Design of an AAR Prone Concrete Mix for Large Scale Testing

Task 1-A

Final Report

by

Victor Saouma Robb Sparks David Graff

University of Colorado, Boulder

NRC-HQ-60-14-G-0010: Reports

1-A: Design of an AAR Prone Concrete Mix for Large Scale Testing

1-B: AAR Expansion; Effect of Reinforcement, Specimen Type, and Temperature 1-C: Effect of AAR on Shear Strength of Panels 2: Diagnosis & Prognosis of AAR in Existing Structures 3-a: Risk Based Assessment of the Effect of AAR on Shear Walls Strength 3-b: Probabilistic Based Nonlinear Seismic Analysis of Nuclear Containment Vessel Structures with AAR

Page 1 | 93

Page 2|93

| 1 | Ove | erview | 8 |
|---|-------|---|----|
| | 1.1 I | dentification of Reactive Aggregates | 8 |
| | 1.2 (| Overview of ASTM C1567 Mortar Bar Test | 10 |
| | 1.3 N | Narrative of Aggregate Testing | 10 |
| | 1.4 7 | Fest Results | 17 |
| | 1.5 (| Conclusion | 21 |
| 2 | Cor | ncrete Testing Program | 22 |
| | 2.1 M | Material Properties | 22 |
| | 2.1. | 1 Cement Mill Reports | 22 |
| | 2.2 A | Aggregate Tests | 25 |
| | 2.2. | 1 Coarse Aggregate Relative Density | 25 |
| | 2.2. | 2 Fine Aggregate Relative Density | 26 |
| | 2.2. | Coarse Aggregate Bulk Density | 28 |
| | 2.2. | Sleve Analysis and Fineness Modulus Detrographic Study | 29 |
| | 2.22 | 2.2.5.1 Introduction | 31 |
| | 2 | 2.2.5.2 Preparation of Thin Sections | |
| | 2 | 2.2.5.3 Results of the Petrographic Analysis | 32 |
| | 2 | 2.2.5.4 Photographic Documentation | 32 |
| | 2 | 2.2.5.5 Final Remarks | 33 |
| | 2.3 M | Mixing Procedure | 38 |
| | 2.4 H | Freshly-Mixed Concrete Tests | 40 |
| | 2.4. | 1 Temperature | 40 |
| | 2.4. | 2 Slump | 40 |
| | 2.4. | 3 Air Content and Unit Weight | 41 |
| | 2.5 0 | Cured-Concrete Tests | 43 |
| | 2.5. | 1 Compressive Strength | 43 |
| | 2.5. | 2 ASR Expansion | 46 |
| | 2.6 I | Doping the Cement with Alkali | 49 |
| | 2.6. | 1 Reason | 49 |
| | 2.6. | 2 Procedure | 49 |
| | 2.7 M | Mitigation of ASR by Lithium Nitrate | 50 |
| | 2.7. | 1 Control Concrete | 50 |
| | 2.7. | 2 Procedure | 50 |
| 3 | Mix | x Design Objectives | 51 |
| 4 | Cor | ncrete Mix Designs | 52 |
| | 4.1 (| Concrete Mix Design 1 | 52 |
| | 4.1. | 1 Test Results | 53 |
| | | | |

Page 3 | 93

| | 4 | .1.2 Discussion | 54 |
|---|-----|-----------------------------------|------|
| | 4.2 | Mix Design 2, Reactive | 55 |
| | 4 | .2.1 Test Results | 55 |
| | 4 | .2.2 Discussion | 57 |
| | 4.3 | Mix Design 2, Nonreactive | 58 |
| | 4 | .3.1 Test Results | 58 |
| | 4 | .3.2 Discussion | 60 |
| | 4.4 | Mix Design 3, Reactive | 60 |
| | 4 | .4.1 Test Results | 60 |
| | 4 | .4.2 Discussion | 62 |
| | 4.5 | Mix Design 4, Reactive | 63 |
| | 4 | .5.1 Test Results | 63 |
| | 4 | .5.2 Discussion | 65 |
| | 4.6 | Mix Design 5, Reactive | 65 |
| | 4 | .6.1 Test Results | 66 |
| | 4.7 | Temperature Effects | 68 |
| 5 | S | pecimens | . 70 |
| | 5 1 | From Dustations to Madal | 70 |
| | 5.1 | From Prototype to Model | |
| | 5.2 | Reinforcement | 73 |
| | 5 | .2.1 Reinforcement Construction | 74 |
| | 5.3 | Formwork | 78 |
| | 5.4 | Aggregate | 79 |
| | 5.5 | Casting Plan | 80 |
| | | | |
| 6 | C | Casting Results | . 82 |
| | 6.1 | Final Mixes | 82 |
| | 6.2 | 7 and 28 Day Compressive Strength | 84 |
| | 6 | 0.2.1 Concrete strengths | 84 |
| | 6 | .2.2 Stress-Strain Curves | 87 |
| 7 | А | Acknowledgments | . 92 |
| | 71 | Fall Line Testing & Inspection | 92 |
| | 7.1 | | |
| | 7.2 | Whitewater Building Materials | 93 |
| | 7.3 | Brady Trucking | 93 |
| | 7.4 | Holcim | 93 |
| | 7.5 | Grace Concrete Products | 93 |
| | | | |

| Figure 1 - Whitewater Building Materials, Whitewater, Colorado | 8 |
|--|----|
| Figure 2 - 3/4" Gravel storage bin at Whitewater Building Materials | 9 |
| Figure 3 - Grand Junction Ready Mix, Grand Junction, Colorado | 9 |
| Figure 4 - 3/8" Gravel storage bin at Grand Junction Ready Mix | 9 |
| Figure 5 - Aggregate crusher in use at University of Colorado materials laboratory | 11 |
| Figure 6 - Mechanical shaker with sieves at Fall Line laboratory | 11 |
| Figure 7 - The contents of each sieve were separated into steel bowls | 11 |
| Figure 8 - Each grade was washed three to four times in tap water and decanted | 12 |
| Figure 9 – Dana Schwartz of Fall Line placing washed material in drying oven | 12 |
| Figure 10 - Balance used to weigh dried aggregate | 12 |
| Figure 11 - Oiled molds | 13 |
| Figure 12 - Assembled molds ready for mortar | 13 |
| Figure 13 – Mixer with measured cement, water, and aggregate | 14 |
| Figure 14 - Time was kept with a stopwatch during mixing | 14 |
| Figure 15 - Tamping mortar into molds | 15 |
| Figure 16 - Mortar bars placed in curing room and covered with plastic | 15 |
| Figure 17 – This mortar bar broke during extraction | 16 |
| Figure 18 - Initial readings were taken before bars were placed in water bath | 16 |
| Figure 19 - After zero readings, bars were placed in 1N aqueous NaOH | 17 |
| Figure 20- Summary of 16-day ASTM C1567 results | 18 |
| Figure 21 - Mortar bar expansion vs. time. Whitewater sand | 19 |
| Figure 22 - Mortar bar expansion vs. time. Whitewater 3/8" aggregate | 19 |
| Figure 23 - Mortar bar expansion vs. time. Whitewater 3/4" aggregate | 20 |
| Figure 24 - Mortar bar expansion vs. time. Grand Junction Ready Mix sand | 20 |
| Figure 25 - Mortar bar expansion vs. time. Grand Junction Ready Mix 3/8" aggregate | 21 |
| Figure 26 - Mortar bar expansion vs. time, Grand Junction 3/4" aggregate | 21 |
| Figure 27 Mill report for Holcim cement batch 1 | 23 |
| Figure 28 Mill report from Holcim cement batch 2 | 24 |
| Figure 29 - Soaked coarse aggregate is spread on a towel for drying to saturated surface-dry condition | 25 |
| Figure 30 – Close up of coarse aggregate at saturated surface-dry condition | 25 |
| Figure 31 - Coarse aggregate at SSD is weighed | 26 |
| Figure 32 - Aggregate is immersed and weighed a second time. This apparatus allows the same scale to be | |
| used for both weighings | 26 |
| Figure 33 - Filling cone mold to check surface moisture of fine aggregate | 27 |
| Figure 34 - Saturated surface-dry conditions are reached when sample slumps slightly when cone mold is | |
| removed. This sample does not slump and is still too wet. | 27 |
| Figure 35 - A 500g sample of fine aggregate at SSD is weighed | 27 |
| Figure 36 - Adding fine aggregate to volumetric flask. | 28 |
| Figure 37 - The volumetric flask is rotated to remove trapped air | 28 |
| Figure 38 – Coarse aggregate is shoveled into the measure in three lifts. | 29 |
| Figure 39 - Each lift is rodded 25 times | 29 |
| Figure 40- Washing aggregate in preparation for sieve analysis | 30 |
| Figure 41 Sieve stack ready for coarse aggregate analysis | 30 |
| Figure 42 - Cleaning a screen during coarse aggregate sieve analysis | 30 |
| Figure 43 - Concrete sample. Fluorescent light. Magnification: x12.5 | 33 |
| Figure 44 - Concrete sample - the same area as in Figure 43. Plane polarized light. Magnification: x12.5 | 34 |
| Figure 45 - Concrete sample – the same area as in Figure 43 and Figure 44. Cross polarized light. | |
| Magnification: x12.5. | 34 |

Page 5 | 93

| Figure 46 - | Mortar-bar with crushed coarse aggregate: Fluorescent light. Magnification: x25 | 35 |
|-------------|---|----|
| Figure 47 - | Mortar-bar with crushed coarse aggregate - the same area as in Figure 46. Plane polarized light | t. |
| Magn | ification: x25 | 35 |
| Figure 48 - | Mortar-bar with crushed coarse aggregate – same area as in Figure 46 and Figure 47. Cross | |
| polari | zed light. Magnification: x25 | 36 |
| Figure 49 - | Mortar-bar with fine aggregate. Fluorescent light. Magnification: x25 | 36 |
| Figure 50 - | Mortar-bar with fine aggregate – the same area as in Figure 49. Plane polarized light. | |
| Magn | ification: x25 | 37 |
| Figure 51 - | Mortar-bar with fine aggregate – the same area as in Figure 49 and Figure 50. Cross polarized | |
| light. | Magnification: x25 | 37 |
| Figure 52 - | Buttering the mixer | 38 |
| Figure 53 - | Discarding butter | 38 |
| Figure 54 - | Charging the mixer | 39 |
| Figure 55 - | Mixer is capped to prevent moisture loss during rest period | 39 |
| Figure 56 - | Discharging freshly-mixed concrete | 39 |
| Figure 57 - | Placing temperature probe | 40 |
| Figure 58 - | - The slump mold is filled in three layers, each tamped 25 times | 40 |
| Figure 59 - | The slump mold is lifted in one smooth motion | 41 |
| Figure 60 - | Measuring slump | 41 |
| Figure 61 - | - The air meter bowl is filled in two layers, each rodded 25 times | 42 |
| Figure 62 - | After filling, the air meter bowl is struck off. Note the mallet used for tapping the bowl after each | ch |
| layer | is rodded | 42 |
| Figure 63 - | The top portion of the air meter is installed | 42 |
| Figure 64 - | A small quantity of water and isopropyl alcohol is added just until it begins to run out the | |
| weepl | 10le | 43 |
| Figure 65 - | Filling compression test cylinder molds | 43 |
| Figure 66 - | Striking off cylinder molds | 44 |
| Figure 67 - | Cylinders were allowed to rest 30 minutes before capping. Notice minimal bleeding | 44 |
| Figure 68 - | Measuring cylinder diameter | 45 |
| Figure 69 - | Installing sulfur mortar end caps | 45 |
| Figure 70 - | Compression test apparatus | 45 |
| Figure 71 - | Fractured cylinder after testing | 46 |
| Figure 72 - | 4-inch square prism molds ready for casting | 47 |
| Figure 73 - | 4 inch prisms are placed in fog room to cure 24 hours after casting | 47 |
| Figure 74 - | Demolding a concrete prism. | 48 |
| Figure 75 - | Concrete prism in container with 1N NaOH. | 48 |
| Figure 76 - | - Taking length comparometer readings of a concrete prism | 48 |
| Figure 77 - | ASR expansion of Mix 1 | 54 |
| Figure 78 - | Elongation of Mix 2 Reactive, non-accelerated conditions | 57 |
| Figure 79 - | Elongation of Mix 2 Reactive, accelerated conditions | 57 |
| Figure 80 - | Elongation of Mix 2 Nonreactive, non-accelerated conditions | 59 |
| Figure 81 - | Elongation of Mix 3, Reactive, under nonaccelerated conditions | 62 |
| Figure 82-l | Elongation of Mix 3, Reactive under accelerated conditions | 62 |
| Figure 83 - | Elongation of Mix 4, Reactive, under nonaccelerated conditions. Note the dip between 50 days | |
| and 7 | 0 days. During this time period only one of three Hydrofogger units were operational | 64 |
| Figure 84 - | Elongation of Mix 4, Reactive under accelerated conditions | 65 |
| Figure 85 - | Elongation of Mix 4, Reactive, under nonaccelerated conditions | 67 |
| Figure 86 - | Elongation of Mix 5, Reactive under accelerated conditions | 67 |

Page 6 | 93

| Figure 87 - Curve fitting data from concrete mix 5 (80C, 1M NaOH) to Larive's kinetic model |
|---|
| Figure 88 - Expansion vs time for 80C (blue line) and 38C (red line) |
| Figure 89 - Prototype system (right) with model system (left)71 |
| Figure 90 - Model system showing eight experimental specimens taken just above grade level |
| Figure 91 - Specimens are rotated 180 degrees about azimuthal direction71 |
| Figure 92 - Rebar in model system is oriented in the axial (red) and azimuthal (blue) directions |
| Figure 93 Specimen internal reinforcement and applied forces72 |
| Figure 94 - Shear specimen showing concrete and end plates. Note scoring line coincides with shear plane73 |
| Figure 95 - Shear specimen with concrete hidden. Red bars correspond to axial reinforcement and resist |
| shear by dowel action. Blue bars correspond to the azimuthal reinforcement and are not engaged in |
| shear73 |
| Figure 96 - A threaded 'DoughNUT' connection manufactured by BarSplice Products of Dayton, OH. This is |
| one of the options considered for terminating rebar. Unfortunately, it is too large to fit in the narrow |
| shear sample74 |
| Figure 97 - Cutting wooden blocks for the welding jig |
| Figure 98 - Wooden blocks ready for installation in the jig75 |
| Figure 99 - Joining wooden blocks to jig base with wood glue and steel brad nails76 |
| Figure 100 - Cutting reinforcing bar76 |
| Figure 101 - Rebar layout and welding using the wooden jig77 |
| Figure 102 - Rebar cages ready to be welded to sample end plates77 |
| Figure 103 - MIG-welded joint between #6 rebar and sample end plates |
| Figure 104 – Example shear specimen formwork78 |
| Figure 105 - Assembled and painted formwork. Seventeen forms were built in total |
| Figure 106 - Offloading aggregate |
| Figure 107 - Mixing fine aggregate and forming into pile |
| Figure 108 - The three-cubic yard mixer at Fall Line |
| Figure 109 - Close view of mobile batch plant at Fall Line. Note load cells for weighing hopper contents 82 |
| Figure 110 - Measuring Cylinders for Compression Test85 |
| Figure 111 - Concrete cylinder with rubber caps85 |
| Figure 112 - Cylinder installed in MTS machine |
| Figure 113 - Broken Cylinder in MTS Machine86 |
| Figure 114 - Fall Line Testing & Inspection, LLC., Denver, Colroado |
| |

1 OVERVIEW

Concrete mix design and evaluation is complete. We have produced and tested five experimental designs and subjected each to a battery of tests evaluating strength, workability, and reactivity (among other properties). The latest design, Mix 5 meets or exceeds all design objectives.

We have received the 10 tons of aggregate required to cast experimental samples. Concrete formwork and reinforcement for all 16 shear samples are complete. Construction of cubical block forms and fog room renovation are ongoing and expected to be complete within one week of this writing.

Casting is scheduled for April 25th and 27th, to be followed immediately by transportation to the University structures laboratory for storage in the fog room.

1.1 IDENTIFICATION OF REACTIVE AGGREGATES

Two potential sources of reactive aggregates were brought to the project's attention:

- 1. Whitewater Building Materials
- 2. Grand Junction Ready Mix

Both suppliers obtain aggregate from quarries along the Gunnison River in western Colorado, near Grand Junction.

On January 21, 2015, both sites were visited, and 5-gallon bucket of each aggregate type (3/4, 3/8 and sand) were brought for initial testing. Whitewater samples were obtained directly from storage bins.



Figure 1 - Whitewater Building Materials, Whitewater, Colorado



Figure 2 - 3/4" Gravel storage bin at Whitewater Building Materials

Grand Junction ready mix had prepared samples of sand and 3/4" gravel, while the 3/8" gravel was pulled from a storage bin.



Figure 3 - Grand Junction Ready Mix, Grand Junction, Colorado



Figure 4 - 3/8" Gravel storage bin at Grand Junction Ready Mix

ASTM describes two standard test methods to assess aggregate reactivity: C1293 and C1567. The former lasting about 52 weeks, the second was selected as it tests the potential for aggregate reactivity in only 2 weeks.

Page 9 | 93

1.2 Overview of ASTM C1567 Mortar Bar Test

ASTM 1567 identifies the presence of ASR by measuring the elongation of three 1" X 1" X 10" mortar bars after storage in a solution of 1N aqueous sodium hydroxide at 80°C for fourteen days. Expansion beyond 0.1% is considered sufficient to identify problematic expansion due to ASR.

The mortar mix must be prepared according to ASTM C305, using a precise mix of gradations as described in Table 1.

| Sieve | e Size | Mass % | Mass (g) |
|-----------------|------------------|--------|----------|
| Passing | Retained on | | |
| 4.75 mm (No. 4) | 2.36 mm (No. 8) | 10 | 99.0 |
| 2.36 mm (No. 8) | 1.18mm (No. 16) | 25 | 247.5 |
| 1.18mm (No. 16) | 600 µm (No. 30) | 25 | 247.5 |
| 600 μm (No. 30) | 300 µm (No. 50) | 25 | 247.5 |
| 300 µm (No. 50) | 150 μm (No. 100) | 15 | 148.5 |

Table 1 - Proportions of aggregate by size and mass required for 3 mortar bars

Cement and water are proportioned according to the relative density of the aggregate.

| Cement (g) | Water (mL) |
|------------|------------|
| 440.0 g | 207 |
| | |

Table 2 - Cement and water required for 3 mortar bars.

Bars are cured for 24 ± 2 hours in a fog room with relative humidity of at least 50% and temperature maintained at 23 ± 2 °C. An initial length measurement is taken after curing is completed. Bars are then submerged in water at 23 °C and the bath is transferred to an oven at 80.0 ± 2 °C for a further 24 ± 2 hours.

The bath is then removed from the oven and a zero length measurement is taken. Each bar is removed from the bath, dried, and measured within 15 ± 5 seconds. Bars are then transferred to aqueous solution of 1N NaOH at 80.0 ± 2 °C and replaced in the oven within at most ten minutes from the time the water bath was removed. Samples are stored in this manner for a further 14 days. During this time, at least three interim measurements are taken, each at the same time of day and following a procedure similar to that of the zero measurement. The final measurement is taken 14 days after the zero reading (16 days after casting).

The difference between the zero comparometer reading and the 14-day reading is calculated to within 0.001%. Expansions more than 0.10% indicate potentially deleterious expansion.

1.3 NARRATIVE OF AGGREGATE TESTING

The distribution of grades required by ASTM 1567 was obtained by sieving sand samples directly. However, coarse aggregates were crushed using a machine in the University materials laboratory. After crushing, all samples were sieved using a mechanical shaker and calibrated sieves at the Fall Line laboratory. Even with the mechanical shaker, it is easy to overload the

sieves. Each sieve-shaking cycle takes 7 minutes and 30 seconds, and 10-12 cycles were required to obtain the required mass of each grade.



Figure 5 - Aggregate crusher in use at University of Colorado materials laboratory



Figure 6 - Mechanical shaker with sieves at Fall Line laboratory

After each sieve-shaking cycle, the contents of each sieve were emptied into steel bowls. Each was then washed in water and carefully decanted. At least three separate washes were used for coarser grades, while finer grades typically required a fourth wash.



Figure 7 - The contents of each sieve were separated into steel bowls.

Page 11 | 93



Figure 8 - Each grade was washed three to four times in tap water and decanted.

Washed material was then transferred into drying trays and allowed to dry in an oven at 230° F overnight.



Figure 9 – Dana Schwartz of Fall Line placing washed material in drying oven.

Dried aggregate and cement was then weighed using a laboratory balance. Water was measured with a graduated cylinder.



Figure 10 - Balance used to weigh dried aggregate

Steel molds were prepared by cleaning and oiling prior to assembly. Two release agents were tried. One was a silicon-based spray lubricant that performed poorly. A generous coat of '3 In 1' oil proved more effective. Slight corrosion was noticed on the molds (visible Figure 11), although they remained smooth to the touch. While we initially thought that such minor corrosion would have a negligible effect, it made the cured mortar bars quite difficult to extract. This was corrected by mechanically polishing the bars using a hand drill with cotton wheel and abrasive compound.



Figure 11 - Oiled molds



Figure 12 - Assembled molds ready for mortar

Mortar was mixed according to ASTM C305. Water was placed in mixer bowl and entire quantity of cement was added. Water and cement were then mixed at low speed (140 ± 5 rpm) for 30 seconds. Graded aggregate was then added slowly the next 30 seconds. The mixer was then switched to medium speed (285 ± 10 rpm) for 30 seconds. Then the mixer was switched off and the mortar scraped down from the paddle and bowl edges. After sitting 90 seconds, the mixer was switched back on at medium speed and mixing continued for a further 60 seconds.



Figure 13 – Mixer with measured cement, water, and aggregate



Figure 14 - Time was kept with a stopwatch during mixing

After mixing was complete, one person half-filled each mold with mortar while a second person tamped. Special care was required to ensure no void remained near the measuring studs visible in Figure 12. The molds were then filled with a second layer of mortar and tamped again. Finally, each mold was struck using a wetted magnesium trowel. ASTM calls for this process to be complete within two minutes and fifteen seconds from completion of mixing. This proved difficult to achieve, even with the two-person method described. Filling and tamping all three molds took us between two minutes, thirty seconds and two minutes, forty-five seconds consistently.



Figure 15 - Tamping mortar into molds



Figure 16 - Mortar bars placed in curing room and covered with plastic

Molds were then placed in the Fall Line curing room and covered with a plastic sheet. After curing for 24 ± 2 hours, the mortar bars were extracted from the molds. As mentioned previously, this proved to be a difficult task. Any corrosion on the mold caused the mortar bars to stick, even though the molds felt smooth to the touch. One bar molded from Grand Junction Ready Mix sand and two bars of Grand Junction Ready Mix 3/8" gravel broke, requiring that new sets of three bars be molded for each of these samples.



Figure 17 – This mortar bar broke during extraction

Once extracted from their molds, an initial reading of each bar was taken using a length comparometer. The comparometer was zeroed before the first measurement and after between each group of three bars. Bars were then placed in a water bath at 23°C and the bath was placed in an oven at 80 °C for another 24 ± 2 hours. The following day, a zero reading was taken. Each bar was removed from the water bath, quickly dried with a towel (ensuring no water remained on the measuring studs) and measured on the length comparometer. It was not difficult to complete each measurement well within the 15 \pm 5 seconds permitted by ASTM C1567.



Figure 18 - Initial readings were taken before bars were placed in water bath.

After zero readings were taken, bars were placed in a solution of 1N aqueous sodium hydroxide and returned to the oven. This solution was prepared in advance, stored in a sealed plastic bin, and kept in the oven until the zero readings were complete.



Figure 19 - After zero readings, bars were placed in 1N aqueous NaOH.

Over the next 14 days, four measurements were taken following a similar procedure to the zero reading. The determination of ASR reactivity was obtained by determining the average expansion percentage 14 days after the zero reading (16 days after casting). Additional measurements were taken roughly once per week after the 14-day mark to evaluate continued expansion over a longer term.

1.4 TEST RESULTS

All samples expanded well beyond the threshold of 0.10% at 16 days after casting established by ASTM as indicative of ASR. Whitewater 3/4" gravel exhibited the greatest expansion at 1.02%, over ten times the threshold value. Whitewater 3/8" gravel proved least expansive at 0.68%. The expansion of Grand Junction Ready-Mix samples fell between these extremes.

| Pe | rcent expansion, 16 days | after casting |
|---------|----------------------------------|-----------------------------|
| | Whitewater Building Materials | Grand Junction Ready Mix |
| Sand | 0.69 | 0.98 |
| 3/8" | 0.68 | 0.74 |
| 3/4" | 1.02 | 0.77 |
| Average | 0.80 | 0.83 |

Table 3 – Summary of ASTM C1567 initial test results



Figure 20- Summary of 16-day ASTM C1567 results

All samples continued to expand after the 16-day measurement used to establish reactivity. Expansion of each sample vs. time is plotted in Figure 21 - Figure 26 below. Note that expansion during the first ten days is rapid for all samples, followed by a long period of expansion at a reduced rate. Expansion typically begins slowing around 100 days, and most samples reach their maximum elongation after approximately 150 days.







Figure 22 - Mortar bar expansion vs. time, Whitewater 3/8" aggregate



Figure 23 - Mortar bar expansion vs. time, Whitewater 3/4" aggregate



Figure 24 - Mortar bar expansion vs. time, Grand Junction Ready Mix sand



Figure 25 - Mortar bar expansion vs. time, Grand Junction Ready Mix 3/8" aggregate



Figure 26 - Mortar bar expansion vs. time, Grand Junction 3/4" aggregate

1.5 CONCLUSION

Considering the reactivity data presented above, it is evident that both Whitewater and Grand Junction Ready Mix products produce significant expansion. Whitewater sand and 3/4" were

Page 21 | 93

selected for production of experimental concrete due both to the reactivity of the aggregates and the generous support Whitewater Building Materials staff.

2 CONCRETE TESTING PROGRAM

Evaluation of candidate concrete mixes requires the adoption of a test regimen that adequately characterizes the mix in a reasonable period of time and allows for the mix to be reproduced despite small variances in aggregate or cement. Preference was given to standardized test procedures that can be easily duplicated.

Each shipment of Whitewater aggregate was subjected to the tests in Table 4 before use in candidate concrete mixes. All tests except C566 were conducted as soon as practical after receiving aggregate. Moisture was not measured until immediately before casting.

| Test | Standard |
|-----------------------------------|-----------|
| Coarse aggregate relative density | ASTM C127 |
| Fine aggregate relative density | ASTM C128 |
| Coarse aggregate bulk density | ASTM C29 |
| Fineness modulus / gradation | ASTM C136 |
| Moisture content | ASTM C566 |

Table 4 - Aggregate testing program

Candidate concrete mixes were subjected to the tests in Table 5 below. The most important of these are C173, C39, and the ASR test. The remainder of the tests are taken the sake of completeness and to monitor against any unintended environmental effects.

| Test | Standard |
|----------------------|------------|
| Slump | ASTM C173 |
| Unit Weight | ASTM C138 |
| Air Content | ASTM C231 |
| Temperature | ASTM C1064 |
| Compressive Strength | ASTM C39 |
| ASR Expansion | N/A |

Table 5 – Concrete mix testing program

Each of these tests procedures is summarized briefly in the following sections.

2.1 MATERIAL PROPERTIES

2.1.1 <u>Cement Mill Reports</u>

Two batches of cements were donated by Holcim (a total of 5,000 lbs), and the mill reports are shown in Figure 27 and Figure 28. We note that the first batch (2,500 lbs) had an equivalent alkaline content of 0.88%, and the second 0.91%. Thus proper adjustments was made during batching.

| Material: | Po | ortland | Cement | Г | est Period: | 06-J | un-201 |
|--|----------------------------------|---------------------------------|---|---|-------------------------------|--------------------|--|
| Туре: | - | I(MH) | | | To: | 06-J | un-201 |
| | | | Certifi | cation | | | |
| | This Holcim cemen | t meets th | e specificatio | ins of ASTM C150 for | ^r Type I-II(MH) ce | ment. | |
| | | | General Ir | nformation | | | |
| Supplier: | Holcim (US) Inc. | | | Source Location: | Hagerstown Pla | nt | |
| Address: | 1260 Security Road | 740 | | | 1260 Security R | oad | |
| Talaakaaa | (240) 452 4008 | 142 | | Orabash | Hagerstown, IVIL | J 21742 | |
| Telephone: | (240) 452 4908 | | | Contact: | AnnaLisa Homa | n | |
| Date Issued: | 31-Aug-2015 The following | information | ie haeed on a | warana tast data durin | in the test period | | |
| | The data is typi | ical of cem | ent shipped by | Holcim; individual sh | ipments may vary | | |
| | Test | s Data c | on ASTM S | Standard Require | ements | | |
| | Chemical | | | | Physical | | |
| Item | | Limit ^A | Result | ltem | 1 | Limit ^A | Result |
| SIO ₂ (%) | | - | 19.8 | Air Content (%) | | 12 max | 9 |
| Fe ₂ O ₃ (%) | | 6.0 max | 4.2 | Biaine Fineness (m²/k | g) | 200-430 | 387 |
| CaO (%) | | - | 62.0 | | | | |
| MgO (%) | | 6.0 max | 4.1 | Autoclave Expansion | (%) (C151) | 0.80 max | 0.06 |
| SU ₃ (%) | | 3.0 max ⁸ 3.0 max | 3.6 | Compressive Strength | MPa (psi): | | |
| Insoluble Residue (%) | | 0.75 max | 0.42 | 3 davs | | 10.0 (1450) min | 28.1 (4070 |
| CO ₂ (%) | | - | 1.2 | 7 days | | 17.0 (2470) min | 33.3 (4830 |
| Limestone (%) | 2/1 | 5.0 max | 3.1 | 28 days | | 28.0 (4060) min | 40.3 (5850 |
| Inorganic Processing | Mition (%) | 70 min 50 max | 86 0.0 | initial vicat (minutes) | | 45-375 | 110 |
| Potential Phase Comp | ositions ^c : | 0.0 max | 0.0 | Mortar Bar Expansion | (%) (C1038) | | |
| C ₃ S (%) | | - | 57 | | | | |
| $C_2S(\%)$ | | - | 13 | Heat of Hydration: kJ/l | kg (cal/g)D | - | 336 (80) |
| C ₃ A (%) | | a max | 7 | / Days (for informa | auonai purposes) | | |
| C ₃ S + 4.75C ₃ A (%) | | 100 max | 90.3 | | | | |
| | Test | ts Data d | on ASTM (| Optional Require | ements | | |
| Item | Gheimear | Limit ^A | Result | Item | riiysiodi | Limit ^A | Result |
| Equivalent Alkalies (% |) | - | 0.91 | False Set (%) | | 50 min | 63 |
| Notes | | | | | | | |
| A Dashes in the limit / res | ult columns mean Not Applica | able. | | | | | |
| ^B It is permissible to exceed | ed the specification limit prov | ided that AST | M C1038 Mortar Ba | ar Expansion does not exceed | 0.020 % at 14 days. | | |
| Adjusted per Annex A1. | 6 of ASTM C150 and AASHT | O M85. | alian anti- Arrol - | of Lines of Liverstee in the | an opping of the America | naun Chalde H | |
| This data may have been | reported on previous mill cer | idea for inform rtificates. | auon only. Analysi | or near or hydration has be | en carried out by CTLG | гоцр, экокіе, IL. | |
| | | | | | | | |
| N=20 0.40% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na2O 0.16% K2O 1.14% | | | | | | | |
| Na20 0.16% K20 1.14% | а | | | | | | |
| Na20 0.16% K20 1.14% Additional Dat | a nic Processing Addi | ition Data | | Base | Cement Phase 0 | Composition | |
| Na20 0.16% K20 1.14% Additional Dat Inorga | a nic Processing Addi item | ition Data | Result ^a | Base | Cement Phase C | Composition | Result |
| Na20 0.16% K20 1.14% Additional Dat Inorga | a nic Processing Add Item | ition Data | Result ⁴ | Base C ₂ S (%) | Cement Phase (Item | Composition | Result 59 |
| Na20 0.16% K20 1.14% Additional Dat Inorga Type Amount (%) SIO ₂ (%) | a nic Processing Add Item | ition Data | Result ^A | Base C ₃ S (%) C ₂ S (%) C ₂ A (%) | Cement Phase 0 Item | Composition | Result 59 13 7 |
| Na20 0.16% K20 1.14% Additional Dat Inorga Type Amount (%) SiO ₂ (%) Al ₂ O ₃ (%) | a nic Processing Addi Item | ition Data | Result ⁴ - - - | Вазе С ₃ S (%) С ₂ S (%) С ₃ A (%) С ₄ AF (%) | Cement Phase (Item | Composition | Result 59 13 7 7 |
| Na20 0.16% K20 1.14% Additional Dat Inorga Type Amount (%) SIO ₂ (%) Al ₂ O ₃ (%) Fe ₂ O ₃ (%) | a nic Processing Add Item | ition Data | Result ^A - - - - - | Base C ₂ S (%) C ₂ S (%) C ₃ A (%) C ₄ AF (%) | Cement Phase (Item | Composition | Result 59 13 7 7 7 |
| Na20 0.16% K20 1.14% Additional Dat Inorga Type Amount (%) SIO ₂ (%) Fe ₂ O ₃ (%) Fe ₂ O ₃ (%) CaO (%) | a nic Processing Addi Item | ition Data | Result ^a - - - - - - | Base C ₅ S (%) C ₂ S (%) C ₂ A (%) C ₆ AF (%) | Cement Phase 0 Item | Composition | Result 59 13 7 7 |

Figure 27 Mill report for Holcim cement batch 1

| ^C Holcir | n NSF | 2 | | Materi | al Certifi | cation F | Repor |
|--|---|---|---|--|--|--|------------------------------|
| Material: | : P | ortland | Cement | | Test Period: | SA BA | |
| туре. | | | 0 | | 10. | | |
| | | | Certif | cation | | | |
| | This Holcim ceme | nt meets th | e specificatio | ons of ASTM C150 fo | or Type I-II(MH) c | ement. | |
| | | | General Ir | nformation | | | |
| Supplier: | Holcim (US) Inc. | | | Source Location: | Hagerstown Pla | ant | |
| Address: | 1260 Security Roa | d | | | 1260 Security F | Road | |
| | Hagerstown, MD 2 | 21742 | | | Hagerstown, M | D 21742 | |
| Telephone: | (240) 452 4908 | | | Contact: | AnnaLisa Homa | an | |
| Date Issued: | 01-Apr-2016 | | | | | | |
| | The following | information | is based on a | average test data duri | ng the test period | | |
| | | to Doto (| | Standard Requir | ipments may var | y. | |
| | Chemical | is Dala C | IT AS TWIC | lanuaru Requii | Physical | | |
| Item | n onemical | Limit ^A | Result | lter | n | Limit ^A | Result |
| SiO ₂ (%) | · | - | 19.8 | Air Content (%) | | 12 max | 8 |
| Al ₂ O ₃ (%) | | 6.0 max | 4.3 | Blaine Fineness (m ² / | kg) | 260-430 | 408 |
| Fe₂O₃ (%) CaO (%) | | 6.0 max | 2.2 | | | | |
| MgO (%) | | - 6.0 max | 4.1 | Autoclave Expansion | (%) (C151) | 0.80 max | 0.11 |
| SO ₃ (%) | | 3.0 max ^B | 3.5 | Compressive Strengt | h MPa (psi): | | |
| Loss on ignition (%) Insoluble Residue (%) | 6 | 3.0 max 0.75 max | 1.7 | 3 days | | 10.0 (1450) min | 28.2 (4080) |
| CO ₂ (%) | | - | 0.8 | 7 days | | 17.0 (2470) min | 33.7 (4890) |
| Limestone (%) | 0/.) | 5.0 max | 2.2 | Initial Maat (minutes) | | 45 075 | 444 |
| Inorganic Processing / | Addition (%) | 5.0 max | 0.0 | initial vicat (minutes) | | 45-375 | 114 |
| Potential Phase Comp | positions ^C : | | 50 | Mortar Bar Expansion | n (%) (C1038) | | 0.008 |
| C ₃ S (%) C ₂ S (%) | | | 58 12 | Heat of Hydration: kJ | /kg (cal/g)D | _ | 350 (84) |
| C ₃ A (%) | | 8 max | 8 | 7 Days (for inform | national purposes) | | |
| C ₄ AF (%) C ₂ S + 4 75C ₂ A (%) | | - | 7 | | | | |
| 030 - 4.70034 (78) | Ter | sts Data (| on ASTM (| Dotional Requir | ements | | |
| | Chemical | no Build I | | | Physical | | |
| | | Limit ^A | Result | ltem | | Limit ^A | Result |
| Item | 1 | | | | | | |
| Item Equivalent Alkalies (% | <u>າ</u> ຈ) | - | 0.88 | | | | |
| Item Equivalent Alkalies (% Notes | ו א) | | 0.88 | Annal | iaa Haman | | |
| Item Equivalent Alkalies (% Notes * Dashes in the limit / resu |) j) ult columns mean Not Appl | - cable. | 0.88 | AnnaL | isa Homan | 9 Ourslite | |
| Item Equivalent Alkalies (% Notes ³ Dashes in the limit / resu ³ It is permissible to exces |) ult columns mean Not Appl ad the specification limit pro | - cable. wided that ASTI | 0.88 M C1038 Mortar Ba | AnnaL ar Expansion Manage | isa Homan er, Process | & Quality | |
| Item Equivalent Alkalies (% Notes ^A Dashes in the limit / resu ^a It is permissible to excee ^A Adjusted per Annex A1 f ^a Test result reconcents m |) i) uit columns mean Not Appl ad the specification limit pr 6 of ASTM C150 and AASH vost recent value and is not | - cable. ovided that ASTI ITO M85. | 0.88 M C1038 Mortar Ba | ar Expansion AnnaLi Manage Holcim | isa Homan er, Process (US) Inc. | & Quality | |
| Item Equivalent Alkalies (% Notes ⁴ Dashes in the limit / resu ⁸ It is permissible to excee ² Adjuated per Annex A1 f ² Test result represents m This date may have been | ult columns mean Not Appl ed the specification limit pro 5 of ASTM C150 and AASH vost recent value and is pro reported on previous mill c | - ovided that AST ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | AnnaL ar Expansion Manage s of Heat of H Hagers | isa Homan er, Process (US) Inc. town Plant | & Quality | |
| Item Equivalent Alkalies (% Notes ^A Dashes in the limit / ress. ¹ Adjusted per Annex A1 (² Test result represents m This data may have been | ult columns mean Not Appl ad the specification limit pro 6 of ASTM C150 and AASH nost recent value and is pro reported on previous mill c | - ovided that ASTI iTO M85. wided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysia | AnnaLi ar Expansion s of Heat of H Hagers 1260 S | isa Homan er, Process (US) Inc. town Plant ecurity Road | & Quality | |
| Item Equivalent Alkalies (% Notes ^A Dashes in the limit / ress. ¹ A its permisable to excee ² Adjusted per Annex A1 (² Test result represents m This data may have been | ult columns mean Not Appl ad the specification limit pr 6 of ASTM C150 and AASH nost recent value and is pro reported on previous mill c | - ovided that ASTI ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | AnnaLi ar Expansion s of Heat of Holcim Hagers 1260 S Hagers | isa Homan er, Process (US) Inc. town Plant ecurity Road | & Quality | |
| Item Equivalent Alkalies (% Notes ^A Dashes in the limit / ress. ^B his permissible to excee ^A adjusted per Annex A1.5 ¹ Test result represents in This data may have been Na2O 0.15 |) i) ult columns mean Not Appl ad the specification limit pr 6 of ASTM C150 and AASH reported on previous mill c | - svided that ASTI TO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Bation only. Analysi | AnnaLi ar Expansion s of Heat of Halders 1260 S Hagers | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 | & Quality 1 1742 | |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ² Test result represents m This data may have been Na2O 0.15 K2O 1.1 |) i) uit columns mean Not Appl ed the specification limit pr 6 of ASTM C150 and AASH reported on previous mill c | - ovided that ASTI TO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | AnnaLi Manage s of Heat of Hagers 1260 S Hagers Phone: | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 | & Quality 1742 2 4908 | |
| Item Equivalent Alkalies (% Notes Dashes in the limit / ress. ¹ its permissible to axcee Adjusted per Annex A1 f ² Test result represents m This data may have been Na2O 0.15 K2O 1.1 | b) wit columns mean Not Appl ad the specification limit pro 6 of ASTM C150 and AASH nost recent value and is pro- reported on previous mill c | - vided that ASTI tTO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | AnnaLi Manage s of Heat of s of Heat of Hagers 1260 S Hagers Phone: Fax: | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 | & Quality 1 1742 2 4908 9 6708 | |
| Item Equivalent Alkalies (% Notes Dashes in the limit / ress. ⁸ It is permissible to excee Adjusted per Annex A1.6 ⁹ Test result represents m This data may have been Na2O 0.15 K2O 1.1 | ult columns mean Not Appl ed the specification limit pr 6 of ASTM C150 and AASH osst recent value and is pro- reported on previous mill c | cable. svided that ASTI tTO M85. vided for inform. ertificates. | 0.88 VI C1038 Mortar Br | ar Expansion s of Heat of Hagers 1260 S Hagers Phone: Fax: annalis | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 ⁻¹ +1 240 45 +1 301 73 a.homan@h | & Quality 1742 2 4908 9 6708 1001cim.com | |
| Item Equivalent Alkalies (% Dashes in the limit / ress. ¹ its permissible to axcee Adjusted per Annex A1 f ² Test result represents m This data may have been Na2O 0.15 K2O 1.1 | ult columns mean Not Appl ad the specification limit pro 6 of ASTM C150 and AASH nost recent value and is pro reported on previous mill c | - vided that ASTI ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | ar Expansion s of Heat of S of Heat of Holcim Hagers 1260 S Hagers Phone: Fax: annalis WWW.ho | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h olcim.us | & Quality 1742 2 4908 9 6708 iolcim.com | |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ⁴ It is permissible to excee Adjusted per Annex A1 f ⁷ Test result represents m This data may have been Na2O 0.15 K2O 1.1 | ult columns mean Not Appl ad the specification limit pro 6 of ASTM C150 and AASH nost recent value and is pro reported on previous mill c | - vvided that ASTI tTO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Ba ation only. Analysi | AnnaLi Manage s of Heat of Holcim Hagers 1260 S Hagers Phone: Fax: <u>annalis</u> www.he | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 iolcim.com | |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ⁹ Adjusted per Annex A1 6 ¹ Test result represents in This data may have been Na2O 0.15 K2O 1.1 Additional Dat | ult columns mean Not Appl ad the specification limit pro 6 of ASTM C150 and AASH nost recent value and is pro reported on previous mill c | - vided that ASTI ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Bi ation only. Analysi | AnnaLi Manage s of Heat of H s of Heat of H Hagers 1260 S Hagers Phone: Fax: <u>annalis</u> www.hc | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 tolcim.com | |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ⁹ Adjusted per Annex A1 6 ¹ Test result represents in This data may have been Na2O 0.15 K2O 1.1 Additional Data Inorga | ult columns mean Not Appl ed the specification limit pro 6 of ASTM C150 and AASH rost recent value and is pro reported on previous mill c | - vided that ASTI ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Bi ation only. Analysi | AnnaLi AnnaLi Manage Holcim Hagers 1260 S Hagers Phone: Fax: annalis www.ho | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h olcim.us | & Quality 1742 2 4908 9 6708 tolcim.com | |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ² Adjusted per Annex A1 6 ² Test result represents in This data may have been Na2O 0.15 K2O 1.1 Additional Dat Inorga | a b) uit columns mean Not Appl ed the specification limit pr 6 of ASTM C150 and AASH nost recent value and is pro- reported on previous mill c reported on previous mill c and a processing Add Item | - vided that ASTI ITO M85. vided for inform ertificates. | 0.88 M C1038 Mortar Bi ation only. Analysi Mesult ^A | AnnaLi Manage s of Heat of H Base Base Base | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 tolcim.com | Result |
| Item Equivalent Alkalies (% Notes Dashes in the limit / ress. Adjusted per Annex A1 f Test result represents m This data may have been Na2O 0.15 K2O 1.1 Additional Date Inorga | a b) ult columns mean Not Appl ed the specification limit pn 6 of ASTM C150 and AASt nost recent value and is pro- reported on previous mill c reported on previous mill c is a nic Processing Add Item | - vided that ASTI TTO M85. vided for inform ertificates. | 0.88 M C1038 Mortar B. ation only. Analysi | AnnaLi Manage Holcim Hagers 1260 S Hagers Phone: Fax: annalis www.ho | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 iolcim.com | Result 60 12 |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ¹ is permissible to excet ² Test result represents m This data may have been Na2O 0.15 K2O 1.1 Additional Date Inorga Type Amount (%) SiO ₂ (%) | a) b) ult columns mean Not Appl ed the specification limit pn 6 of ASTM C150 and AAS+ nost recent value and is pro- reported on previous mill c b) c) | - avided that ASTI iTO M85. wided for informa ertificates. | 0.88 M C1038 Mortar B. ation only. Analysi Analysi Result ^A - | AnnaLi Manage Holcim Hagers 1260 S Hagers Phone: Fax: annalis www.ho C ₂ S (%) C ₂ S (%) C ₂ A (%) | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 iolcim.com | Result 60 12 8 |
| Item Equivalent Alkalies (% Notes ¹ Dashes in the limit / ress. ¹ its permissible to axcet ² Test result represents in This data may have been Na2O 0.15 K2O 1.1 Additional Data Inorga Type Amount (%) SiO ₂ (%) Al ₂ O ₃ (%) Fe ₂ O ₃ (%) Fe ₂ O ₃ (%) | tion tion tion tion tion tion tion tion | - ovided that ASTI iTO M85. wided for inform ertificates. | 0.88 M C1038 Mortar B. ation only. Analysi example Result ^A - - - | AnnaLi Manage Manage Holcim Hagers 1260 S Hagers Phone: Fax: annalis www.hc C ₂ S (%) C ₂ S (%) C ₂ A (%) C ₄ AF (%) | isa Homan er, Process (US) Inc. town Plant ecurity Road town, MD 2 +1 240 45 +1 301 73 a.homan@h blcim.us | & Quality 1742 2 4908 9 6708 iolcim.com | Result 60 12 8 7 |

Figure 28 Mill report from Holcim cement batch 2

2.2 Aggregate Tests

2.2.1 <u>Coarse Aggregate Relative Density</u>

Relative density (specific gravity) of coarse aggregate is readily evaluated following ASTM test procedure C127. A sample of washed coarse aggregate is soaked in water for 24 ± 4 hours. After soaking, the sample is spread onto a towel and rolled against the cloth until visible surface water film is removed. Once the sample has reached saturated surface-dry (SSD) conditions it is weighed on a laboratory scale. The sample is then immersed in 23° C water and weighed again. The sample is then dried overnight in an oven at 110° C and weighed a third time.



Figure 29 - Soaked coarse aggregate is spread on a towel for drying to saturated surface-dry condition.



Figure 30 – Close up of coarse aggregate at saturated surface-dry condition



Figure 31 - Coarse aggregate at SSD is weighed



Figure 32 - Aggregate is immersed and weighed a second time. This apparatus allows the same scale to be used for both weighings.

| The results of C127 | for the first s | ample shipment | of aggregate a | re as follows. |
|----------------------|------------------|-----------------------|----------------|----------------|
| 110 100 0100 01 0121 | 101 0110 11100 0 | winpite simplifierite | | |

| 2.604 |
|-------|
| 2.641 |
| 2.705 |
| 1.433 |
| |

Table 6 - Whitewater 3/4" (Coarse Aggregate) Specific Gravity

2.2.2 Fine Aggregate Relative Density

The relative density of fine aggregate is found by following ASTM C128. Similar to C127 described above, a sample of fine aggregate is first soaked for 24 ± 4 hours. It is then spread on a tray and allowed to dry at room temperature until it just reaches saturated surface-dry condition. Surface moisture is tested by filling a standard cone mold to overflowing and lightly tamping 25 times. The tamper is held 5 mm above the heaped aggregate surface and allowed to fall freely under the influence of gravity. SSD condition is reached when the molded aggregate slumps slightly when the cone mold is removed.



Figure 33 - Filling cone mold to check surface moisture of fine aggregate.



Figure 34 - Saturated surface-dry conditions are reached when sample slumps slightly when cone mold is removed. This sample does not slump and is still too wet.

A 500 mL volumetric flask is weighed empty and weighed again filled to the line with water at 23 °C. A portion of the water is removed and a weighed sample of 500 g of fine aggregate at SSD is added. The flask with aggregate is again filled to the line with water and gently swirled to remove all trapped air. Finally, the de-aerated flask with water and aggregate is weighed.



Figure 35 - A 500g sample of fine aggregate at SSD is weighed

Page 27 | 93



Figure 36 - Adding fine aggregate to volumetric flask.



Figure 37 - The volumetric flask is rotated to remove trapped air.

The results of ASTM C128 are summarized below.

| Oven Dry Bulk Specific Gravity | 2.583 |
|--------------------------------|-------|
| SSD Bulk Specific Gravity | 2.623 |
| Apparent Bulk Specific Gravity | 2.690 |
| Absorption (%) | 1.551 |

Table 7 - Whitewater Sand (Fine Aggregate) Specific Gravity

2.2.3 <u>Coarse Aggregate Bulk Density</u>

Bulk density of coarse aggregate is measured by following ASTM C29. A sample of coarse aggregate is shoveled into a 1/2 ft³ measure (a bucket-like steel container of known volume). The measure is filled in three lifts. Each lift is levelled by hand and rodded 25 times using a cylindrical steel tamping rod 5/8 inch in diameter and 24 inches long.



Figure 38 – Coarse aggregate is shoveled into the measure in three lifts.



Figure 39 - Each lift is rodded 25 times.

Results of ASTM C29 testing are summarized below.

| Bulk Specific Gravity | 2.641 |
|-----------------------|-------|
| Bulk Density (pcf) | 100.9 |
| Void (%) | 39% |
| | |

Table 8 - Whitewater 3/4" (Coarse Aggregate) Bulk Density

2.2.4 Sieve Analysis and Fineness Modulus

A sieve analysis as described in ASTM C136 permits determination of the fineness modulus as well as the grade classification of the aggregate. The procedure for both fine and coarse aggregate is similar. A sample of about 1500 g of aggregate is weighed and washed. After washing, it is oven-dried at 110 ± 5 °C before being weighed again. The sample is then divided into portions of about 300 g each and each portion is introduced separately to the sieve stack. Fine aggregate is separated using 3/8 inch, #4, #8, #16, #30, #50, #100, and #200 sieves. Coarse aggregate is separated using 1 inch, 3/4 inch, 1/2 inch, 3/8 inch and #4 sieves. The sieves are mechanically shaken for 7 minutes using a "Sally Mae" sieve shaker which vibrates, taps, and rotates the sieve stack automatically. The material retained on each sieve is removed and combined with the corresponding retained material from the other portion runs.



Figure 40- Washing aggregate in preparation for sieve analysis



Figure 41 Sieve stack ready for coarse aggregate analysis



Figure 42 - Cleaning a screen during coarse aggregate sieve analysis

| Sieve | | |
|------------------|------------------|-----------------|
| Size | Percent Retained | Percent Passing |
| 3/8" | 0.0 | 100.0 |
| #4 | 2.8 | 97.2 |
| #8 | 15.3 | 84.7 |
| #16 | 31.0 | 69.0 |
| #30 | 40.6 | 59.4 |
| #50 | 67.8 | 32.2 |
| #100 | 91.1 | 8.9 |
| #200 | 98.3 | 1.7 |
| Fineness Modulus | | 2.5 |

Table 9 - Whitewater Sand (Fine Aggregate) Sieve Analysis

| Sieve | | |
|-------|------------------|-----------------|
| Size | Percent Retained | Percent Passing |
| 1" | 0.0 | 100.0 |
| 3/4" | 6.0 | 94.0 |
| 1/2" | 56.4 | 43.6 |
| 3/8" | 77.8 | 22.2 |
| #4 | 97.9 | 2.1 |

Table 10 - Whitewater 3/4" (Coarse Aggregate) Sieve Analysis

2.2.5 <u>Petrographic Study</u>

A petrographic study from a concrete sample was performed by the Technical University of Denmark and submitted by

- Chief Consultant Bent Grelk (Grelk Consult and Technical University of Denmark)
- Associate Professor Kurt Kielsgaard Hansen (Technical University of Denmark)
- PhD-student Ricardo Antonio Barbosa (Technical University of Den- mark)

Their report Petrographic Analysis of Potential ASR Reactive Concrete Prism and Mortar Bars is hereby included.

2.2.5.1 Introduction

The purpose of the petrographic analysis is to:

- Verify whether the longitudinal expansion measured on the received concrete prism and mortar bars are caused by alkali-silica reaction (ASR)
- Determine which rock types that may be reacting in the concrete sample and in the mortar bars

The petrographic analysis is conducted on thin sections prepared from one of concrete prism and from two of the mortar bars. The authors have been informed by our colleagues at the University of Colorado Boulder that one of the mortar bars was cast with potentially ASR reactive fine aggregate (sand) and the second mortar bar was cast with potentially crushed ASR reactive coarse aggregate.

Page 31 | 93

By the naked eye, both mortar-bars had visually more cracks than the concrete prism. To the authors knowledge all the received samples have been exposed to a NaOH solution at 80 degrees Celsius in accordance with ASTM C1260 "Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)".

The authors are not aware of the exposure time of the samples in the NaOH solution. Additionally, the authors are not aware of the concrete mix used for the samples and the rock types used in the concrete mix.

2.2.5.2 Preparation of Thin Sections

The thin sections were prepared from a slice of the concrete prism and a slice of each of the mortar bars.

The thin sections are made by 1) vacuum impregnation of the slices cut from the samples with an epoxy resin containing a fluorescent dye, and 2) the impregnated slices are mounted on glass plates and grinded and polished to a thickness of 0.02 mm. The thin sections are examined in a polarizing optical microscope using plane polarized light, crossed polarized light and blue transmitted light with a yellow blocking filter (fluorescent mode). The vacuum impregnation of the samples with fluorescent dye causes all voids and cavities to be filled with fluorescent epoxy. By transmitting blue light through the thin section in the microscope, the fluorescent epoxy in the various porosities will emit a yellow light that makes voids, cavities and cracks easy to identify.

The thin sections are examined using a polarization microscope in accordance with the Danish test method "TI-B 5 (87) Structure analysis of hardened concrete" and ASTM C856-04 "Standard Practice for Petrographic Examination of Hardened Concrete".

2.2.5.3 <u>Results of the Petrographic Analysis</u>

The petrographic analysis verifies that the measured longitudinal expansion in the concrete prism and in the mortar bars is caused by ASR. In all samples there is on-going harmful ASR. The harmful reaction is linked to a reactive mix of porous and semi porous flint like grains, metamorphic, sedimentary and magmatic rock types. The metamorphic and sedimentary rock types consist typically of areas with reactive microcrystalline quartz. Since many different rock types are reacting in the concrete prism and in the mortar bars, the reactive rock types will not be distinguished in this report.

The ASR reactions are observed in both the fine aggregate fraction and in the coarse aggregate fraction. For the coarse aggregate fraction, reactions are mainly seen in the mortar-bar where the coarse aggregate fraction has been crushed. In thin sections prepared from the concrete prism only few reactions in the coarse aggregate have been observed. In our opinion there may be a potential for further expansion in the concrete prism, since mostly the fine aggregate fraction is reacting. However, by the petrographic analysis it is not possible to give a quantitative evaluation of the rate and extend of the reaction – only a rough qualitative evaluation.

2.2.5.4 Photographic Documentation

The following photographic documentation shows a representative selection of the reactive rock types in the samples. The photographic documentation shows different harmful reactive rock types including crack formation and ASR gel formation. The presented photos are taken in different light configurations which give the reader a better opportunity to identify the reactive rock types.

Generally, on the following fluorescent light photos the on-going ASR reactive rock types are marked with a red circle. The ASR induced cracks in the cement paste are locally marked with an arrow.

2.2.5.5 Final Remarks

The petrographic analysis is a powerful tool to reveal on-going harmful ASR.



Figure 43 - Concrete sample. Fluorescent light. Magnification: x12.5



Figure 44 - Concrete sample - the same area as in Figure 43. Plane polarized light. Magnification: x12.5



Figure 45 - Concrete sample – the same area as in Figure 43 and Figure 44. Cross polarized light. Magnification: x12.5.



Figure 46 - Mortar-bar with crushed coarse aggregate: Fluorescent light. Magnification: x25



Figure 47 - Mortar-bar with crushed coarse aggregate – the same area as in Figure 46. Plane polarized light. Magnification: x25.

Page 35 | 93



Figure 48 - Mortar-bar with crushed coarse aggregate – same area as in Figure 46 and Figure 47. Cross polarized light. Magnification: x25



Figure 49 - Mortar-bar with fine aggregate. Fluorescent light. Magnification: x25

Page 36 | 93


Figure 50 - Mortar-bar with fine aggregate – the same area as in Figure 49. Plane polarized light. Magnification: x25



Figure 51 - Mortar-bar with fine aggregate – the same area as in Figure 49 and Figure 50. Cross polarized light. Magnification: x25

Page 37 | 93

2.3 MIXING PROCEDURE

As an illustrative example, we consider that a 2 ft³ sample of concrete must be prepared in accordance with ASTM C192.

The concrete mixer is prepared by buttering with a sufficient quantity of water, cement and sand to thoroughly coat the inside surface of the mixer. The ratios of water, cement, and sand, are chosen to approximately match the proportions of the concrete mixture. The butter mixture is rotated for about a minute and discarded.



Figure 52 - Buttering the mixer



Figure 53 - Discarding butter

The mixer is loaded by first adding about 1/3 of the water and all of the coarse aggregate. Rotation is then started and the entire quantities of by fine aggregate and cement are added, along with remaining water. Mixing continues for 3 minutes. Rotation is then halted and the concrete allowed to rest for 3 minutes. The mixer is capped during rest to minimize moisture loss. The mixer is restarted and mixing continues for a final 2 minutes. The freshly-mixed concrete is then discharged into a wetted wheel barrow.



Figure 54 - Charging the mixer



Figure 55 - Mixer is capped to prevent moisture loss during rest period



Figure 56 - Discharging freshly-mixed concrete

2.4 FRESHLY-MIXED CONCRETE TESTS

2.4.1 <u>Temperature</u>

The temperature of freshly-mixed concrete is measured in accordance with ASTM C1064. A digital thermometer with remote probe is used. Immediately after discharging the fresh concrete into the wheelbarrow, the temperature probe is placed in the concrete such that its tip is 3 inches below the concrete surface but not in contact with the walls or base of the wheelbarrow. The temperature is read to the nearest 1 °F between 2 and 5 minutes after probe placement.



Figure 57 - Placing temperature probe

2.4.2 <u>Slump</u>

Slump is measured following the method of ASTM C143. A tapered cylindrical mold is wetted and placed small-end up on a flat surface. While standing on the mold handles, the tester fills the mold with freshly-mixed concrete in three roughly equal-volume layers. Each layer is rodded 25 times. While rodding the topmost layer, extra concrete is heaped about the opening to ensure the mold remains filled. After rodding, the top of the mold is struck off using the tamping rod.



Figure 58 – The slump mold is filled in three layers, each tamped 25 times.

After the mold is struck off, spilled concrete is cleared away from the base of the mold. The tester then removes his feet from the mold handles and lifts the mold in one smooth motion

taking 5 ± 2 seconds to lift the mold 12 inches above the concrete. The entire process from filling to mold removal is completed in 2.5 minutes or less.



Figure 59 - The slump mold is lifted in one smooth motion.

Slump is measured by placing the mold next to the concrete and laying the tamping rod across the top of the mold. The distance between the bottom of the tamping rod and the displaced original center of the concrete specimen.



Figure 60 - Measuring slump

2.4.3 Air Content and Unit Weight

Air content of freshly-mixed concrete is determined using an air meter in accordance with ASTM C173. The unit weight is measured in accordance with ASTM C138 using the air meter bowl, which is of known volume. The inside of the air meter bowl is wetted slightly and weighed. It is then filled with freshly-mixed concrete in two equal layers. Each layer is rodded 25 times and the container is tapped 10-15 times with a rubber mallet after each rodding step. The top surface of the wet concrete is struck off and any excess concrete wiped away. The air meter bowl with concrete is weighed.



Figure 61 – The air meter bowl is filled in two layers, each rodded 25 times.



Figure 62 - After filling, the air meter bowl is struck off. Note the mallet used for tapping the bowl after each layer is rodded.

The top portion of the air meter is wetted and installed on the measuring bowl. A small quantity of water and isopropyl alcohol is added through the fill port of the air meter, just until it begins to run out the weephole. The fill port and weephole are closed and the meter is tilted and rolled to allow trapped air to escape the concrete. Air content is read using the dial pressure gage and comparing with a table provided by the air meter manufacturer.



Figure 63 - The top portion of the air meter is installed.

Page 42 | 93



Figure 64 - A small quantity of water and isopropyl alcohol is added just until it begins to run out the weephole

2.5 CURED-CONCRETE TESTS

2.5.1 <u>Compressive Strength</u>

Compressive strength of concrete is evaluated in accordance with ASTM C39. Six standard 4-inch diameter, 8-inch long cylinders are prepared after completion of the freshly-mixed concrete tests described in sections 2.4.1 - 2.4.3. Concrete is scooped into plastic molds and struck off with a wetted magnesium trowel. Cylinders are permitted to rest for 30 minutes before capping in order to evaluate bleed. After capping, cylinders are placed in a fog room to cure for 24 -48 hours. After initial cure, molds are removed and the cylinders replaced in the fog room.



Figure 65 - Filling compression test cylinder molds



Figure 66 - Striking off cylinder molds



Figure 67 - Cylinders were allowed to rest 30 minutes before capping. Notice minimal bleeding.

Two of the cylinders are destructively tested for compression strength after curing for 8 days and two more tested after 28 days. The final two cylinders are reserved for potential future testing.

Compression testing begins with measurement of cylinder diameter and length using calipers. The average of three measurements is accepted. Cylinders are then weighed using a laboratory scale. These measurements permit calculation of circular area, cylinder volume, and concrete density.



Figure 68 - Measuring cylinder diameter

Cylinders are then capped with either sulfur mortar or unbonded rubber end caps. Generally, sulfur mortar is preferred, but both methods are acceptable. Mix 1 was tested using sulfur end caps, but all subsequent mixes were tested with unbonded rubber caps.



Figure 69 - Installing sulfur mortar end caps



Figure 70 - Compression test apparatus

The cylinders are then mounted in a compression testing machine and tested to destruction. The load at failure is recorded and strength in force per unit area calculated.

Page 45 | 93



Figure 71 - Fractured cylinder after testing

2.5.2 ASR Expansion

The accelerated mortar bar tests described above (ASTM C1567) are valid for establishing aggregate reactivity. However, that test is performed using crushed aggregate mortar. In order to characterize the expansion of a given concrete mix, a modified version of ASTM C1293 was adopted.

ASTM C1293 is intended to evaluate the efficacy of fly ash or pozzolans in controlling ASR when reactive aggregates are used. This standard specifies measuring the elongation of 4"x4"x10" concrete prisms that have been cured at 38 ± 2 °C while suspended above water in an enclosed container. C1293 requires use of a standard concrete mix ($420 \pm 10 \text{ kg/m}^3$ cementitious materials, water-to-cement ratio of 0.42-0.45 by mass, coarse aggregate content of 0.70 ± 0.02 of its dry-rodded bulk density, and adjustment of total alkalinity to 1.25% as Na₂O by doping with NaOH. Measurements are taken periodically up to 24 months after casting.

Measuring pozzolan efficacy is not relevant to the current study. In order to obtain a characteristic value representing the expansivity of a given concrete mix, ASTM C1293 is modified in the following ways.

- 1. Instead of testing a standard concrete mix, variation is permitted to allow testing of candidate mixes as-designed.
- 2. Aggregates are not grade-separated and oversize coarse aggregate is not crushed; all materials are employed as-delivered.
- 3. Two groups of samples are produced. The first is subjected to the storage environment specified in C1567. Specifically, curing temperature is increased to 80°C and samples are immersed in 1N aqueous NaOH.
- 4. The second group of samples is stored in a fog room at >90% relative humidity and 22 ± 2°C

Specimens are cast using carbon steel molds of standard dimensions. Before casting, the molds are cleaned and scoured using coarse steel wool. Each surface of the mold is coated with a generous layer of "3-in-1" oil, which acts as both a release agent and as a protectant for the steel. The molds embed two studs in the long axis of each sample, which allows length measurement using the same comparometer described in section 1.3.



Figure 72 - 4-inch square prism molds ready for casting

Molds are filled in two layers. Each layer is rodded at least 25 times. Special care is taken to ensure that concrete is well placed below the measurement stud. Filled molds are placed in a fog room at 21°C and permitted to cure 24 hours. All samples are then demolded.



Figure 73 - 4 inch prisms are placed in fog room to cure 24 hours after casting.

Typically, half of the samples are returned to the fog room where they are stored uncovered in the fog room at 21°C for the duration of the study. An important variation from this procedure occurred with Mix 2R, which is discussed in more depth below.

The other half of the samples are placed into tap water baths ambient temperature after demolding. The water baths are then placed in an oven at 80 $^{\circ}$ C for a further 24 hours. 48 hours after casting, specimens are removed from water baths and placed in 1 aqueous NaOH solution at 85 $^{\circ}$ C.



Figure 74 - Demolding a concrete prism.



Figure 75 - Concrete prism in container with 1N NaOH.



Figure 76 – Taking length comparometer readings of a concrete prism.

Length measurements are taken with a comparometer approximately weekly 4 days for a period of two weeks. Subsequent measurements are taken approximately weekly.

2.6 DOPING THE CEMENT WITH ALKALI

Adapted from: MCPT Test and its Round Robin Evaluation. Federal Highway Administration. Solicitation No. DTFH61-08-R-00010. Principal Investigator – Dr. Prasad Rangaraju, Clemson University

2.6.1 <u>Reason</u>

In order to accelerate the reaction, the concrete mix must have an Na₂O_{eq} of 1.25-1.6%. The higher value was recommended by Experts from the Danish Technical University and is equivalent to 5-6 kg/m³.

Part of the Na₂O_{eq} is provided by the cement whose alkalinity is provided in the Mill Report, and the rest must be provided by doping the water with Sodium Hydroxide.

2.6.2 <u>Procedure</u>

Let us assume that Type I Portland Cement having an alkali content of $0.53 \pm 0.1\%$ Na₂O_{eq} (as supplied to us by the Ash Grove Cement Company of Midlothian, TX) should be used and that the alkali content of the concrete should be further boosted to 1.25% by weight of cement by adding adequate reagent grade NaOH to the mix water in order to achieve approximately 1M NaOH, such that the hydroxyl ion concentration in the pore solution is similar to that of the external soak solution (1M NaOH). Also, the impact of alkali leaching from the concrete test specimens during the initial storage of test specimens in water for 1 day is minimized.

The alkali content of concrete is calculated based only on the mass of the cement and not that of the supplementary cementitious materials. This assumes that the alkali content of the supplementary cementitious materials is not greater than 4% by mass of the supplementary cementitious material.

Example Calculation for determining the amount of NaOH to be added to the mixing water to increase the alkali content of the cement from 0.90% to 1.25%.

| = 365 kg |
|---------------------|
| = 365 kg |
| = 365 kg x 0.53% |
| = 1.93 kg |
| = 365 kg x 1.25% |
| = 4.56 kg |
| = 4.56 kg – 1.93 kg |
| = 2.63 kg |
| |

The 2.63 kg of alkali (i.e. the difference) is the amount of alkali, expressed as Na₂Oequivalent, to be added to the mix water.

The conversion factor to convert Na₂O equivalent to NaOH is 1.291, derived as follows:

| $Na_2O + H_2O$ | → 2 NaOH |
|---------------------------------------|---|
| 1 mole of Na ₂ O | \rightarrow 2 moles of NaOH |
| 61.98 grams/mole of Na ₂ O | \rightarrow 2 x 39.997 grams/mole of NaOH |

Therefore, 2 x 39.997 / 61.98 = 1.291.

Therefore, NaOH required to achieve an a total alkali content of 1.25% of Na₂O in 1 m³ of concrete = $1.291 \times 2.63 = 3.39 \text{ kg/m}^3$

2.7 MITIGATION OF ASR BY LITHIUM NITRATE

2.7.1 <u>Control Concrete</u>

In order to evaluate the degradation of concrete shear strength due to ASR, it is necessary to generate control specimens that are as similar as possible to the ASR-affected (experimental) specimens, with the single exception that the experimental concrete has experienced ASR while the control concrete has not. Ideally, these reactive and nonreactive concretes should be generated from the same materials (or as similar as practical) and exhibit the same mechanical properties absent the effects of the reaction itself. The following strategies were considered for generating a control concrete.

- 1. Substitute nonreactive aggregate
- 2. Employ an ASR-mitigating admixture
 - a. Fly ash / pozzolan
 - b. Lithium nitrate

The use of a nonreactive aggregate for control concrete was rejected due to concerns that doing so would produce unacceptable variation in mechanical properties between the reactive and control specimens. Addition of fly ash was also rejected despite its proven efficacy at mitigating ASR. According to conversations with Whitewater Building Materials, approximately 25% replacement of cement with fly ash has proven effective at limiting reactivity of their aggregates. Such a modification to mix design would require reduction of water-to-cement ratio to maintain desired strength. This change in turn would likely necessitate the inclusion of a water reducing agent to maintain workability. The end result is a control concrete that varies significantly from the experimental concrete in both composition and mechanical properties.

Lithium nitrate is an alternative to fly ash for control of ASR. Its effectiveness is widely accepted, though it is less commonly used for construction than fly ash due to its increased price. The advantage of lithium for production of a control concrete is that it is dosed as an aqueous solution that that replaces a portion of the mix water and is effective in small quantities. This allows the control concrete to be nearly identical to the experimental concrete.

2.7.2 Procedure

A commercially-prepared lithium nitrate admixture supplied by Grace Concrete Products called *RASIR* was selected. This is a 30% solution of lithium nitrate. Dosing is calculated per manufacturer's recommendation.

Lithium admixture dose
$$\left(\frac{L}{m^3}; \frac{gal}{yd^3}\right) = \frac{\alpha * \beta * \gamma}{100}$$

where

$$\alpha = Cement \ content \ of \ concrete \ \left(\frac{kg}{m^3}; \frac{lb}{yd^3}\right)$$

Page 50 | 93

$$\beta = Alkali \text{ content of cement in } \% \text{ as } Na_2O$$

$$\delta = Coefficient \left(4.6 \text{ for } \frac{L}{m^3}; \ 0.55 \text{ for } \frac{gal}{yd^3}\right)$$

Because the lithium nitrate solution is a liquid, mix water is reduced proportional to the amount of *RASIR added*.

 $w = w_0 - 0.84 * (LiNO_3 admixture volume (L; gal))$

where

 $w = adjusted \ concrete \ mix \ water \ volume \ (L; gal)$

 $w_0 = original \ concrete \ mix \ water \ volume \ (L; \ gal)$

Example Calculation for determining the amount of Grace RASIR and adjusted water content of a control concrete mix.

| Alkali content of cement | = 0.91 % as Na ₂ O |
|----------------------------|-------------------------------|
| Cement content of Concrete | $= 365 \text{ kg/m}^3$ |
| δ Coefficient | =4.6 |
| LiNO ₃ Dose | $=15.3 \text{ L/m}^{3}$ |
| Water content of concrete | $=208 L/m^{3}$ |
| Water reduction factor | =0.84 |
| Adjusted water content | $= 195 \text{ L/m}^3$ |

3 MIX DESIGN OBJECTIVES

Concrete mix design plays a significant role in the progression of the alkali silica reaction. Even a concrete mixed with highly reactive aggregates may exhibit slow or small expansion due to inadequate alkali content to attack reactive minerals, insufficient water to maintain ionic mobility and hydrate ASR gels, or excessively high or low permeability.

It is important to note that no effort was made to precisely duplicate the concrete used in the Seabrook reactor containment vessel. The authors have access to neither the aggregates nor cement that were convenient to its builders. Furthermore, construction concretes are never designed with the intention of magnifying ASR effects; these are exclusively deleterious and unintended. Therefore, responsible designers avoid reactive aggregates or include pozzolans to mitigate their effects if such aggregate is unavoidable.

However, the purpose of this study is to deliberately create a concrete which will exhibit rapid, vigorous expansion. Therefore, best practices for producing durable concrete must be deliberately avoided. The best that can be hoped is to produce a model concrete that is reasonably similar to a construction material.

It is believed that prototype concretes were designed to have a compressive cylinder strength of at least 4000 psi, with actual strength likely closer to 4,500 psi. The model concrete experimental concrete to be vibrated between the closely-spaced shear studes of the sample end plates is also required. A slump of at least 5" is considered sufficient. Finally, the expansion

target for our reactive samples is 0.5%. This is an ambitious target, and will require some experimentation to achieve.

There are a number of factors which may be adjusted to increase expansion while maintaining workability and strength. The most important of these is the alkali content of the cement. Sufficient alkali must be provided to fully activate reactive aggregate minerals. This may be achieved by selecting cements with high natural alkalinity and by artificially boosting alkalinity through addition of sodium hydroxide.

Considering that experimental samples must reach their expansion target in six months or less, it is probable that the bulk of expansion is due to the reactivity of the fine aggregates. Indeed, the role of the coarse aggregates may be quite small. Thus increasing the ratio of fine aggregate to coarse aggregate may reasonably be expected to increase expansion. However, it is unrealistic to boost sand content excessively without producing more of a model mortar than a concrete.

Both very low and high air content can inhibit expansion. Excessively high air content allows a large pore volume for expanding ASR gels to fill without inducing gross strain. Low air content, such as is present in high-performance concrete occurs with low water-to-cement ratios. The lack of water in such concrete can limit ionic mobility and hydration of ASR gel. Thus there is likely an ideal w/c ratio that we hope to find by experimentation.

| Compressive Strength | 4,500 psi | 31.0 MPa | |
|----------------------------------|--------------|----------|--|
| Slump | 4.5-6.5 " | 11-14 cm | |
| Expansion | 0.5% | | |
| Air Content | Less than 3% | | |
| Table 11 Concrete mix objectives | | | |

Table 11 - Concrete mix objectives

4 CONCRETE MIX DESIGNS

The concrete mix used for experimental specimens should be representative of those used in construction of US nuclear power plant containment vessels, while exhibiting measurable ASR expansion and adequate workability. These goals are obviously contradictory since construction concrete typically includes admixtures intended to reduce undesirable expansion when reactive aggregates must be used. However, it is our hope that adherence to the objectives stated in section 3 will permit production of a concrete mix that is reasonably representative of prototype systems while providing the desired expansion.

Several mix designs were considered and tested until objectives for strength, workability, and expansion were met.

4.1 CONCRETE MIX DESIGN 1

The first mix was designed by following ACI 211.1, Chapter 6 with the intent meeting the objectives for mechanical properties described in 3. The first mix would also form a baseline against which future refinements could be compared. Recall that admixtures such as water-reducing agents or retarders were avoided in order to minimize the number of variables that might affect ASR expansion. Of particular concern was obtaining adequate workability to allow thorough penetration of concrete between the closely-spaced shear studs in our sample end plates.

Slump was therefore selected at 5 inches, one inch greater than ACI recommendations in Table 6.3.1 for beams, reinforced walls, and building columns. Maximum aggregate size is limited to 3/4 the clear distance between reinforcement members. Considering the 1.5 inches of clear space between shear studs in sample end plates, maximum aggregate size of 3/4 inches was selected. Water to cement ratio was estimated following Table 6.3.4 at 0.57, which is consistent with suggestions by researchers in Switzerland (EMPA, Holcim, and EPFL) that w/c should be no less than 0.45 in order to avoid inhibition of ASR by pore-space desiccation during hydration.

| Material | lbs/yd3 | kg/m3 |
|-------------------------------------|---------|-------|
| Portland Cement, Type 1 | 614 | 365 |
| Fine Aggregate: Manufactured Sand | 1187 | 705 |
| Coarse Aggregate: 3/4" Crushed Rock | 1771 | 1052 |
| Water | 350 | 208 |
| w/c | 0.57 | 0.57 |

| 7 | ahle | 12 | - Mix | 1 | Reactive | concrete | desian |
|---|------|----|-------|----|----------|----------|--------|
| ' | ubic | 12 | IVIIA | 1, | ncucuvc | CONCICIC | ucsign |

4.1.1 Test Results

A 1 ft³ sample of concrete Mix 1 was prepared on June 11, 2015 and subject to the battery of freshly-mixed concrete tests described above in section 2.4. The results of these tests are summarized below.

| Temperature of freshly-mixed concrete (°F) | 77 |
|--|-------|
| Ambient temperature (°F) | 75 |
| Slump (in) | 6.25 |
| Air Content (%) | 0.7% |
| Unit Weight (lbs/ft ³) | 147.6 |

Table 13 - Freshly-mixed concrete testing results: Mix 1

Reviewing these results, we find that this mix exceeds the target slump value by 1.25", but easily meets the desired air content. The higher-than-anticipated slump is deemed acceptable, since a higher slump provides acceptable workability, provided no bleed occurs. Mix 1 was found to bleed very slightly (refer to Figure 67 below), less than is typically considered problematic according to Dana Schwartz.

Compression testing was conducted on June 19th, 2015 and on July 9th, 2015, 8- and 28-days after casting, respectively. The 28-day cylinder strength of Mix 1 is nearly ideal.

| Age | |
|--------|----------------|
| (Days) | Strength (psi) |
| 8 | 4170 |

| | 28 | 4430 | |
|---|------------------|--------------------------|---|
| Τ | able 14 - Compre | essive strength of Mix . | 1 |

Expansion was measured using the modified version of ASTM C1293 described in section 2.5.2, with the exception that no samples from Mix 1 were stored in the fog room. All Mix 1 samples were kept immersed in 1 M NaOH and stored in an oven at 80C. Results are summarized in Figure 77 below.

| Curing Conditions | Elongation | Age |
|--------------------------|------------|---------|
| 80°C, 1M NaOH | 0.247% | 65 days |



Table 15 - Elongation of 4x4x10 prisms, Mix 1, Reactive

Figure 77 - ASR expansion of Mix 1

4.1.2 <u>Discussion</u>

Contrasting Figure 77 with Figure 21 suggests that concrete prisms are much less expansive than mortar bars mixed using the same aggregates. Mix 1 concrete bars expanded only 0.27% after 100 days while mortar bars cast with the same fine aggregate expanded roughly 1.15% after 100 days under the same curing conditions. Ultimate elongation of Mix 1 is 0.32%, reached after about 180 days.

Subsequent mix designs focus on improving reactivity while maintaining acceptable mechanical properties.

4.2 MIX DESIGN 2, REACTIVE

The goal of the second reactive mix design (called hereafter Mix 2R) is to evaluate the potential for increasing ASR expansion by boosting alkalinity. The cement used for the first mix was provided by Midlothian of Ash Grove, Texas and has alkalinity of 0.45% as Na₂O. For mix 2R, a different cement provided by Holcim of Hagerstown, Maryland with alkalinity of 0.91% as Na₂O was used. The alkalinity of this mix was further increased to 1.25% as Na₂O by addition of sodium hydroxide as described in section 2.6 above. Mix 2 is similar in all other ways to mix 1 above.

| Material | lbs/yd³ | kg/m ³ |
|-------------------------------------|--------------------|-------------------|
| Portland Cement, Type 1, Holcim | 614 | 365 |
| Fine Aggregate: Manufactured Sand | 1,205 | 716 |
| Coarse Aggregate: 3/4" Crushed Rock | 1,753 | 1,041 |
| Water | 350 | 208 |
| w/c | 0.57 | 0.57 |
| Admixtures | kg/yd ³ | kg/m ³ |
| NaOH(s) Doping Additive | 1.22 | 1.6 |

| Table 16 - | Mix 2, | Reactive | concrete | design |
|------------|--------|----------|----------|--------|
|------------|--------|----------|----------|--------|

4.2.1 Test Results

A 2.5 ft^3 batch of concrete mix 2R was produced on August 31, 2015. The results of testing on the freshly-mixed concrete are summarized below.

| Property | Value |
|--------------------------|-----------|
| Slump | 8.3 in |
| Air content | 2.1% |
| Unit weight | 145.7 pcf |
| Wet-concrete temperature | 83.4 °F |
| Ambient temperature | 78.2 °F |

Table 17 - Freshly mixed concrete testing results: Mix 2 Reactive

Note that the slump of mix 2R is excessively high. Some variance in properties is to be expected when changing a major component like cement. The air content of this mix is higher than ideal.

The compressive cylinder strength of mix 2R was tested by on September 8th, 2015, and again on September 25th, 2015.

| Property | Value |
|-------------------------|-----------|
| Cylinder strength at 8 | |
| days | 3,920 psi |
| Cylinder strength at 28 | |
| days | 4,760 psi |

| Table | 18 | -Comp | ressive | strength | of | Mix | 2, | Reactive |
|-------|----|-------|---------|----------|----|-----|----|----------|
| | | | | | | | | |

Note that the compressive strength of mix 2R after 28 days of curing is quite close to our target value.

Unlike previous the elongation test, prisms of mix 2R, were divided into two groups and were stored under different conditions. Immediately after casting, molds with fresh concrete were placed in a fog room to cure 24 hours. Samples were then demolded and placed into water baths which were themselves moved to an 80°C oven. Twenty-four hours later, all samples were transferred to 1M NaOH at 80°C and returned to the oven. Eight days after casting, two bars were removed from the oven and the NaOH soak containers and placed in the fog room 21°C. The remaining two bars were left undisturbed in the oven at 80°C and immersed in 1M NaOH.

| Curing Conditions | Elongation | Age |
|--------------------------|------------|---------|
| 80°C, 1M NaOH | 0.478% | 65 days |
| 21°C, Fog Room | 0.191% | 65 days |

Table 19 - Elongation of 4x4x10 prisms, Mix 2 Reactive

Note that mix 2R is significantly more reactive than mix 1. However, reducing cure temperature to near-ambient greatly retards expansion. Even after many months, samples stored in the fog room reached only 0.251% elongation, while those in the oven reached 0.688%.



Figure 78 - Elongation of Mix 2 Reactive, non-accelerated conditions



Figure 79 - Elongation of Mix 2 Reactive, accelerated conditions

4.2.2 Discussion

The slump of mix 2R is higher than the target and should be reduced. Air content is also slightly high. Elongation of bars stored at 80 °C exceeded target values of 0.5% after about 70

days, but those stored at 21 °C did not. Since it is difficult to anticipate how the larger shear samples will expand, subsequent designs will attempt to further increase expansion.

It is important to note that measures taken to boost reactivity in subsequent designs were successful only for those bars stored at high temperature and immersed in NaOH. Samples of later, more reactive mixes which were stored in the fog room underperformed those of mix 2R. It appears that the initial 6 days these unaccelerated mix 2R samples spent soaking in 1M NaOH had a lasting effect on expansion. This idea is developed in more detail below.

4.3 MIX DESIGN 2, NONREACTIVE

It is beneficial produce a control concrete mix with composition as similar as possible to reactive concrete with the single exception that the control mix does not undergo ASR expansion. To evaluate the efficacy of using aqueous lithium nitrate to prevent ASR expansion in an otherwise reactive mix, a nonreactive version of mix 2 was produced (hereafter called mix 2NR). A 30% solution of lithium nitrate manufactured by Grace Concrete Products called *Rasir* was used to produce nonreactive concrete.

| Material | lbs/yd ³ | kg/m ³ |
|-------------------------------------|---------------------|-------------------|
| Portland Cement, Type 1, Holcim | 614 | 365 |
| Fine Aggregate: Manufactured Sand | 1,227 | 729 |
| Coarse Aggregate: 3/4" Crushed Rock | 1,786 | 1,786 |
| Water | 329 | 195 |
| w/c | 0.57 | 0.54 |
| Admixtures | kg/yd ³ | kg/m ³ |
| Lithium Nitrate Additive | 11.7 | 15.3 |

Table 20 - Mix 2, Nonreactive concrete design

4.3.1 Test Results

A 2 ft³ batch of concrete Mix 2NR was produced on September 1st, 2015.

| Property | Value |
|---------------------|-----------|
| Slump | 7.0 in |
| Air content | 1.7% |
| Unit weight | 145.4 pcf |
| Wet-concrete | |
| temperature | 81.6 °F |
| Ambient temperature | 82.1 °F |

Table 21 - Freshly mixed concrete testing results: Mix 2 nonreactive

The slump of Mix 2NR is one inch lower than that of mix 2R. However, considering that it is rarely practical to attempt to control slump to better than ± 1 inch, this may be a result of statistical uncertainty.

The compressive cylinder strength of mix 2, reactive was tested on September 8th, 2015, and again on September 25th, 2015.

| Property | Value |
|-------------------------|-----------|
| Cylinder strength at 7 | |
| days | 4,160 psi |
| Cylinder strength at 27 | |
| days | 5,030 psi |

Table 22 –Compressive strength of Mix 2, Nonreactive

The compressive strength of the nonreactive version of Mix 2 is somewhat higher than that of Mix 1. It may be that the water-to-cement ratios of the control and experimental mixes must differ in order to achieve similar strengths.

All samples of Mix 2, Nonreactive were cured in a fog room and were never soaked in 1M NaOH. Because this mix is only intended for use as a control, no effort was made to accelerate ASR in samples produced from it. The goal of the nonreactive mix is to limit expansion to nearly zero.

| Curing Conditions | Elongation | Age |
|-------------------|------------|---------|
| 20°C, Fog Room | 0.006% | 65 days |

Table 23 - Elongation of 4x4x10 prisms, Mix 2 Nonreactive

Mix 2 nonreactive exhibits very little expansion, as desired.



Figure 80 - Elongation of Mix 2 Nonreactive, non-accelerated conditions

4.3.2 <u>Discussion</u>

Lithium nitrate is ideal for the purpose of generating a control concrete. Expansion of mix 2NR is essentially zero. Simultaneously, mechanical properties were altered only slightly. Contrasting results of mix 2R and 2NR, we find that slump declined 16%, strength increased 6%, and expansion declined 97%. It is doubtful that any other method of ASR control, such as fly ash, kaolinite, or other pozzolans could outperform LiNO₃ in terms of halting ASR expansion without drastically influencing mechanical properties.

4.4 MIX DESIGN 3, REACTIVE

Increasing the alkalinity of mix 2R relative to mix 1 corresponded to an increase in ASR expansion. The objective of Mix 3 is to further increase reactivity without adversely affecting other properties. This is achieved by adjusting the following parameters.

- Because fine aggregate exposes a much greater surface area to caustic pore water solution than an equal mass of coarse aggregate, it is reasonable to assume that sand is the primary driver of elongation within the timescale of the study. Thus, increasing the proportion of sand to gravel in the mixture should increase gel formation and elongation. For Mix 3, a volume proportion of about 34.5% fine aggregate to total concrete was used.
- 2. Increasing the amount of fine aggregate in the mix should increase expansion, provided sufficient alkalinity is available to drive the reaction. Thus, alkalinity of the reactive mix is increased to 1.6% as Na₂O_(s), which corresponds to 5.84 kg/m^3 .
- 3. Water-to-cement ratio was reduced to about 0.50. The intent of this change is to reduce slump and offset any strength reduction due to increasing the content of fine aggregate.

| Material | lbs/yd ³ | kg/m ³ |
|-------------------------------------|---------------------|-------------------|
| Portland Cement, Type 1, Holcim | 614 | 365 |
| Fine Aggregate: Manufactured Sand | 1,525 | 906 |
| Coarse Aggregate: 3/4" Crushed Rock | 1536 | 912 |
| Water | 310 | 184 |
| w/c | .50 | .50 |
| Admixtures | kg/yd ³ | kg/m ³ |
| NaOH(s) Doping Additive | 2.48 | 3.25 |

Making these adjustments to the prior reactive concrete mix yields the following design.

Table 24 - Mix 3 Reactive concrete design

4.4.1 <u>Test Results</u>

On November 16th, 2015 a 1 ft³ sample of Mix 3, reactive was prepared at Fall Line Testing. Aggregate moisture is measured the morning before mixing a test batch and the mix is adjusted accordingly. Unfortunately, I made a mistake in my calculations which caused the as-mixed water content to be less than the 310 lbs/yd³ specified above.

| Volume of test batch (ft ³) | 1.0 |
|---|-------|
| Material | lbs |
| Portland Cement, Type 1, Holcim | 22.7 |
| Fine Aggregate: Manufactured Sand | 58.2 |
| Coarse Aggregate: 3/4" Crushed Rock | 56.7 |
| Water | 10.0 |
| Admixtures | g |
| NaOH(s) Doping Additive | 92.03 |

Table 25 - Mix 3 Reactive, test batch actual weights

Considering the observed moisture content of the fine aggregate pile on the day of mixing was 4.46 % and that of the coarse aggregate pile was 1.10%, one may note that adding 12.95 lbs of water would produce the design water content, but only 10.0 lbs were added to the test batch.

The combination of adding much more sand than Mix 2R coupled with lower-than-design water content caused Mix 3R to have an unacceptably low slump.

| Property | Value |
|--------------------------|--------|
| Slump | 2.5 in |
| Air content | 2.8% |
| Wet-concrete temperature | 68 °F |
| Ambient temperature | 65 °F |

Table 26 - Freshly mixed concrete testing results: Mix 3 Reactive







Figure 82-Elongation of Mix 3, Reactive under accelerated conditions

4.4.2 <u>Discussion</u>

Mix 3R was rejected based both on its unacceptably low slump and on my mistake mixing the test batch properly. While the elongation data may be useful for developing a sense for the effects of fog-room conditioning vs conditioning in hot sodium hydroxide solution.

4.5 MIX DESIGN 4, REACTIVE

The objective for Mix 4R were to correct the mistakes which caused Mix 3 to exhibit unacceptably low slump, while retaining the high sand and alkali content. Comparison of the asmixed content of Mix 2R to that of Mix 3R suggested that the increased proportion of fine aggregate to coarse in Mix 3R contributed somewhat to slump reduction. Therefore, the water content of Mix 4 is between those of Mix 2 and Mix 3.

| Material | lbs/yd ³ | kg/m ³ |
|-------------------------------------|---------------------|-------------------|
| Portland Cement, Type 1, Holcim | 636 | 378 |
| Fine Aggregate: Manufactured Sand | 1,585 | 941 |
| Coarse Aggregate: 3/4" Crushed Rock | 1362 | 809 |
| Water | 350 | 208 |
| w/c | .55 | .55 |
| Admixtures | kg/yd ³ | kg/m ³ |
| NaOH(s) Doping Additive | 2.57 | 3.37 |

Table 27 – Mix 4, Reactive concrete design

4.5.1 <u>Test Results</u>

A test batch of Mix 4R, reactive was produced on December 21, 2015 at Fall Line Testing.

| Property | Value |
|--------------------------|-----------|
| Slump | 4.5 in |
| Air content | 2.7% |
| Unit weight | 144.7 pcf |
| Wet-concrete temperature | 69.8 °F |
| Ambient temperature | 64.2 °F |

Table 28 - Freshly mixed concrete testing results: Mix 4 Reactive

Note that slump is within just within the target range, providing sufficient workability to vibrate concrete between the closely-spaced shear studs of the sample end plates. The air content is slightly higher than ideal, closer to that of Mix 3R than Mix 2R. This may be an unavoidable consequence of increased sand content.

Two 4x8" cylinders were tested to failure in compression 7 days after casting and four more after 28 days. While the 7-day results were acceptable, the 28 day results were slightly below the target of 4000 psi. Of particular concern, two of the 28-day cylinders failed at less than 3800 psi. Mix 4 must be rejected on the basis of strength.

| Property | Value |
|---|----------|
| Cylinder strength at 7 days | 3500 psi |
| Cylinder strength at 27 days | 3958 psi |
| Table 29- Compressive strenath of Mix 4. Reactive | |

Similar to previous cases, two prisms were conditioned in hot $1M \text{ NaOH}_{(aq)}$ and two prisms in a fog room at ambient temperature. Plotting the expansion curves allows interpolation of the 65-day elongation figures.

| Curing Conditions | Elongation | Age |
|--------------------------|------------|---------|
| 80°C, 1M NaOH | 0.587% | 65 days |
| 20°C, Fog Room | 0.030% | 65 days |



Table 30-Elongation of 4x4x10 prisms, Mix 4 Reactive

Figure 83 - Elongation of Mix 4, Reactive, under nonaccelerated conditions. Note the dip between 50 days and 70 days. During this time period only one of three Hydrofogger units were operational.



Figure 84 - Elongation of Mix 4, Reactive under accelerated conditions

4.5.2 <u>Discussion</u>

Note that, under accelerated conditions, expansion of Mix 4R exceeds that of Mix 2R and exceeds the target value of 0.5% after 65 days. This suggests that the provision of additional sand and alkali are effective at boosting reactivity.

However, elongation of specimens conditioned in the fog room is significantly retarded in comparison to Mix 2. I suspect that the unexpectedly slow expansion may be due to partial drying of the specimens in the fog room. Normally, the fog room at Fall Line uses three Hydrofogger units to maintain humidity, however, only one unit was found to be operational on when measurements were taken on December 23rd. It is conceivable that humidity may have dropped over the holidays, allowing some degree of shrinkage to occur. Once all three Hydrofogger units were brought back online on January 4th, expansion accelerated and returned to trend.

4.6 MIX DESIGN 5, REACTIVE

Considering the failure of Mix 4R to reach strength targets after 28 days, a fifth mix was designed with the objective of increasing strength without altering other properties. A new w/c ratio was interpolated between tabulated values and the measured results of Mix 4R, resulting in a decrease of 0.02. Likewise, water content was also interpolated to maintain workability, resulting in an increase of 3 lbs/yd³.

| Material | lbs/yd ³ | kg/m ³ |
|-------------------------------------|---------------------|-------------------|
| Portland Cement, Type 1, Holcim | 666 | 396 |
| Fine Aggregate: Manufactured Sand | 1,552 | 922 |
| Coarse Aggregate: 3/4" Crushed Rock | 1,362 | 809 |
| Water | 353 | 210 |
| w/c | .53 | .53 |
| Admixtures | kg/yd³ | kg/m ³ |
| NaOH(s) Doping Additive | 2.69 | 3.52 |

Table 31 – Mix 5, Reactive concrete design

4.6.1 <u>Test Results</u>

A two-ft³ batch of Mix 5, reactive was produced on January 22, 2016 at Fall Line Testing.

| Property | Value |
|--------------------------|-----------|
| Slump | 6.5 in |
| Air content | 1.7% |
| Unit weight | 146.4 pcf |
| Wet-concrete temperature | 68.7 °F |
| Ambient temperature | 66.2 °F |

Table 32 - Freshly mixed concrete testing results: Mix 4 Reactive

All initial test results are positive. While the slump is at the high end of acceptability, no bleeding was evident in cylinders or prism molds

The compressive strength of mix 5R was tested on January 29th, 2016 and again on February 29th, 2016. Its 28-day strength is higher than our target value of 4500 psi. However, it is common for construction concretes to exceed their rated strength and it is not unreasonable to consider a strength of 5100 psi to be representative of a construction concrete rated at 4000 psi.

| Property | Value |
|-------------------------|----------|
| Cylinder strength at 7 | |
| days | 3700 psi |
| Cylinder strength at 27 | |
| days | 5100 psi |

Table 33- Compressive strength of Mix 5, Reactive

Similar to previous cases, two prisms were conditioned in hot 1M NaOH_(aq) and two prisms in a fog room at ambient temperature.

| Curing Conditions | Elongation | Age |
|--------------------------|------------|---------|
| 80°C, 1M NaOH | 0.590% | 65 days |
| 20°C, Fog Room | 0.033% | 65 days |



Table 34-Elongation of 4x4x10 prisms, Mix 5 Reactive





Figure 86 - Elongation of Mix 5, Reactive under accelerated conditions

Continuing a pattern first observed in Mix 4R, the accelerated samples readily exceed the expansion target within a relatively short period of time. However, the non-accelerated samples significantly underperform.

As remarked above in the discussion for Mix 4R, the results of the Hydrofogger failure is evident as a dip in elongation between age of 20 days and 40 days. In fact, not only did the drying effect of lower humidity arrest the expansion of Mix 5, these samples shrank somewhat during the drying period. This serves as a reminder of the critical nature of moisture control in quantification of ASR.

4.7 **TEMPERATURE EFFECTS**

The temperature dependence of ASR can be estimated using Larive's kinetic model (Larive, 1998), which described concrete expansion as a function of both absolute temperature (T) and time (t).

$$\varepsilon(t,T) = \frac{1 - e^{\left(\frac{1}{\tau_c(T)}\right)}}{1 + e^{\left(\frac{1 - \tau_L(T)}{\tau_c(T)}\right)}}$$

The two parameters τ_L and τ_C are the latency and characteristic times of the sigmoidal strain function, respectively. Each of these parameters may be calculated for some given temperature if they are known for some other temperature (T_0) as follows (Ulm, Coussy, Li, & Larive, 2000):

$$\tau_C(T) = \tau_C(T_0) e^{\left(U_c\left(\frac{1}{T} - \frac{1}{T_0}\right)\right)}$$

$$\tau_L(T) = \tau_L(T_0) e^{\left(U_L\left(\frac{1}{T} - \frac{1}{T_0}\right)\right)}$$

The values of $\tau_C(T_0)$ and $\tau_L(T_0)$ may be obtained from curve fitting experimental data. Note that reducing storage temperature increases the time for concrete to achieve maximum strain, but does not reduce maximum strain. However, reducing pore humidity does alter maximum strain. Thus, this conversion is only valid for scenarios in which humidity is unchanging.

The most reasonable data set to use for this purpose is that obtained using concrete mix 5 under accelerated conditions Figure 86. The accelerated prisms were stored immersed in 1M NaOH, and thus experienced nearly infinite pore humidity. Experimental shear specimens are stored under a constant flow of 1.0M NaOH, and therefore also remain very nearly saturated. Furthermore, the caustic wash solution prevents alkali leaching in both scenarios.

Results of curve fitting generously provided by Dr. M. Hariri-Ardebili of the University of Colorado are presented below. The result is latency time $\tau_L(353 K) \approx 0$ and characteristic time $\tau_c(353 K) = 7.7 days$ with acceptable goodness-of-fit $R^2 = 0.9$.



Figure 87 - Curve fitting data from concrete mix 5 (80C, 1M NaOH) to Larive's kinetic model.

Using these values, characteristic times at 38°C may be obtained: $\tau_L(311 K) \approx 0$ and $\tau_c(353 K) = 60.8 days$. The resulting expansion curve is presented in Figure 88. Observe that reducing storage temperature does not alter maximum strain, but does extend the time required to reach it. Inspection of this plot reveals that approximately 145 days are required to achieve the target expansion of 0.5%. This is acceptable, as it is quite likely the target value of 0.5% expansion will be reached within the 6-month period permitted by the overall project schedule.



Figure 88 - Expansion vs time for 80C (blue line) and 38C (red line)

Page 69 | 93

5 Specimens

5.1 FROM PROTOTYPE TO MODEL

It is important to not only visualize but also understand the inter-relationship between the actual nuclear containment vessel and the concrete block that are tested. Given the constraint imposed by the testing apparatus (blocks being 42x30x10 inches), and a representative nuclear containment vessel having the dimensions shown in Table 35 (NUREG/CR-6706),

| Inner radius (ft) | 63 |
|--|-----|
| Wall thickness (ft) | 4.5 |
| Wall height (ft) | 122 |
| Foundation thickness (ft) | 10 |
| Grade level (ft above | |
| foundation) | 56 |
| Table 25 Destations contraction to a share of dise | |

Table 35 - Prototype contaminant vessel dimensions

Hence, the specimens tested are representative of a 0.56 model of the actual prototype whose dimensions are shown in Table 36.

| Scale Factor | 0.56 |
|---------------------------|------|
| Inner radius (ft) | 35 |
| Wall thickness (ft) | 2.5 |
| Wall height (ft) | 68 |
| Foundation thickness (ft) | 5.6 |
| Grade level (ft above | |
| foundation) | 31 |

Table 36 - Model containment vessel dimensions

The scaled down model is shown in Figure 89. Figure 90, and Figure 91 show the interrelationship between sample and model. Finally, it should be noted that the container has hoop reinforcements shown in blue, and vertical ones shown in red, Figure 92.



Figure 89 - Prototype system (right) with model system (left)



Figure 90 - Model system showing eight experimental specimens taken just above grade level



Figure 91 - Specimens are rotated 180 degrees about azimuthal direction

Page 71 | 93



Figure 92 - Rebar in model system is oriented in the axial (red) and azimuthal (blue) directions

A better visualization of the specimen layout with respect to the container wall is shown in Figure 93.



Figure 93 Specimen internal reinforcement and applied forces

Page 72 | 93
5.2 REINFORCEMENT

Experimental samples are cast using steel endplates with shear studs to permit shear load in the laboratory vertical axis. Reinforcement is provided in two directions, corresponding to the axial and circumferential directions in the prototype system. Axial reinforcement (shown as red bars in Figure 95) aids in resisting shear forces via dowel action. Azimuthal reinforcement (shown as blue bars in Figure 95) is not engaged by shear forces



Figure 94 - Shear specimen showing concrete and end plates. Note scoring line coincides with shear plane.



Figure 95 - Shear specimen with concrete hidden. Red bars correspond to axial reinforcement and resist shear by dowel action. Blue bars correspond to the azimuthal reinforcement and are not engaged in shear.

Shear specimen reinforcement is provided such that the total reinforcement ratio in both axial and azimuthal directions is 1% (which is to say that the reinforcement ratio of each layer of bars is 0.5%). Selecting #6 bars for the axial reinforcement and #7 bars for the circumferential allows a reasonable distribution of steel throughout the sample cross section without overcrowding. Considering that 11 reinforced samples are required, 242 short #7 bars and 88 longer #6 bars are required.

| (All dimensions in inches) | Bar Number | Bar Diameter | Bar Length | Number of bars per layer | Bar spacing (center to center) | Pactual | Total bars required |
|----------------------------------|---------------|-----------------|---------------|--------------------------------|--------------------------------------|---------|---------------------------|
| Circumferential Reinforcement | 7 | 0.875 | 8 | 11 | 2.813 | 0.52% | 242 |
| Axial Reinforcement | 6 | 0.75 | 42 | 4 | 2.083 | 0.59% | 88 |

Table 37 - Reinforcement plan

Insufficient length is available for either the axial or circumferential steel to develop its full tensile strength. Considering the large strains anticipated due to ASR expansion, it is necessary to provide for some type of anchorage at the bar terminations. A number of options were considered, including hooks and threaded terminations. Unfortunately, the standard hook size for a #7 bar is 10.5" with a minimum bend diameter of 7". Considering that these bars are only 8" long, attempting to use standard hooks would deform the circumferential reinforcement geometry to an extent that it would bear little resemblance to the prototype structure. Furthermore, the minimum development length even with hooks is 19" which exceeds the out-to-out thickness of the sample (10").

We also considered using threaded bar terminations onto which a 'donut' could be connected. These are also not suitable, due to the size of the terminations and donuts.



Figure 96 - A threaded 'DoughNUT' connection manufactured by BarSplice Products of Dayton, OH. This is one of the options considered for terminating rebar. Unfortunately, it is too large to fit in the narrow shear sample.

Ultimately, we decided to weld axial bars to the sample end plates, and weld circumferential bars to the axial bars at each intersection. While welding rebar is not typically best practice, no other practical option exists for developing tensile strength in such a confined volume as the shear samples. The sample end plates provide development for the axial bars, while the axial bars themselves act as hooks for the circumferential bars. While certainly not ideal, this solution allows for at least some tensile development without drastically altering the reinforcement scheme of the prototype system.

5.2.1 <u>Reinforcement Construction</u>

To facilitate rapid construction of the sixteen required rebar cages, we first built a wooden jig to hold the loose bars during the welding process. The desired locations of the bars were computed and carefully laid out on a piece of plywood. Sixty small wooden blocks sized to fit in the areas between the steel bars with 1/32" clearance were then cut. These wooden blocks were then affixed to the plywood base using wood glue and 18-gauge brad nails.

Page 74 | 93



Figure 97 - Cutting wooden blocks for the welding jig.



Figure 98 - Wooden blocks ready for installation in the jig.



Figure 99 - Joining wooden blocks to jig base with wood glue and steel brad nails.

Steel bars were primarily sourced from stock on-hand in the structures laboratory. All were cut to size using a steel saw and ground to final dimension with a bench grinder. This proved to be a time-consuming process, as a total of 330 bars are required.



Figure 100 - Cutting reinforcing bar



Figure 101 - Rebar layout and welding using the wooden jig.

The jig allowed rapid layout and welding of the rebar cages. The jig also provided a simple way to verify that all bars were cut to proper length. Any long or short bars would not fit properly into the jig and could be ground down or replaced.

Bars were MIG-welded to one another at each intersection. Care was taken to make small welds in order to minimize the size of heat-affected regions in the substrate bars.



Figure 102 - Rebar cages ready to be welded to sample end plates.

Completed cages were then connected to the sample end plates by tack-welding the #6 bars to the plates.



Figure 103 - MIG-welded joint between #6 rebar and sample end plates.

5.3 FORMWORK

Specimens are cast horizontally, in order to better facilitate concrete penetration between closely-spaced shear studs. Thus a simple form was designed using 21/32" oriented-strand board and 2x4 studs. The top brace of the stud was elevated somewhat from the top surface of the concrete to allow a trowel to pass under during finishing. Corners are strengthened with steel brackets and the entire assembly is joined with deck screws. The form rests on its 2x4 braces, which allow it to be moved via forklift without extra blocking.



Figure 104 – Example shear specimen formwork

While we considered building a small number of reusable forms using more durable *Plyform* in lieu of OSB, our casting schedule is sufficiently compressed that one form is required per sample. Since each form need survive only one use, OSB is sufficiently durable. Seventeen forms were built, one for the dummy sample and sixteen for the experimental shear samples. To

Page 78 | 93

mitigate water absorption by the wood from the fresh concrete, the inside of each form was given two coats of oil-based primer.



Figure 105 - Assembled and painted formwork. Seventeen forms were built in total.

5.4 Aggregate

Casting this volume of concrete requires a significant supply of material, summarized in Table 38.

| Material | lbs | kg |
|-------------------------------------|--------|-------|
| Portland Cement, Type 1, Holcim | 4,200 | 2,500 |
| Fine Aggregate: Manufactured Sand | 10,100 | 6,100 |
| Coarse Aggregate: 3/4" Crushed Rock | 8,600 | 5,300 |
| Admixtures | | Unit |
| NaOH(s) Doping Additive (kg) | | 12.2 |
| Lithium Nitrate Additive (L) | | 34.5 |

Table 38 - Estimate of materials required based on Mix 5R and Mix 5NR

The required aggregate was delivered to the Fall Line laboratory on February 1st, 2016. Using a conveyor and skid-steer loader, the aggregate was formed into piles, wet slightly, and mixed. Each pile was then covered with plastic sheet until casting.



Figure 106 - Offloading aggregate



Figure 107 - Mixing fine aggregate and forming into pile.

5.5 CASTING PLAN

A listing of concrete specimens to be produced is provided in Table 39. The most significant effort is preparation of the 16 shear samples, which are 42"x30"x10" prisms, discussed in greater detail in section 5.1. Additionally, 15 cubical specimens measuring 14"x14"x14" referred to as "blocks" have been produced. These blocks will allow extraction of cores from the center of their centers, thereby avoiding any undesirable surface effects such as alkali leaching. A third type of sample is intended for a wedge splitting test. A significant number of cylinders are necessary to measure compressive strength and tensile splitting strength. Finally, a small number of 4"x4"x12" prisms were produced which allow monitoring of expansion using a

demountable strain gauge (DEMEC). In total, 6.3 cubic yards of concrete was cast, which is a significant quantity to produce without the benefit of a commercial facility.

| Required Concrete Volume | | |
|--------------------------------|--------|-----------------|
| | Number | |
| | Number | yd ³ |
| Reactive | | |
| Shear specimens with rebar | 9 | 2.43 |
| Shear specimens without | | 0.81 |
| rebar | 3 | 0.01 |
| Wedge splitting test | 3 | 0.02 |
| Cylinder, 4"x8" | 36 | 0.08 |
| Cylinder, 6"x12" | 12 | 0.09 |
| Blocks without rebar | 6 | 0.35 |
| Blocks with rebar | 3 | 0.18 |
| Prism 4"x4"x12" | 6 | 0.06 |
| Wastage factor | 15.0% | |
| Total | 4.5 | 54 |
| Non-Reactive | | |
| Shear specimens with rebar | 2 | 0.54 |
| Shear specimens without | | 0.54 |
| rebar | 2 | 0.54 |
| Wedge splitting test | 3 | 0.02 |
| Cylinder, 4″x8″ | 12 | 0.03 |
| Cylinder, 6"x12" | 4 | 0.03 |
| Blocks without rebar | 3 | 0.18 |
| Blocks with rebar | 3 | 0.18 |
| Prism 4"x4"x10" | 3 | 0.03 |
| Wastage factor | 15. | 0% |
| Total | 1.7 | 73 |
| Grand Total (yd ³) | 6.2 | 27 |

Table 39- Sample requirements

Casting of experimental samples took place at the Fall Line laboratory on April 27th and May 4th. Concrete mixing utilized a three-cubic yard mixer provided by Fall Line. The mixer is charged using a mobile batch plant. The batch plant is a trailer equipped with two hoppers for coarse and fine aggregate and a water tank. The hoppers are emptied onto a built-in conveyor which charges the mixer. Each hopper and tank is equipped with load cells to monitor weight change as aggregates are loaded into the mixer. The hoppers can be filled using a skid-steer loader.



Figure 108 - The three-cubic yard mixer at Fall Line



Figure 109 - Close view of mobile batch plant at Fall Line. Note load cells for weighing hopper contents.

Concrete is transported from the mixer to the formwork using a forklift-borne hopper. Concrete is shoveled or troweled into forms and vibrated into place in two lifts. Shear specimens, blocks, and prisms will receive a trowel finish while cylinders are capped.

6 CASTING RESULTS

6.1 FINAL MIXES

The tables below outline the final mixes used for each batch of specimens. Note that Mix 1 uses the cement from Figure 27 and the subsequent mixes use the cement from Figure 28.

| Mix 1 – Mix Date: April 27, 2016 | | | |
|----------------------------------|----------|----------------|---------------------------|
| Cement: | 934 lb | Slump: | 5.5 in |
| Water: | 349.8 lb | Air Content: | 2.0 % |
| Fine: | 1615 lb | Unit Weight: | 145.61 lb/ft ³ |
| Coarse: | 1375 lb | Ambient Temp: | 49.5 °F |
| Sodium Hydroxide: | 3779.7 g | Concrete Temp: | 69.5 °F |

| Fine Moisture Content: | 4.80 % | Water Cement Ratio: | 0.53 |
|--------------------------|--------|---------------------|-----------------------|
| Coarse Moisture Content: | 1.66 % | Actual Yield: | 29.35 ft ³ |
| Table 40 - Mix 1 | | | |

After Mix 1 was completed and poured into the forms, there was a lower yield than expected. Mix two was adjusted by following ACI 211.1-15 (Equation 7.2.8, Step 8). For the next mix, the water to cement ratio and coarse aggregate were kept the same as the previous mix. The cement, water, and fine aggregate were increased to get the desired volume to fill all the forms originally scheduled for Mix 2 and all the forms that were not filled with Mix 1.

| Mix 2 – Mix Date: April 27, 2016 | | | |
|----------------------------------|----------|---------------------|---------------------------|
| Cement: | 1039 lb | Slump: | 2.25 in |
| Water: | 587.8 lb | Air Content: | 3.1 % |
| Fine: | 4125 lb | Unit Weight: | 143.11 lb/ft ³ |
| Coarse: | 3125 lb | Ambient Temp: | 60.6 °F |
| NaOH | 5473 g | Concrete Temp: | 70.5 °F |
| Fine Moisture Content: | 4.80 % | Water Cement Ratio: | 0.53 |
| Coarse Moisture Content: | 1.66 % | Actual Yield: | 64.16 ft ³ |

Table 41 - Mix 2

The initial slump of the concrete was 1.75 in. In attempt to increase the workability, the concrete was returned to the mixer and more water was added to give a final slump of 2.25 in.

On the next day of pouring, the mix design was adjusted from Mix 2 per ACI 211.1-15 Section 6.3.9.1 to increase the slump of the next mix. The water was increased by 10 lb for each inch of slump that needed to be increased from the original 1.75 in. Thus, an extra 32.5 lb of water was added per cubic yard of concrete.

| Mix 3 – Mix Date: May 4, 2016 | | | |
|-------------------------------|---------|---------------------|----------------------------|
| Cement: | 1076 lb | Slump: | 6.0 in |
| Water: | 504 lb | Air Content: | 2.3 % |
| Fine: | 2240 lb | Unit Weight: | 144.406 lb/ft ³ |
| Coarse: | 1849 lb | Ambient Temp: | 71.4 °F |
| NaOH | 4542 g | Concrete Temp: | 71.2 °F |
| Fine Moisture Content: | 4.65 % | Water Cement Ratio: | 0.53 |
| Coarse Moisture Content: | 0.36 % | Actual Yield: | 39.20 ft ³ |

Table 42 - Mix 3

This mix provided an adequate yield with an acceptable slump. Thus, the per yard mix was held the same.

| Mix 4 – Mix Date: May 4, 2016 | | | |
|-------------------------------|--------|--------------|---------|
| Cement: | 861 lb | Slump: | 4.75 in |
| Water: | 366 lb | Air Content: | 2.2 % |

| Fine: | 2790 lb | Unit Weight: | 144.29 lb/ft ³ |
|--------------------------|---------|---------------------|---------------------------|
| Coarse: | 2295 lb | Ambient Temp: | 75.0 °F |
| Lithium Nitrate: | 41.7 lb | Concrete Temp: | 69.6 °F |
| Fine Moisture Content: | 4.65 % | Water Cement Ratio: | 0.53 |
| Coarse Moisture Content: | 0.36 % | Actual Yield: | 43.75 ft ³ |

| Table | 43 - | Mix 4 |
|---------------|------|-------|
| <i>i</i> ubic | | |

6.2 7 AND 28 DAY COMPRESSIVE STRENGTH

6.2.1 <u>Concrete strengths</u>

Below are the results of compressive strength tests adhering to ASTM C39. Tests were performed 7 and 28 days after casting.

| Mix # | Cylinder # | f'c (ksi) |
|-------|------------|-----------|
| | 1 | 3.672 |
| 1 | 2 | 2.356 |
| | 3 | 1.877 |
| | 1 | 3.932 |
| 2 | 2 | 4.249 |
| _ | 3 | 4.216 |
| | 1 | 3.615 |
| 3 | 2 | 3.662 |
| | 3 | 3.732 |
| | 1 | 5.053 |
| 4 | 2 | 4.626 |
| | 3 | 4.821 |

Table 44 - 7 Day Compressive Strength

| Mix # | Cylinder # | f'c (ksi) |
|-------|------------|-----------|
| | 1 | 5.950 |
| 1 | 2 | 5.969 |
| | 3 | 6.044 |
| | 1 | 4.931 |
| 2 | 2 | 4.924 |
| | 3 | 5.092 |
| | 1 | 3.928 |
| 3 | 2 | 4.192 |
| | 3 | 4.500 |
| | 1 | 5.561 |
| 4 | 2 | 5.751 |
| | 3 | 5.816 |

Table 45 - 28 Day Compressive Strength



Figure 110 - Measuring Cylinders for Compression Test



Figure 111 - Concrete cylinder with rubber caps



Figure 112 - Cylinder installed in MTS machine



Figure 113 - Broken Cylinder in MTS Machine

6.2.2 <u>Stress-Strain Curves</u>

Below are the graphs generated from MATLAB plotting the stress vs. strain from the 7 and 28-day compression tests for each mix. The title gives the mean and standard deviation of the data The legend gives the concrete strength of each cylinder which corresponds to the results given in Table 44 & Table 45. It should be noted that the curve does not immediately increase linearly due to the compression of the rubber caps (seen in Figure 111) before the force is complete transferred to the concrete cylinder.





P a g e 88 | 93



Page 89 | 93



P a g e 90 | 93



P a g e 91 | 93



7 ACKNOWLEDGMENTS

7.1 FALL LINE TESTING & INSPECTION

We were fortunate enough to enter partnership with Fall Line Testing and Inspection, LLC. The manager of Fall Line's Denver Laboratory, Dana Schwartz, provided outstanding counsel and support throughout the project.



Figure 114 - Fall Line Testing & Inspection, LLC., Denver, Colroado

7.2 WHITEWATER BUILDING MATERIALS

Whitewater Building Materials generously donated several batches of sand and crushed rock over the course of the project. Special thanks go to Doug Wolfe, who coordinated the donations and provided invaluable guidance throughout the project.

7.3 BRADY TRUCKING

Brady Trucking Inc. of Vernal, Utah Grand Junction, Colorado provided trucking from the Whitewater mixing plant to Denver. Sam Rendon and Tony Spafford coordinated the shipping effort and driver Tim *** made a hazardous crossing of Vail pass in icy weather to complete the delivery. Sincere thanks go to the entire team at Brady Trucking.

7.4 HOLCIM

Holcim of Hagerstown, Maryland has generously donated 6 barrels of cement for project use.

7.5 GRACE CONCRETE PRODUCTS

Grace has donated two three and half-gallon buckets of Lithium Nitrate.