# Accelerated Test for Measuring Sulfate Resistance of Hydraulic Cements for Caltrans LLPRS Program

Report Prepared for

**CALIFORNIA DEPARTMENT OF TRANSPORTATION**

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## 1.0 EXECUTIVE SUMMARY

Many California soils are rich in sulfates, which can have deleterious effects on the cements used in rigid pavements. Field experience has demonstrated that sulfate attack usually manifests itself in the form of loss of adhesion and strength. In order to assess the sulfate susceptibility of cements submitted to the Caltrans Long Life Pavement Rehabilitation Strategy (LLPRS) program, an accelerated test program was undertaken. In the accelerated test method employed, changes in cement paste strength after periods of sulfate exposure relative to the strength after seven days hydration served to indicate the sulfate resistance of the cement in question.

Five cements submitted to Caltrans by four different manufacturers, and four portland cement mixes prepared in the laboratory were tested by an accelerated test method in which pH and sulfate concentration remained constant. Conditions provided in this accelerated test method are similar to field conditions, and results should be indicative of field performance. The cements were classified into three categories: portland cements and blends (PC), calcium aluminate cements and blends (CA), and calcium sulfoaluminate cements (CSA). After 28 and 63 days of exposure to a 4 percent $Na_2SO_4$ solution in which the pH was maintained at 7.2, performance of the nine cements and blends tested can be described by the following two rankings:

At 28 days,

> CA3 = CSA2 = Type I/II > Type V = Type III > CA1 > Type III/silica fume/flyash > CA2 > CSA1

At 63 days,

CA3 = Type I/II = CSA2 > Type III = Type V > Type III/silica fume/flyash >
CA2 > CA1 > CSA1

Based on a failure criterion of 25 percent strength reduction after sulfate exposure for 28 days, only one cement was considered not to be sulfate resistant (CSA1) and three cements failed the criterion at 63 days (CA1, CA2, CSA1). Recommendations are given to quickly identify sulfate susceptible cements.

## 2.0 INTRODUCTION

Caltrans engineers and policymakers have identified a need to develop lane replacement strategies that will not require long-term closures that are associated with the use of PCC (portland cement concrete), and provide longer lives than the current assumed PCC design life of 20 years. The Caltrans LLPRS-Rigid committee has developed strategies for rehabilitation of concrete pavements that are intended to meet the following objectives:

1. Provide 30+ years of service life
2. Require minimal maintenance, although zero maintenance is not a stated objective,
3. Have sufficient production to rehabilitate or reconstruct about 6 lane-kilometers within a construction window of 67 hours (10 a.m. Friday to 5 a.m. Monday).

These objectives provide the motivation for the investigation of advanced cementitious materials for pavement construction. The cementitious materials under consideration by Caltrans may be classified into four categories: portland cements and blends, calcium aluminate cements and blends, calcium sulfoaluminate cements, and fly ash-based cements. To achieve a 30+ year design life, it is essential to select materials that are expected to exhibit long-term durability. Since most of the cementitious materials under consideration have not been extensively used for pavement construction in the United States, it is essential to characterize the long-term durability of each material.

A previously submitted report, “Analysis of Durability of Advanced Cementitious Materials for Rigid Pavement Construction in California” (*1*), provided a state-of-the-art review of deleterious reactions that may affect concrete pavements in California. The reactions addressed were sulfate attack, alkali-aggregate reaction, corrosion of reinforcing steel and dowels, and freeze-thaw action. The report describes, based on available literature, the expected performance of portland cements and blends, calcium aluminate cements and blends, calcium sulfoaluminate cements, and fly ash-based cements with regard to each of the deleterious reactions listed. The report also presented a recommended test program, including a detailed description of an accelerated test for measuring sulfate resistance of cement paste. For more details on the hydration products formed by sulfate attack, see Reference (*1*).

Frequently, soils and groundwater sources in the Western United States are rich in sulfates (*2*). An accelerated test program was undertaken to assess the sulfate attack susceptibility of cements submitted to the CAL/APT Program LLPRS-Rigid project. Sulfate ions present in soil, groundwater, seawater, decaying organic matter, and industrial effluents are known to have an adverse effect on the long-term durability of concrete. When exposed to an aggressive sulfate environment, the resistance of concrete to the sulfate ions is dependent upon the permeability of the concrete and the composition of the hydrated cement paste. Once sulfate ions ingress into the concrete, the form of the sulfate attack, and therefore the effects of the attack, depend upon the amounts of monosulfate hydrate, calcium aluminate hydrate, unhydrated tricalcium aluminate, and calcium hydroxide in the cement paste. Note: It is important to distinguish between attack by external sulfates, which is caused by reaction of hydration products with sulfates that permeate the concrete from the surrounding environment, and delayed ettringite formation, which is thought to result from a combination of steam curing and exposure. (*3*)

The two main forms of sulfate attack known to exist are the following:

- Reaction with monosulfate hydrate, calcium aluminate hydrate, and/or unhydrated tricalcium aluminate to produce ettringite; and
- Reaction with calcium hydroxide to produce gypsum, which results in a decrease in pore solution alkalinity.

Expansion may occur during sulfate attack as a result of the formation of ettringite and gypsum. In addition, the loss of calcium hydroxide through the production of gypsum can decrease the pore solution alkalinity, resulting in decalcification and loss of calcium-silicate hydrate, the primary strength-giving component of the cement paste. External sulfate attack on concrete may lead to cracking, spalling, increased permeability, and strength loss. Sulfate damage to concrete typically starts at the surface exposed to the sulfate environment and sulfate containing salt that forms on the concrete surface. (*4*) To ensure long periods of satisfactory performance, concrete pavements in contact with sulfate-containing soil or water must be resistant to sulfate attack.

Currently, no published information has been discovered in the literature or internally at Caltrans to suggest that concrete pavements in California have failed or deteriorated significantly because of sulfate attack. It is difficult to determine whether a sulfate problem exists in Caltrans pavements for three reasons:

1. Caltrans pavement surveys only record cracking of the slab, not whether the cracking is durability or load associated;
2. Caltrans has primarily used a moderately to highly sulfate-resistant cement (Type I/II) for concrete pavement construction; and

3. concrete pavements (highway) were not in direct contact with sulfate-containing soils and/or in contact continuously with water.

In a recently published NCHRP report, Zollinger states that sulfate attack is of major concern for portland cement concretes used for projects in which the pavement is opened to traffic early. (*5*) If new cementitious materials (i.e., other than portland cement based) used for highway construction perform poorly in sulfate-rich environments, sulfate attack may potentially be a failure mechanism. Robinson reported many sulfate-related failures of concrete slabs on grade in housing areas in southern California, specifically in Los Angeles County. (*6*) Dry concrete in dry sulfate-bearing soils will not be attacked (*7*), while alternating wetting and drying increases the severity of sulfate attack. (*7, 8*) To repair or avoid sulfate attack on concrete slabs on grade, Robinson recommends Type V cement (highly sulfate-resistant cement), vapor barrier, 0.65 m of a gravel base, and adequate drainage to remove water from under the concrete slab. (*6*)

This report presents the results from the accelerated sulfate testing of cements submitted to Caltrans as part of the CAL/APT Program, LLPRS-Rigid Phase III project. Five proprietary cements submitted by four different manufacturers and four portland cement blends mixed in the laboratory were tested. In this accelerated test method, changes in cement paste strength after sulfate exposure as compared to the strength after 7 days hydration serve as a measurement of sulfate resistance.

## 3.0 ACCELERATED TEST TO MEASURE SULFATE RESISTANCE

Currently, ASTM describes two accelerated test methods to evaluate the performance of hydraulic cements in sulfate-rich environments: ASTM C452 Standard Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate and ASTM C1012 Test Method for Length Changes of Hydraulic-Cement Mortars Exposed to a Sulfate Solution. Both ASTM test methods have been the subject of much criticism. Cohen and Mather identified improvements in sulfate testing as a top priority, stating "the current need for reliably predicting long-term durability performance and service life that are based on short-term accelerated tests is more than ever justified." (*9*) Many researchers, including Mehta and Gjorv, 1974; Mehta, 1975; Brown, 1981; Cohen and Mather, 1991; Idorn, *et al.*, 1992; Mehta, 1992; and Tumidajski and Turc, 1995; have expressed concern that existing accelerated test methods do not adequately predict field performance. (*10-12, 9, 13-15*) Specific criticisms of these test methods with the corresponding references are listed in Table 1. Much of the criticism centers on the specimen size, specimen curing, form of sulfate exposure, duration of test, and assessment of sulfate resistance by expansion measurements.

An example of the inadequacy of the accepted test methods is described by Mehta *et al.* (*16*). Alite cements ($C_3S$) tested using ASTM C452 exhibited minimal expansion because the cements contained no $C_3A$, and therefore would not produce ettringite upon exposure to sulfate ions. However, after six years of curing, spalling occurred, and it was discovered the C-S-H had converted to aragonite ($CaCO_3$) and gypsum. The spalling observed was attributed to the crystallization of gypsum caused by sulfate attack. It is important to note the deterioration most

**Table 1 Criticism of ASTM Sulfate Durability Tests (ASTM C452 and ASTM C1012)**

| Criticism | Reference |
|---|---|
| Cause of expansion not determined by the tests; tests do not address formation of ettringite during storage and expansion | Idorn, Johansen, and Thaulow (1992) (*13*) |
| Addition of sulfate in ASTM C452 is not representative of field conditions; both ASTM C452 and ASTM C1012 are too sensitive to specimen size and geometry. | Tumidajski and Turc (1995) (*15*) |
| ASTM C452 does not represent field conditions because inadequate curing of cement results in anhydrous cement compounds being exposed to attack; softening-spalling attack is ignored by both ASTM C452 and ASTM C1012. | Mehta and Gjorv (1974) (*10*) |
| ASTM specifications for sulfate testing of cements (ASTM C452 and C1012) generally ignore the acid-type sulfate attack, which is more typical in the field. | Mehta (1975) (*11*) |
| Cement composition affects the rate of consumption of sulfate ion and introduces variability in ASTM C1012; pH during testing is 3 to 5 orders of magnitude different from field conditions; C1012 tests are protracted because of decreasing amount of sulfate ion in solution. | Brown (1981) (*12*) |

often reported in the field is *not* caused by ettringite formation; rather it is due to the decomposition of CH and C-S-H to gypsum by sulfate ions, and conversion of these hydration products to aragonite (presumably due to carbonation). Neither of the currently accepted ASTM test methods predicts this form of damage. Because both ASTM C452 and C1012 use mortar bar expansion as a measure of sulfate resistance, only the ettringite form of sulfate attack is considered. The accelerated test method described by Mehta and Gjorv uses loss in strength as a measure of sulfate resistance. (*9, 10*) Cracking caused by ettringite or gypsum formation and ettringite expansion as well as loss of C-S-H will adversely affect compressive strength. Thus, both forms of sulfate attack are considered when loss in strength is used as the measure of damage experienced during sulfate exposure.

Contributing to the need for an improved accelerated sulfate resistance test method is the growing interest in construction using cements other than portland cement, such as calcium aluminate and calcium sulfoaluminate cements. The very high early strength and rapid setting qualities of these cements make them attractive for certain applications. However, little information is available concerning the long-term durability of these cements in sulfate environments, particularly in the case of calcium sulfoaluminate cements and blends of calcium aluminates. Before such cements are used widely, it is critical to evaluate their sulfate resistance by a reliable test method.

In this investigation, the sulfate resistance of portland cements, calcium sulfoaluminate cements, and calcium aluminate cement and blends was assessed using an accelerated test method based upon a method proposed by Mehta and Gjorv. (*9, 10*) Fundamentally, the test is different from ASTM standard tests because the pH and sulfate concentration of the solution are maintained throughout the test period, and changes in strength (not expansion) serve as a measure of sulfate resistance. Previously, the method has been used to assess the resistance of portland cement and portland-pozzolan blends. (*10*)

The purpose of the testing is to evaluate the suitability of this accelerated test method for measuring sulfate resistance of non-portland cements for LLPRS projects. A non-portland cement which performs similarly to a known sulfate-resistant portland cement (Type I/II or Type V) would most likely perform satisfactorily in the field for 30 years.

The range of cements tested includes 9 cements:

- 4 portland cements: Type I/II, Type III, Type V, and Type III with pozzolans; referred to as PC1, PC2, PC3, and PC4, respectively.

- 3 calcium aluminate cements, referred to as CA1, CA2, and CA3 in this report.
- 2 calcium sulfoaluminate cements, referred to as CSA1 and CSA2 in this report.

## 3.1 Test Method

12.7-mm hydrated cement paste cubes are immersed in a circulating 4 percent $Na_2SO_4$ solution maintained at a pH of 7.2. The sulfate solution pH of 7.2 is suitable for simulating sulfate attack under slightly acidic conditions typically present in the field. Given that specimen dimensions along with permeability affect ingress of sulfate ions into a specimen, dimensions were selected to maximize the surface-to-volume ratio, thus increasing the potential for reaction with the sulfate solution. With a high surface-to-volume ratio, the testing time required to determine a cements susceptibility is reduced to between four and nine weeks. The lower the surface-to-volume ratio, the less likely the sulfate ions will permeate throughout the specimen and potentially react to cause a strength reduction.

A schematic of the test apparatus is shown in Figure 1; a photograph is provided in Figure 2. Under the control of three pumps, sulfate solution circulates among two 22.7-liter capacity polypropylene tanks. One of the polypropylene tanks contains approximately 14L of sulfate solution and acts as a reservoir. Sulfate solution is pumped from the reservoir through 12.7-mm chemical-resistant polyethylene tubing at a rate of 19 L/min by a variable speed pump drive (Cole Parmer model 75225-10) with attached MICROPUMP® pump (model 101-000-010). A microprocessor-based water treatment controller (Pulsatrol model MCT1208) constantly monitors the pH of the solution before it flows into the tank containing the specimens. If the pH measures below 7.2, aliquots of 0.1N $H_2SO_4$ are pumped, by an electronic metering pump (Pulsatron model LPH6EA-PTC3), into the reservoir tank. In the second 22.7-liter polypropylene tank, the specimens are arranged on 10-mm polycarbonate mesh, which allows the solution to flow around the specimens and increases potential for reaction between the cement paste and the sulfates. The sample tank contains approximately 19L of sulfate solution.

Because the pH is maintained constantly, changes in the sulfate solution pH caused by leaching of CH from the paste are minimized. In addition, Brown demonstrated that controlling the pH through additions of sulfuric acid ensures that the sulfate ion concentration of the solution remains constant over time. (*12*) The conditions provided in this accelerated test method are more representative of field conditions because, unlike other accelerated methods, the sulfate concentration and the pH of the solution remain constant as these parameters do in the field.

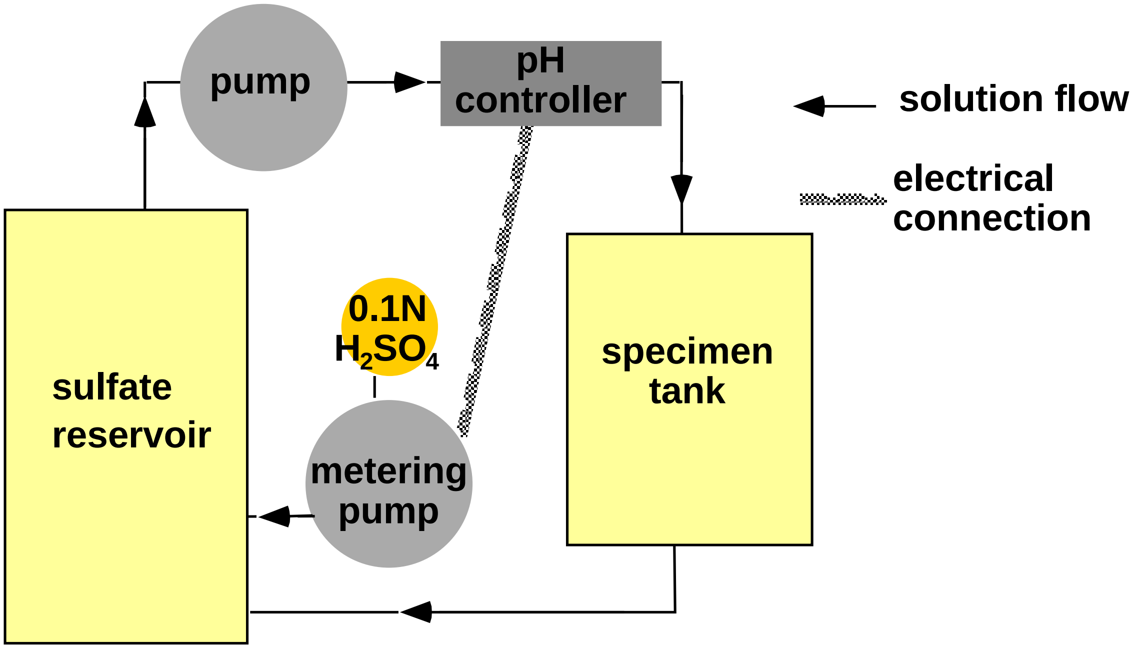


**Figure 1. Schematic of pH-controlled accelerated test apparatus to measure sulfate resistance of cement paste.**

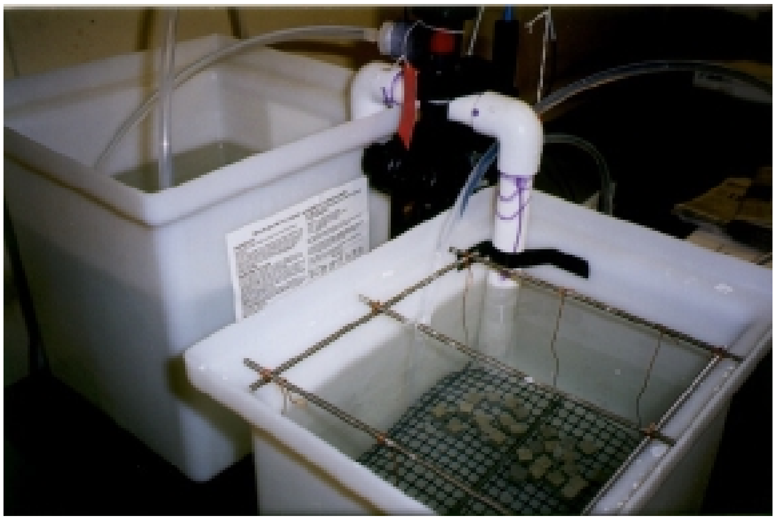


**Figure 2. Photograph of the accelerated test apparatus showing the sample tank (right) and the solution reservoir (left).**

## 3.2 Test Program

The sulfate resistance of three classes of cements was evaluated: portland cements and blends (PC), calcium aluminate cements and blends (CA), and calcium sulfoaluminate cements (CSA). Commercially available cements conforming to ASTM Type I/II, Type III, and Type V designations were included in the test program. Loss of strength for Type I/II, III, and V portland cement cubes will serve to distinguish between cements that perform well in a sulfate-rich environment (Type V and Type I/II) and those that typically perform poorly (Type III). Type I/II was developed as a moderately sulfate-resistant cement whereas Type V was originally manufactured for high sulfate resistance. Type III cements typically do not perform well in high-sulfate environments due to their higher percentage of $C_3A$ (e.g., as much as 15 percent) in the clinker.

Many of the cement manufacturers have requested that the trade names of the cements submitted to Caltrans for testing be concealed. Hence, the cements are referenced by a designation based upon their classification, as shown in Table 2. In addition, cement compositions were not provided by some of the manufacturers. To gain further information about the chemical composition of these cements, chemical oxide analyses were performed. Results from the chemical oxide analysis are also provided in Table 2.

Estimations of the cement composition can be made from the chemical oxide analysis. For portland cements, the calculations are straightforward and are described by the Bogue equations (ASTM C 150). However, no such equations exist for calcium aluminate cements and blends nor for calcium sulfoaluminate cements. Approximations of the composition of these cement types may be obtained by modifying the Bogue equations. The approximate

**Table 2 Designation, Classification, and Chemical Oxide Analysis of Cements Tested for Sulfate Resistance.**

| | **Portland Cements and Blends** | | | | **Calcium Aluminate Cements and Blends** | | | **Calcium Sulfoaluminate Cements** | |
|---|---|---|---|---|---|---|---|---|---|
| **Component** | **PC1** | **PC2** | **PC3** | **PC4*** | **CA1** | **CA2** | **CA3** | **CSA1** | **CSA2** |
| SiO2 | 21.24 | 21.05 | 20.50 | 20.50 | 9.06 | 18.76 | 5.60 | 15.59 | 15.40 |
| Al2O3 | 3.57 | 3.79 | 4.15 | 4.15 | 26.29 | 10.54 | 48.66 | 13.96 | 12.88 |
| Fe2O3 | 3.82 | 4.02 | 3.70 | 3.70 | 3.44 | 2.94 | 5.49 | 1.49 | 2.63 |
| CaO | 64.72 | 64.22 | 64.27 | 64.27 | 36.97 | 53.78 | 37.04 | 50.19 | 53.02 |
| MgO | 1.69 | 1.26 | 1.27 | 1.27 | 0.97 | 1.99 | 0.39 | 1.35 | 2.03 |
| TiO2 | 0.31 | 0.36 | 0.34 | 0.34 | 1.17 | 0.60 | 2.33 | 0.53 | 0.70 |
| Mn2O3 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 | 0.05 | 0.05 | 0.04 |
| P2O5 | 0.32 | 0.35 | 0.28 | 0.28 | 0.07 | 0.10 | 0.12 | 0.14 | 0.01 |
| Cr2O3 | 0.04 | 0.03 | 0.04 | 0.04 | 0.04 | 0.04 | | 0.04 | 0.03 |
| ZrO2 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.54 | | 0.05 | 0.06 |
| Na2O | 0.18 | 0.23 | 0.20 | 0.20 | 1.24 | 0.34 | 0.06 | 0.23 | 0.39 |
| K2O | 0.10 | 0.13 | 0.20 | 0.20 | 0.03 | 0.22 | 0.23 | 0.44 | 0.32 |
| SO3 | 2.27 | 2.45 | 2.33 | 2.33 | 8.73 | 6.81 | 0.03 | 14.20 | 10.81 |
| Ig. Loss | 1.43 | 1.83 | 2.42 | 2.42 | 12.19 | 3.18 | | 2.17 | 1.96 |
| Total | 99.72 | 99.77 | 99.73 | 99.73 | 100.33 | 99.90 | 100.00 | 100.42 | 100.27 |

* Composition provided is for the cement only. PC4 is Type III cement, fly ash, and silica fume.

compositions of the cements included in the accelerated test program are given in Table 3.

As shown in Table 3, Type III cement has a low percentage of $C_3A$. ASTM C 150 states that Type III cements with less than 5 percent $C_3A$ are highly sulfate resistant. This is characteristic of a California manufactured Type III cement. Typically, Type III cements have $C_3A$ contents of 10 percent. When comparing Type I/II, III, and V, similar sulfate resistance should be expected because all three of these cements have similar chemical compositions.

Cement paste was prepared from each of the cements being tested using a water-to-cement ration of 0.50. The pastes were formed into 12.7-mm cubes in watertight molds. The water-to-cement ratio of 0.50 increases the permeability of the hydrated cement paste cubes and

**Table 3 Approximate Compositions of the Cements Included in the Accelerated Test Program.**

| | Description | Bogue Composition |
|---|---|---|
| PC1 | Type I/II: moderate sulfate resisting portland cement | $C_3S$ =66.13, $C_2S$ = 11.00, $C_3A$=3.00, $C_4AF$ =11.62 |
| PC2 | Type V: sulfate resisting portland cement | $C_3S$ =63.27, $C_2S$ = 12.62, $C_3A$=3.24, $C_4AF$ =12.23 |
| PC3 | Type III: rapid setting portland cement | $C_3S$ =66.03,$C_2S$ = 8.96, $C_3A$=4.74, $C_4AF$ =11.26 |
| PC4 | Blend Type III cement and 10% Class F fly ash and 8% silica fume by weight | N/A |
| CA1 | Portland Cement, CA, C | N/A |
| CA2 | Portland Cement, CA, C | N/A |
| CA3 | Primarily CA | N/A |
| CSA1 | $C_2S$, $C_3S$, $C_4AF$, $C_3A$, C , and $C_4A_3$ | N/A |
| CSA2 | Primarily $C_2S$ and $C_4A_3$ | N/A |

C = CaO; S = $SiO_2$; A = $Al_2O_3$; F = $Fe_2O_3$; = $SO_3$

allows for better comparison between various cement types. If different water-to-cement ratios were used for each cement type, then it would be difficult to compare the final strength results because of the differences in the hydrated cement paste permeabilities.

The cubes were cured in the molds for 24 hours followed by moist curing at 50 C for 6 days. Even slow-hydrating cement, such as blended, pozzolan-containing, or slag-containing cement, will be well hydrated after 7 days moist curing at 50 C. (*11*) Because heat and moisture are known to accelerate the conversion of calcium aluminate cements (CAC), half of the CAC samples were cured at 20 C for 6 days, and the other half were cured at 50 C for 150 days in a moist (~100 percent relative humidity) environment to induce conversion. Table 4 provides details about the curing regimen for the cements tested.

After curing, control samples were tested at 7 days to measure compressive strength prior to sulfate immersion. For all compression testing, load was applied at a rate of 2.7 kN/min. After 28 days and 63 days of immersion, compressive strength was measured and compared to the initial 7-day strength. Because a one-to-one correlation between 28-day sulfate exposure and long term field performance doesn't exist, nine weeks (63 days) of exposure was also included to verify the results of the 28-day exposure. In addition, the strength of the calcium aluminate

**Table 4 Description of Curing Schedule for Cements Evaluated.**

| Curing Regimen | | | |
|---|---|---|---|
| Temp. | Humidity | Time | Cements |
| 20C | moist | 6 days | CA1, CA2, CA3 |
| 50C | moist | 6 days | PC4, CSA1, CSA2, Type I/II, Type III, Type V |
| 50C | moist | 150 days | CA1, CA2, CA3 |

cements was measured after 150 days at 50 C and approximately 100 percent relative humidity. This strength was compared to the initial 7-day strength.

Accelerated sulfate testing was performed on converted calcium aluminate cement cubes, with one cement exhibiting a significant decrease in strength resulting from conversion (i.e. CA3; see Section 4.0). Table 5 shows the number of samples tested at each time interval. The number of samples tested at each interval depended upon the agreement of the compression test data, and ranged from 7-12 samples per test period. Samples exhibiting noticeable defects (e.g., surface defects or voids) were not compression tested.

**Table 5** **Number of Samples for Each Cement Tested in Compression at Each Time Interval.**

| | **Age** | | | | |
|---|---|---|---|---|---|
| **Cement Designation** | **7 days Moist Cure at 50 C and 20 C for CA.** | **35 days (28 days exposure + 7 day moist cure at 50 C and 20 C for CA)** | **70 days (63 days exposure + 7 day moist cure at 50 C and 20 C for CA)** | **157 days (150 days conversion period at 50 C, ~100%RH + 7 days moist cure)** | **185 days (28 days sulfate exposure after conversion + 7 days moist cure)** |
| PC1 | 8 | 8 | 7 | NA | NA |
| PC2 | 10 | 10 | 10 | NA | NA |
| PC3 | 12 | 11 | 10 | NA | NA |
| PC4 | 12 | 10 | 9 | NA | NA |
| CSA1 | 10 | 0* | 0* | NA | NA |
| CSA2 | 10 | 11 | 11 | NA | NA |
| CA1 | 10 | 11 | 0* | 4 | 0** |
| CA2 | 8 | 11 | 11 | 7 | 0** |
| CA3 | 8 | 7 | 8 | 9 | 12 |

* specimens too damaged for testing

** testing not performed

Note: Calcium aluminate cements were tested as both unconverted cement paste and as converted cement paste.

## 4.0 RESULTS AND DISCUSSION

Results show a wide range of performance, with the strength of some cement pastes relatively unaffected by sulfate exposure, while others experienced enough damage during exposure that they could not be tested in compression. Figures 3a and 3b show that some cement paste cubes, such as those prepared from CA3, did not appear to experience significant damage. Others, such as those prepared from CSA1, experienced significant sulfate attack.

**Figure 3a. CA3 after 63 days of exposure to 4 percent $Na_2SO_4$ solution maintained at a pH of 7.2.**

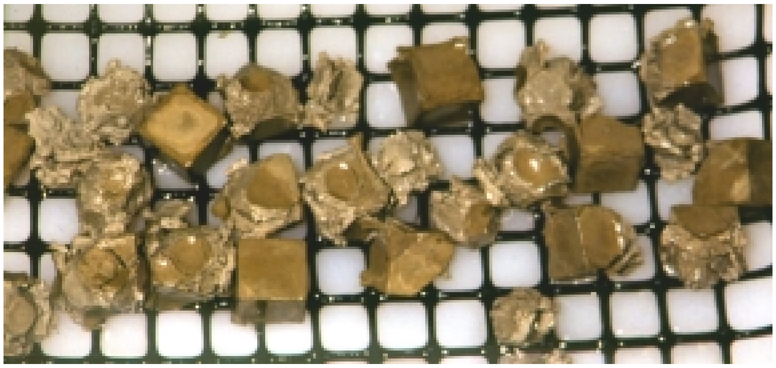


**Figure 3b. CSA1 after 28 days of exposure to 4 percent $Na_2SO_4$ solution maintained at a pH of 7.2.**

Figures 4 and 5 show the relative change in average compressive strength ($\Delta f$) measured after 4 weeks ($f_{28d}$) and 9 weeks ($f_{63d}$) of exposure, respectively. The change in strength is calculated as a percentage of the strength of each cement paste measured after 7 days hydration ($f_{7d}$):

$$\Delta f(\%) = \frac{f_{28d} - f_{7d}}{f_{7d}} \cdot 100$$

or

$$\Delta f(\%) = \frac{f_{63d} - f_{7d}}{f_{7d}} \cdot 100$$

Loss in strength during the exposure period can be the result of cracking caused by expansion of ettringite and gypsum formation and the loss of C-S-H. Some cements may continue to hydrate in the sulfate solution and may experience an increase in strength. Susceptibility to sulfate attack can only be interpreted through strength loss. Increases in strength do not provide any information about sulfate resistance; such results only show that the cement continues to hydrate in sulfate solution over the test period.

Figure 4 shows the effect of 28 days of exposure to sulfate solution on the compressive strength, as compared to strength measured after 7 days of hydration. Only three cements showed a strength reduction after 28 days of sulfate exposure. The strength of CA2 and PC4 (the portland/pozzolan blend) declined moderately, 17.5 and 16.9 percent, respectively. After 28 days of exposure, CSA1 samples had deteriorated to such an extent that they could not be compression tested (See Figure 3b). Note: Type I/II, Type III, and Type V showed similar sulfate resistance.

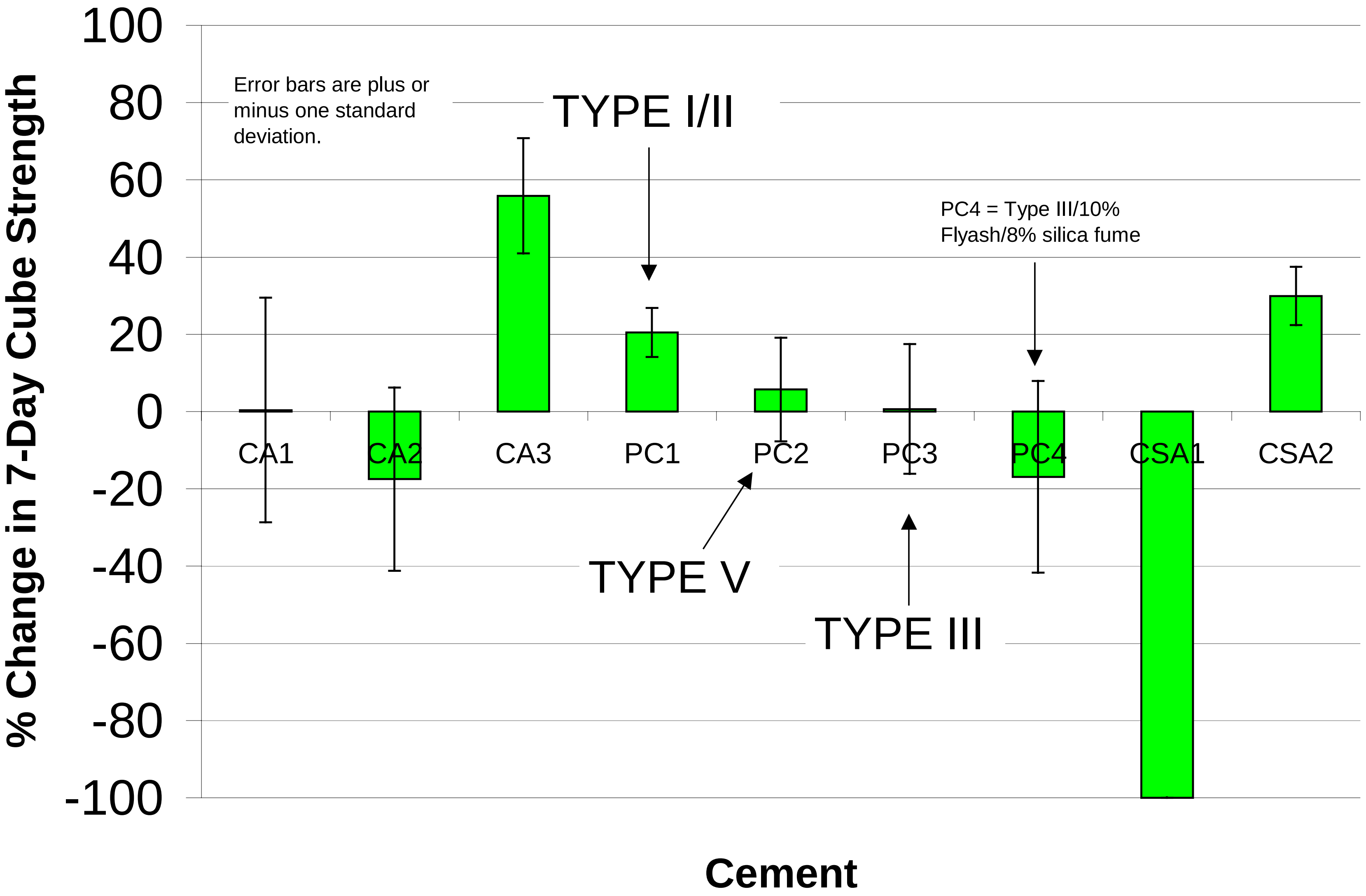
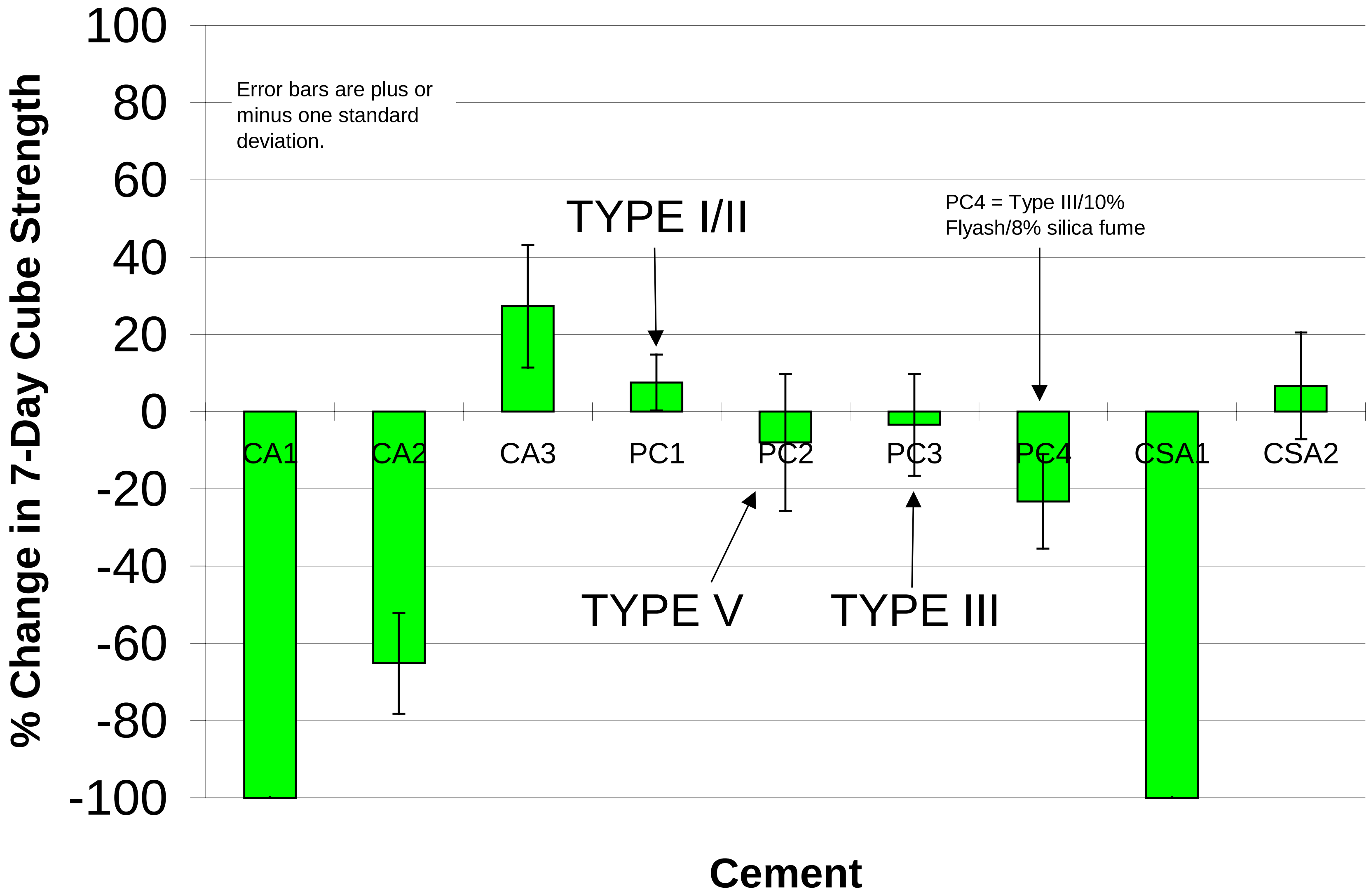


**Figure 4. Results of accelerated sulfate test after 4 weeks (28 days) of exposure. Change in strength is based upon strength measured at 7 days of age, prior to exposure, for each cement.**



Error bars are plus or minus one standard deviation.

PC4 = Type III/10% Flyash/8% silica fume

**Figure 5. Results of accelerated sulfate test after 9 weeks (63 days) of exposure. Change in strength is based upon strength measured at 7 days of age, prior to exposure, for each cement.**

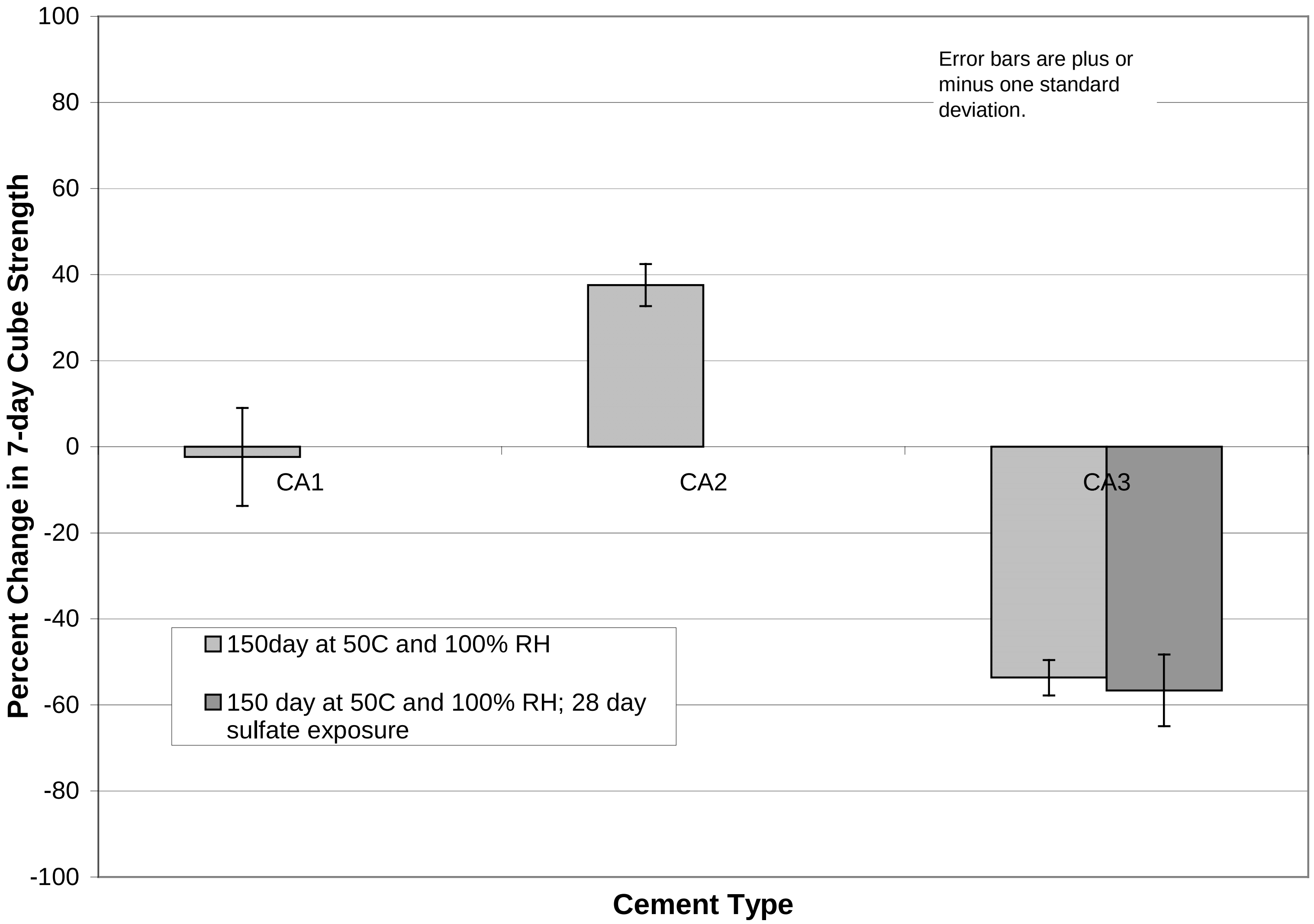
Figure 5 shows the effect of 63 days of exposure to sulfate solution on the compressive strength, as compared to strength measured after 7 days of hydration. Four cements performed less satisfactorily than Type III/Type V cement. The strength of PC4 was again moderately (23.3 percent) less than its strength at 7 days. CA2 experienced a more substantial strength loss of 65.2 percent. Cement paste cubes prepared from CA1 and CSA1 were too damaged to test.

Figure 6 shows the effect of 150 days of exposure to high temperature (50 C) and moisture (approximately 100 percent relative humidity), conditions expected to induce conversion in calcium aluminate cements. Change in strength ($\Delta f$) due to conversion was calculated from the strength at 157 days of age ($f_{157d}$), after 150 days of high temperature/high humidity exposure relative to the 7 day strength ($f_{7d}$):

$$\Delta f\,(\%) = \frac{f_{157d} - f_{7d}}{f_{7d}} \cdot 100$$

Of the calcium aluminate cements and blends tested, only the strength of CA3 was significantly decreased by this exposure condition (53.6 percent). Cubes of converted CA3 were subjected to 28 days of the accelerated sulfate test. As shown in Figure 6, the sulfate exposure did not appear to affect the strength of the converted cement paste CA3.

Overall, the test results show the sulfate resistance of these cements should be examined individually because no general trends were apparent based upon cement classification. While some calcium aluminate or calcium sulfoaluminate cements performed well under sulfate exposure (CA3, CSA2), other cements experienced notable damage. Monosulfate hydrate and calcium hydroxide are the cement hydration products that react directly with sulfate ions. The volumes of these products formed during cement hydration are directly linked to the chemical



Error bars are plus or minus one standard deviation.

**Figure 6. Results of compression testing after 5 months of exposure to conditions expected to induce conversion (50 C and approximately 100 percent relative humidity). Change in strength is based upon strength measured at 7 days of age, prior to exposure, for each cement. CA3 experienced a significant decrease in strength due to conversion, and converted cubes of that cement were subsequently exposed to sulfate solution for 28 days.**

composition of the cement. The chemical composition may vary substantially within a class of cements, as with calcium sulfoaluminate cements. CSA1 had an inferior sulfate resistance probably due to the presence of $C_3A$ in its chemical composition.

From the 28- and 63-day sulfate resistance test results, performance of the nine cements and blends tested can be described by the following ranking:

At 28 days,

CA3 = CSA2 = Type I/II > Type V = Type III > CA1 > PC4 > CA2 > CSA1

At 63 days,

CA3 = Type I/II = CSA2 > Type III = Type V > PC4 > CA2 > CA1 > CSA1

Performance of CA1 is ranked higher than CSA1 because CSA1 failed earlier.

Type V was expected to perform better than Type I/II. However, as indicated in Table 3, Type I/II had a slightly lower amount of $C_3A$ than Type V, leading to improved sulfate resistance. Higher amounts of $C_3A$ in cement decreases its sulfate resistance because the $C_3A$ can later react with external sulfate ions. As shown in Appendix A, Type V had a much higher coefficient of variation compared to Type I/II. This could also be another reason for the discrepancy in the sulfate resistance of Type I/II and Type V. As seen in Figures 4 and 5, Type III cement performed similarly to Type I/II and Type V. As stated earlier, the main reason this specific California Type III cement performed similarly to Type I/II and Type V was its low $C_3A$ content (< 5 percent). PC4, which consisted of a Type III cement, 10 percent Type F fly ash, and 8 percent silica fume, was expected to perform better than the Type III cement alone. Two reasons for the poor performance of PC4 were inadequate curing time for the Type F fly ash in the blend and difficulty in uniformly dispersing the silica fume in such a small batch without any superplasticizer.

The cube strength results for CA1 and CA2, shown in Appendix B, were lower than the other cements.  The one likely reason for their low strength at 7 days was that both were calcium aluminates blended with portland cement.  Given that CA1 and CA2 were cured at 20 C and 100 percent humidity to prevent conversion at 50 C and 100 percent relative humidity, this curing regime for a blended calcium aluminate may have resulted in insufficient hydration by 7 days. Appendix B and Figure 6 show that CA1 did not significantly gain or lose strength when cured for 150 days at 50 C and 100 percent relative humidity.  This indicates that more curing before sulfate exposure probably would not have affected the CA1 results differently.  However, the CA2 results may have been affected if cured at 50C for 6 days instead of at 20C.

# 5.0 PROPOSED TESTING GUIDELINE FOR SUSCEPTIBILITY OF CEMENT TO SULFATE ATTACK

A flowchart has been created to outline the steps necessary to determine if a cement is susceptible to sulfate attack, as shown in Figure 7. The steps in Section 5.1 correspond to this flowchart. Note: The sulfate test employed in this research report should be used to determine cement sulfate susceptibility after 7 days of curing and 28 days of sulfate exposure.

## 5.1 Outline of Test Procedure

1. When testing for the sulfate resistance of any cement type (portland or other hydraulic cement material), it is necessary to identify whether calcium aluminate exists at a level high enough to cause conversion of the hydrated paste. The proposed cement should be tested for thermal stability using ASTM C 109. As per Caltrans existing thermal stability specification for FSHCC, mortar cubes should be cast and cured for 24 hours in their molds in a moist curing room at 23 C. Half the specimens should be cured one day in water at 23 C followed by two days in water at 50 C. The other cube specimens should be cured for three days in water at 23 C.
2. The thermal stability of the cement is defined as the ratio of the 3-day strength of the specimens cured at 50 C to the 3-day strength of the specimens cured at 23 C. Caltrans currently specifies 90 percent as the minimum thermal stability of any hydraulic cement to be used in FSHCC projects. (The existing Caltrans specification

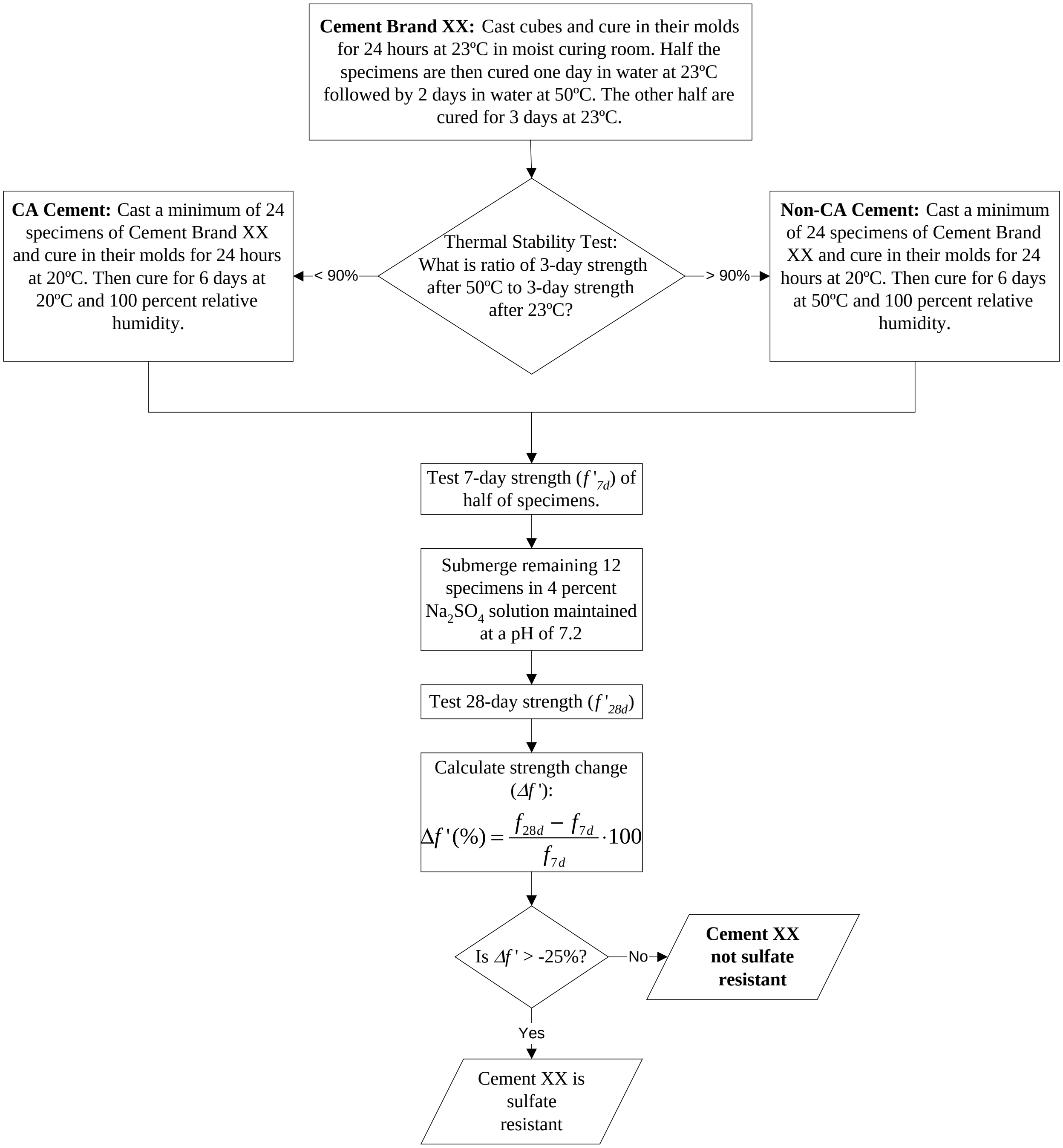


**Figure 7. Basic flowchart showing the steps for the proposed accelerated test for sulfate resistance of cement.**

precludes use of high calcium aluminate cements at this time. Caltrans may wish to consider their use for pavement applications in the future, provided they are structurally designed using the converted strength.) If the cement has a thermal stability less than 90 percent, then it is considered to be dominated by calcium aluminate. Note: some proprietary cements have calcium aluminate in them (e.g., CA1 and CA2), but the amounts are insufficient to cause conversion, as shown in Figure 6.

3. From the thermal stability test, a proposed cement can be classified as calcium aluminate cement (CAC) or non-CAC.
4. A minimum of 24 specimens should then be cast and cured in their molds for 24 hours at 23 C.
5. If the cement was determined to be CAC, the 24 specimens are then cured at 20 C and 100 percent relative humidity for six days. If the cement was determined to be non-CAC, the 24 specimens are then cured at 50 C and 100 percent relative humidity for six days.
6. The cube strengths for 12 of the 24 specimens (half the total replicates) are measured at 7 days (i.e., after the curing described in Step 5). This yields the 7-day strength ($f'_{7d}$).
7. The remaining 12 specimens (whether CAC or non-CAC) are submerged in a 4 percent $Na_2SO_4$ solution with a constant pH of 7.2 for 28 days.

8. The cube strengths for the sulfate-exposed 12 specimens are measured after the 28-day sulfate exposure. A material will be considered sulfate susceptible if the average reduction in cube strength is greater than 25 percent:

$$\Delta f(\%) = \frac{f_{28d} - f_{7d}}{f_{7d}} \cdot 100$$

## 5.2 Other Considerations

Other researchers have recommended using a 25 to 30 percent reduction in original strength as an indication of poor sulfate resistance. (*17-19*) With this strength reduction specification, only one cement tested for this project would not have passed after 28 days exposure to the 4 percent $Na_2SO_4$ solution.

If a cement shows borderline performance at 28 days, Caltrans may decide to require a 9-week (63-day) sulfate exposure to verify the 28-day results. This would require casting 36 cube specimens initially.

For cements being tested for the first time, it is recommended that the strength reduction at 4 and 9 weeks of sulfate exposure be determined.

Given that Caltrans is embarking on a large capital expenditure on their urban freeways, it is imperative that these FSHCCs provide 30 years service life without chemical degradation if they are going to be used for LLPRS concrete projects.

## 6.0 SUMMARY

Results were presented from accelerated sulfate resistance testing of five cements submitted to Caltrans by four different manufacturers, one portland/pozzolan blend (PC4) prepared in the laboratory, and three standard portland cement types. The cements were classified into three categories: portland cements and blends (PC), calcium aluminate cements and blends (CA), and calcium sulfoaluminate cements (CSA). After 28 and 63 days of exposure to a 4 percent $Na_2SO_4$ solution in which the pH was maintained at 7.2, performance of the nine cements and blends tested can be described by the following order:

At 28 days,

CA3 = CSA2 = Type I/II > Type V = Type III > CA1 > PC4 > CA2 > CSA1

At 63 days,

CA3 = Type I/II = CSA2 > Type III = Type V > PC4 > CA2 > CA1 > CSA1

This laboratory testing has shown that several cementitious products may be susceptible to sulfate attack (CA1, CA2, CSA1). Oxide analyses of the portland cements show that Type I/II, Type III, and Type V had similar tricalcium aluminate contents (< 5 percent). The accelerate sulfate testing results show that all three cement types were sulfate resistant, as is expected from the chemical oxide analysis. One calcium aluminate and one calcium sulfoaluminate cement type demonstrated excellent sulfate resistance.

Due to the lack of evidence of existing sulfate attack on California highway pavements, it is can be assumed that the existing cement being utilized by Caltrans (Type I/II) is sulfate resistant.  The sulfate resistance of Type I/II cement is considered the baseline for evaluation of other cements in this report.  It is possible that sulfate attack exists, but that it not been detected or correctly identified.  Finally, sulfate attack may not be a problem because concrete pavements are typically not in continuous contact with sulfate rich soils and/or groundwater.

Little to no durability experience is available in pavement applications using many new cements being proposed for highway reconstruction.  Considering the large investment Caltrans will be making in urban freeway reconstruction, the cost of requiring sulfate resistant concrete materials is small, and could be considered “cheap insurance.”

## 7.0 RECOMMENDATIONS

It is recommended that Caltrans adopt and enforce the sulfate resistance guidelines for 100 percent portland cement concretes, as defined by ACI Building Code 318/318-95 (section 4.3 sulfate exposures, p. 37-38). (*20*) Table 6 shows the ACI sulfate resistance guidelines and summarizes the appropriate types of cement and maximum water-to-cementitious materials ratio for concrete exposed to injurious concentrations of sulfates from soil and water. The building code recommends maximum water-to-cement ratios and minimum compressive strength for different sulfate exposure levels. The building code does not address cement types other than portland cement.

If a contractor proposes to use a different type of cementitious material not covered by the ACI Building Code 318/318-37, evidence should be provided that the material in question is sulfate-resistant. The proposed cementitious material should have similar performance to ASTM

**Table 6 Requirements for concrete exposed to sulfate-containing solutions. (*20*)**

| **Sulfate Exposure** | **Water soluble sulfate in soil, percent by weight** | **Sulfate in water ($SO_4$), ppm** | **Cement Type** | **Maximum water-cementitious materials** | **Minimum compressive strength, psi** |
|---|---|---|---|---|---|
| Negligible | 0.00-0.10 | 0-150 | --- | --- | --- |
| Moderate | 0.10-0.20 | 150-1500 | II, IP (MS), IS (MS), P (MS), I(PM) (MS), I(SM) (MS) | 0.50 | 4000 |
| Severe | 0.20-2.00 | 1500-10,000 | V | 0.45 | 4500 |
| Very Severe | Over 2.00 | Over 10,000 | V plus pozzolan | 0.45 | 4500 |

Note: Type I/II should be limited to 8% $C_3A$ under ASTM C 150 and Type V should be limited to 5% $C_3A$ under ASTM C 150

Type I/II cement in the accelerated sulfate resistance test described in this report. Any proposed cementitious material should have less than 25 percent loss of strength at 28 days and 63 days of sulfate exposure relative to the 7-day strength of the material. The procedure described in Section 5.0 and Figure 7 should be followed.

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# APPENDICES

## Appendix A

### Summary of Cube Strength of Portland Cements (psi)

| | **PC1 (7-Day)** | **PC1 (35-Day)** | **PC1 (70-Day)** | **PC2 (7-Day)** | **PC2 (35-Day)** | **PC2 (70-Day)** |
|---|---|---|---|---|---|---|
| | 1138 | 1810 | 1576 | 1428 | 1364 | 1352 |
| | 1550 | 1904 | 1546 | 1390 | 1602 | 1568 |
| | 1332 | 1746 | 1504 | 1160 | 1178 | 1102 |
| | 1238 | 1914 | 1656 | 1340 | 1482 | 1328 |
| | 1444 | 1704 | 1386 | 1450 | 1298 | 1060 |
| | 1634 | 1802 | 1740 | 1156 | 1610 | 924 |
| | 1652 | 1636 | 1624 | 1342 | 1560 | 1050 |
| | 1740 | 1618 | | 1396 | 1560 | 1362 |
| | | | | 1260 | 1100 | 1212 |
| | | | | 1318 | 1246 | 1270 |
| Average: | 1466 | 1767 | 1576 | 1324 | 1400 | 1223 |
| Std. Dev.: | 215 | 112 | 114 | 103 | 188 | 191 |
| C.O.V.: | 15% | 6% | 7% | 8% | 13% | 16% |
| | *PC1 = Type I/II Portland Cement* | | | *PC2 = Type V Portland Cement* | | |

| | **PC3 (7-Day)** | **PC3 (35-Day)** | **PC3 (70-Day)** | **PC4 (7-Day)** | **PC4 (35-Day)** | **PC4 (70-Day)** |
|---|---|---|---|---|---|---|
| | 1842 | 1888 | 1560 | 1738 | 1082 | 1128 |
| | 1616 | 1808 | 1506 | 1622 | 1120 | 1034 |
| | 2012 | 1596 | 1490 | 1630 | 1380 | 1320 |
| | 1722 | 2282 | 2054 | 1520 | 1076 | 1376 |
| | 1802 | 2008 | 1510 | 1308 | 700 | 1270 |
| | 1576 | 1322 | 2076 | 1576 | 1184 | 1114 |
| | 1578 | 1876 | 1664 | 1342 | 1600 | 1240 |
| | 1904 | 2054 | 1944 | 1592 | 1764 | 946 |
| | 2004 | 1940 | 1694 | 1520 | 1612 | 1320 |
| | 1892 | 1310 | 1700 | 1644 | 1426 | |
| | 1512 | 1642 | | 1540 | | |
| | 1916 | | | 1650 | | |
| Average: | 1781 | 1793 | 1720 | 1557 | 1294 | 1194 |
| Std. Dev.: | 176 | 301 | 226 | 125 | 321 | 146 |
| C.O.V.: | 10% | 17% | 13% | 8% | 25% | 12% |
| | *PC3 = Type III Portland Cement* | | | *PC4 = Type III Portland Cement with10% Type F Fly Ash and 8% Silica Fume* | | |

## Appendix B

### Summary of Cube Strength of Calcium Aluminate Cements (psi)

| | **CA1 (7-Day)** | **CA1 (35-Day)** | **CA1 (70Day)** | **CA2 (7-Day)** | **CA2 (35-Day)** | **CA2 (70-Day)** |
|---|---|---|---|---|---|---|
| | 832 | 958 | no | 1208 | 1224 | 508 |
| | 880 | 1226 | test | 1356 | 1468 | 474 |
| | 905 | 1030 | done | 1329 | 1264 | 404 |
| | 863 | 1048 | cubes | 1390 | 1214 | 516 |
| | 911 | 722 | were | 1340 | 908 | 478 |
| | 1045 | 708 | too | 926 | 1034 | 452 |
| | 895 | 690 | deteriorated | 1324 | 776 | 400 |
| | 760 | 812 | | 1369 | 1146 | 474 |
| | 670 | 874 | | | 1104 | 310 |
| | 460 | 674 | | | 904 | 450 |
| | | 338 | | | 580 | 440 |
| Average: | 822 | 825 | | 1280 | 1057 | 446 |
| Std. Dev.: | 161 | 240 | | 153 | 250 | 58 |
| C.O.V.: | 20% | 29% | | 12% | 24% | 13% |

| | **CA3 (7-Day)** | **CA3 (35-Day)** | **CA3 (70-Day)** |
|---|---|---|---|
| | 2780 | 3600 | 3610 |
| | 2990 | 3340 | 3660 |
| | 2590 | 5060 | 2240 |
| | 2440 | 3970 | 3330 |
| | 1880 | 4630 | 3040 |
| | 2620 | 3760 | 3240 |
| | 2650 | 3880 | 3990 |
| | 2750 | | 3240 |
| Average: | 2588 | 4034 | 3294 |
| Std. Dev.: | 328 | 603 | 521 |
| C.O.V.: | 13% | 15% | 16% |

## Appendix B *continued*

**150 Days at 150°C and 100% Relative Humidity.**

| | CA1 (7-Day) | CA1 (157-Day) | CA2 (7-Day) | CA2 (157-Day) |
|---|---|---|---|---|
| | 832 | 804 | 1208 | 1762 |
| | 880 | 914 | 1356 | 1696 |
| | 905 | 690 | 1329 | 1814 |
| | 863 | 802 | 1390 | 1774 |
| | 911 | | 1340 | 1892 |
| | 1045 | | 926 | 1620 |
| | 895 | | 1324 | 1770 |
| | 760 | | 1369 | |
| | 670 | | | |
| | 460 | | | |
| Average: | 822 | 803 | 1280 | 1761 |
| Std. Dev.: | 161 | 91 | 153 | 86 |
| C.O.V.: | 20% | 11% | 12% | 5% |

| | CA3 (7-Day) | CA3 (157-Day) | CA3 (187-Day) 30-day Sulfate Exposure |
|---|---|---|---|
| | 2780 | 1174 | 1018 |
| | 2990 | 1208 | 1040 |
| | 2590 | 1144 | 1152 |
| | 2440 | 1208 | 1026 |
| | 1880 | 1200 | 1128 |
| | 2620 | 1296 | 1104 |
| | 2650 | 1174 | 1014 |
| | 2750 | 1248 | 1328 |
| | | 1142 | 1116 |
| | | | 1220 |
| | | | 1160 |
| | | | 1170 |
| Average: | 2588 | 1199 | 1123 |
| Std. Dev.: | 328 | 49 | 93 |
| C.O.V.: | 13% | 4% | 8% |

## Appendix C

### Summary of Cube Strength of Calcium Sulfoaluminate Cements (psi)

| | CSA1 (7-Day) | CSA1 (35-Day) | CSA1 (70-Day) | CSA2 (7-Day) | CSA2 (35-Day) | CSA2 (70-Day) |
|---|---|---|---|---|---|---|
| | 1210 | Cubes | Cubes | 1712 | 2050 | 1650 |
| | 1456 | were | were | 1602 | 1900 | 1440 |
| | 1248 | no | no | 1466 | 2120 | 1476 |
| | 1272 | longer | longer | 1440 | 1738 | 1948 |
| | 1242 | in | in | 1474 | 1928 | 1264 |
| | 1220 | testing | testing | 1538 | 2168 | 1456 |
| | 1302 | condition | condition | 1414 | 1968 | 1674 |
| | 1336 | | | 1648 | 2156 | 1940 |
| | 1354 | | | 1660 | 1986 | 1932 |
| | 1278 | | | 1452 | 1808 | 1686 |
| | | | | | 2198 | 1612 |
| Average: | 1292 | | | 1541 | 2002 | 1643 |
| Std. Dev.: | 74 | | | 107 | 152 | 227 |
| C.O.V.: | 6% | | | 7% | 8% | 14% |