petrographic microscope equipped with a universal stage or more commonly by x-ray diffraction with a pole figure goniometer. More specialized techniques are neutron and electron diffraction. Whereas electron diffraction can provide a grain-by-grain analysis of orientation, neutron diffraction provides an analysis of the bulk material.

Pole figures for biotite and quartz are shown in Figures 2 and 3 respectively. These were obtained from an earlier analysis by Wenk and Pannetier.(5) The figures are contoured to show multiples of random distribution and are represented in equal area projections. Table 2 shows the maxima and minima of the pole figures in multiples of random distribution.

Table 2 Maxima and Minima in Pole Figures in Multiples of Random Distribution(5)

Material	hkl	Granodiorite	Mylonite	Phyllonite
Biotite	001	2.3 to 0.5	5.8 to 0.0	14.5 to 0.0
Quartz	1120	2.5 to 0.4	2.4 to 0.5	1.1 to 0.8

Quartz pole figures do not indicate a development of preferred orientation with increasing deformation. Rather, texture is best developed as granodiorite is deformed into mylonite. This preferred orientation is partially destroyed upon further deformation. The texture development in quartz is due to the response to stress. As granite deforms into mylonite, dislocation assisted subgrain rotation (intracrystalline slip) is the primary mechanism of stress accommodation. This results in a reduction of grain size by a factor of 10-100. In phyllonite, a switch to other mechanisms such as grain boundary sliding occurs resulting in partial destruction of texture.

Texture analysis indicates a strong development of preferred orientation in biotite as the rocks become progressively deformed. Similar to quartz, biotite preferred orientation strengthens as granodiorite deforms to mylonite. However, unlike quartz, the preferred

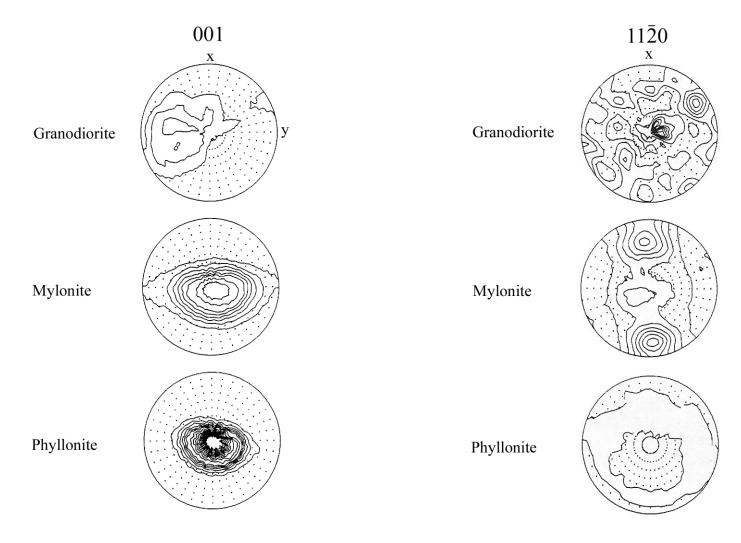


Figure 2. Pole figures of biotite. Contour intervals are 0.5 multiples of random distribution. Dotted portions represent areas below 1 multiple of a random distribution.(5)

Figure 3. Pole figures of quartz. Contour intervals are 0.25 multiples of random distribution. Dotted portions represent areas below 1 multiple of a random distribution.(5)

orientation of biotite strengthens as mylonite deforms to phyllonite. The mechanisms that lead to preferred orientation of biotite include rotation, intracrystalline slip, and dynamic recrystallization. The alignment of phylosillicate grains leads to the development of foliation, which is thought to increase the reactivity of a rock. This is enhanced by the morphology of biotite, which is belongs to the family of sheet silicate minerals.

2.3 **Description of Cements**

Five types of cement from four different manufacturers were submitted for testing. The cement types were portland cement types I/II, and III, calcium sulfoaluminate cement, and calcium aluminate cement. Because the cement manufacturers have requested that the trade names of their products not be revealed, the cements are referred to by the following designation:

Portland Cement Type I/II: OPC I

Portland Cement Type III: OPC II

Calcium Sulfoaluminate Cements: CSA I, CSA II

Calcium Aluminate Cements: CA

The chemical compositions of these cements were obtained through chemical oxide analysis. The results are listed in Table 3.

2.4 **Description of Testing Method ASTM C 1260**

ASTM C 1260 requires that the aggregate be crushed to a prescribed grade and that mortar bars $(2.5 \times 2.5 \times 25 \text{ cm})$ be cast with an aggregate-to-cement ratio of 2.25 by mass, and a water-to-cement ratio equal to 0.47 by mass.(9) Three mortar prisms are required for each sample. The mortar prisms are cured for 24 hours in a fog room at $21 \pm 2^{\circ}$ C, demoulded, and immersed in water in a closed container maintained at 80°C. After 24 hours, the length of the

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Table 3 Chemical Oxide Analysis of Cements Used for Testing(8)

	Cement				
Oxide	OPC I	OPC II	CSA I	CSA II	CA
SiO ₂	21.24	20.50	15.40	15.59	5.60
Al_2O_3	3.57	4.15	12.88	13.96	48.66
Fe ₂ O ₃	3.82	3.70	2.63	1.49	5.49
CaO	64.72	64.27	53.02	50.19	37.04
MgO	1.69	1.27	2.03	1.35	0.39
TiO_2	0.31	0.34	0.70	0.53	2.33
Mn_2O_3	0.04	0.04	0.04	0.05	0.05
P_2O_5	0.32	0.28	0.01	0.14	0.12
Cr ₂ O ₃	0.04	0.04	0.03	0.04	0.00
ZrO_2	0.00	0.00	0.06	0.05	0.00
Na ₂ O	0.18	0.20	0.39	0.23	0.06
K_2O	0.10	0.20	0.32	0.44	0.23
SO_3	2.27	2.33	10.81	14.20	0.03
Ignition Loss	1.43	2.42	1.96	2.17	-
Total	99.72	99.73	100.27	100.42	100.00

specimens is measured. They are then immersed in a closed container of 1M sodium hydroxide solution maintained at 80°C. The specimens are removed periodically from the containers and measured before significant cooling can occur. The length change of the prisms is measured up to 16 days after casting.

If the observed expansion exceeds 0.20 percent after 16 days after casting, the aggregate tested should be considered potentially reactive. If the expansion measured lies between 0.10 and 0.20 percent after 16 days after casting, additional testing, supplemental information, and/or field performance data should be evaluated. If the expansion measured is less than 0.10 percent after 16 days after casting, it is indicative of innocuous behavior in most cases.

The main disadvantage of this test is the use of high temperature and high alkali concentration, which induce the reactions under different conditions than those in practice. The main advantage of the test is the ability to quickly obtain fairly reliable results.

3.0 RESULTS

3.1 Selection of Aggregates for Testing of Cement ASR Resistance

Prior to testing how well the different cements affect ASR resistance, it was necessary to find one highly reactive and one mildly reactive aggregate sample. To limit the possible causes of expansion, rocks from the Santa Rosa Mylonite Zone in Southern California were chosen for this experiment. These rocks were chosen because x-ray fluorescence and petrologic mineral norm calculations show that there is no bulk chemical or mineralogical change associated with these rocks. The only difference in these rocks is the physical characteristics of the mineral grains. By using these rocks, it could be possible to address the question of what causes some forms of quartz to be reactive while others are not. The result of ASTM C 1260 using Type I/II portland cement is shown in Figure 4. Granodiorite shows levels of expansion that is considered innocuous by ASTM.

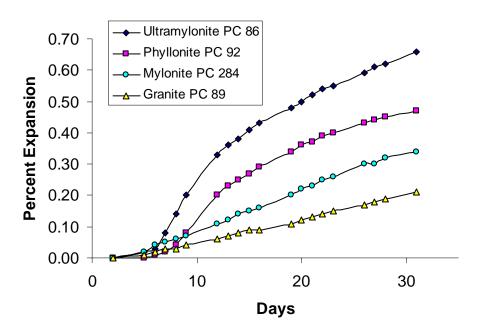


Figure 4. Results of ASTM C 1260 for various deformed granites using Type I/II portland cement.

Mylonite shows levels of expansion that would require further investigation to determine whether or not it would cause deleterious expansion. Samples of phyllonite and ultramylonite show unacceptable levels of expansion.

Photomicrographs of the rock samples show the difference in grain size and texture of the minerals in these rocks. For detailed explanation of these rocks, please refer to Section 1.1.

Electron probe microanalysis done on sections of mortar bar after varying exposure to the sodium hydroxide solution shows the progress of deterioration of quartz grains. Before exposure to the bath solution, quartz grains appear without major cracking or defects. As time progressed to 10 days, pits and cracks start to develop. At 20 days, the pits and cracks are interconnected and form an extensive network. Reaction product can be seen existing in the cracks of quartz grains and in the air voids present in the matrix. Quantitative analysis shows that the reaction product has a variable chemical composition consisting of calcium, sodium, silicon, hydrogen, and oxygen.

3.2 Cements Tested for ASR Resistance

Five types of cement from four different manufacturers were submitted for testing. The cement types were portland cement types I/II and III, calcium sulfoaluminate cement, and calcium aluminate cement. These cements were tested using phyllonite and granite aggregate.

As discussed in Section 2.1, according to ASTM C 1260, phyllonite was reactive and granite was not reactive.

3.3 Results of ASTM C 1260 on Aggregate-Cement Combinations

The accelerated test ASTM C 1260 is used to investigate the affect different types of cement have on the reactivity of an aggregate. The results are shown in Figures 5 and 6 for the tests using the non-reactive (granite) and reactive aggregates (phyllonite), respectively.

As expected, regardless of the type of aggregate used, calcium aluminate cement exhibited exceptional resistance to chemical attack. The high strength and low porosity of calcium aluminate cement prevents the hydroxides in the alkali bath from reaching the aggregate.

However, due to the known variability of results obtained through ASTM C 1260, it is not possible to ascertain what effect, if any, portland cement type I/II, type III, or calcium sulfoaluminate have on the expansion of aggregates. The results were expected to show little or no difference in the expansion of these cements given that this specific test was not designed to test the effect of different types of cement, rather it was designed to test different aggregates. Even though it is yet to be proven whether this test has any correlation with concrete or field performance, the timely manner in which results are obtained from this particular test is most beneficial.

4.0 DISCUSSION

ASTM C 1260 is a test that is commonly used to test the reactivity of an aggregate. This test was developed to provide a way to identify reactive aggregates in a cost-effective and timely manner. Being able to obtain results within 14 days, this test is quick and economical. However, it has not been able to escape criticism.

Many have argued that the test is irrelevant. However, this test was developed from Stanton in the 1940's based on data from California aggregates. The goal was to find a test that provides results more quickly than standard tests, which can require as much as a year to

Cement Type Using Granitic Aggregate

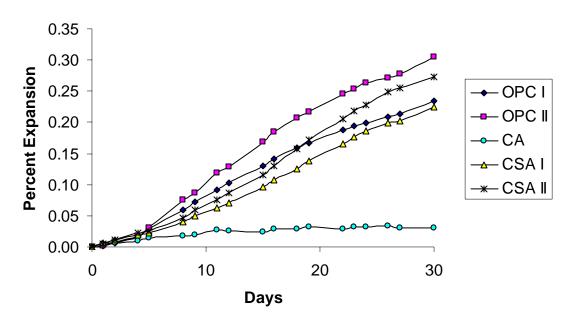


Figure 5. ASTM C 1260 test results for different types of cement using granitic aggregate PC89 that was shown earlier to not be reactive for type I/II portland cement.

Cement Type Using Phyllonitic Aggregate

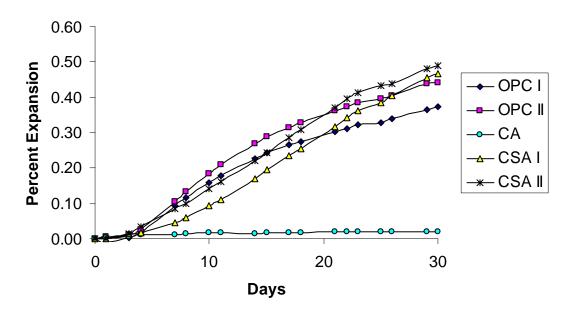


Figure 6. ASTM C 1260 test results for different types of cement using phyllonitic aggregate PC92 that was shown earlier to be reactive for type I/II portland cement.

complete. By correlating data of the accelerated test to the non-accelerated test, the following ranges were obtained:

If the observed expansion exceeds 0.20 percent after 16 days after casting, the aggregate tested should be considered potentially reactive.

If the expansion measured lies between 0.10 and 0.20 percent after 16 days after casting, additional testing, supplemental information, and/or field performance data should be evaluated.

If the expansion measured is less than 0.10 percent after 16 days after casting, it is indicative of innocuous behavior in most cases.

As for using ASTM C 1260 to test the reactivity of deformed granites, the results show a nice progression of reactivity for the aggregates based on the level of deformation that the rock has been exposed to. It was once thought that the deformation of a rock, and hence its reactivity, could be correlated with the undulatory extinction angle of quartz. However, this has been thoroughly disproved. A correlation was found that could indicate the reactivity of deformed granites is by using texture analysis. Texture analysis was performed on the minerals of quartz and biotite for each of the rocks. A comparison in the results from ASTM C 1260 to the pole figures indicates a very strong correlation of the texture of biotite to the reactivity of the rock.

However, many concerns related to the differences between testing conditions and field conditions exist. First among these concerns is the test's applicability to cements other than portland cement (PC). ASTM C 1260 specifies that an expansion greater than 0.1 percent signifies that the aggregate is reactive and therefore that the use of such aggregate will cause the concrete in question to fail, exceeding its elastic limit.

Tensile strength can be approximated by as 10 percent of the compressive strength.

Further, 80 percent of peak compressive strength can be assumed to be the maximum value

within the elastic region, that is, the strain at which the specimen would fail (i.e., exceed the elastic limit). Table 4 presents strain estimates using these assumptions for the cements considered in this study.

Table 4 Modulus and Peak Strength of Different Cements Used to Calculate Strain Values

Cement	Modulus	80 Percent Peak Strength	Strain	Percent OPC I
OPC I	26895	419	0.0156	100
CSA I	33130	906	0.0274	176
CSA II	30976	832	0.0269	173
CA	29873	730	0.0244	157

Non-PC cements can take up to 75 percent more strain than PC cement before plastic deformation occurs. This could signify that the limits placed on portland cement may not be applicable to non-PC cement.

The test requires that the mortar bars be immersed in a 1 M NaOH solution. A question may arise as to whether this saturation could cause false results. The hydroxide concentration in calcium sulfoaluminate and portland cements were measured and found to be the same.

Measurements were taken of freshly mixed cement paste with a water-to-cement ratio of 0.47 at intervals of every 3 minutes for 15 minutes. The results are shown in Table 5.

 Table 5
 Average pH Value of Freshly Mixed Cement Pastes

Sample	Average pH
OPC I	13.1
OPC II	13.2
CSA I	13.0
CSA II	13.1
CA	12.1

Calcium aluminate cement was found to have 10 times less hydroxide than both portland cement and calcium sulfoaluminate cement, yet displayed the least expansion under testing using

ASTM C 1260. This shows that, although hydroxide was added by means of a bath solution, there was no effect on the expansion results.

ASTM C 1260 specifies that the water-to-cement ratio be 0.47, but ready mix manufacturers suggestions are in the range of 0.35 to 0.38. The lower water-to-cement ratio creates a denser (less porous) and stronger cement matrix, and could possibly lower the magnitude of expansion. However, the manufacturers' suggested water-to-cement ratios are often exceeded in the field, therefore the high water-to-cement ratio could be more representative of actual performance.

ASTM C 1260 is a test that is favored because results can be obtained quickly and is cost effective. However, this test may only be good for identifying aggregates that are *not* reactive because the harsh conditions of the test may induce reactivity that may otherwise not be found in field conditions. For aggregates that do not pass ASTM C 1260, it is suggested that other tests be performed which are more indicative of field conditions, such as non-accelerated concrete prism tests.

5.0 SUMMARY

Results were presented from the accelerated test for determining the susceptibility of aggregates to ASR (ASTM C1260). Results were also presented for ASTM C 1260 testing the effects of different cements on the expansion of aggregates. The aggregates used for testing were granite, mylonite, phyllonite, and ultramylonite. The cements used for testing were portland cements type I/II and III, two calcium sulfoaluminate cements, and calcium aluminate cement. All tests were performed following ASTM C 1260.

From accelerated tests on different rock samples, it was found that the reactivity of the rocks could be described in the following manner:

Granite < Mylonite < Phyllonite < Ultramylonite

A result of interest is the correlation of texture analysis with the reactivity of the rocks. Pole figures of the mineral grains indicate that a correlation exists between the orientation of biotite and the reactivity of the rock. Due to the mechanism of deformation involved with quartz, this correlation does not exist for quartz. If a correlation exists between the reactivity of a rock and its texture, then texture analysis could be used as a quantitative technique to determine rock reactivity.

Granite was chosen as the rock to be used as an innocuous aggregate in subsequent testing. Phyllonite was chosen as a deleterious aggregate based on the results of accelerated testing. Testing to determine the effect different cements have on the expansion of these aggregates showed that calcium aluminate cement has excellent resistance to chemical attack. The results were inconclusive for the other three cement types due to the known deviance of results from ASTM C 1260.

Whether an aggregate is reactive cannot be solely based on ASTM C 1260. This test is an accelerated test that subjects the aggregate to harsh conditions that may not be indicative of field conditions. At best, the aggregates that passed the test can be considered not reactive. It is suggested that other tests be run on aggregates that do not pass the test.

6.0 PROPOSED SPECIFICATION

Figure 7 illustrates the current specification for testing for ASR. Figures 8 and 9 show the proposed specification for testing ASR in portland and non-portland cements, respectively.

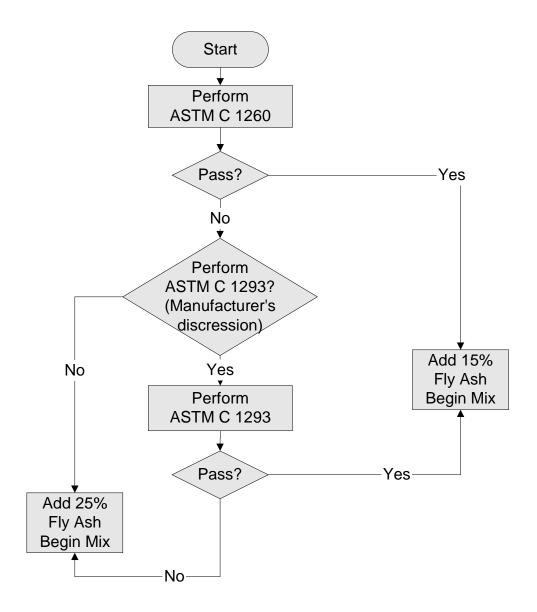


Figure 7. Current ASR testing specification.

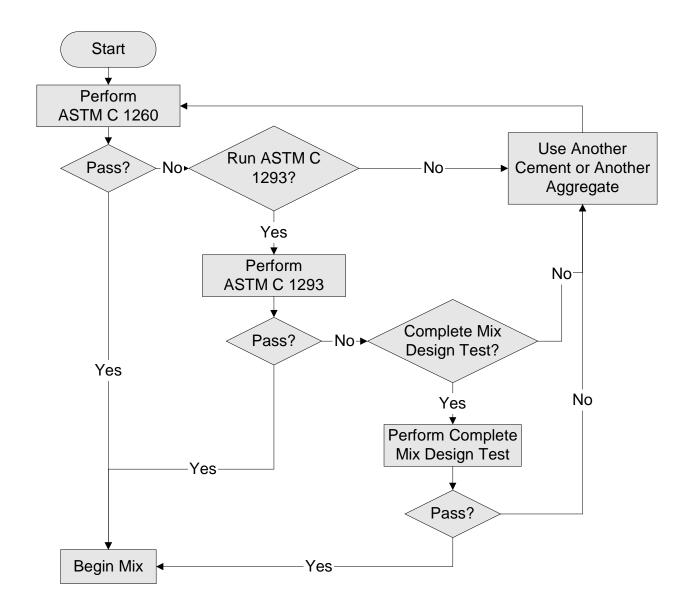


Figure 8. Proposed ASR testing specification for portland cements..

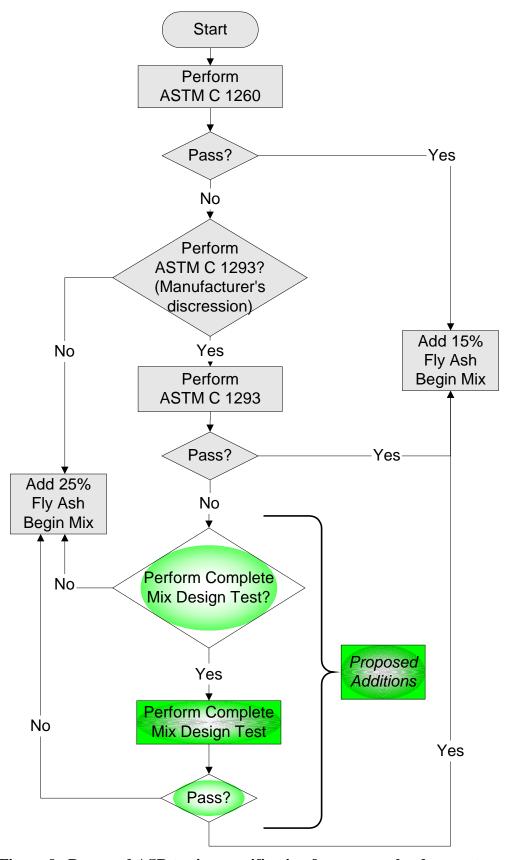


Figure 9. Proposed ASR testing specification for non-portland cements.

6.1 **ASTM C 1260**

Aggregate reactivity will first be tested using ASTM C 1260 with the following specifications. In this test, the aggregate in question will be crushed and graded to the requirements in Table 6 below. This aggregate is to be washed and oven dried before use.

Table 6 Aggregate Grading Requirements of ASTM C 1260

Passing Sieve Size	Retained on Sieve Size	Percent of Total Mass
4.75 mm	2.36 mm	10
2.36 mm	1.18 mm	25
1.18 mm	600 μm	25
600 μm	300 μm	25
300 μm	150 μm	15

The ratio of cement to aggregate will be 1 to 2.25 by mass. For each batch of mix to make three specimens, 440 g of cement and 990 g of aggregate will be used, along with a water-to-cement ratio equal to 0.47 by mass. It is required that the type of cement used in this test will be the same cement used with the aggregate in any proposed structure or pavement.

The mortar will be cast into bars that are $1 \times 1 \times 11.25$ in. The mortar is to stay in the molds for 24 ± 2 hours. The specimens are then removed from the molds and placed in a sealed container that contains tap water kept at 80 ± 2 °C. The specimens are to remain in this condition for a period of 24 hours. The specimens are then removed from the water one at a time and their length is measured. This reading will be reported as the zero reading. After the specimens length is recorded, the specimen is placed in a sealed container containing a solution of 1M NaOH. This solution is to be kept at 80°C. Readings of length change will be taken approximately daily for 32 days after casting.

If the average expansion of a batch of mortar bars is 0.10 percent or less at 16 days after casting, then the aggregate is satisfactory. If the length change is more than 0.10 percent at 16 days after casting, then the aggregate has failed this portion of the test.

Aggregates that failed 16 days after casting will continue to be tested until 32 days have elapsed since testing. If the average expansion of the batch of mortar bars is less than 0.25 percent at 32 days after casting, then the aggregate is satisfactory. If the length change is more than 0.25 percent at 32 days after casting, then the aggregate has failed. Failed aggregates at this point will be tested according to the second phase of the specification.

6.2 ASTM C 1293 (10)

Aggregates that failed under ASTM C 1260 will be tested by using ASTM C 1293 with the following specifications. If the aggregate that failed the first part of the specification is to be used as a fine aggregate in the pavement or structure, then the grading that is to be tested will be the same as the grading to be used in the pavement or structure to be built. When testing the reactivity of fine aggregate, a non-reactive coarse aggregate must be used. Non-reactive is defined as having an expansion equal to or less than 0.10 percent as tested by the first part of this specification.

If the aggregate that failed the first part of the specification is to be used as a coarse aggregate in the pavement or structure, then the grading presented in Table 7 will be used.

 Table 7
 Aggregate Grading Requirements of ASTM C 1293

Passing Sieve Size	Retained on Sieve Size	Percent of Total Mass
19.0 mm	12.5 mm	33
12.5 mm	9.5 mm	33
9.5 mm	4.75 mm	33

When testing the reactivity of coarse aggregate, a non-reactive fine aggregate must be used. Non-reactive is defined as having an expansion equal to or less than 0.10 percent as tested by the first part of this specification.

The cement used in this test will be the same as the cement used in the pavement or structure. The cement content of the concrete will be $420 \pm 10 \text{ kg/m}^3$. The water-to-cement ratio will be in the range of 0.42 to 0.45 by mass and adjusted according to saturated surface dry aggregate condition. NaOH is to be added to the mix water such that the alkali content of the cement will be 1.25 percent by mass.

The volume of coarse aggregate per unit volume of concrete will be 0.70 ± 0.2 percent using the oven-dry-rodded unit volume.

The concrete mixed will yield three specimens from the batch with the dimensions of $3 \times 3 \times 11.25$ in. After casting, the molds will be placed in a room with 100 percent humidity at 25° C for 23.5 ± 0.5 hours. Afterwards, the specimens will be measured for the initial length, and then transferred to a sealed container that is kept at $38.0 \pm 2^{\circ}$ C. Subsequent measurements will be taken at 7 days, 28 days, 56 days, 3 months, 6 months, 9 months, and 12 months. Before each measurement, the containers holding the specimens must be removed from the $38.0 \pm 2^{\circ}$ C environment and placed in a moist room or cabinet for 16 ± 4 hours.

If the average length change of the specimen is equal to or less than 0.04 percent after one year, then the aggregate has passed and is suitable for use. All aggregate that fails to meet these criteria will be rejected.

Questions about this testing procedure and its relevance to actual mix designs are the same as for ASTM C 1260, as discussed in Section 3.0. In addition, ASTM C 1293 may be more relevant if the actual amount of aggregate in the manufacturer's mix design were used instead of

the prescribed amount. Changing this part of the specification would make the test take a lot of time because it would need to be run for all the different mixes. It would be more feasible for the manufacturers to do instead of Caltrans.

6.3 Proposed ASR Specification

The goal is to have a specification that protects Caltrans, but is also fair to the manufacturers. Currently, Caltrans is using or will be using a specification for testing portland cement mixes that requires using ASTM C 1260 and/or ASTM C 1293. If the aggregate passes either test, then the mix design will have 15 percent fly ash. If the aggregate fails both tests, then 25 percent fly ash will be added to the mix. The proposed specification includes these requirements, but expands the testing to include non-portland cements/aggregate combinations.

In the proposed specification, at any time, the manufacture may choose not to test and add 25 percent fly ash to portland cement mixes. However, this cannot be done by users of non-PC mixes because it is unclear as to whether or not fly ash mitigates ASR in these mixes.

Therefore, a non-PC mix must pass either ASTM C 1260 or ASTM C 1293 before being allowed to be used.

Other additions to the current specification include the complete mix design test and a Strubble/ASTM C 441 type test. However, the objective of the Strubble test is to test different cements, and to use it in conjunction with ASTM C 1260 to create a matrix prescribing which cement and aggregate combinations are acceptable. This scenario should be avoided because it amounts to nothing more than certifying materials. Since these materials change from location to location, or over time, it is not possible to guarantee the certification of materials for periods of time.

7.0 REFERENCES

- 1. Stanton, T. E. "Expansion of concrete through reaction between cement and aggregate." *Proceedings*, ASCE, vol. 66, pp. 1781-1811, 1940.
- 2. Carman, P. C. "Constitution of Colloidal Silica." *Transactions*, Faraday Society, vol. 36, pp. 964-973, 1940.
- 3. Bérube, M. A. and Fournier, B. "Testing for Alkali-Aggregate Reactivity in Concrete." *Proceedings*, Durabilidad Del Concreto, Monterrey, N.L. Mexico, 1993.
- 4. Wenk, H.R. "Deformation of Mylonites in Palm Canyon, California, Based on Xenolith Geometry." *Journal of Structural Geology*, Vol. 20, No. 5, pp. 559-571, 1998.
- 5. Wenk, H.R., Pannetier, J. "Texture Development in Deformed Granodiorites from the Santa Rosa Mylonite Zone, Southern California." *Journal of Structural Geology*, Vol. 12, No. 2, pp. 177-184, 1990.
- 6. Goodwin, L.B., Wenk, H.R. "Development of Phyllonite from Granodiorite: Mechanisms of Grain-size Reduction in the Santa Rosa Mylonite Zone, California." *Journal of Structural Geology*, Vol. 17, No. 5, pp. 689-707, 1995.
- 7. Kocks, U.F. *Texture and Anisotropy: Preferred Orientations in Polycrystals and Their Effect on Material Properties.* Cambridge University Press, 1998.
- 8. Monteiro, P.J.M., Roesler, J., Kurtis, K.E., Harvey, J. *Accelerated Test for Measuring Sulfate Resistance of Hydraulic Cements for Caltrans LLPRS Program.* Report prepared for the California Department of Transportation. Report No. FHWA/CA/OR-2000/03. University of California at Berkeley, Institute of Transportation Studies, Pavement Research Center, 2000.
- 9. American Society for Testing and Materials. "Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method), ASTM C 1260-94." *Annual Book of ASTM Standards*, Vol. 04.02, Philadelphia, 1994.
- 10. American Society for Testing and Materials. "Standard Test Method for Concrete Aggregates by Determinations of Length Change of Concrete Due to Alkali-Silica Reaction, ASTM C 1293-95. *Annual Book of ASTM Standards*, Vol. 04.02, Philadelphia, 1995.