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Geotechnical and Structures Laboratory
U.S. Army Engineer Research and Development Center
3909 Halls Ferry Road
Vicksburg, MS 39180

Craig A. Rutland

U.S. Air Force Civil Engineer Center
Tyndall Air Force Base, FL 32403

Final report

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Under Project JCL6F0, "Expedient Evaluation of Alkali-Silica Reactivity (ASR) of Aggregates"

Abstract

This study focused on development and evaluation of a novel autoclaving procedure for the rapid evaluation of concrete aggregates for alkali-silica reaction susceptibility. The use of high pressure and temperature autoclaving methods was selected to speed evaluation timeframes from months to a few days. A total of 30 aggregates of varying mineralogy and reactivity were evaluated as part of the study that was divided into two phases. The first phase of research focused on development and optimization of the autoclaving procedures, including temperature, alkali loading, and test duration. Results from Phase 1 were used to identify optimal testing parameters that were then used during a Phase 2 round robin testing study, which included internal laboratory testing as well as four external laboratories. Strong correlation was observed between autoclave expansion results and the results of ASTM C1260 and C1293 standard test methods. In addition, within- and multi-laboratory variability were within acceptable bounds provided by ASTM standards. Overall, the research indicated that autoclaving is a viable option for rapid evaluation of aggregates using testing procedures that can be completed in approximately three days.

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Preface

This study was conducted for the Air Force Civil Engineer Center under Project JCL6F0, “Expedient Evaluation of Alkali-Silica Reactivity (ASR) of Aggregates.” The technical monitor was Dr. Robert D. Moser.

The work was performed by the Concrete and Materials Branch (GMC), of the Engineering Systems and Materials Division (GM), U.S. Army Engineer Research and Development Center, Geotechnical and Structures Laboratory (ERDC-GSL). At the time of publication, Mr. Christopher M. Moore was Chief, CEERD-GMC; Mr. Jeffrey G. Averett was Acting Chief, CEERD-GM; and Mr. R. Nicholas Boone, CEERD-GZT, was Technical Director for Force Projection and Maneuver Support. The Deputy Director of ERDC-GSL was Mr. Charles W. Ertle II, and the Director was Mr. Bartley P. Durst.

COL Ivan P. Beckman was the Commander of ERDC, and Dr. David W. Pittman was the Director.

Unit Conversion Factors

Multiply	By	To Obtain
cubic feet	0.02831685	cubic meters
cubic inches	1.6387064 E-05	cubic meters
cubic yards	0.7645549	cubic meters
degrees Fahrenheit	(F-32)/1.8	degrees Celsius
feet	0.3048	meters
gallons (U.S. liquid)	3.785412 E-03	cubic meters
inches	0.0254	meters
mils	0.0254	millimeters
ounces (mass)	0.02834952	kilograms
ounces (U.S. fluid)	2.957353 E-05	cubic meters
pounds (force)	4.448222	newtons
pounds (force) per square inch	6.894757	kilopascals
pounds (mass)	0.45359237	kilograms
pounds (mass) per cubic foot	16.01846	kilograms per cubic meter
yards	0.9144	meters

1 Introduction

1.1 Background

Rapid test methods have been developed with the goal of reducing the time needed to obtain results for determining potential alkali-silica reaction (ASR) in concrete aggregates. The most commonly used test methods for this purpose are the ASTM C1293 (ASTM International 2015) concrete prism test and the ASTM C1260 (ASTM International 2014) accelerated mortar bar test. ASTM C1293 requires one year to acquire expansion results using ordinary portland cement concrete. If testing the effectiveness of mitigation measures, such as the use of supplementary cementitious materials, the duration of testing is extended to two years. ASTM C1293 is widely accepted as the most reliable standardized laboratory test method for aggregate reactivity and, therefore, serves as a benchmark for accelerated test methods (Lu et al. 2008). However, due to its long duration, it is used primarily as a research tool and not as a routine test method in practice. ASTM C1260 requires only 16 days to complete the test. Despite its rapidity and frequent use in industry, ASTM C1260 fails to identify some aggregates known to be reactive, a situation referred to as a false negative. Furthermore, ASTM C1260 produces a large number of false positives, meaning the aggregate appears reactive in the test but has not proven to be reactive otherwise.

Even the relatively short duration of the ASTM C1260 test method is often too long to support military construction operations. In some cases, the timeline between project identification, contracting, design and specification development, and construction may not allow for multiple weeks or months for materials testing. In the case of contingency military concrete construction operations, even more rapid evaluation of materials is desired. The result of these application-centric drivers is the constant need for rapid evaluation of materials through novel test methods that provide data in a matter of days rather than weeks or months.

A number of autoclave test methods have been developed for ultra-rapid testing for ASR in concrete aggregates (Tang et al. 1983; Tamura 1987; Nishibayashi et al. 1987; Fournier et al. 1991; Nishibayashi et al. 1996; Giannini and Folliard 2013). These methods satisfy the timelines desired for rapid military construction, with testing completed in days, including aggregate preparation, mixing, curing, and testing. Some of these methods emphasized testing a large number of aggregates while others focused on evaluating the effects of several different test parameters on specimen expansions while testing a relatively small number of aggregates. Alkali loadings in these studies were between 1.5 to 3.5% by mass of cement, and autoclaving temperatures were between 111 and 150°C. All of the test methods limited the autoclaving durations to between 2 and 6 hr for mortar bars and between 4 and 24 hr for concrete prisms. The method described by Fournier et al. (1991) and Bérubé et al. (1992) was found to be particularly promising. The single-operator coefficient of variation was very low (4.4%) for the first phase of the development of the test method, which included 40 aggregates. The test method produced reactivity classifications in agreement with ASTM C1293 81% of the time based on a total of 106 aggregates tested and was the basis for the testing performed in Part II of this study.

1.2 Research scope

This study is divided into two parts. The objective of Part I was to determine appropriate parameters for alkali loading, autoclaving temperature, and autoclaving duration for silica dissolution in aggregates of different mineralogies and reactivity classifications in autoclave testing for ASR in mortar bars. Initially, 20 aggregates were tested according to ASTM C1260 and the 5-hr autoclaved mortar bar test described by Fournier et al. (1991). Eight of those aggregates plus one additional aggregate were tested in the autoclave using different test parameters including alkali loadings between 0.52 and 4.5%, autoclaving temperatures ranging from 105 to 130°C, and autoclaving durations between 5 and 48 hr. These test parameters and their effects on specimen expansion expand upon what is documented in the literature. Recommendations for autoclaved mortar bar testing are provided based on expansion results in this study.

Part II involved a multi-laboratory autoclave study with the objective of determining whether or not the autoclave method is a suitable test method for identifying alkali-silica reactive aggregates used in rapid construction of short-life structures. Additionally, the reproducibility of the test method was evaluated. Mortar bars with boosted equivalent alkali contents were autoclaved for 5 hr, and expansions were measured. The research was led by the U.S. Army Engineer Research and Development Center's (ERDC's) Geotechnical and Structures Laboratory. Four other laboratories at The University of Alabama, The University of Texas at Austin, Clemson University, and CTL Group also participated in the study.

2 Materials and Methods

2.1 Materials

2.1.1 Aggregates

The aggregates were provided by ERDC and are listed in Table 1, including the aggregate abbreviations, size fractions, mineralogies based on x-ray diffraction (XRD), results of ASTM C1260 tests performed at ERDC, and ASTM C1293 expansions from ERDC and the literature (Folliard et al. 2006, Leśnicki et al. 2013, Latifee and Rangaraju 2015). To test the coarse aggregates in mortar bars, the aggregates were crushed into fine fractions. Reactivity classifications based on ASTM C1778 guidance on interpreting 14-day ASTM C1260 expansions are also provided in the table.

Table 1. Summary of aggregates used in this study.

Aggregate Abbreviation	Coarse/ Fine	Rock Type; Mineralogy	ASTM C1260 Expansion, %		ASTM C1293 Expansion, %
			14 Days	28 Days	
Non-reactive[1]					
NR1	Coarse	Oolitic to dense limestone; calcite, dolomite, quartz	0.022	0.035	0.030
NR2	Coarse	Dolostone; dolomite	0.013	0.026	0.034
NR3	Fine	Feldspar-rich gravel; anorthite, albite, quartz, microcline, biotite	0.085	0.117	...
Moderately Reactive[1]					
MR1	Coarse	Dense limestone; calcite, dolomite, quartz, trace feldspar	0.098	0.156	0.106
MR2	Coarse	Intermediate to felsic igneous rock; anorthite, quartz, magnesiohornblende, albite, orthoclase, biotite, clinopyroxene	0.117	0.229	...
MR3	Fine	Quartz sand; quartz	0.169	0.324	...
MR4	Fine	Quartz sand; quartz, kaolinite	0.217	0.337	0.109
MR5	Fine	Quartz gravel; quartz, orthoclase, iron	0.226	0.322	...
MR6	Coarse	Green schist, quartzite, and granite mixture; quartz, clinochlore, albite, muscovite, anorthite, fluor-riebeckite	0.256	0.418	0.144
MR7	Coarse	Quartz limestone; quartz, orthoclase, dolomite	0.293	0.451	...

Aggregate Abbreviation	Coarse/ Fine	Rock Type; Mineralogy	ASTM C1260 Expansion, %		ASTM C1293 Expansion, %
			14 Days	28 Days	
Highly Reactive[1]					
HR1	Coarse	Metapelite green schist; muscovite, clinochlore, quartz, albite, calcite, orthoclase	0.325	0.482	0.192
HR2	Fine	Complex gravel with intermediate to felsic composition aggregates; quartz, orthoclase, albite, clinochlore, muscovite, dolomite, actinolite, calcite	0.349	0.489	...
HR3	Coarse	Dense limestone; calcite, dolomite, quartz	0.399	0.524	0.204
HR4	Coarse	Dolostone; dolomite, calcite, quartz	0.399	0.487	...
HR5	Fine	Quartz gravel; quartz, microcline	0.405	0.537	...
HR6	Coarse	Complex alluvial gravel with granite/rhyolite, basalt, welded tuff, chert, amber; microcline, quartz, calcite, albite, muscovite, cristobalite, hematite	0.413	0.613	0.590
Very Highly Reactive[1]					
VHR1	Fine	Quartz sand; quartz, albite	0.458	0.625	...
VHR2	Coarse	Dense dolostone; dolomite, quartz, calcite, microcline	0.527	0.756	0.167
VHR3	Fine	Complex gravel; quartz, calcite, anorthite, orthoclase, cristobalite, stilbite, clinochlore	0.576	0.767	...
VHR4	Coarse	Quartzite and granite mixture; albite, quartz, muscovite, orthoclase, calcite, cristobalite, actinolite	1.016	1.255	0.251

[1] ASTM C1778 expansion limits for mortar bars in the ASTM C1260.

NR – Non-reactive: expansion < 0.10%

MR – Moderately-reactive: 0.10% ≤ expansion < 0.30%

HR – Highly-reactive: 0.30% ≤ expansion < 0.45%

VHR – Very-highly-reactive: 0.45% ≤ expansion

2.1.2 Cements

Two ASTM C150 Type I/II cements with similar equivalent alkali contents (Na_2O_{eq}) of 0.52 and 0.56% by mass of cement were used for all mixtures. Two cements were used in this study due to the limited supply of one of the cements. Previous work demonstrated the negligible effect of cement alkalinity on expansion in autoclaved concrete prism testing (Wood et al. 2016). Results of the cement oxide analyses are provided in Table 2.

Table 2. Cement oxide analysis.

Oxide Notation	% Weight	
	Cement 1	Cement 2
SiO_2	18.99	20.2
Al_2O_3	4.44	4.7
Fe_2O_3	3.34	2.9
CaO	67.72	62.8
MgO	2.63	2.4
SO_3	3.44	3.0
Loss on Ignition	3.06	2.5
Na_2O_{eq}	0.52	0.56

2.2 Methods

2.2.1 Autoclave test method

2.2.1.1 Part I: examination of test parameters

The mixture proportions, aggregate gradations, w/cm (0.47), and specimen size (25 x 25 x 285 mm) matched those specified by ASTM C1260. Sodium hydroxide (NaOH) was added to the mixing water to boost the Na_2O_{eq} to the desired alkali loading. Four bars were cast for each mixture with metal studs embedded in the ends for length-change measurements per ASTM C157. All mortar bars were cured for 48 hr inside a moist curing room before they were demolded, measured, and autoclaved over a reservoir of deionized water in a Yamato SQ510C sterilizer. Once the autoclave temperature cooled to 90°C, the bars were removed and cooled to 23°C in a tap-water bath over a period of approximately 15 min. The lengths of the mortar bars were measured once again to determine the expansion after autoclaving. Giannini and Folliard (2013) provided details on the autoclaving procedure followed in this study.

The influence of three test parameters on the reactivities of the aggregates, as indicated by the expansion of the mortar bars, were investigated, i.e., (1) total alkali loading, or Na_2O_{eq}, (2) autoclaving temperature, and (3) autoclaving duration. Test parameter variations included alkali loadings of 0.52, 0.56, 2.0, 2.5, 3.0, 3.5, and 4.5%, autoclaving temperatures of 105, 110, 115, 120, and 130°C, and autoclaving durations of 5, 8, 12, 16, 24, and 48 hr. Autoclaving duration is the amount of time the autoclave remained at peak temperature.

2.2.1.2 Part II: Round robin study

Aggregates were graded and proportioned by ERDC in accordance with ASTM C1260 and provided to the other laboratories. The material proportions, i.e., w/cm of 0.47 and specimen size of 25 by 25 by 285 mm, were also in accordance with ASTM C1260. The Na_2O_{eq} of the mixtures was boosted to 3.5% by mass of cement by adding sodium hydroxide (NaOH) to deionized mixing water. Four bars were formed from each mixture, and metal gauge studs were embedded in the ends to measure expansions using a length-change comparator in accordance with ASTM C157.

Immediately after casting, the mortar bars were cured for 48 hr inside a moist curing room at 23°C before they were demolded. An initial length-change measurement was made at 23°C after demolding the mortar bars. The bars were then autoclaved for 5 hr at 130°C (0.17 MPa gauge pressure), following the same parameters used by Fournier et al. (1991).

Once the autoclave cooled to 90°C, the mortar bars were removed, placed in a pot of hot water, and cooled to 23°C by overflowing the pot with running water over a period of approximately 15 min, similar to the cooling step described in ASTM C151. The temperature of the hot water as the cooling process began was approximately 90°C. Final length-change measurements were taken after cooling the bars to 23°C.

The amount of alkali leaching for select sets of mortar bars was determined at Laboratory 2 by sampling the autoclave water after autoclaving and measuring the concentration of alkalis via inductively coupled plasma optical emission spectrometry (ICP-OES). The concentrations were compared to the initial alkali loading of the mortar bars.

2.2.2 Aggregate characterization

2.2.2.1 X-ray diffraction (XRD)

The mineralogy of the aggregate samples was determined using X-ray diffraction (XRD) analysis. In preparation for XRD analysis, a portion of the sample was ground in a Pulverisette (Fritsch Co., Idar-Oberstein, Germany) and passed through a 45-µm (No. 325) sieve. Random orientation powder mounts of bulk samples were analyzed using XRD to determine the mineral constituents present in each sample. XRD patterns were gathered from an X-Pert Pro Multipurpose Powder Diffractometer system that used standard techniques for phase identification (Panalytical

Inc.). The run conditions included Co-Kα radiation, scanning from 2 to 70 ° 2θ with a step size less than 0.02 and collection of the diffraction patterns accomplished using the PC-based Windows version of X-Pert Pro Data Collector, and analysis of the patterns using the Jade2010 program (Materials Data Inc.).

2.2.2.2 Petrographic analysis

The samples were examined in accordance with ASTM C 295, *Standard Guide for Petrographic Examination of Aggregates for Concrete* with the modification on the number of particles examined. ASTM C 295 recommends that a minimum of 150 particles in each aggregate sieve fraction should be studied and their mineralogy identified. Generally, 300 particles per sieve fraction were identified, when available, to decrease the statistical degree of error. The Rock Color Chart from the Geological Society of America was used to determine the color of the aggregates. The colors were identified and then labeled using the same identification as the Rock Color Chart. The identification of the aggregate colors is helpful for future examination of hardened concrete.

2.2.2.3 Refractive index

The samples were placed in immersion oil with a 1.544 refractive index. For fine aggregates, portions of the 600-µm (No. 30) sieve size were ground into powder mounts and examined to determine whether the quartz grains had an index of refraction (IOR) greater than or less than 1.544. Some of the more common phases that may be potentially reactive in portland cement concrete include fused quartz (IOR=1.4583), pure chalcedony (IOR=1.537), and chalcedonic chert (IOR=1.530 - 1.535). Commonly, these phases are identified with quartz. Since the indices of refraction for these siliceous phases are lower than that of quartz (IOR=1.544), this analysis is a common way to discern these phases.

2.2.3 Alkali leaching

Following autoclave testing, water from the bottom of the autoclave was collected and analyzed to determine the amount of alkalis that had leached from specimens during testing. Alkali metal contents (i.e., Na, K, and Ca) were measured using inductively coupled plasma spectroscopy methods. The concentration of alkalis measured was extrapolated to the total mass of alkalis in solution to compare to the total alkali loading in test specimens.

3 Part I: Examination of Test Parameters

3.1 Mortar bar expansions

Average mortar bar expansions under the different test parameters for all aggregates except NR1 and HR3, the benchmark aggregates, are provided in Table 3, along with coefficients of variations (CVs), and are further illustrated in Figures 1-3. The autoclave expansion results for the first six aggregates were compared to the ASTM C1260 expansions for these aggregates at 14 days. The average expansions and CVs were single-operator except where they were based on expansion data from two laboratories for the initial 5-hr autoclave test. Some tests used only 2 or 3 mortar bars because specimens occasionally broke during demolding. In the figures, the horizontal dashed lines signify the expansion limits provided by Fournier et al. (1991). Because these limits were developed for autoclave testing using specific parameters (3.5% alkali loading at 130°C for 5 hr), they are used in this paper as merely a guide when different test parameters were applied. Expansions less than 0.150% suggest the aggregates are non-reactive (NR). Expansions equal to or greater than 0.150% indicate reactive (R) aggregates. Expansions greater than or equal to 0.250% denote highly reactive (HR) aggregates. The vertical dashed lines in the figures indicate the expansion limits recommended by ASTM C1778 based on 14-day expansions in ASTM C1260. Error bars show the range of expansion for each set of mortar bars. Because test parameters used for NR1 and HR3 mortar bars are dissimilar from those used for testing the other aggregates, data for those aggregates are presented and discussed separately.

Table 3. Test parameters, average mortar bar expansions, and coefficients of variation (CVs).

Alkali Loading, Temperature, Duration	Average Expansion, % and CV, %					
	NR2	MR1	MR2	HR1	HR2	VHR1
2.5%, 130 °C, 5h	0.038, 5.5	0.065, 2.3	0.033, 3.0[b]	0.232, 2.0	0.061, 4.7	0.532, 4.4
3.5%, 110 °C, 5h	0.045, 1.3[b]	0.085, 1.7	0.225, 2.2	0.440, 17.8	0.195, 4.6	0.669, 4.1[b]
3.5%, 120 °C, 5h	0.055, 1.7	0.134, 2.1[b]	0.277, 14.6	0.492, 3.9	0.316, 2.4	0.808, 1.9
3.5%, 130 °C, 5h	0.082, 5.4*	0.188, 8.3*	0.307, 8.2*	0.566, 7.0*	0.371, 10.4*	0.796, 3.7*
3.5%, 130 °C, 24h	0.337, 5.7	0.272, 3.9	0.416, 4.4	0.745, 7.6	0.660, 10.0[b]	1.003, 5.4
4.5%, 130 °C, 5h	0.092, 1.9[b]	0.255, 2.1	0.311, 3.0[a]	0.620, 2.3[b]	0.448, 2.5	0.769, 2.2

* Expansion values and CVs based on data from two laboratories.

[a] Two mortar bars tested.

[b] Three mortar bars tested.

Figure 1. Autoclave expansions versus 14-Day ASTM C1260 expansions. Alkali loading, autoclaving temperature, and autoclaving duration were (a) 2.5%, 130°C, and 5 hr and (b) 3.5%, 110°C, and 5 hr.

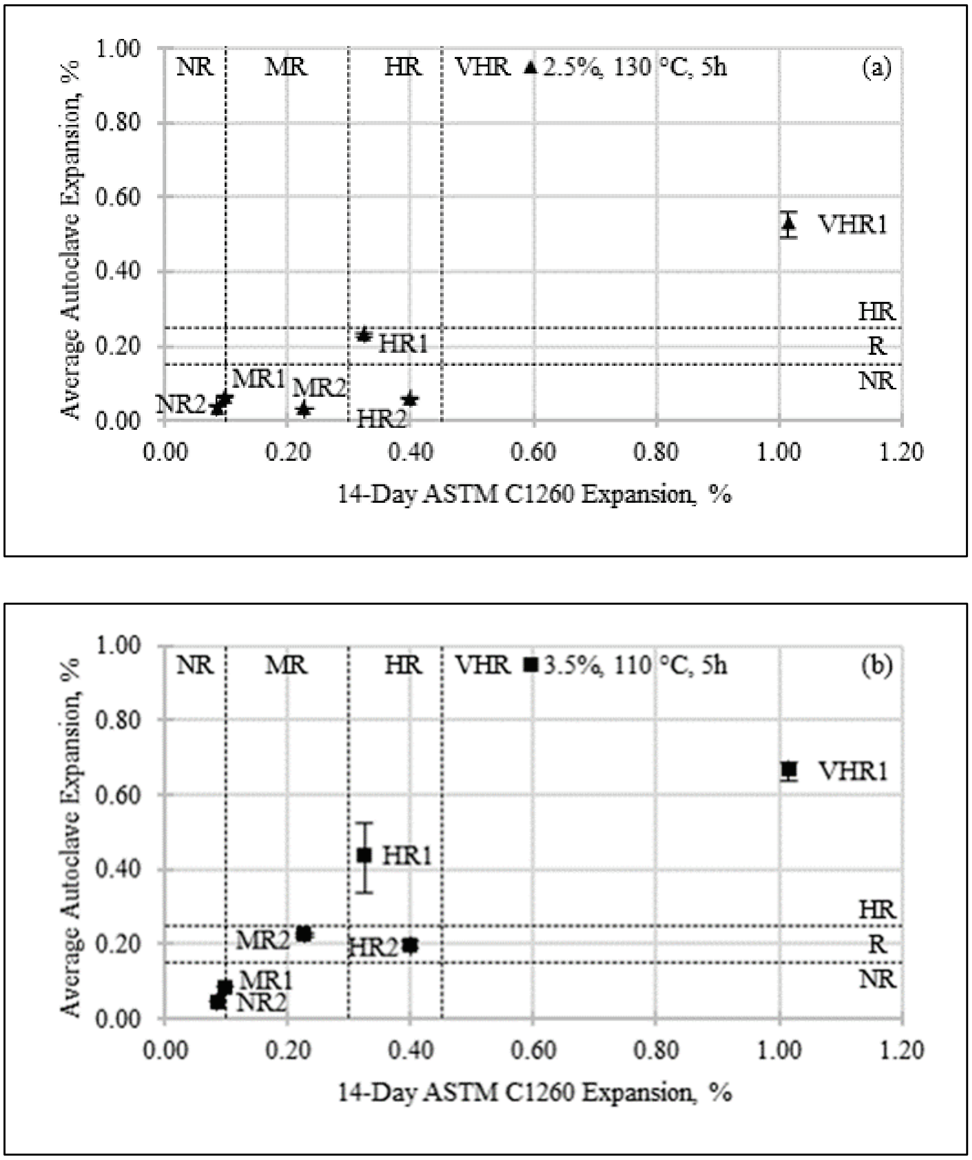
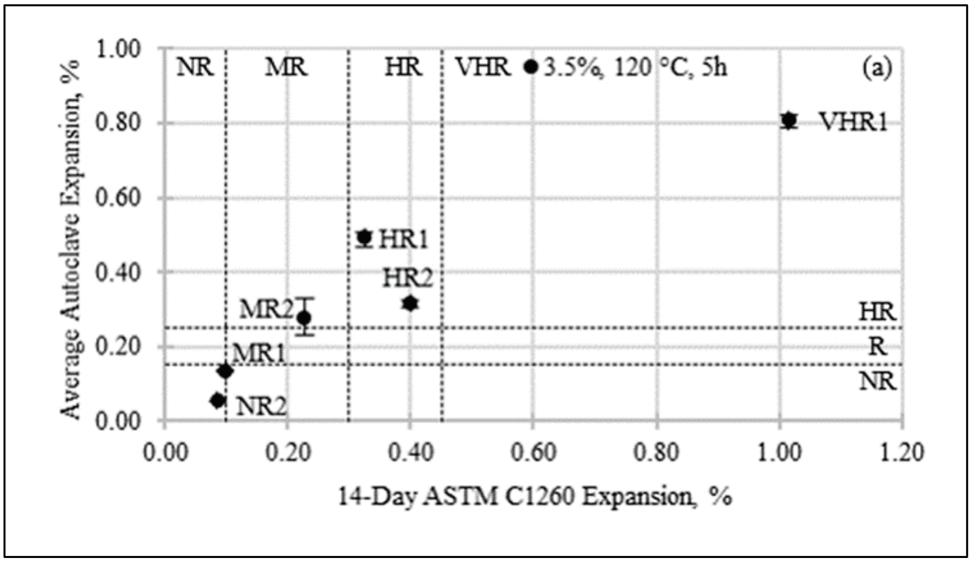


Figure 2. Autoclave expansions versus 14-Day ASTM C1260 expansions. Alkali loading, autoclaving temperature, and autoclaving duration were (a) 3.5%, 120°C, and 5 hr and (b) 3.5%, 130°C, and 5 hr.



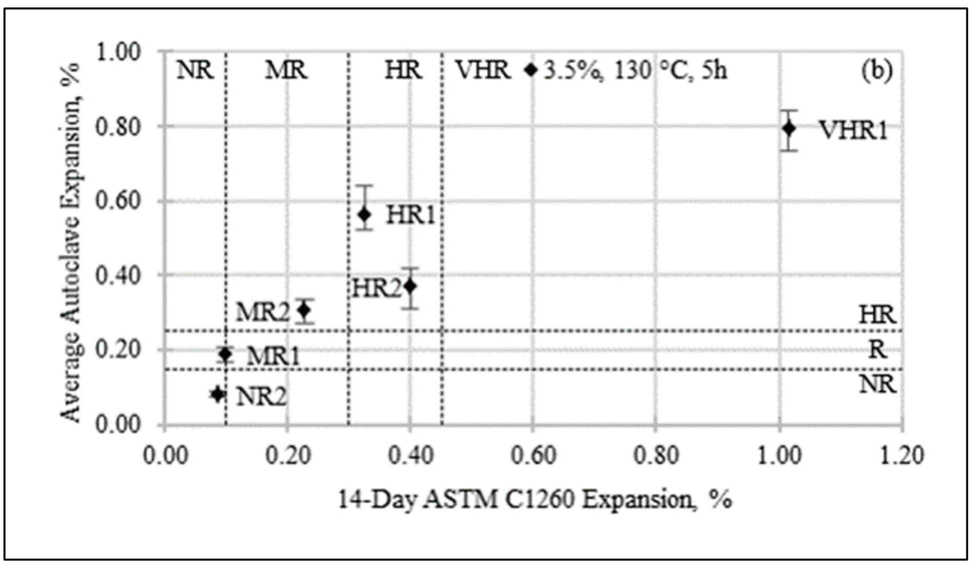
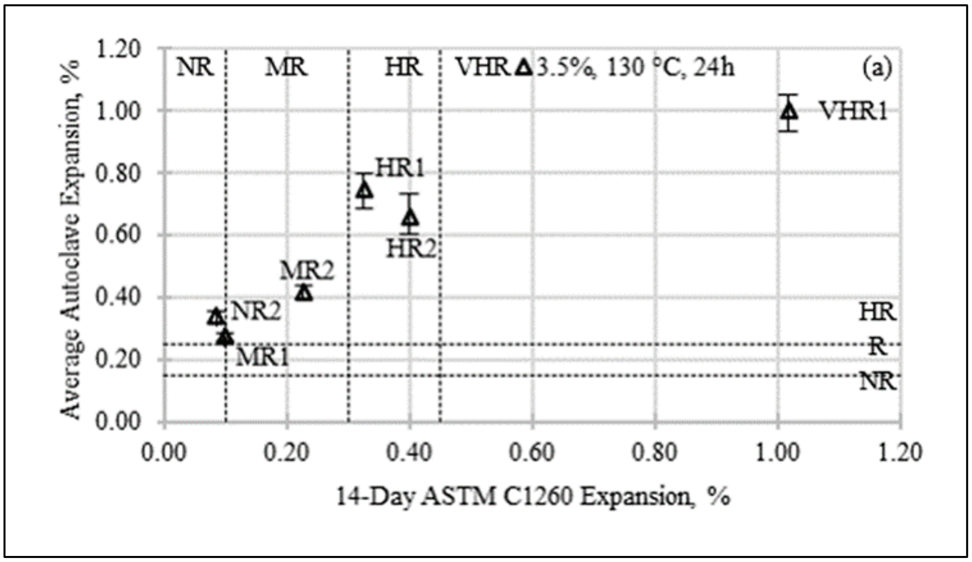


Figure 3. Autoclave expansions versus 14-Day ASTM C1260 expansions. Alkali loading, autoclaving temperature, and autoclaving duration were (a) 3.5%, 130°C, and 24 hr and (b) 4.5%, 130°C, and 5 hr.



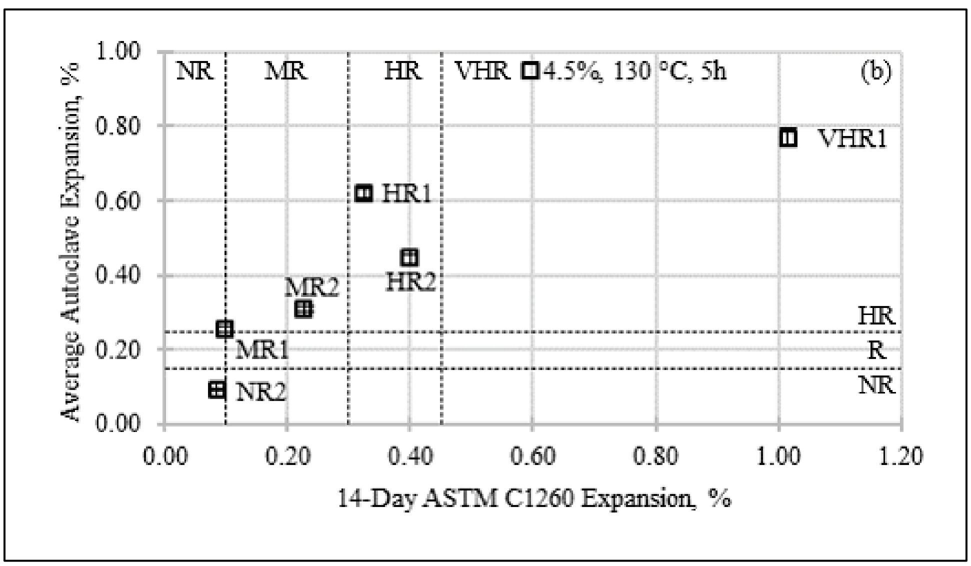


Figure 1 shows that 2.0% alkali loading and 110°C autoclaving temperature failed to distinguish between reactive and non-reactive aggregates. When 2.0% Na_2O_{eq} was used, MR1, MR2, and HR2 all appeared to be non-reactive with expansions less than 0.10%. At 110°C, even with 3.5% Na_2O_{eq}, MR1 expansion was less than 0.10%, and MR2 expansion was higher than HR2 expansion.

The test parameters shown in Figure 2 were more in line with ASTM C1260 classifications for these aggregates. Figures 2a and 2b are quite similar, but the difference between the effects of 120 and 130°C autoclaving temperatures is revealed in the expansion of MR1. At 120°C, MR1 exhibited non-reactive expansion, but at 130°C, the aggregate produced expansion that was more definitively reactive. Of the sets of parameters used in this study for these six aggregates, using 3.0% Na_2O_{eq}, 130°C autoclaving temperature, and 5 hr autoclaving duration most effectively sorted aggregates by their ASTM C1260 performance.

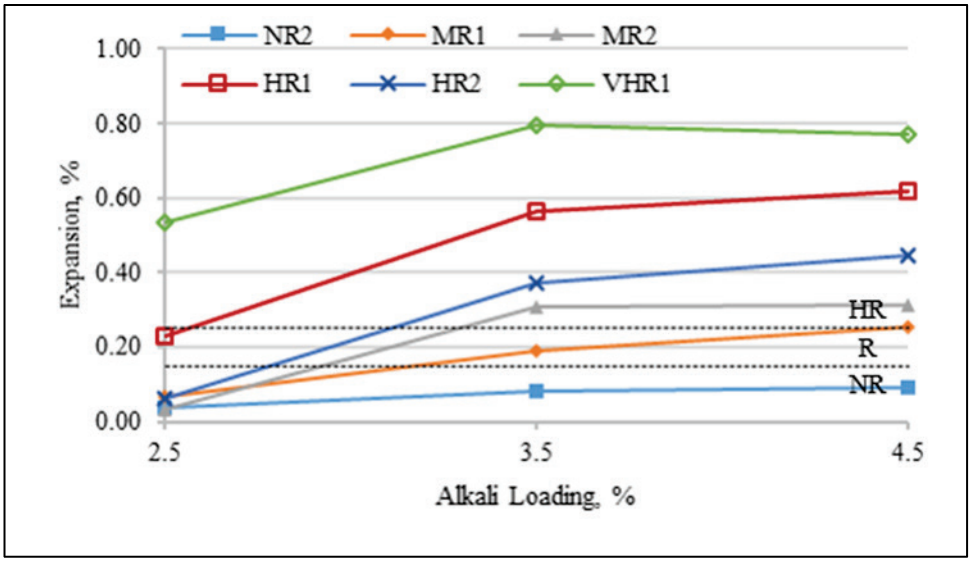
Figure 3 shows mortar bar expansions based on more severe test parameters. When the mortar bars were conditioned for 24 hr, all six of the aggregates expanded beyond 0.25%, and NR2 expansion surpassed MR1 expansion. Increasing the alkali loading to 4.5% produced higher expansions for aggregates MR1 and MR2, but expansion of NR2 remained relatively low.

In every set of test parameters except for the sets involving 2.0% alkali loading and 110°C autoclaving temperature, aggregate MR2 generated expansions higher than expected. Another sample of aggregate from the same quarry produced higher ASTM C1260 expansion at 14 days (0.405%) and was categorized as highly reactive. Both samples exhibited similar autoclave expansions. It is possible that ASTM C1260 does not achieve consistent results for this aggregate.

3.2 Influence of alkali loading

Figure 4 shows the influence of alkali loading on mortar bar expansion when autoclaving temperature and duration remain constant at 130°C and 5 hr. As in the previous figures, the horizontal dashed lines represent the 0.150 and 0.250% expansion limits proposed by Fournier et al. (1991). The solid lines connecting data points exist as guides only and do not indicate linear relationships among the data.

Figure 4. Influence of alkali loading on mortar bar expansion when autoclaving temperature and duration remain constant at 130°C and 5 hr.



The most influential parameter modification on expansion for these aggregates was the increase from 2.5 to 3.5% alkali loading at 130°C for 5 hr; mortar bar expansions at 3.5% alkali loading were 4.07 times greater, on average, compared to expansions at 2.5% alkali loading. Aggregate VHR1 exhibited the least amount of change, increasing in expansion by a factor of 1.50; aggregate MR2 exhibited the greatest change, increasing in expansion by a factor of 9.32. Fournier et al. (1991) observed an increase in mortar bar expansions when the Na_2O_{eq} was increased from 2.5 to 3.5% for reactive aggregates but practically no effect on mortar bars with non-reactive aggregates. For aggregates in this study, except the least reactive (NR2) and most reactive (VHR1), an alkali loading of 2.5% failed to discriminate between non-reactive and reactive aggregates and is considered to be too low for autoclave testing for ASR at these temperatures and durations.

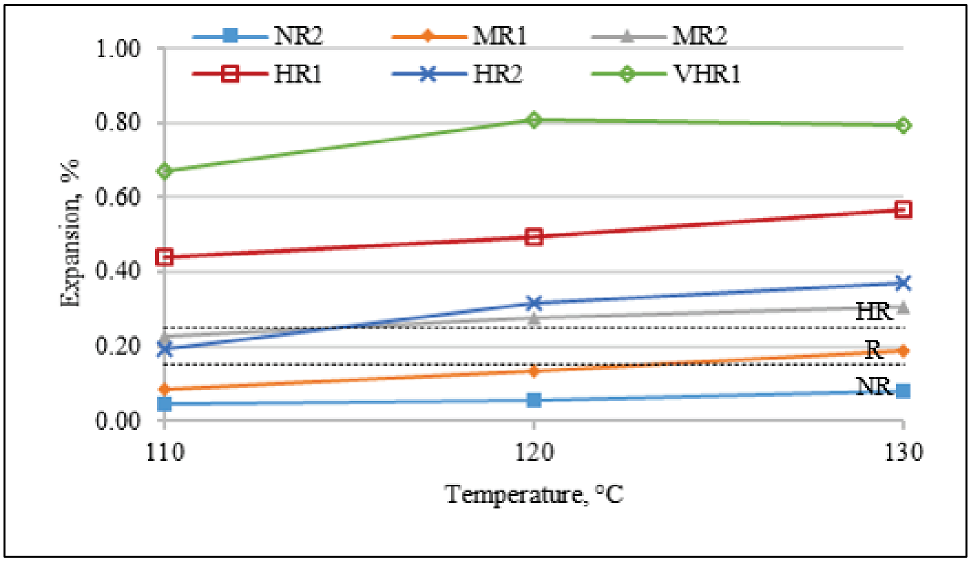
Increasing the alkali loading from 3.5 to 4.5% at 130°C for 5 hr had significantly less impact on the resulting mortar bar expansions. For the six aggregates tested, expansions at 4.5% alkali loading were, on average, increased by a factor of 1.13 compared to 3.5% alkali loading. One aggregate, VHR1, exhibited a slightly reduced expansion for this increase in overall alkalinity. Aggregate MR1 was the only aggregate to produce a somewhat significant increase in expansion (factor of 1.36) when the alkali loading was raised from 3.5 to 4.5%. Although the effect of elevating the Na_2O_{eq} of the mixtures from 3.5 to 4.5% had minimal effect on expansions for most aggregates, the modification may be the difference between yielding reactive or highly reactive classifications, as it was for aggregate MR1, which is an incorrect classification of this aggregate. Because increasing the alkali loading to 4.5% proved to be generally ineffective and in one case, extreme, it is suggested alkali loadings not exceed 3.5% in autoclave tests.

In the literature, there are documented pessimum alkali concentrations for storage solutions, which are generally 1 mol/L NaOH or NaCl for storage temperatures between 20 and 60°C (Kuroda et al. 2000; Kuroda et al. 2012; Chatterji and Christensen 1990). Kagimoto et al. (2014) studied alkali loadings ranging from about 1.1 to 3.0% in concrete prisms stored at 40°C for 182 days and observed pessimum behavior at 2.2% alkali loading. Alkali loadings in this part of the study, however, exceeded 2.2%, and it appears that a pessimum alkali loading was not yet reached for most of the aggregates tested. The literature does not address pessimum behavior with alkali loadings this high. Additionally, temperatures used in this study exceed those in the literature. The higher temperatures appear to have increased the pessimum alkali loading beyond those used here.

3.3 Influence of autoclaving temperature

Figure 5 shows the influence of autoclaving temperature on expansion when alkali loading and autoclaving duration remain constant at 3.5% and 5 hr. For these six aggregates, there was an average increase in mortar bar expansion by a factor of 1.33 when the temperature of the autoclave was increased from 110 to 120°C at 3.5% Na_2O_{eq} for 5 hr. The expansions of aggregates MR1, MR2, and HR2 corresponded to less severe reactivity classifications when the autoclaving temperatures were lowered to 110°C. This suggests that autoclaving temperatures at 110°C or less are too low for rapid ASR testing in the autoclave using 3.5% Na_2O_{eq} and a 5-hr duration. The temperature increase from 120 to 130°C improved discrimination between non-reactive and reactive aggregates, particularly for aggregate MR1 whose expansion increased by a factor of 1.40.

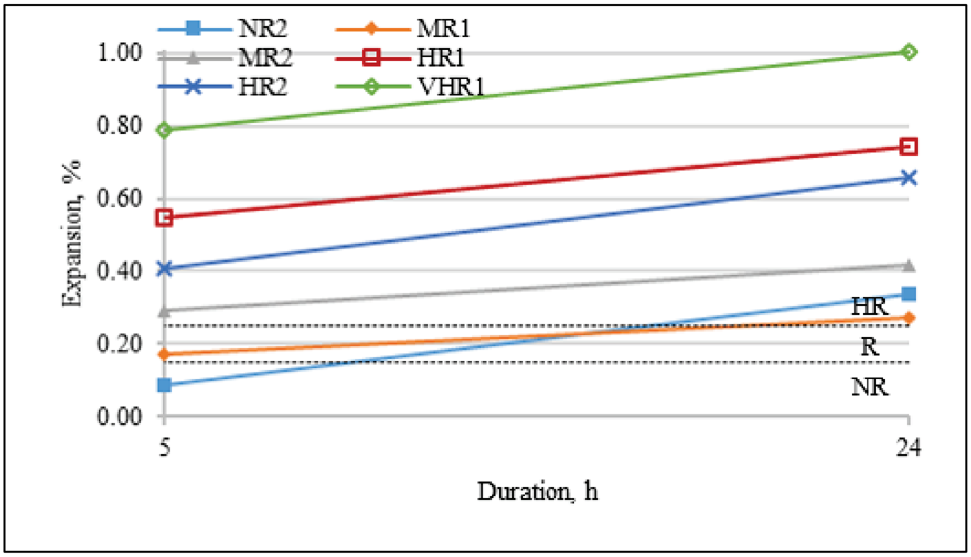
Figure 5. Influence of autoclaving temperature on mortar bar expansion when alkali loading and autoclaving duration remain constant at 3.5% and 5 hr.



3.4 Influence of autoclaving duration

Figure 6 presents the effect of autoclaving duration on mortar bar expansion when alkali loading and autoclaving temperature remained constant at 3.5% and 130°C. When the autoclaving duration was increased from 5 to 24 hr with 3.5% Na_2O_{eq} at 130°C, the mortar bar expansions increased by a factor of 1.88, on average. Aggregate NR2 was most affected by this change in duration with expansion experiencing an increase by a factor of 4.13 and exceeding the expansion of MR1. Because of this lack of distinction between a non-reactive and reactive aggregate, it is suggested that 24-hr autoclaving durations be avoided when testing mortar bars of this size.

Figure 6. Influence of autoclaving duration on mortar bar expansion when alkali loading and autoclaving temperature remain constant at 3.5% and 130°C.



A summary of the changes in expansion resulting from modifications of the test parameters are provided in Table 4. The parameters in bold were altered while the other parameters listed were held constant. For instance, in the second column of the table, the differences in expansion are shown for an increase in alkali loading from 2.5 to 3.5% at 130°C for 5 hr; the factor is the expansion at 3.5% alkali loading divided by the expansion at 2.5% alkali loading.

Table 4. Summary of influence of test parameter modifications on mortar bar expansions.

Aggregate	Change in Expansion, %				
	2.5 to 3.5% 130°C 5 hr	3.5 to 4.5% 130°C 5 hr	3.5% 110 to 120°C 5 hr	3.5% 120 to 130°C 5 hr	3.5% 130°C 5 to 24 hr
NR2	2.16	1.13	1.22	1.48	4.13
MR1	2.90	1.36	1.58	1.40	1.45
MR2	9.32	1.01	1.23	1.11	1.35
HR1	2.44	1.10	1.12	1.15	1.32
HR2	6.11	0.21	1.61	1.17	1.78
VHR1	1.50	0.97	1.21	0.98	1.26
Average	4.07	1.13	1.33	1.21	1.88

3.5 Benchmark aggregates

The average mortar bar expansions of the NR1 and HR3 aggregates, which were tested more extensively, are given in Tables 5 and 6 and are illustrated in Figures 7a and 7b, respectively. As in the previous figures, the horizontal dashed lines define the expansion limits described by Fournier et al. (1991) for the 5-hr autoclave test. The data symbols indicate the levels of alkali loading, e.g., 2.5%, and the autoclaving duration, in hours, is provided next to each data point, e.g., 5h. Test parameters for these aggregates were selected to push the boundaries of their known reactivity classifications. As expansion results were obtained, more or less severe test parameters were used in order to locate threshold values for alkali loading, autoclaving temperature, and autoclaving duration for these aggregates.

Table 5. Test parameters, average mortar bar expansions, and coefficients of variation (CVs) for aggregate NR1.

Alkali Loading, Temperature, Duration	Average Expansion, %	CV, %
2.5%, 105 °C, 16h	0.016[a]	0.0[a]
3.0%, 105 °C, 16h	0.009[b]	17.6[b]
3.0%, 105 °C, 24h	0.034	5.0
3.0%, 105 °C, 48h	0.033	4.6
3.0%, 115 °C, 6h	0.012[a]	11.8[a]
3.0%, 115 °C, 48h	0.049	19.9
3.0%, 130 °C, 5h	0.029	4.3
3.0%, 130 °C, 12h	0.057	6.9
3.0%, 130 °C, 16h	0.105	4.0
3.0%, 130 °C, 24h	0.179	4.9
3.0%, 130 °C, 48h	0.325	3.7
3.5%, 120 °C, 8h	0.037[b]	5.6[b]
3.5%, 130 °C, 8h	0.044[b]	4.5[b]

[a] Two mortar bars tested.

[b] Three mortar bars tested.

Table 6. Test parameters, average mortar bar expansions, and coefficients of variation (CVs) for aggregate HR3.

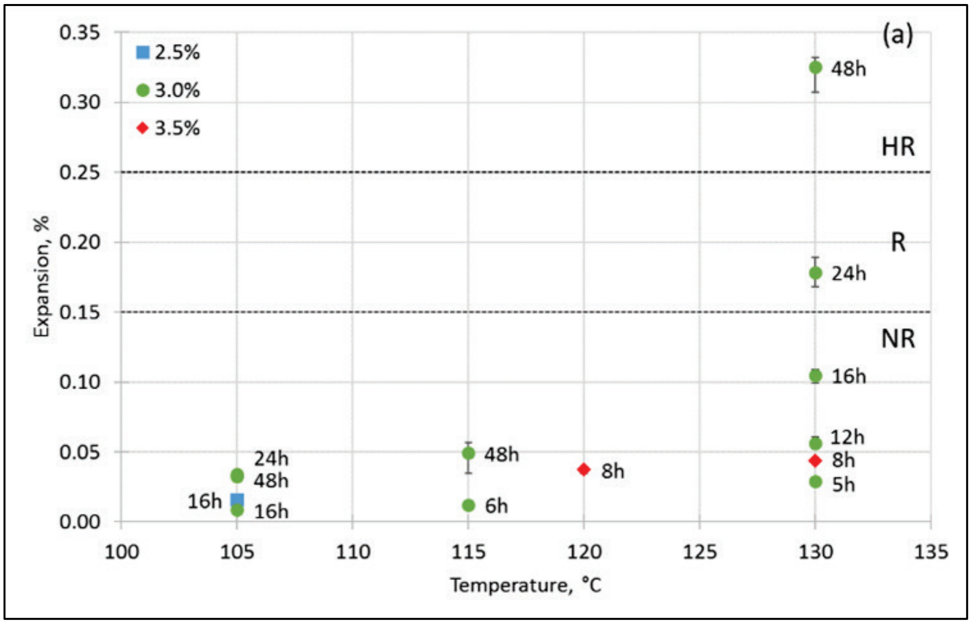
Alkali Loading, Temperature, Duration	Average Expansion, %	CV, %
0.52%, 105 °C, 24h	0.020	6.2
0.52%, 105 °C, 48h	0.018	5.4
0.52%, 115 °C, 48h	0.019	4.3
0.56%, 130 °C, 24h	0.022	5.7
0.56%, 130 °C, 48h	0.025	2.0
1.0%, 130 °C, 24h	0.025	2.0
1.5%, 130 °C, 24h	0.048	2.9
2.0%, 115 °C, 24h	0.407	29.4
2.0%, 130 °C, 5h	0.269	21.3
2.0%, 130 °C, 24h	0.433	5.7
3.0%, 105 °C, 5h	0.332	7.6
3.0%, 105 °C, 16h	0.460[a]	4.3[a]
3.0%, 115 °C, 6h	0.523[b]	8.0[b]
3.0%, 130 °C, 5h	0.531	2.1
3.5%, 120 °C, 8h	0.555[b]	1.1[b]
3.5%, 130 °C, 5h	0.543*	4.8*

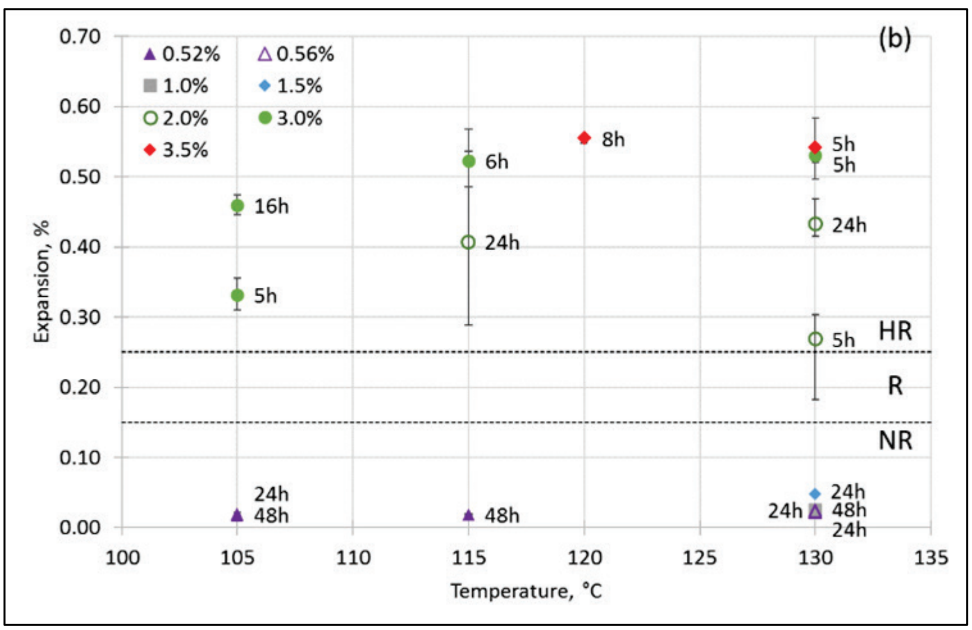
* Expansion value and CV based on data from two laboratories.

[a] Two mortar bars tested.

[b] Three mortar bars tested.

Figure 7. Average mortar bar expansions using different test parameters for (a) NR1 and (b) HR3.





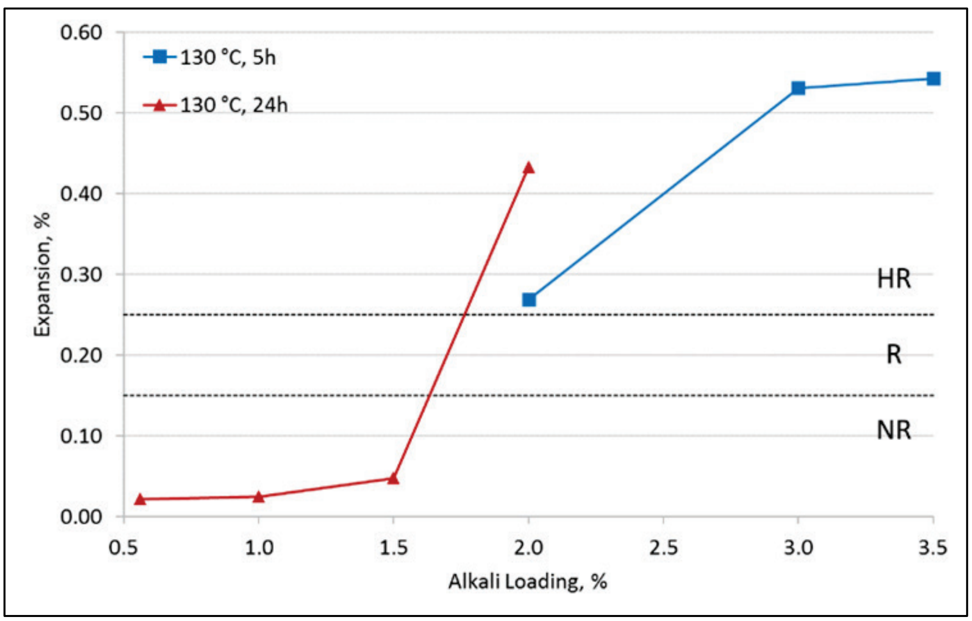
The highest average mortar bar expansion observed for aggregate NR1 (0.325%) occurred at 130°C with 3.0% Na_2O_{eq} conditioned for 48 hr. This was one of only two instances in which NR1 reached levels of expansion exceeding the proposed threshold for reactive aggregates in this study; it also appeared to be reactive when tested at 130°C using 3.0% Na_2O_{eq} for 24 hr.

3.5.1 Influence of alkali loading

Mortar bars with NR1 showed an average reduction in expansion by a factor of 0.56 when alkali loading was increased from 2.5 to 3.0% at 105°C for 16 hr. This reduction is not considered to be significant because both expansion values are quite small (< 0.02%).

Figure 8 demonstrates the effect of alkali loading on mortar bar expansion for aggregate HR3. An increase in alkali loading from 3.0 to 3.5% at 130°C for 5 hr had essentially no effect on expansion for this aggregate. However, in order to produce expansions for HR3 that fall in the reactive ranges, it is clear that some amount of alkali loading is necessary. Expansions remained well below 0.010% until alkali loading was increased from 1.5 to 2.0%, resulting in an increase in expansion by a factor of 8.48. Raising the alkali loading from 2.0 to 3.0% at 130°C for 5 hr increased the expansion by a factor of 1.97. The alkali loading affected the expansion of HR3 more than temperature or autoclaving duration.

Figure 8. Influence of alkali loading on expansion of mortar bars containing HR3.



Although alkali leaching was not measured in this study, mortar bars with aggregate HR3 and alkali boosting up to 1.5% may have leached alkalis, resulting in insufficient alkalis in the mortar bars to promote ASR. A previous study by the authors showed alkali leaching between 4 and 11% for autoclaved concrete prisms (Wood et al. 2016).

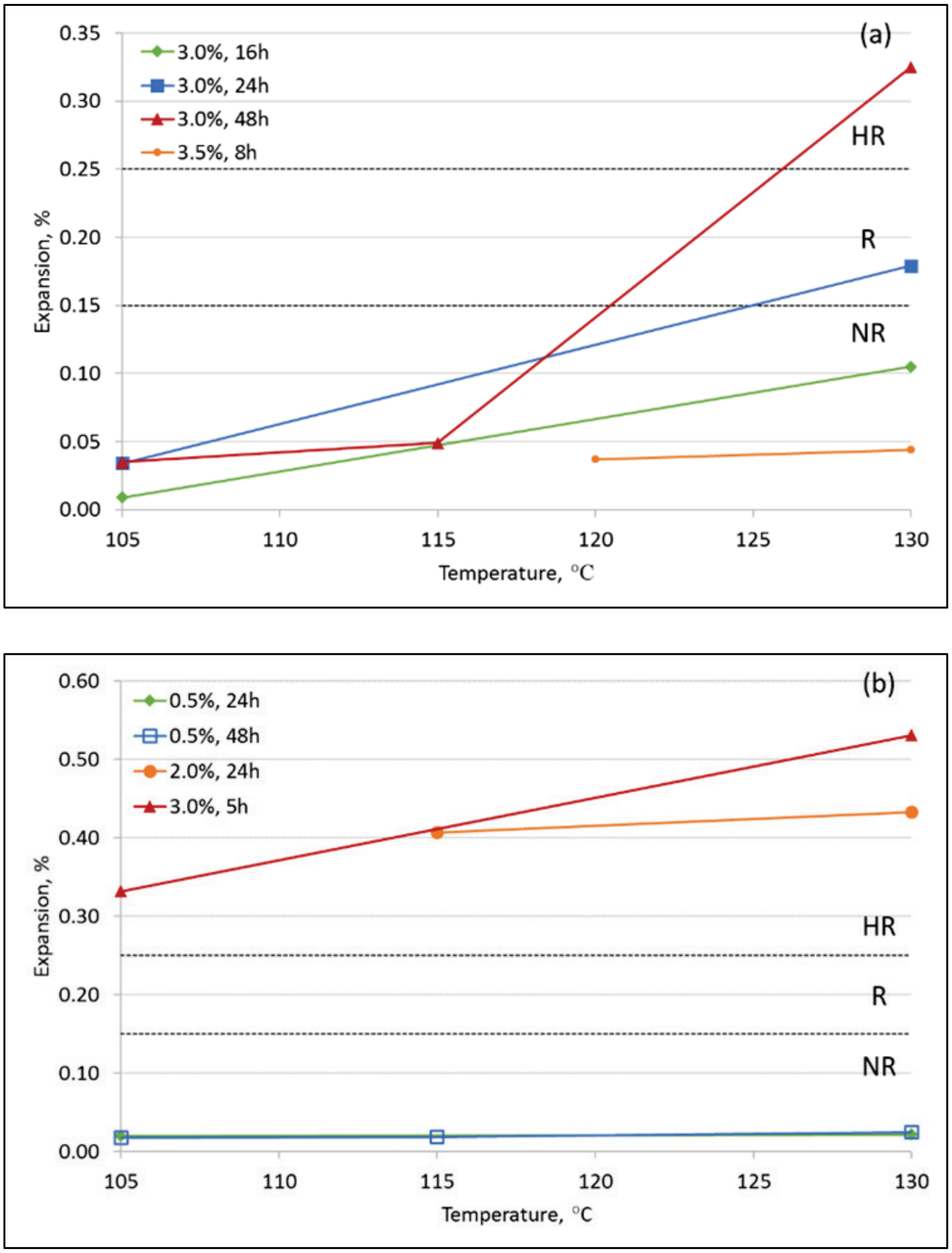
3.5.2 Influence of autoclaving temperature

Figure 9 shows the influence of autoclaving temperature on mortar bar expansion for (a) aggregate NR1 and (b) aggregate HR3. Aggregate NR1 is more sensitive to changes in temperature than to other parameter modifications. When the autoclaving duration was sequentially decreased from 16 to 6 to 5 hr using 3.0% Na_2O_{eq} at 105, 115, and 130°C, respectively, the mortar bar expansions continued to increase. Expansion increased by a factor of 5.26 when the autoclaving temperature was raised from 105 to 130°C with 3.0% Na_2O_{eq} for 24 hr and by a factor of 9.85 when the duration was 48 hr for this temperature difference and alkali loading. Even when the temperature was increased from 115 to 130°C using 3.0% Na_2O_{eq} for 48 hr, the increase in expansion was a substantial factor of 6.63.

The fact that the non-reactive, crystalline quartz sand expands at all in the autoclave is likely due to thermal expansion. Mu et al. (1996) observed unusual expansion values for mortars containing calcite and subjected to similar test parameters to those used in this study. The expansion was determined to have been heat-induced. Other studies showed that the thermal conductivity of siliceous aggregates is higher than that of calcareous aggregates (Razafinjato et al. 2016; Kizilkanat et al. 2013). Therefore, it is reasonable to assume that small expansions (<0.06%) of mortars containing the non-reactive siliceous aggregates in this study were caused by thermal effects rather than ASR. However, it is possible that, in cases where the expansion approached 0.10%, some ASR had formed. Aggregate NR1, for instance, reached 0.105% expansion using 3.0% Na_2O_{eq} at 130°C for 16 hr.

When the autoclaving temperature was increased from 105 to 115°C using no added alkalis (0.52% Na_2O_{eq}) for 48 hr, an average expansion increase by a factor of only 1.06 was observed for HR3. Expansion increased by a factor of just 1.10 when the temperature was raised from 105 to 130°C for 24 hr with no alkali boosting. At 3.0% Na_2O_{eq}, however, an increase in expansion by a factor of 1.60 was observed for the same temperature increase using 5 hr of autoclaving. Changes in temperature did not have a significant effect on the reactivity of HR3. This change in temperature could, however, result in a more severe reactivity classification for this aggregate if following the expansion thresholds for reactivity levels set by Fournier et al. (1991).

Figure 9. Influence of autoclaving temperature on expansion of mortar bars containing (a) NR1 and (b) HR3.



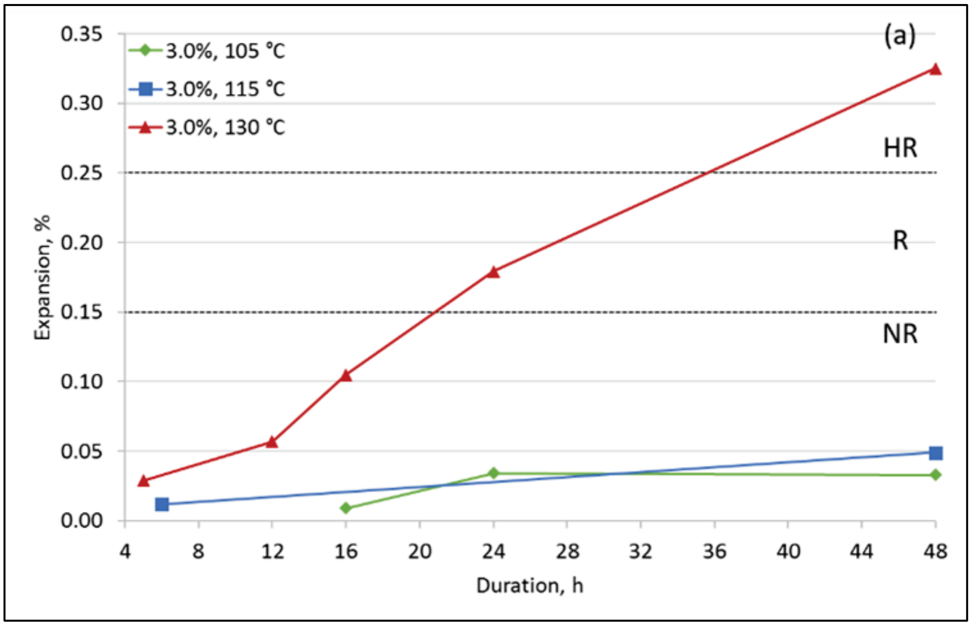
3.5.3 Influence of autoclaving duration

The effects of autoclaving duration on mortar bar expansions with aggregates NR1 and HR3 are presented in Figures 10a and 10b, respectively. For aggregate NR1, there was an average reduction in expansion by a factor of 0.97 when autoclaving duration was increased from 24 to 48 hr at 105°C with 3.0% alkali loading. The impact on expansion due to prolonged durations of autoclaving was relatively small until a high autoclaving temperature (130°C) was used. When the autoclaving duration was raised from 16 to 24 hr at 130°C with 3.0% Na_2O_{eq}, expansion increased by a factor of 1.70. With alkali loading and temperature held constant, a duration increase from 24 to 48 hr resulted in an increase in expansion by a factor of 1.82. Expansions exceeding 0.15% for NR1 are likely due to some degree of ASR in the mortar bars, and further work is planned to study the extent of ASR in autoclaved mortar bars with non-reactive aggregates.

When no alkalis were added, increasing the autoclaving duration from 24 to 48 hr had no effect on expansion of aggregate HR3. Increases in expansion were observed when the autoclaving duration was increased using 2.0 and 3.0% alkali loadings, but expansions were already high (> 0.25%) even for an autoclaving duration of 5 hr.

A summary of the percent changes in expansion resulting from modifications of the test parameters are provided in Tables 7 and 8 for the benchmark aggregates. The parameters in bold were altered while the other parameters listed were held constant.

Figure 10. Influence of autoclaving duration on expansion of mortar bars containing (a) NR1 and (b) HR3.



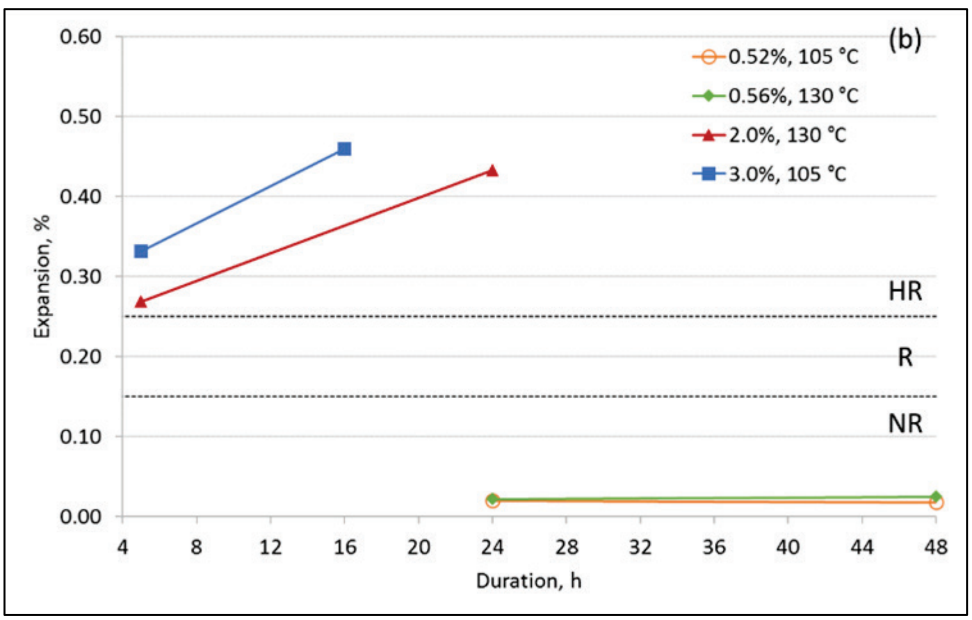


Table 7. Summary of influence of test parameter modifications on NR1 expansion.

Alkali Loading, Temperature, Duration	Change in Expansion, by factor
2.0 to 3.0%, 105 °C, 16h	0.56
3.0%, **105 to 115 °C**, 48h	1.48
3.0%, **105 to 130 °C**, 24h	5.26
3.0%, **105 to 130 °C**, 48h	9.85
3.0%, **115 to 130 °C**, 48h	6.63
3.0%, 105 °C, **16 to 24h**	3.78
3.0%, 105 °C, **24 to 48h**	0.97
3.0%, 115 °C, **6 to 48h**	4.08
3.0%, 130 °C, **16 to 24h**	1.70
3.0%, 130 °C, **24 to 48h**	1.82

Table 8. Summary of influence of test parameter modifications on HR3 expansion.

Alkali Loading, Temperature, Duration	Change in Expansion, by factor
0.56 to 1.0%, 130 °C, 24h	1.14
1.0 to 1.5%, 130 °C, 24h	1.92
1.5 to 2.0%, 130 °C, 24h	8.48
2.0 to 3.0%, 130 °C, 5h	1.97
3.0 to 3.5%, 130 °C, 5h	1.02
0.5%, **105 to 130 °C**, 24h	1.10
2.0%, **115 to 130 °C**, 24h	1.06
3.0%, **105 to 130 °C**, 5h	1.60
0.52%, 105 °C, **24 to 48h**	0.90
0.56%, 130 °C, **24 to 48h**	1.14
2.0%, 130 °C, **5 to 24h**	1.61

3.5.4 Variability in test results

A total of 72 autoclaved mortar bar tests involving 267 mortar bars were performed in this study. Of those, 62 tests resulted in single-operator expansion CVs less than 10.0% for a single set of mortar bars. The highest CV was 29.4%. The average single-operator CV for all tests was 5.7%. Wood et al. (2016) reported an average single-operator CV of 5.6% for the autoclaved concrete prism test. The average multi-laboratory CV, generated by data from two laboratories for the standard 5-hr autoclave test, was 6.8% with the highest value being 10.4%. Other autoclave studies in the literature did not provide expansion variability data for mortar bars or concrete prisms.

4 Part II: Round Robin Test Study

4.1 Expansion results and reactivity classifications

Table 9 presents average mortar bar expansions measured after autoclaving for 5 hr, along with reactivity classifications based on expansion limits proposed by Fournier et al. (1991), which are also provided in the table. Some tests used only three mortar bars because specimens occasionally broke during demolding.

Multi-laboratory autoclave expansion results were compared to results from ASTM C1260 for these aggregates in Figure 11. The vertical dashed lines indicate the expansion limits provided by ASTM C1778 for 14-day expansions in ASTM C1260. The horizontal dashed lines denote expansion limits suggested by Fournier et al. (1991) for the 5-hr autoclave test. Vertical error bars show the range of single-bar expansions measured across all five laboratories.

Table 9. Average autoclave mortar bar expansion for each laboratory and reactivity classifications based on autoclave results.

Aggregate	Average Autoclave Expansion, %						Autoclave Reactivity Classification[2]
	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Multi-laboratory Average	
NR1	0.049	0.062	0.053	0.062	0.038	0.053	NR
NR2	0.053	0.052	0.045	0.073	0.032	0.051	NR
NR3	0.086	0.078	0.066	0.062	0.051	0.069	NR
MR1	0.173	0.202	0.220	0.177	0.160	0.186	R
MR2	0.092	0.101	0.066	0.105	0.052	0.083	NR
MR3	0.091[a]	0.098[a]	0.055	0.083	0.055	0.076	NR
MR4	0.217	0.194[a]	0.176	0.189	0.233	0.202	R
MR5	0.293	0.330[a]	0.274	0.279	0.220	0.279	HR
MR6	0.349	0.312	0.328	0.295[a]	0.200	0.297	HR
MR7	0.197	0.160	0.155	0.226	0.141	0.176	R
HR1	0.544	0.590	0.453	0.536	0.546	0.534	HR
HR2	0.214	0.228[a]	0.175	0.167	0.162	0.189	R
HR3	0.386[a]	0.383[a]	0.341	0.335	0.282	0.345	HR
HR4	0.400	0.336	0.303	0.324	0.280	0.329	HR
HR5	0.260[a]	0.279[a]	0.259	0.249	0.218	0.253	HR
HR6	0.559	0.526	0.503	0.433	0.490	0.502	HR

Aggregate	Average Autoclave Expansion, %						Autoclave Reactivity Classification[2]
	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Multi-laboratory Average	
VHR1	0.228	0.158	0.161	0.329	0.151	0.205	R
VHR2	0.452	0.428	0.416	0.395	0.360	0.410	HR
VHR3	0.108[a]	0.152[a]	0.054	0.251	0.085	0.130	NR
VHR4	0.788	0.810	0.740	0.759	0.908	0.801	HR

[2] Expansion limits provided by Fournier et al. (1991) for 5-hr autoclave test.

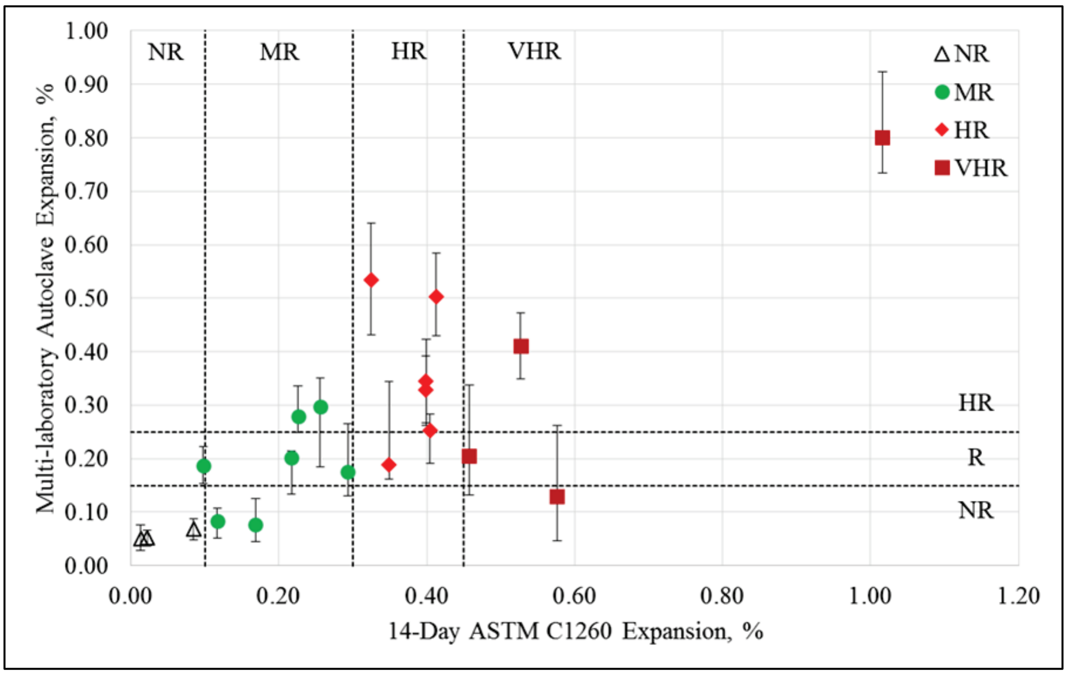
NR – expansion < 0.150%

R – expansion ≥ 0.150%

HR – expansion ≥ 0.250%

[a] Three bars tested.

Figure 11. Average multi-laboratory autoclave expansions versus 14-day ASTM C1260 expansions.



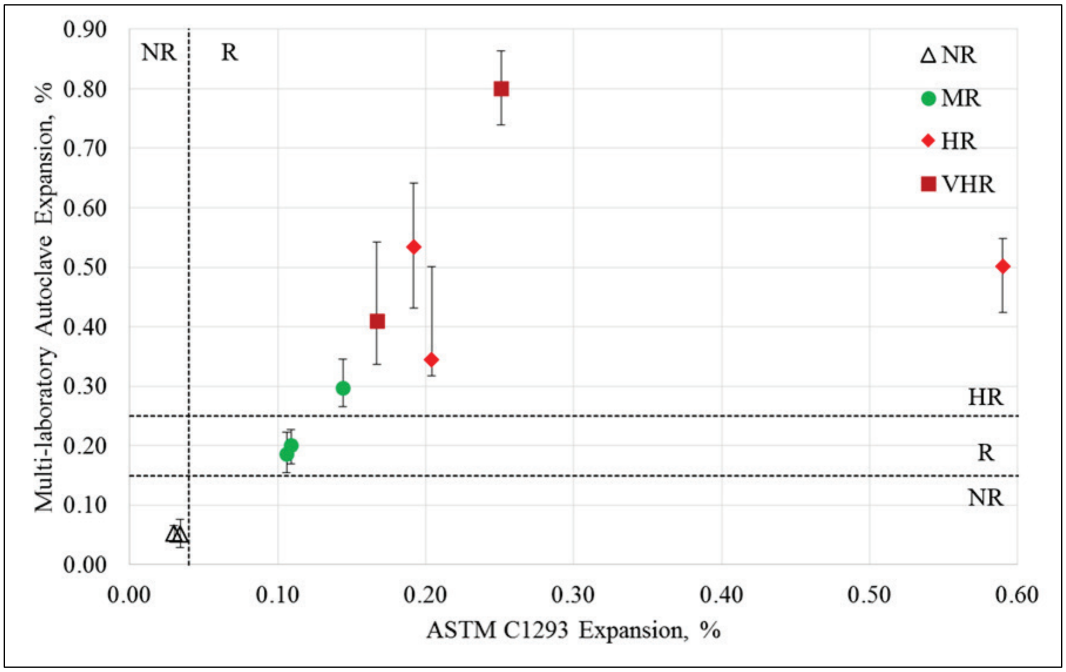
The three aggregates determined to be non-reactive by ASTM C1260 were also classified as non-reactive in the autoclave test, with no laboratory reporting average expansions greater than 0.086%. However, three aggregates categorized as reactive to some degree in ASTM C1260 (MR2, MR3, and VHR3) exhibited expansions in the autoclave test that would classify them as non-reactive. Fournier et al. (1991) observed the same phenomenon for two aggregates that did not appear reactive in the autoclave test but expanded more than 0.040% at one year in ASTM

C1293. The authors stated the reason was unknown, but both aggregates exhibited non-reactive expansions in the NBRI test method (Hooton and Rogers 1993). However, field data were not available for those aggregates, nor were they available for the low-expanding aggregates here.

Two aggregates deemed moderately reactive in ASTM C1260 (MR5 and MR6) appeared to be highly reactive in the autoclave, and two aggregates showing high and very high reactivity in ASTM C1260 (HR2 and VHR1) did not exhibit expansions consistent with a highly reactive classification in the autoclave test. Aggregates HR1, HR6, and VHR4 produced expansions in excess of 0.50% in the autoclave test. All three aggregates are well-documented for being reactive in field structures and the laboratory (Ideker et al. 2004; Giannini et al. 2014; Tremblay et al. 2012). Nevertheless, the other two aggregates classified as very highly reactive in ASTM C1260, VHR1 and VHR3, were indistinguishable from moderately reactive aggregates in the autoclave test. ASTM C1293 and field performance data are not available for these two aggregates, so it is uncertain whether the autoclave test is able to accurately classify their reactivity.

Overall, the two test methods demonstrated 85% agreement when determining whether or not an aggregate was non-reactive or reactive to some degree. In the study by Bérubé et al. (1992), which included the results from Fournier et al. (1991), the autoclave test method demonstrated a better overall effectiveness (81%) than the NBRI test method (75%) in categorizing aggregate reactivities based on ASTM C1293 data or field performance of those aggregates. Because ASTM C1260 is known to provide a significant number of false positive and false negative results, caution should be taken when comparing results from a new test method with ASTM C1260 results. ASTM C1293 data were available for some of the aggregates tested in this study and are presented in Table 1. Average multi-laboratory expansions in the autoclave were compared to ASTM C1293 expansions and are shown in Figure 12. Vertical error bars show the range of single-bar expansions measured across all laboratories.

Figure 12. Average multi-laboratory autoclave expansions versus ASTM C1293 expansions.



There was agreement between the autoclave method and ASTM C1293 for 10 out of the 10 aggregates shown. Average expansions for aggregates HR1, VHR2, and VHR4 were considerably larger than the expansions they produced in ASTM C1293. The coarse fractions of these aggregates were used in ASTM C1293 and had to be crushed into fine aggregates for the autoclave test. Crushing could cause some reactive silica in the aggregates to become more readily available for ASR (Latifee and Rangaraju 2015), altering its reactivity. This would explain the significant difference in expansion of aggregates HR1, VHR2, and VHR4 between the two test methods.

Several aggregates in this study were classified at a lower reactivity level based on their autoclave test results compared to their original classification based on ASTM C1260 results. VHR3 is the most notable disparity (non-reactive per autoclave results), but MR2, MR3, HR2, and VHR1 also would be characterized as being less-reactive based on the autoclave test. It is possible that modifying the parameters (temperature, duration, and alkali loading) of the autoclave test may improve the agreement between its results and that of other standardized tests, including ASTM C1260. The behavior of various silica morphologies under autoclaving conditions with very high alkali loadings is not well-understood, and the authors have made progress through investigating the effects of varying the test parameters (Wood et al. 2018). Additionally, a more in-depth comparison of aggregate petrography against the autoclave test results may provide valuable information. Lastly, it should be noted that ASTM C1260 is a severe test that can yield false positive results. In the absence of corroborating field performance data, it is unclear which test (ASTM C1260 or the autoclave) has yielded overly conservative results for these five aggregates.

4.2 Within-laboratory precision and variability

Statistical calculations were based on ASTM C802. However, more than one operator performed tests in some laboratories. Therefore, the statistical data for a laboratory are not necessarily single-operator as they are in the ASTM standard.

The within- and multi-laboratory standard deviations and coefficients of variation (CVs) for all mortar bar expansions are provided in Table 10. The average within-laboratory CV was 5.9%. The 10% single-operator, within-laboratory CV limit proposed by Grattan-Bellew (1997) was exceeded for only 2 of the 20 aggregates tested.

Table 10. Within- and multi-laboratory standard deviations and coefficients of variation for mortar bar expansions.

Aggregate	Within-laboratory Standard Deviation	Within-laboratory CV, %	Multi-laboratory Standard Deviation	Multi-laboratory CV, %
NR1	0.003	4.9	0.010	19.5
NR2	0.003	6.2	0.015	29.7
NR3	0.002	3.0	0.014	20.1
MR1	0.007	3.9	0.025	13.4
MR2	0.003	4.1	0.023	28.0
MR3	0.014	18.6	0.024	31.0
MR4	0.010	4.8	0.024	12.1
MR5	0.018	6.4	0.043	15.2
MR6	0.013	4.4	0.059	19.8
MR7	0.025	14.4	0.041	23.4
HR1	0.027	5.1	0.055	10.3
HR2	0.009	4.6	0.031	16.2
HR3	0.011	3.3	0.044	12.6
HR4	0.018	5.5	0.048	14.6
HR5	0.014	5.6	0.025	10.1
HR6	0.015	3.1	0.049	9.7
VHR1	0.016	8.0	0.077	37.5

Aggregate	Within-laboratory Standard Deviation	Within-laboratory CV, %	Multi-laboratory Standard Deviation	Multi-laboratory CV, %
VHR2	0.019	4.7	0.039	9.4
VHR3	0.007	5.5	0.077	59.0
VHR4	0.022	2.7	0.068	8.5
Average	0.013	5.9	0.040	20.0

Although earlier studies on autoclave test methods do not provide precision data or statements, it is possible to compare the results of this study to the precision of standardized tests for ASR. A study by Rogers showed that, for aggregates with average expansions in ASTM C1260 greater than 0.1% at 14 days, the within-laboratory CV was 2.94% (Rogers 1999). The within-laboratory precision for concrete prisms with average expansions greater than 0.014% in ASTM C1293 was 12% (ASTM International 2015).

4.3 Multi-laboratory precision and variability

Multi-laboratory standard deviations and CVs are provided in Table 10. Of the multi-laboratory CVs, 75% exceed the 12% limit suggested by Grattan-Bellew (1997). The average multi-laboratory CV was 20.0%.

In comparison to standardized tests, Rogers (1999) found the ASTM C1260 multi-laboratory CV to be 15.4% for mortar bars with average expansions above 0.1% at 14 days. In ASTM C1293, for average concrete prism expansions greater than 0.014%, the multi-laboratory CV was 23% (ASTM International 2015).

Although the within-laboratory CV for aggregate VHR3 was less than 10%, the multi-laboratory CV was the highest among mortar bars containing other aggregates (59.0%). Two laboratories reported difficulty in casting bars with mortar containing VHR3; the mortar was described as drier than the other mortars and crumbling. The reason for the unusual consistency of the mortar is unknown, but it could be that the aggregate was highly absorptive or had a specific gravity less than 2.45. In accordance with ASTM C1260, if the specific gravity of an aggregate is less than 2.45, the equation in Section 8.3.1 in ASTM C1260 is used, and the proportion of aggregate is less than it would be if the specific gravity were at or above 2.45. It was assumed in this study that each aggregate had a specific gravity at or above 2.45. Therefore, it is possible that the mortar mixture for VHR3 contained more aggregate than required, resulting in a relatively dry mixture.

Among the highest-expanding aggregates (HR1, HR6, and VHR4), the multi-laboratory CVs were relatively low (between 8.5 and 10.3%). As previously mentioned, these aggregates have been well-documented for their expansive behavior.

For unknown reasons, average expansions reported by Laboratory 5 were generally lower than expansions reported by the other laboratories. Excluding data from Laboratory 5 reduced the average multi-laboratory CV to 17.7%.

Four aggregates (MR4, MR6, HR2, and HR5) produced expansions within each of the three reactivity categories set forth by Fournier et al. (1991). Three aggregates (MR7, VHR1, and VHR3) exhibited expansions that fell within two of the reactivity categories. These minimum and maximum expansions recorded among the five laboratories are indicated by the error bars in Figure 11. The instances involving aggregates MR4 and MR6 were attributed to Laboratory 5 being an outlier. In cases such as these in which aggregates may be classified in more than one reactivity category, it is recommended that additional testing be performed on the aggregates in question.

4.4 Alkali leaching

Table 11 presents the results of the alkali leaching measurements taken at Laboratory 2. In some cases, more than one aggregate was tested in the autoclave simultaneously. For this reason, multiple aggregates are sometimes listed together. The percentage of leached alkalis was calculated based on the initial alkali content of the mortar, which was 21 kg/m^3 Na_2O_{eq} for all mixtures. This value assumes that the specific gravity of each aggregate was 2.60. The concentrations of sodium ions (Na^+) and potassium ions (K^+) were used to calculate the Na_2O_{eq} of the autoclave water in mg/L. That number was then multiplied by the volume of deionized water initially added to the autoclave (7 L) and divided by the number of mortars bars being simultaneously autoclaved, giving a mass of alkalis (Na^+ and K^+) per prism in the autoclave water, which was compared to the initial alkali content. For the purposes of this study, it is assumed that mortar properties were relatively uniform and did not significantly affect the amount or rate of alkali leaching. The same assumption is true for aggregate properties.

Table 11. Alkali concentrations in autoclave water samples and calculated leached alkalis.

Aggregate	Number of Mortar Bars	Autoclave Water Concentration, mg/L			Leached Alkalis, %
		$[Na^+]$	$[K^+]$	$[Na_2O_{eq}]$	
MR1	4	124.49	31.86	193.21	8.7
MR3	3	93.82	19.69	142.21	8.6
MR4, MR7	7	150.78	32.83	229.48	5.9
HR3	3	82.96	21.28	128.81	7.8
NR1, HR2, HR5	10	291.32	64.61	444.30	8.4

Alkali leaching in these samples ranged from 6 to 9% per mortar bar. In a study using autoclaved concrete prisms, alkali leaching was between 4 and 11% per prism (Wood et al. 2016). Rivard et al. (2007) determined alkali leaching in ASTM C1293 to be between 12 and 25% of the total alkalis per prism. The relatively shorter duration of the autoclave test is likely the reason alkali leaching is so low. If the duration were to be extended, it is expected that the amount of alkali leaching would increase because it typically occurs over longer periods of time.

Aggregate MR3 was one of three aggregates classified as reactive by ASTM C1260 but did not produce reactive expansions in the autoclave. In Laboratory 2, the only laboratory to measure alkali leaching, the mortar bars with aggregate MR3 leached 8.6% on average, which is not significant enough to slow or halt expansion caused by ASR. It is possible that this aggregate produces a false positive result in ASTM C1260. No amount of alkali leaching measured in this study would have affected the progress of ASR in these specimens, so false negative autoclave test results due to alkali leaching should not be expected.

5 Conclusions/Summary

Based on the evaluation of modified test parameters used to test aggregate alkali-silica reactivity in this study, the following conclusions can be made:

- An alkali loading of 2.5% produced smaller expansions than expected when autoclaving for 5 hr at temperatures up to 130°C. Based on previous autoclave studies (Fournier et al. 1991, Giannini and Folliard 2013), 2.5% Na_2O_{eq} may not be suitable for rapid testing for ASR, especially if autoclaving durations are relatively short.
- Increasing the Na_2O_{eq} from 3.5 to 4.5% had no significant effect and even reduced expansion for some aggregates.
- Increasing the Na_2O_{eq} from 3.0 to 3.5% also had essentially no effect on aggregate HR3.
- For the autoclaving temperatures, autoclaving durations, and cement alkalinity employed in this study, at least some alkali boosting is required, even for highly reactive aggregates such as HR3.
- Temperatures at or below 110°C in the autoclave were too low when Na_2O_{eq} was 3.5% and autoclaving took place over 5 hr.
- A 24-hr autoclaving duration at 130°C with 3.5% Na_2O_{eq} was too severe for aggregates known to be non-reactive or moderately reactive and resulted in higher reactivity classifications. This duration was also too severe for aggregate NR1 when alkali loading was reduced to 3.0%.
- The following ranges for test parameters are recommended when testing for ASR potential of aggregates in 25- x 25- x 285-mm mortar bars:
  - $3.0 \leq Na_2O_{eq} \leq 3.5\%$
  - 120 ≤ Temperature ≤ 130°C
  - 5 ≤ Duration ≤ 12 hr

This study investigated the effectiveness of the 5-hr autoclaved mortar bar test method in identifying ASR potential in concrete aggregates. Five laboratories participated in testing, and within- and multi-laboratory statistical data were determined. Based on the results of this study, the following conclusions can be made:

- For the 20 aggregates tested, agreement between the autoclave test and ASTM C1260 was 85% in determining whether or not an aggregate was reactive to some degree.

- For the 10 aggregates for which ASTM C1293 expansions were obtained, agreement was 100% between the autoclave test and ASTM C1293 in determining whether or not an aggregate was reactive to some degree.
- The average within-laboratory CV was 5.9% for the autoclave test. This value falls between the within-laboratory CVs of ASTM C1260 (2.94%) and ASTM C1293 (12%). Statistical calculations for the autoclave test were not based on single-operator conditions.
- The average multi-laboratory CV for the autoclave test was 20.0%. This value is between the multi-laboratory CVs of ASTM C1260 (15.4%) and ASTM C1293 (23%).
- Alkali leaching in the autoclave test (6 to 9%) was comparable to alkali leaching in the autoclaved concrete prism test (4 to 11%) and less than the amount of leaching in ASTM C1293 (12 to 25%).

Improvements for further evaluating the repeatability and reproducibility of the 5-hr autoclave test method would include employing a single operator in each laboratory, eliciting the participation of a statistically significant number of laboratories, and obtaining more information regarding aggregate behavior in ASTM C1293 and in field structures. Nonetheless, the 5-hr autoclave test method appears to be a viable alternative to traditional ASR test methods to support the needs of rapid materials evaluation of military contingency concrete construction operations.

6 Provisional Test Method

6.1 Scope

This provisional test method provides an assessment of the potential for alkali-silica reaction of aggregate in mortar bars using an autoclave.

6.2 Procedure

Preparation of mortar:

- Testing will utilize same mixture proportions as ASTM C1260 mortar bars.
- Aggregate and cement have been provided for each material to be tested.
- The materials provided are sufficient to produce (4) 1- x 1- x 11.25-in. mortar bars.
- Materials provided include 1,320 g of aggregate that has been prepped to the correct gradation and 587 g of Type I-II portland cement (Holcim – St. Genevieve) with Na_2O_{eq} of 0.54%.
- Volume of DI mix water is 276 mL.
- Boost alkali loading in mortar to 3.5% by weight of cement by addition of 22.44 g of NaOH to water.
- Prep molds and place gauge studs according to ASTM C1260.
- Label molds according to aggregate serial number and sample number for each batch.
- Mix batch according to provisions of ASTM C1260/ASTM C305.
- First, add water to the bowl then introduce cement and mix for 30 sec on low.
- Then slowly add test aggregate and mix for 30 sec.
- Then change mixer to medium speed and mix an additional 30 sec.
- Then stop the mixer and scrape the bowl with a spatula for 15 sec, taking care to mix the bottom of the mortar.
- Allow mortar to sit for 90 sec with the cover on.
- Finally, remove the cover and mix for 60 sec at medium speed.
- The clearance between the lower end of the paddle and the bottom of the bowl should be 5.1 mm.
- Fill all molds within 2 min and 15 sec after completion of the mixing.
- Cure specimens in moist cabinet for 48 hr at 23°C before demolding.

Autoclave mortar bar expansion test:

- Remove samples from moist cabinet and demold.
- Relabel with aggregate serial number and sample number.
- Take photographs of specimens.
- Make initial length measurements of mortar bars at room temperature using a length-change comparator.
- Place mortar bars into autoclave with 500 mL of H_2O added (same as for cement soundness measurement using autoclave expansion technique). If using an automated steam autoclave, ensure that reservoir has sufficient water to perform test.
- Pressurize/heat autoclave to reach temperature of 130°C. This should correspond to pressure of ~25 psi. Maintaining correct temperature is most important. **This is different from ASTM C151**. If using an automated autoclave, this can be set to the desired temperature.
- Once a stable temperature/pressure is reached, continue testing for 5 hr. **This is different from ASTM C151.**
- Cool autoclave and vent (same as for cement soundness test). If using an automated autoclave, use provided cooling cycle followed by 15 min of cooling in water prior to making final length-change measurement.
- When sample temperature has equilibrated to room temperature, measure length change using comparator.
- Take post-test photographs of specimens. Note any damage observed visually.
- Store sample in plastic bag for characterization. Label bag according to aggregate name/serial number and “mortar bar autoclave test.”

References

ASTM International. 2014. *Standard test method for potential alkali reactivity of aggregates (mortar-bar method)*. West Conshohocken, PA: ASTM International.

ASTM International. 2015. *Standard test method for determination of length change of concrete due to alkali-silica reaction*. West Conshohocken, PA: ASTM International.

Bérubé, M. A., B. Fournier, N. Dupont, P. Mongeau, and J. Frenette. 1992. *A simple autoclave mortar bar method for assessing potential alkali-aggregate reactivity in concrete*. 9th International Conference on Alkali-Aggregate Reaction in Concrete. London, England.

Chatterji, S., and P. Christensen. 1990. Studies of alkali-silica reaction. part 7. Modelling of expansion. *Cement and Concrete Research* 20:285-290.

Folliard, K. J., R. Barborak, T. Drimalas, L. Du, S. Garber, J. H. Ideker, T. Ley, S. Williams, M. Juenger, B. Fournier, and M. D. A. Thomas. 2006. *Preventing ASR/DEF in new concrete: Final report*. Austin, TX: Center for Transportation Research.

Fournier, B., M. A. Bérubé, and G. Bergeron. 1991. A rapid autoclave mortar bar method to determine the potential alkali-silica reactivity of St. Lawrence lowlands carbonate aggregates. *Cement and Concrete Research* 13(1):58-71.

Giannini, E. R., A. F. Bentivegna, and K. J. Folliard. 2014. Strain gradients in concrete affected by alkal-silica reaction: A laboratory simulation. *Advances in Civil Engineering Materials* 3(1):388-403.

Giannini, E. R., and K. J. Folliard. 2013. *A rapid test to determine alkali-silica reactivity of aggregates using autoclaved concrete prisms*. Skokie, IL: Portland Cement Association.

Grattan-Bellew, P. E. 1997. A critical review of ultra-accelerated tests for alkali-silica reactivity. *Cement and Concrete Composites* 19:402-414.

Hooton, R. D., and C. A. Rogers. 1993. Development of the NBRI rapid mortar bar test leading to its use in North America. *Construction and Building Materials* 7(3):145-148.

Ideker, J., K. J. Folliard, M. Juenger, and M. D. Thomas. 2004. *Laboratory and field experience with ASR in Texas, USA*. 12th International Conference on Alkali-Aggregate Reaction in Concrete. Beijing, China.

Kagimoto, H., Y. Yasuda, and M. Kawamura. 2014. ASR expansion, expansive pressure and cracking in concrete prisms under various degrees of restraint. *Cement and Concrete Research* 59:1-15.

Kizilkanat, A. B., N. Yuzer, and N. Kabay. 2013. Thermo-physical properties of concrete exposed to high temperature. *Construction and Building Materials* 45:157-161.

Kuroda, T., S. Inoue, and A. Yoshino. 2000. *ASR expansion characteristics of mortar bars immersed in NaOH solution by autoclave method*. 11th International Conference on Alkali-Aggregate Reaction. Québec, Canada.

Kuroda, Tamotsu, Shoichi Inoue, Akira Yoshino, Shinzo Nishibayashi, and Toyoaki Miyagawa. 2012. *Effects of accelerated test conditions on ASR expansion of concrete core dimension*. Japan: Tottori University.

Latifee, E. R., and P. R. Rangaraju. 2015. Miniature concrete prism test: Rapid test method for evaluating alkali-silica reactivity of aggregates. *Materials in Civil Engineering* 27(7).

Leśnicki, K. J., J. Y. Kim, K. E. Kurtis, and L. J. Jacobs. 2013. Assessment of alkali-silica reaction damage through quantification of concrete nonlinearity. *Materials and Structures* 46:497-509.

Lu, D., B. Fournier, P. E. Grattan-Bellew, Z. Xu, and M. Tang. 2008. Development of a universal accelerated test for alkali-silica and alkali-carbonate reactivity of concrete aggregates. *Materials and Structures* 41(2):235-246.

Mu, X., Z. Xu, M. Deng, and M. Tang. 1996. *Abnormal expansion of coarse-grained calcite in the autoclave method*. 10th International Conference on Alkali-Aggregate Reaction in Concrete. Melbourne, Australia.

Nishibayashi, S., T. Kuroda, S. Inoue, and Y. Okawa. 1996. *Expansion characteristics of AAR in concrete by autoclave method*. 10th International Conference on Alkali-Aggregate Reaction in Concrete. Melbourne, Australia.

Nishibayashi, S., K. Yamura, and H. Matsushita. 1987. *A rapid method of determining the alkali-aggregate reaction in concrete by autoclave*. 7th International Conference on Alkali-Aggregate Reaction in Concrete. Ottawa, ON, Canada.

Razafinjato, R. N., A.-L. Beaucour, R. L. Hebert, B. Ledesert, R. Bodet, and A. Noumowe. 2016. High temperature behaviour of a wide petrographic range of siliceous and calcareous aggregates for concretes. *Construction and Building Materials* 123:261-273.

Rivard, P., M. A. Bérubé, and J. P. Ollivier. 2007. Decrease of pore solution alkalinity in concrete tested for alkali-silica reaction. *Materials and Structures* 40:909-921.

Rogers, C. A. 1999. Multi-laboratory study of the accelerated mortar bar test (ASTM test method C1260) for alkali-silica reaction. *Cement, Concrete, and Aggregates* 21:185-194.

Tamura, H. 1987. *A test method on rapid identification of alkali reactivity of aggregates (GBRC rapid method)*. 7th International Conference on Alkali-Aggregate Reaction in Concrete. Ottawa, ON, Canada.

Tang, M., S. Han, and S. Zhen. 1983. A rapid method for identification of alkali reactivity of aggregate. *Cement and Concrete Research* 13:417-422.

Tremblay, S., B. Fournier, M. D. A. Thomas, T. Drimalas, and K. J. Folliard. 2012. *The Lomas Boulevard road test site, Albuquerque (New Mexico) - A case study on the use of preventative measures against ASR in new concrete*. 14th International Conference on Alkali-Aggregate Reaction in Concrete. Austin, Texas.

Wood, S. G., E. R. Giannini, M. A. Ramsey, and R. D. Moser. 2018. Autoclave test parameters for determining alkalli-silica reactivity of concrete aggregates. *Construction and Building Materials* 168.

Wood, S. G., M. L. Kimble, N. D. Klenke, B. Fiore, J. E. Tanner, and E. R. Giannini. 2016. *Inter-laboratory comparison of expansions from the autoclaved concrete prism test*. 15th International Conference on Alkali-Aggregate Reaction in Concrete. São Paulo, Brazil.

Wood, S. G., A. V. Sutton, and E. R. Giannini. 2016. *Tracking alkali migration and leaching in the autoclaved concrete prism test*. 15th International Conference on Alkali-Aggregate Reactions in Concrete. São Paulo, Brazil.

Appendix A: Multi-Laboratory Round Robin Testing Reports

A.1 Laboratory 1 Final Report – The University of Alabama

Results of Alkali-Silica Reaction Testing Using Autoclave Accelerated Methods

Research Team: The University of Alabama
Stephanie G. Wood and Eric R. Giannini

1. INTRODUCTION

Alkali-silica reaction (ASR) is a deleterious chemical reaction in concrete first documented by Stanton about 75 years ago (1940). It occurs when three conditions exist within the concrete: (1) sufficient alkali hydroxides in the pore solution, (2) reactive siliceous minerals within the aggregate, and (3) sufficient moisture. Alkali hydroxides in the pore solution, primarily provided by the portland cement, react with certain siliceous minerals present in some aggregates. The reaction forms a hydrophilic alkali-silica gel which absorbs water and swells, potentially causing expansion and microcracking of the aggregates and cement paste. This can lead to macro-cracking that propagates to the surface of the structure, loss of mechanical properties, and may render the concrete vulnerable to other forms of attack.

Provided that the reactivity of the aggregates are known, the risk of ASR in new structures can be minimized by employing proper mix designs that limit alkali loading, use SCMs such as fly ash and slag cement, and that use non-reactive aggregates. Accurately identifying reactive aggregates is a critical component of reducing the risk of ASR in new construction. This applies to both testing new areas of existing quarries and pits, and to testing previously unexploited or tested sources. Accurately identifying reactive aggregates is particularly challenging when test results are needed quickly.

Currently, two of the most common test methods for determining the potential for ASR in aggregates are the ASTM C1293 concrete prism test (CPT) and the ASTM C1260 accelerated mortar-bar test (AMBT). The CPT tests concrete prisms over one year for normal concrete and two years when testing the effectiveness of ASR mitigation measures and is generally considered to be the most reliable standardized laboratory test method for aggregate reactivity. As a result, the CPT is frequently used as a benchmark for accelerated test methods (Lu et al. 2008). However, the duration of the CPT has limited its adoption in practice. Expansion results from ASTM C1260 are obtained within 16 days of mixing the mortar bars. Although the AMBT is much shorter than the CPT, the test method fails to identify some aggregates found to be reactive using the CPT or in documented field performance; this situation is referred to as a false negative. Furthermore, the AMBT produces a large number of false positives, meaning the aggregate appears reactive in the test but has not proven to be reactive in the CPT or in the field. False positives in the AMBT are generally linked to the harsh exposure conditions, while many false negatives are potentially linked to aggregate pessimum behavior.

A number of autoclave test methods have been developed for ASR testing, most notably those by Tang et al. (1983), Tamura (1987), Nishibayashi et al. (1987), Fournier et al. (1991), Bérubé et al. (1992), Nishibayashi et al. (1996), and Giannini and Folliard (2013). Most used mortar specimens, but Nishibayashi (1996) and Giannini and Folliard (2013) used concrete prisms. Some of those methods demonstrated promise, but none have come into wider use or been standardized. While using concrete prisms avoids potentially altering coarse aggregate reactivity by reducing the aggregate to sand sizes, it does require significantly more material. The method described by Fournier et al. (1991) and Bérubé et al. (1992) was applied to a total of 106 aggregates and found to be particularly promising. The coefficient of variation was very low (4.4%) and the test method produced reactivity classifications in agreement with the CPT 81% of the time. This method would be the basis for the testing performed in the study described in this report.

2. SCOPE

This inter-laboratory autoclave study was conducted with the objective of determining if the autoclave method is a suitable test method for identifying alkali-silica reactive aggregates used in rapid construction of short-life structures. Mortar bars with boosted equivalent alkali contents were autoclaved for 5 hours, and expansions were measured. The research was led by the US Army Engineer Research and Development Center's (ERDC) Geotechnical and Structures Laboratory. Four other laboratories, including The University of Alabama (UA), participated. UA was funded to perform this work through Battelle Memorial Institute TCN 15-034.

3. METHODS

3.1. Materials

The aggregates provided by ERDC for this study are listed in Table 3-1, which includes the aggregate name, serial number, results of ASTM C1260 tests performed at ERDC, uniformity, and ERDC-assigned reactivity classifications.

Table 3-1: Summary of aggregates used in this study.

Serial Number	ASTM C1260 Expansion, %		Aggregate	Coarse /Fine	Uniformity		Reactivity
	14 Days	28 Days			Homogenous	Heterogeneous	
150094	0.0848	0.1168	Vulcan Atlanta	Fine	X		Low/Non
140040	0.3993	0.4868	Calera, AL	Fine	X		Low/Non
120016	0.5270	0.7563	Rogers Gordonsville, TN	Coarse	X		Borderline
120032	0.0980	0.1560	Rogers Liberty, TN	Coarse	X		Borderline
130038	0.0820	0.1090	Adairsville, GA	Coarse	X		Borderline
120102	0.1170	0.2293	China Lake, CA	Coarse		X	Borderline
120021	0.0223	0.0353	Cookeville, TN	Coarse	X		Moderate
150104	0.1688	0.3235	Clayton, NJ	Fine	X		Moderate
140037	0.4045	0.5368	Green Brothers #2, MS	Fine		X	Moderate
150047	0.3490	0.4893	Wabasha, MN	Fine		X	Moderate
130041	0.2260	0.3220	Green Brothers, MS	Fine		X	Moderate
150096	0.2170	0.3365	LG Everist	Fine	-	-	Moderate
150100	0.2930	0.4510	Geneva	Coarse	-	-	Moderate
150098	0.3990	0.5240	Spratt, ONT	Coarse	X		High
130002	0.3250	0.4820	Gold Hill, NC	Coarse	X		High
140085	0.5763	0.7668	Nevada Test Site	Fine		X	High
150098.1	0.2560	0.4180	Sudbury, ONT	Coarse		X	High
130037	0.4130	0.6130	Jobe, TX	Coarse		X	High
152149	0.4575	0.6248	Arkadelphia	Fine	-	-	High
130006	1.0160	1.2550	Las Placitas, NM	Coarse		X	Very High

An ASTM C150 Type I/II portland cement from Missouri with a 0.524% equivalent alkali content (Na_2O_{eq}) was provided by ERDC and employed for all mixtures in this study. The oxide analysis of the cement is given in Table 3-2.

Table 3-2: Cement oxide analysis.

Oxide Notation	% Weight
SiO_2	18.99
Al_2O_3	4.44
Fe_2O_3	3.34
CaO	67.72
MgO	2.63
SO_3	3.44
Na_2O	0.09
K_2O	0.66
Loss on Ignition	3.06
Na_2O_{eq}	0.524

3.2. Test Procedure

Aggregates were graded and proportioned by ERDC in accordance with ASTM C1260 and provided to UA. The material proportions, w/cm (0.47), and specimen size (25 x 25 x 285 mm) were also in accordance with ASTM C1260. The Na_2O_{eq} of the mixtures was boosted to 3.5% by mass of cement by adding 45.1 g of 50% w/w sodium hydroxide (NaOH) solution to 253.3 g deionized mixing water. Four bars were formed from each mixture, and metal gauge studs were embedded in the ends to measure expansions using a length change comparator in accordance with ASTM C157.

Immediately after casting, the mortar bars were cured for 48 hours inside a moist curing room before they were demolded. An initial length change measurement was made after demolding the mortar bars, and the bars were then autoclaved for 5 hours at 130 °C (0.17 MPa gauge pressure), following the same parameters used by Fournier et al. (1991) and Bérubé et al. (1992). A Yamato SQ-510C with an internal capacity of 50 L was used (Figure 3-1). Figure 3-2 shows the mortar bars standing upright inside the autoclave.

Figure 3-1: Yamato SQ-510 autoclave.

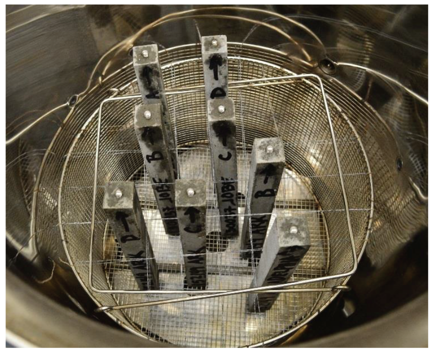


Figure 3-2: Mortar bars positioned upright inside the autoclave.

Once the autoclave cooled to 90 °C, the mortar bars were removed, placed in a pot of hot water, and cooled to 23 °C by overflowing the pot with running water over a period of approximately 15 minutes, similar to the cooling step described in ASTM C151. Final length change measurements were taken after cooling the bars to 23 °C and the expansion calculated. Photographs were taken of each mortar bar set before and after autoclaving, and photographs of one face of each specimen before and after autoclaving are in the appendix; photographs of the specimens containing the Green Brothers aggregate (130041) are missing and are not included in the appendix.

3.3. Alkali Leaching Measurements

Following removal of the mortar bars from the autoclave, water in the autoclave chamber was sampled to assess alkali leaching from the bars. Because only deionized water is added to the autoclave at the start of the test, alkalis present in the water after the test are assumed to be leached from the specimens. The sample was analyzed for sodium and potassium using inductively coupled plasma – optical emission spectroscopy (ICP-OES) in the Analytical Geochemistry Lab in UA's Department of Geological Sciences. Nitric acid was added to the autoclave water samples until they contained 2% nitric acid and the samples were then diluted 10:1 with deionized water immediately prior to performing ICP-OES. The total alkali concentration was then compared to the assumed initial alkali loading in the specimens. This analysis was performed on autoclave water samples from testing eight aggregates.

4. RESULTS

4.1. Expansion Measurements

Table 4-1 presents mortar bar expansions measured after autoclaving for 5 hours, along with the standard deviation and coefficient of variation for each aggregate. Asterisks indicate re-test for a particular aggregate. All four re-tested aggregates initially exhibited very low expansions relative to those measured by other laboratories involved in this study. The cause of error in the initial round of testing of these four aggregates is unknown; however, the initial tests were conducted during the same week, and no other aggregates were tested during that week. However, it does appear that the error was procedural and not related to the actual reactivity of these four aggregates.

When the initial tests of re-tested aggregates are not considered, 5-hour autoclave expansions ranged from 0.052% for Adairsville (130028) to 0.805% for Las Placitas (130006). The average standard deviation within a set of specimens for each aggregate was 0.011%. The average coefficient of variation was 6.1%, and would be 4.7% if the Clayton, NJ (150104) specimens are not included in the calculation.

Visual observations of the mortar bars after autoclaving typically included features such as map-cracking and efflorescence for aggregates exhibiting expansions greater than 0.30%, including Calera (140040), Sudbury (150098.1), Jobe (130037), Spratt (150098), Green Brothers (130041), and Las Placitas (130006). Curving or warping of the Calera, Sudbury, Spratt, and Las Placitas specimens was also observed.

The Nevada specimens (140085) were noted to be difficult to work with; the fresh mortar was much drier than mixtures containing other aggregates, and it proved difficult to consolidate in the molds. Figure 4-1 shows the mortar containing the Nevada aggregate being placed in the molds. Despite this difficulty, it did not lead to a large variation in the results (coefficient of variation = 2.5%).

Table 4-1: Mortar bar expansions from the autoclave test compared to expansions in ASTM C1260 at 14 days. (Asterisk* denotes re-test).

Serial Number	Aggregate	ASTM C1260 14-Day Expansion, %	Average 5-hr Autoclave Expansion, %	Standard Deviation	Coefficient of Variation (%)
150094	Vulcan Atlanta	0.0848	0.078	0.002	2.7
140040	Calera, AL	0.3993	0.336	0.019	6.6
120016	Rogers Gordonsville, TN	0.5270	0.428	0.022	5.1
120032	Rogers Liberty, TN	0.0980	0.202	0.005	2.6
130038	Adairsville, GA	0.0820	0.052	0.005	9.2
120102	China Lake, CA	0.1170	0.101	0.004	4.1
120021	Cookeville, TN	0.0223	0.021	0.001	6.7
120021	Cookeville, TN	0.0223	0.062*	0.004	7.0
150104	Clayton, NJ	0.1688	0.017	0.003	19.1
150104	Clayton, NJ	0.1688	0.098*	0.025	25.4
140037	Green Brothers #2, MS	0.4045	0.019	0.001	4.3
140037	Green Brothers #2, MS	0.4045	0.279*	0.005	2.0
150047	Wabasha, MN	0.3490	0.037	0.002	4.1
150047	Wabasha, MN	0.3490	0.228*	0.008	3.6
130041	Green Brothers, MS	0.2260	0.326	0.010	3.0
150096	LG Everist	0.2170	0.194	0.015	7.9
150100	Geneva	0.2930	0.160	0.002	1.4
150098	Spratt, ONT	0.3990	0.383	0.011	2.9
130002	Gold Hill, NC	0.3250	0.586	0.046	7.9
140085	Nevada Test Site	0.5763	0.152	0.004	2.5
150098.1	Sudbury, ONT	0.2560	0.312	0.016	5.3
130037	Jobe, TX	0.4130	0.526	0.022	4.1
152149	Arkadelphia	0.4575	0.158	0.013	8.0
130006	Las Placitas, NM	1.0160	0.805	0.025	3.1
Average Coefficient of Variation (%)					6.1

Figure 4-1: Nevada Test Site (140085) mixture crumbling during placement.

4.2 Alkali Leaching

Table 4-2 presents the results of the alkali leaching measurements. In some cases, more than one aggregate was tested in the autoclave simultaneously. For this reason, multiple aggregates are sometimes listed together. The percentage of leached alkalis was calculated based on the initial alkali content of the mortar, which was 21 kg/m^3 Na_2O_{eq} for all mixtures. This value assumes that the specific gravity of each aggregate was 2.60. The concentrations of Na^+ and K^+ were used to calculate the Na_2O_{eq} of the autoclave water in mg/L. That number was then multiplied by the volume of deionized water initially added to the autoclave (7 L) and divided by the number of mortars bars being conditioned at once, giving a mass of alkalis (Na^+ and K^+) per prism, which was compared to the initial alkali content.

Table 4-2: Alkali concentration of autoclave water samples and calculated percentage of leached alkalis.

Aggregate	Number of Mortar Bars	Autoclave Water Concentration, mg/L			Leached Alkalis, %
		$[Na^+]$	$[K^+]$	$[Na_2O_{eq}]$	
Rogers Liberty	4	124.49	31.86	193.21	8.7
Spratt	3	82.96	21.28	128.81	7.8
Green Brothers #2, Cookeville, Wabasha	10	291.32	64.61	444.30	8.0
Everist, Geneva	7	150.78	32.83	229.48	5.9
Clayton	3	93.82	19.69	142.21	8.6

Alkali leaching in these samples ranged from 5.9 to 8.7% per mortar bar. It should be noted that autoclave water alkali concentrations shown in Table 4-2 may not be directly compared among sets because the number of bars conditioned in each set varied. For instance, only three mortar bars made with

Spratt aggregate were conditioned while ten mortar bars were conditioned at once in the set involving Green Brothers #2, Cookeville, and Wabasha aggregates.

5. DISCUSSION

Table 5-1 presents a list of the aggregates and potential reactivity classifications. In this table, ASTM C1260 reactivity classifications follow ASTM C1778 guidelines for interpreting ASTM C1260 expansions at 14 days, and are based upon test results provided by ERDC. The 5-hour autoclave reactivity classes are based upon those suggested by Fournier et al. (1991) and Bérubé et al. (1992), and only the expansion data generated at UA during this study were considered.

Based on the reactivity classifications provided in Table 5-1, 16 of 20 aggregates (80%) showed some reactivity in both the ASTM C1260 and the 5-hour autoclave test; 18 aggregates (90%) were considered at least borderline reactive by ERDC designations. ASTM C1260 and the autoclave test were in agreement for 18 of 20 aggregates (90%) when determining if an aggregate was non-reactive or reactive to some degree. The only three aggregates to show non-reactive expansions in both ASTM C1260 and the autoclave test were Vulcan Atlanta (150094), Adairsville (130038), and Cookeville (120021).

Fournier et al. (1991) and Bérubé et al. (1992) did not establish a very highly reactive (VHR) aggregate category in the 5-hour autoclave classification system. However, of the aggregates considered to be highly reactive or very highly reactive in the ASTM C1778 classification system, 7 of those aggregates (70%) were also considered highly reactive by the 5-hour autoclave classification system.

Calera (140040) has generally been considered an innocuous aggregate based upon historical ASTM C1260 results and with no documented field history of ASR. However, recent ASTM C1260 and 5-hour autoclave expansions both indicate a potentially high degree of reactivity. It is possible that this indicates that the sample came from an area of the quarry with a different mineralogy than the material that has historically been mined.

Because ASTM C1260 is known to produce a significant quantity of false negative and positive results, expansions obtained through the 5-hour autoclave test should be compared to more reliable test methods, such as the CPT, or to aggregate field behavior. Such caution is particularly warranted when the test method produces results that are considerably in disagreement with past field performance.

Once comparisons have been made between the autoclave expansions and more reliable test methods/field history, an expansion limit, or set of limits, can be established and should take into account aggregate mineralogy and specimen size. Additionally, more non-reactive aggregates should be tested in order to better evaluate whether or not those can be accurately identified by the autoclave test method.

Alkali leaching in this 5-hour autoclave test was measured to be between 5.9 and 8.7% of the initial alkali loading. This degree of leaching is significantly less than what has been documented for the CPT (12 to 25% per concrete prism) by Rivard et al. (2003, 2007). It is comparable to the 4 to 11% leaching reported by the authors for the 24-hour autoclaved concrete prism test (ACPT) (Wood et al., 2016).

Table 5-1: Comparison of aggregate reactivity classifications.

Serial Number	Aggregate	ERDC-Designated Reactivity	ASTM C1260 Reactivity[1]	5-hr Autoclave Reactivity[2]
150094	Vulcan Atlanta	NR	NR	NR
140040	Calera, AL	NR	HR	HR
120016	Rogers Gordonsville, TN	BR	VHR	HR
120032	Rogers Liberty, TN	BR	NR	R
130038	Adairsville, GA	BR	NR	NR
120102	China Lake, CA	BR	MR	R
120021	Cookeville, TN	MR	NR	NR
150104	Clayton, NJ	MR	MR	NR
140037	Green Brothers #2, MS	MR	HR	HR
150047	Wabasha, MN	MR	HR	R
130041	Green Brothers, MS	MR	MR	HR
150096	LG Everist	MR	MR	R
150100	Geneva	MR	MR	R
150098	Spratt, ONT	HR	HR	HR
130002	Gold Hill, NC	HR	HR	HR
140085	Nevada Test Site	HR	HR	R
150098.1	Sudbury, ONT	HR	MR	HR
130037	Jobe, TX	HR	HR	HR
152149	Arkadelphia	HR	VHR	R
130006	Las Placitas, NM	VHR	VHR	HR

NR – non-reactive ([1] expansion < 0.10%; [2] expansion < 0.100% for silicate aggregates, expansion < 0.150% for sands and gravels, and carbonate aggregates)
R – reactive ([2] 0.100% ≤ expansion for silicate aggregates, 0.150% ≤ expansion for sands and gravels, and carbonate aggregates)
MR – moderately reactive ([1] 0.10% ≤ expansion < 0.30%)
HR – highly reactive ([1] 0.30% ≤ expansion < 0.45%; [2] 0.250% ≤ expansion)
VHR – very highly reactive ([1] 0.45% ≤ expansion)
BR – ERDC designation; undefined expansion limits.

6. SUMMARY

The following conclusions can be made based on this study:

- The correlation between reactivity classifications in ASTM C1778 for ASTM C1260 expansions at 14 days and the 5-hour autoclave test is very good (90% agreement) when determining if an aggregate is non-reactive or reactive to some degree.
- More tests involving known non-reactive aggregates are needed to determine whether or not these aggregate reactivities can be properly identified by the autoclave test method.
- Results from the autoclave method should be compared to results from the CPT, exposure blocks, and/or known field behavior to better determine the efficacy of the autoclave test method in classifying aggregates and to establish expansion limits for the test method.
- Compared to the amount of alkali leaching in the CPT (12 to 25% per prism), the degree of alkali leaching in the 5-hour autoclave test (5.9 to 8.7% per bar) is relatively low.

Concurrent work at UA related to this study includes testing concrete prisms that incorporate some of these aggregates in the 24-hour ACPT, adjusting test parameters such as alkali loading, duration of conditioning, and autoclave temperature to account for aggregate mineralogy and size, and identifying and characterizing any reaction product in petrographic samples from autoclave test specimens.

For future works, the authors suggest testing more known non-reactive aggregates in the autoclave to determine whether or not those aggregates can be properly identified as non-reactive. Additionally, reactivity classifications from the autoclave test should be compared to what has been observed in the CPT and field exposure blocks which use those aggregates.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the efforts of undergraduate research assistants Ashley Sutton, Elliot McCandless, and Matthew Chandler in sample preparation and analysis. Dr. Sid Bhattacharyya in the Department of Geological Sciences conducted the ICP-OES analysis of the autoclave water.

REFERENCES

Bérubé, M.-A., Fournier, B., Dupont, N., Mongeau, P., & Frenette, J. (1992). A Simple Autoclave Mortar Bar Method for Assessing Potential Alkali-Aggregate Reactivity in Concrete. *Proceedings of the 9th International Conference on Alkali-Aggregate Reaction in Concrete*, (pp. 81-91). London.

Fournier, B., Bérubé, M., & Bergeron, G. (1991). A rapid autoclave mortar bar method to determine the potential alkali-silica reactivity of St. Lawrence lowlands carbonate aggregates. *Cement and Concrete Research, 13*(1), 58-71.

Giannini, E. R., & Folliard, K. J. (2013). *A Rapid Test to Determine Alkali-Silica Reactivity of Aggregates Using Autoclaved Concrete Prisms.* Skokie, IL, USA: Portland Cement Association.

Ideker, J., Folliard, K., Fournier, B., & Thomas, M. (2006). The role of "non-reactive" aggregates in the accelerated (60 C) concrete prism test. *Proceedings from the Marc-André Bérubé Symposium on Alkali-Aggregate Reactivity in Concrete, 8th CANMET/ACI International Conference on Recent Advances in Concrete Technology* (pp. 45-70). Montreal: CANMET.

Lu, D., Fournier, B., Grattan-Bellew, P., Xu, Z., & Tang, M. (2008). Development of a universal accelerated test for alkali-silica and alkali-carbonate reactivity of concrete aggregates. *Materials and Structures, 41*(2), 235-246.

Nishibayashi, S., Kuroda, T., Inoue, S., & Okawa, Y. (1996). Expansion Characteristics of AAR in Concrete by Autoclave Method. *Proceedings of the 10th International Conference on Alkali-Aggregate Reaction*, (pp. 370-376). Australia.

Nishibayashi, S., Yamura, K., & Matsushita, H. (1987). A Rapid Method of Determining the Alkali-Aggregate Reaction in Concrete by Autoclave. (P. Grattan-Bellew, Ed.) *Concrete Alkali-Aggregate Reactions*, 299-303.

Rivard, P., Bérubé, M., Ollivier, J., & Ballivy, G. (2003). Alkali mass balance during the accelerated concrete prism test for alkali-aggregate reactivity. *Cement and Concrete Research, 33*(8), 1147-1153.

Rivard, P., Bérubé, M., Ollivier, J., & Ballivy, G. (2007). Decrease of pore solution alkalinity in concrete tested for alkali-silica reaction. *Materials and Structures, 40*(9), 909-921.

Stanton, T. (1940). Expansion of Concrete through Reaction Between Cement and Aggregate. *Publications of the American Society of Civil Engineers, 66*, 1781-1811.

Tamura, H. (1987). A test method on rapid identification of alkali reactivity aggregates (GBRC rapid method). *Proceedings of the 7th International Conference on Alkali-Aggregate Reaction in Concrete (ICAAR)*, (pp. 304-308). Ottawa, ON, Canada.

Tang, M., Han, S., & Zhen, S. (1983). A Rapid Method for Identification of Alkali Reactivity of Aggregate. *Cement and Concrete Research, 13*, 417-422.

Wood, S., Sutton, A., & Giannini, E. (2016). Tracking Alkali Migration and Leaching in the Autoclaved Concrete Prism Test. *Proceedings of the 15th International Conference on Alkali-Aggregate Reaction in Concrete (ICAAR).* São Paulo, Brazil.

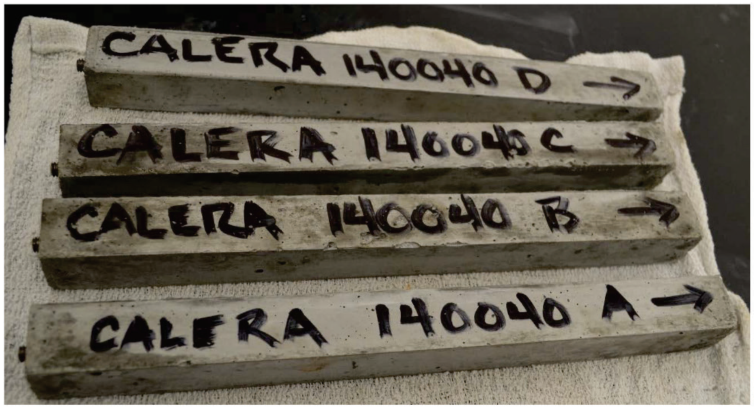
APPENDIX

Photographs of Mortar Bar Sets Before and After Autoclaving

(A)

(B)

Figure A-1: Vulcan Atlanta 150094 (A) before autoclaving and (B) after autoclaving.



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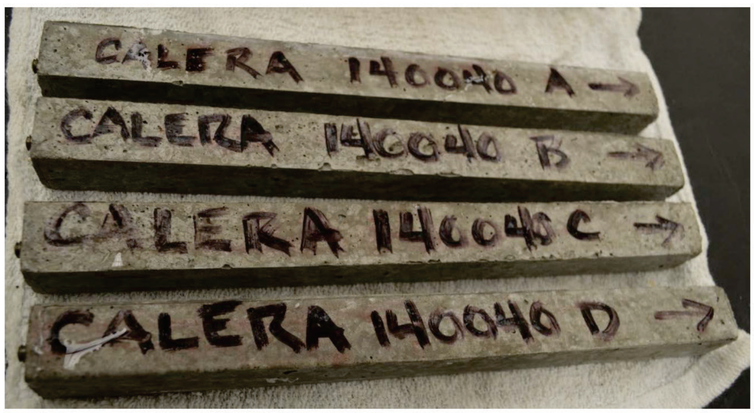


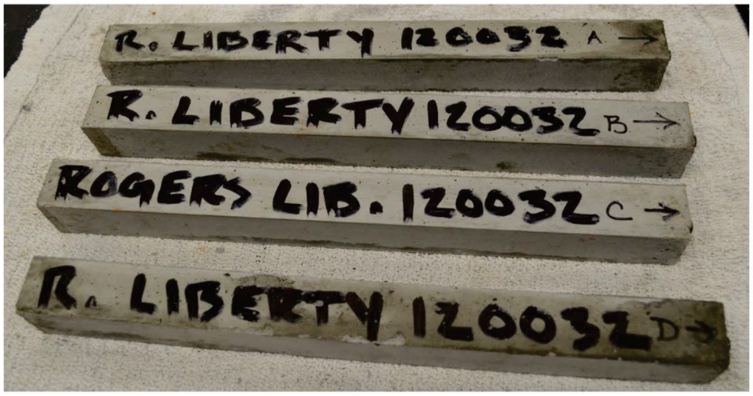
(B)

Figure A-2: Calera 140040 (A) before autoclaving and (B) after autoclaving

(A)

(B)

Figure A-3: Rogers Gordonsville 120016 (A) before autoclaving and (B) after autoclaving



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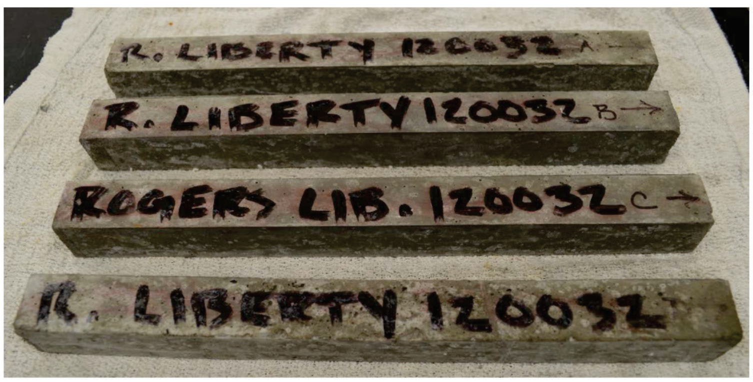


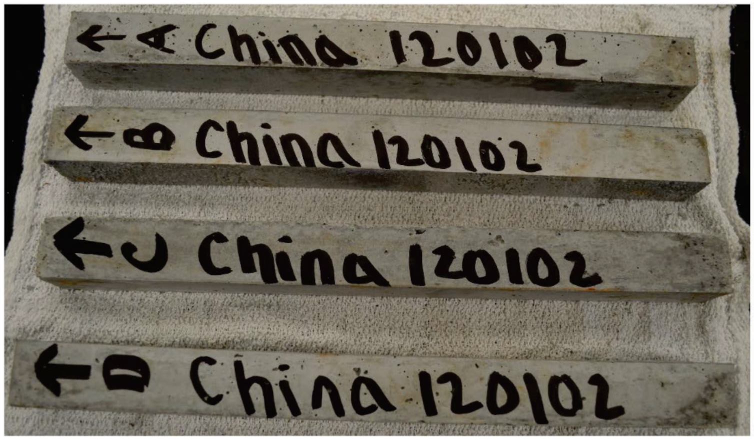
(B)

Figure A-4: Rogers Liberty 120032 (A) before autoclaving and (B) after autoclaving

(A)

(B)

Figure A-5: Adairsville 130038 (A) before autoclaving and (B) after autoclaving



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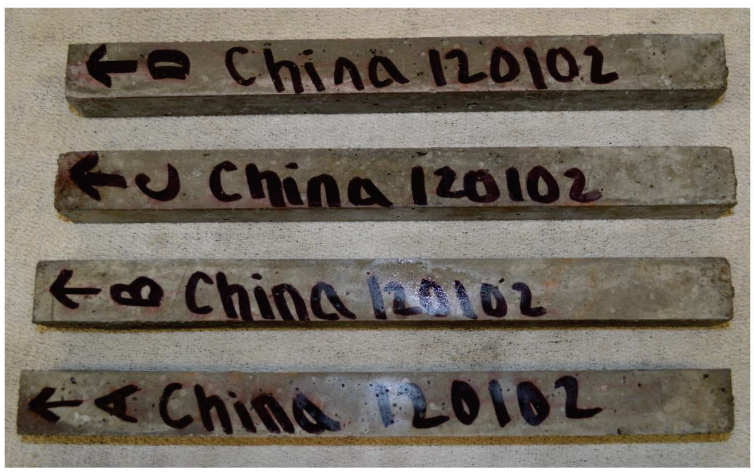
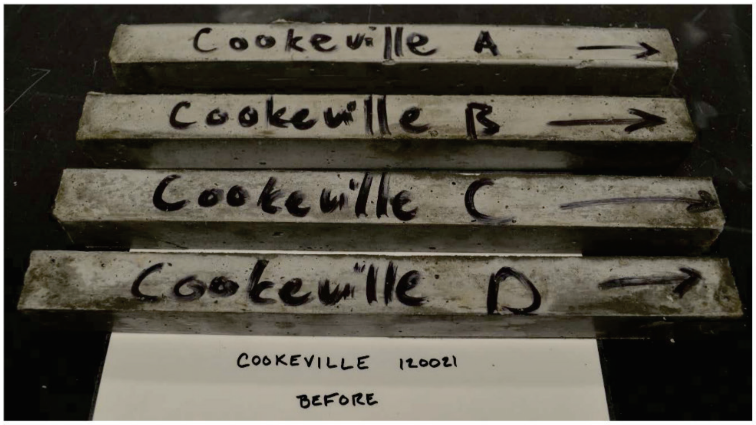


(B)

Figure A-6: China Lake 120102 (A) before autoclaving and (B) after autoclaving



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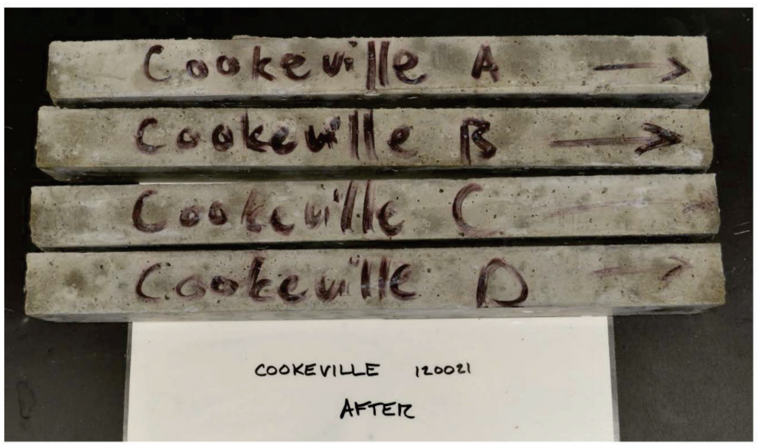
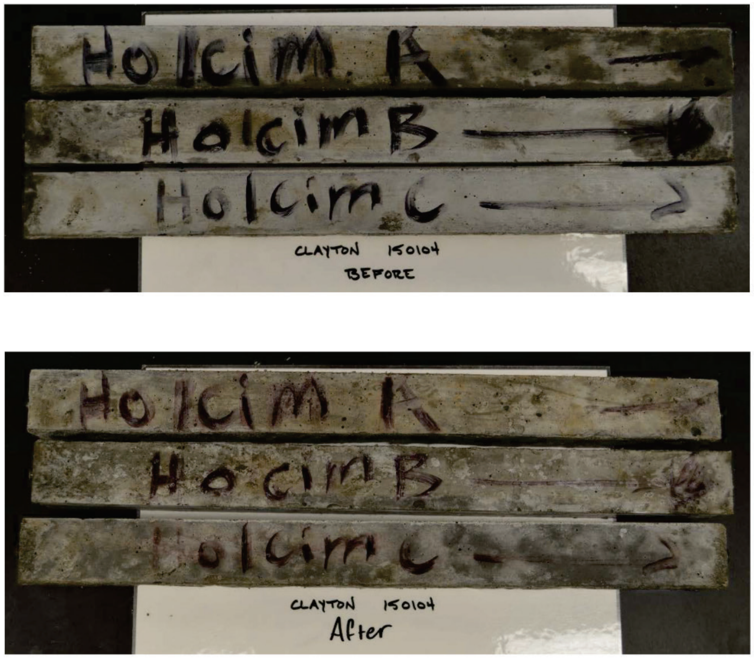


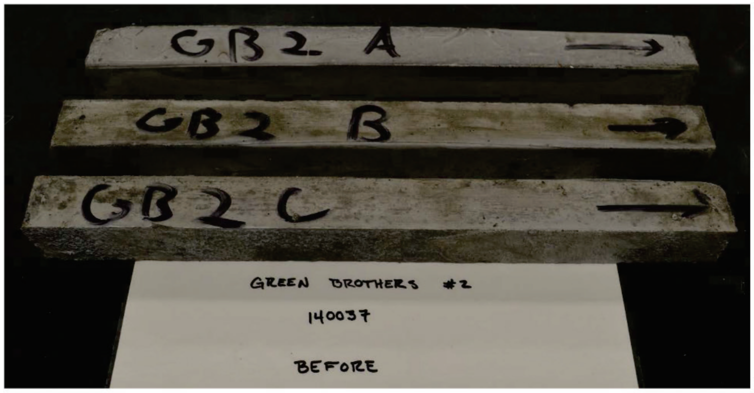
(B)

Figure A-7: Cookeville 120021 (A) before autoclaving and (B) after autoclaving



(A)
(B)

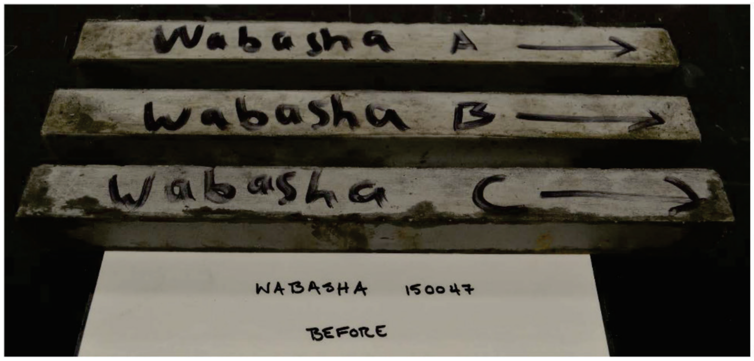
Figure A-8: Clayton 150104 (A) before autoclaving and (B) after autoclaving



(A)

(B)

Figure A-9: Green Brothers #2 140037 (A) before autoclaving and (B) after autoclaving



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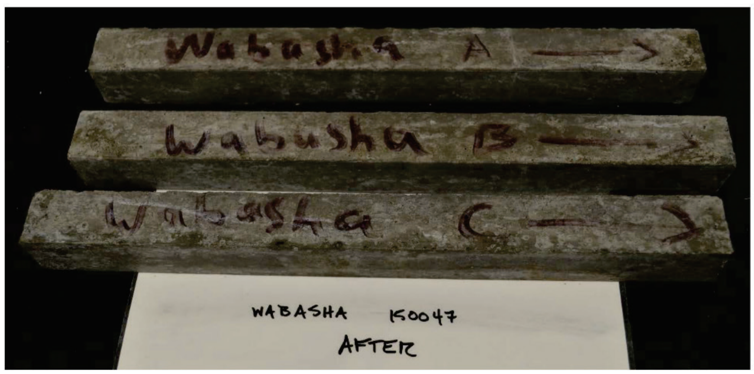
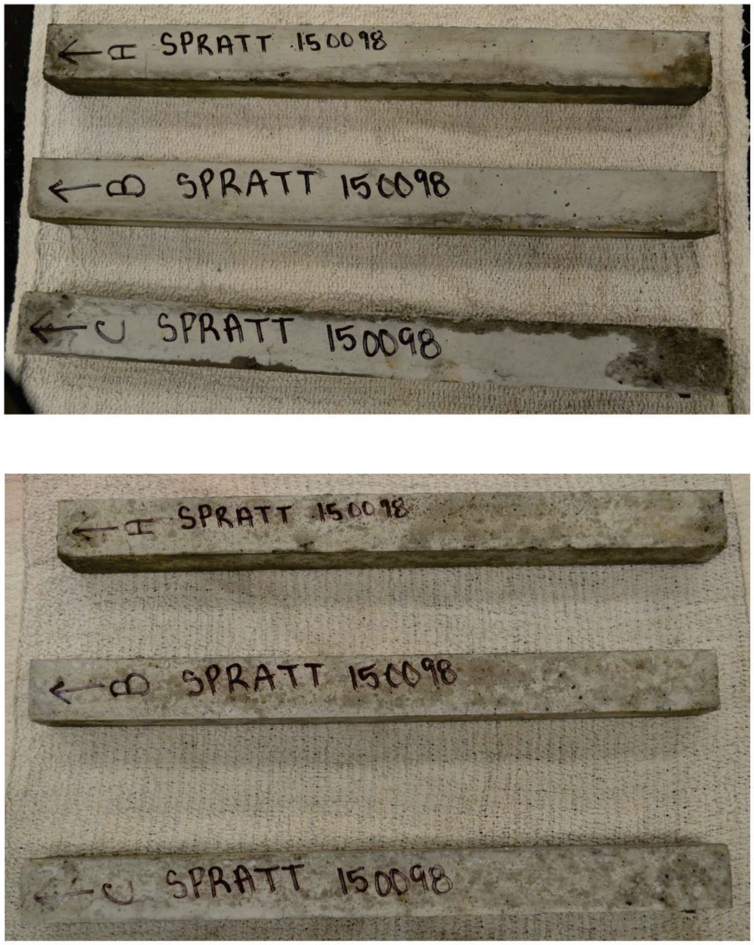


(B)

Figure A-10: Wabasha 150047 (A) before autoclaving and (B) after autoclaving



(A)

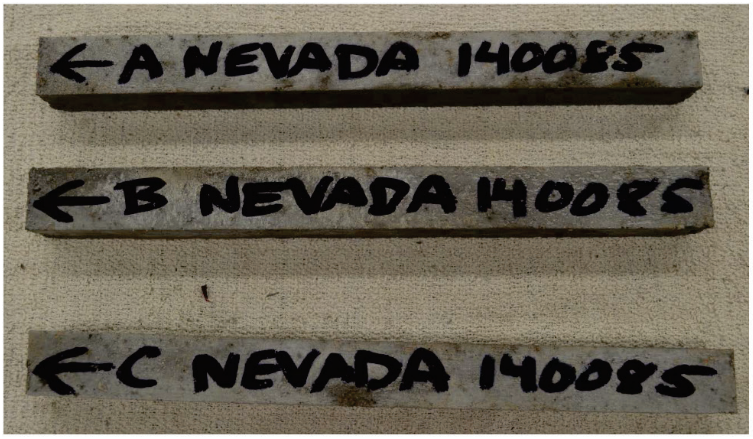
(B)

Figure A-11: Spratt 150098 (A) before autoclaving and (B) after autoclaving

(A)

(B)

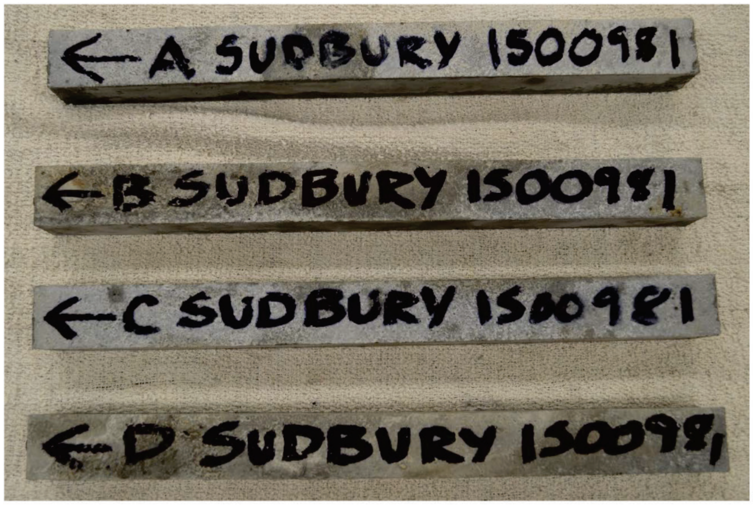
Figure A-12: Gold Hill 130002 (A) before autoclaving and (B) after autoclaving



(A)

(B)

Figure A-13: Nevada Test Site 140085 (A) before autoclaving and (B) after autoclaving



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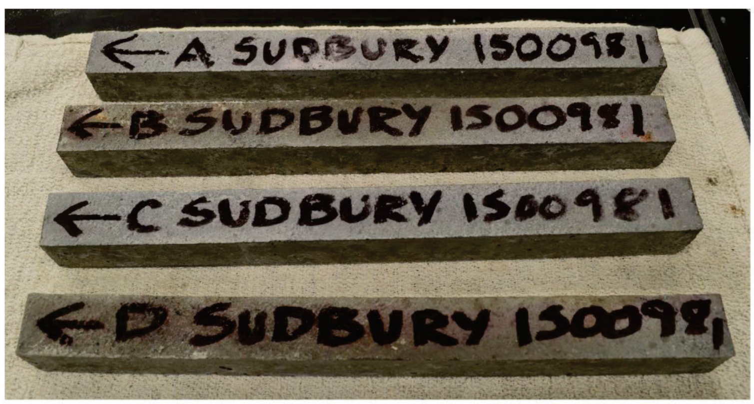

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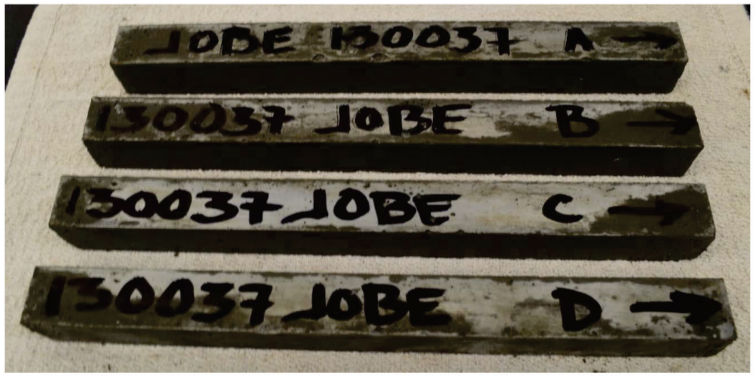
Figure A-14: Sudbury 150098.1 (A) before autoclaving and (B) after autoclaving



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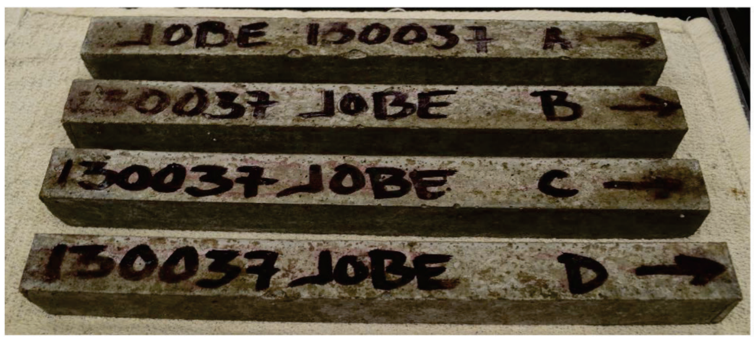


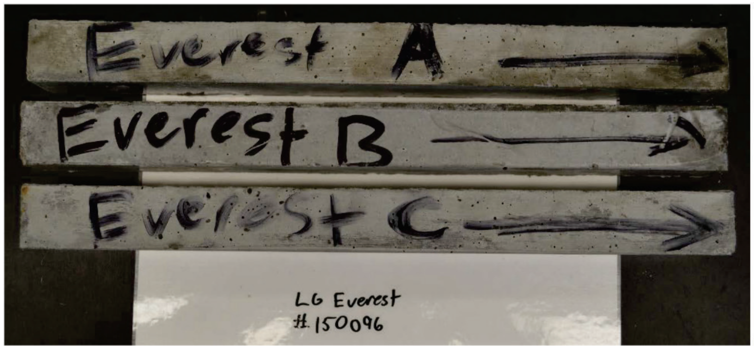
(B)

Figure A-15: Jobe 130037 (A) before autoclaving and (B) after autoclaving

(A)

(B)

Figure A-16: Arkadelphia 152149 (A) before autoclaving and (B) after autoclaving



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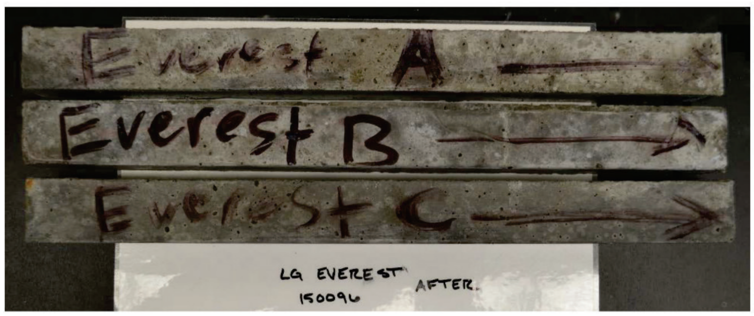
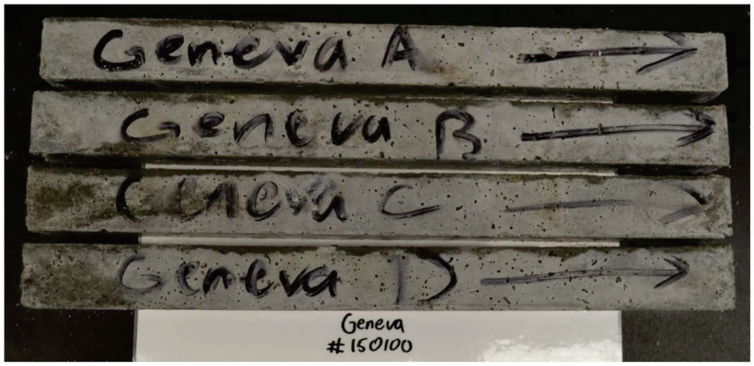
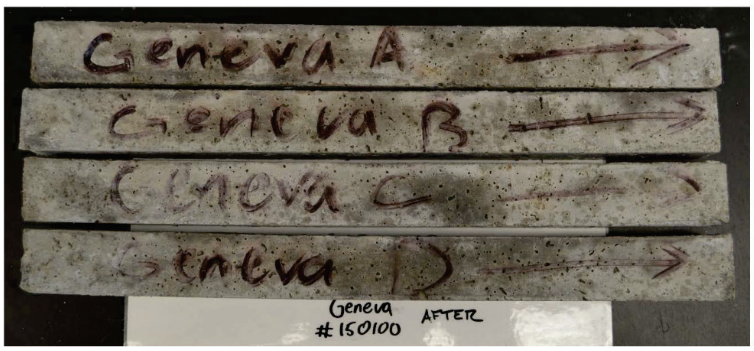


(B)

Figure A-17: LG Everist 150096 (A) before autoclaving and (B) after autoclaving



(A)



(B)

Figure A-18: Geneva 150100 (A) before autoclaving and (B) after autoclaving

(A)

(B)

Figure A-1: Las Placitas 130006 (A) before autoclaving and (B) after autoclaving

A.2 Laboratory 2 Final Report – The University of Texas at Austin

Final Report: SSP TCN 15-032 Round Robin Testing for Alkali-Silica Reactivity of Aggregates using Autoclaving Techniques

University of Texas at Austin

Dr. Thanos Drimalas and Dr. Kevin J. Folliard

August 2016

INTRODUCTION:

This Final Report provides test results for 20 aggregates evaluated for alkali-silica reaction using the accelerated autoclave test method. Aggregates and cement were procured prior to being sent to the University of Texas for testing. All 20 mixtures were cast at the University of Texas at Austin under the supervision of Dr. Thano Drimalas and Dr. Kevin J. Folliard. The goal of this testing was to determine the reactivity of aggregates in order to compare with other laboratories testing the same 20 aggregates. In addition to providing test results, comparisons were made with these test results to existing ASTM C1260 and ASTM C1293 test results to determine the accuracy of the test method.

SCOPE:

The goal of this project was to provide round robin testing of alkali-silica reactivity (ASR) on aggregates (mortar bars) using the rapid autoclave technique. A total of 20 aggregates were evaluated by the Performing Agency to determine their reactivity level, and in order to help determine multi-laboratory precisions. With each of the aggregates the Performing Agency accessed ASR reactivity, measured expansion of samples before and after autoclaving, and photographed samples before and after autoclaving.

METHODS:

Aggregates and cement were sent to the University of Texas at Austin for casting of mortar bars to be evaluated in the autoclave test method. Table 1 provides the aggregates evaluated using the mortar bar autoclave test method. The aggregates were received in ASTM C1260 proportions (1260 grams), and along with 587 grams of a ASTM C150 Type I/II cement. Enough materials were sent to produce four 1X1X11.25" mortar bars.

Prior to mixing, 22.54 grams of NaOH was added to the mixing water (276ml) to boost the alkali loading to 3.5% in the mortar bars. The mixing water was allowed to mix for 1 hour prior to mixing. ASTM C305 was followed for mortar mixing, and once the mixing concluded the mortar was placed into the four mortar bar molds within 2 minutes of completion. The mortar bars were fog cured for 48 hours at 23°C before demolding.

Once removed from the fog room and demolded, the mortar bars were labeled and photographed. Initial length measurements were taken on the four mortar bars at room temperature using a length change comparator. De-ionized water was added to the bottom of the autoclave chamber and the samples were suspended in a basket above the water. Once the samples were placed in the autoclave, the chamber was closed and set to 130°C. Once the temperature and pressure were reached, the testing continued for 5 hours. After the 5 hours, the samples were allowed to air cool for a few minutes and then cooled with tap water for 15 minutes. At this point, the final length change measurement and final photographs were conducted. Samples were then placed into a plastic bag and labeled with aggregate name, serial number and mortar bar autoclave test.

RESULTS:

The autoclave 5-hour test results are shown in Table 1 below. From the 20 aggregates tested, a range of reactivity levels were achieved to compare to other laboratories conducting the same test, and for comparisons with existing test methods such as ASTM C1260 and ASTM C1293. Figures 1 and 2 provide test results comparing the autoclave test results with ASTM C1260 and ASTM C1293 results, respectfully. ASTM C1260 and ASTM C1293 test results were provided to the University of Texas at Austin. An expansion limit of 0.10% was used in Figures 1 and 2 as it seems to provide a good correlation when comparing to existing test methods.

Table 1: Autoclave Test Results

Aggregate Name	Aggregate Serial No.	% Length Change					
		C1260		C1293	Autodave Test	Std. Dev.	COV
		14 Day	28 Day	1 Year	UT		
Cookeville	120021	0.02	0.04	0.12	0.038	0.0007	1.86
Rogers Liberty	120032	0.10	0.16	0.01	0.16	0.0072	4.50
Rogers Gordonville	120016	0.53	0.76	0.31	0.3600	0.0097	2.70
China Lake	120102	0.12	0.23		0.052	0.0008	1.59
Gold Hill, NC	130002	0.33	0.48		0.546	0.0080	1.47
Las Placitas, NM	130006	1.02	1.26	0.17	0.908	0.0119	1.31
Calera	140040.1	0.40	0.49		0.28	0.0213	7.61
Jobe, El Paso TX	130037	0.41	0.61	0.51	0.49	0.0066	1.33
Green Brothers	130041	0.23	0.32		0.22	0.0155	7.05
Green Brothers	140037	0.40	0.54		0.218	0.0181	8.32
Nevada	140085	0.58	0.77		0.085	0.0034	4.03
Wabasha, MN	150047	0.35	0.49		0.162	0.0082	5.06
Adairsville-Atlanta, GA	150093			0.04	0.032	0.0027	8.56
Vulcan-Atlanta, GA	150094	0.08	0.12		0.051	0.0017	3.28
LG Everest Dell Rapids, SD	150096	0.22	0.34	0.15	0.233	0.0059	4.17
Spratt-Canada	150098/130001	0.40	0.52	0.20	0.282	0.0094	3.33
Sudburry-Canada	150098.1/130030	0.26	0.42	0.14	0.2	0.0086	4.29
Clayton/ Yardville	150104				0.055	0.0060	10.89
Arkadelphia	152149	0.46	0.62		0.151	0.0096	6.33
Geneva					0.141	0.0073	5.21

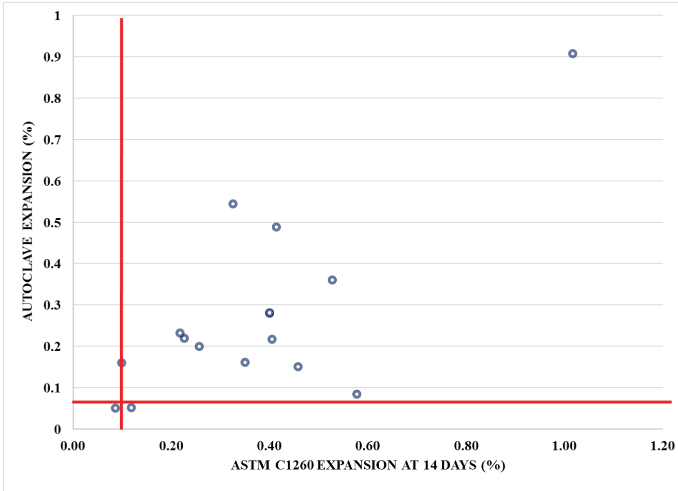


Figure 1: Comparison between ASTM C1260 and Autoclave Expansions

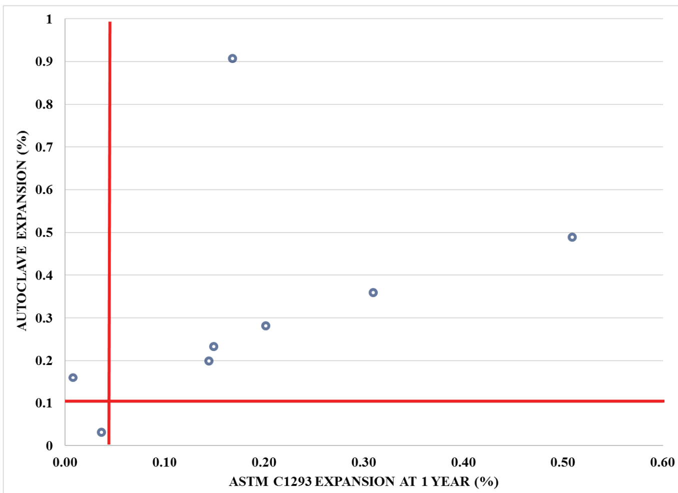


Figure 2: Comparison between ASTM C1293 and Autoclave Expansions

DISCUSSION:

This autoclave test was an easier test method to conduct compared to several other ASTM test methods for determining aggregate reactivity. Overall, the autoclave test does show promising results when compared to ASTM C1260 or ASTM C1293 results as shown in Figures 1 and 2. A wide range of aggregates were chosen with different reactivity levels which allowed for a broad spectrum of expansions. More ASTM C1293 results would benefit these test results to prove the accuracy of the test.

One downfall of the test, is the high temperature and pressure that the mortar bars are placed into during the test. These conditions may result in aggregates that contain certain phases that would normally not react due to them never seeing this high of a temperature. These phases do exist in aggregates such as basalt and may show reactivity in this test method even though they tend to be non-reactive in field concrete. This is also a concern with the ASTM C1260 test method.

With the encouraging results comparing the autoclave test method with some of the existing test methods on aggregate reactivity, future testing that may benefit this test method would be to include mixtures with supplementary cementing materials (SCM's) such as fly ash or slag to determine if they would accurately measure mitigation options for reactive aggregates. It would be of great value to the industry if mitigation measures could be determined within a few days compared to 2 years from some of the existing test methods.

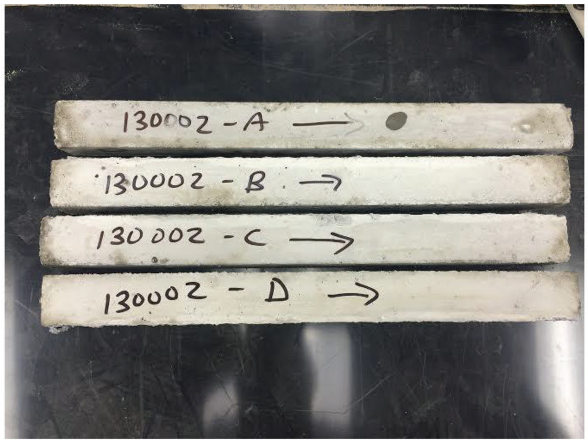
SUMMARY:

The determination of aggregate reactivity was conducted on 20 aggregates using the rapid autoclave technique. The results will be compared to other laboratories to determine multi-laboratory precision on this test method. Results were compared to existing test methods for determine aggregate reactivity and these results were promising in that there was a correlation between them. Overall, this test method can be a promising if it does show good precision between laboratories and good correlation to other test methods and field concrete behavior.

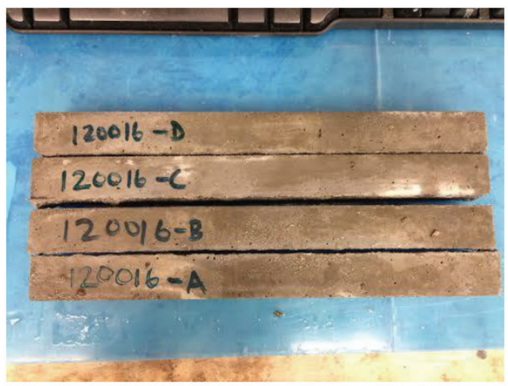
APPENDICES:

Table 2: Raw initial and final measurements for autoclave mixtures

	Initial Raw Measurement				Final Raw Measurement			
Aggregate	a	b	c	d	a	b	c	d
13006	-0.0856	0.0039	-0.0720	-0.0147	0.0037	0.0963	0.0181	0.0767
150104	0.0118	0.0059	0.0034	-0.0210	0.0177	0.0119	0.0079	-0.0153
120032	0.0463	-0.0061	0.1675	-0.1018	0.0618	0.0111	0.1829	-0.0855
130002	0.0438	-0.1110	0.0080	0.0001	0.0975	-0.0556	0.0619	0.0555
140040	-0.0289	0.0080	-0.0211	0.1126	0.0027	0.0342	0.0064	0.1393
152149	-0.0174	0.0052	0.0466	-0.1170	-0.0022	0.0203	0.0603	-0.1006
120021	-0.0206	0.0559	0.0265	0.0933	-0.0167	0.0596	0.0303	0.0971
130037	0.1126	0.0850	0.0451	0.1630	0.1622	0.1344	0.0934	0.2131
120102	0.0422	0.0219	-0.0251	0.0349	0.0475	0.0270	-0.0199	0.0402
120016	0.0354	-0.0214	0.0320	0.0235	0.0703	0.0153	0.0692	0.0587
150098	-0.0164	-0.0173	0.0274	0.0331	0.0128	0.0114	0.0558	0.0598
150098.1	-0.0116	0.0501	0.0101	-0.0367	0.0069	0.0703	0.0305	-0.0160
150093	0.0689	-0.0547	0.0360	0.0980	0.0723	-0.0512	0.0391	0.1008
150094	-0.0317	-0.0537	0.0038	-0.0111	-0.0267	-0.0489	0.0090	-0.0059
140037	-0.0812	-0.0004	-0.0018	-0.0106	-0.0598	0.0237	0.0173	0.0118
Nevada	0.0042	0.0891	0.0513	-0.0222	0.0130	0.0977	0.0599	-0.0143
LG Everest	0.0410	0.0858	-0.0077	-0.0192	0.0558	0.1002	0.0060	-0.0059
Geneva	-0.0329	-0.0267	0.0428	-0.0400	-0.0181	-0.0129	0.0558	-0.0253



Aggregate 130002



Aggregate 120016 Post Autoclave

Aggregate 120021 Post Autoclave

Aggregate 120032 Post Autoclave

Aggregate Geneva Post Autoclave

Aggregate LG Evrest Post Autoclave

Aggregate Nevada Post Autoclave

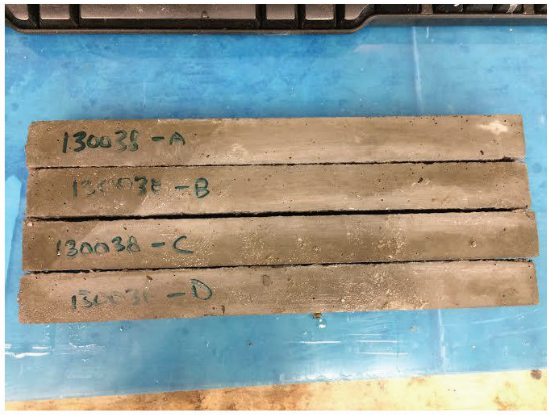
Aggregate 120102 Post Autoclave

Aggregate 13002 Post Autoclave

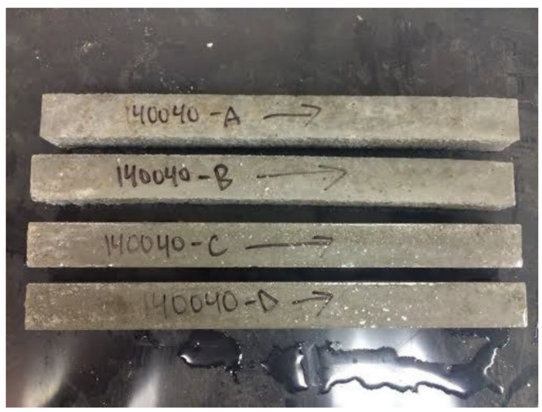
Aggregate 13006 Post Autoclave

Aggregate 13006 Pre-Autoclave

Aggregate 130037 Pre-Autoclave

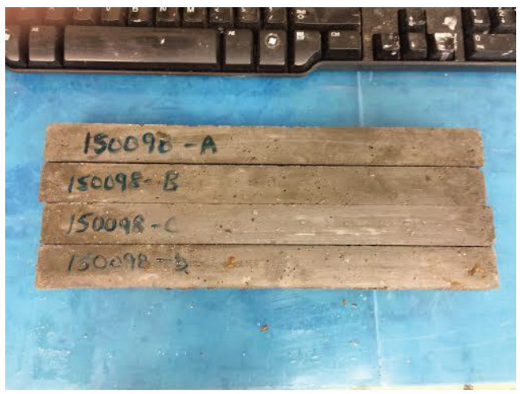


Aggregate 130038 Post Autoclave

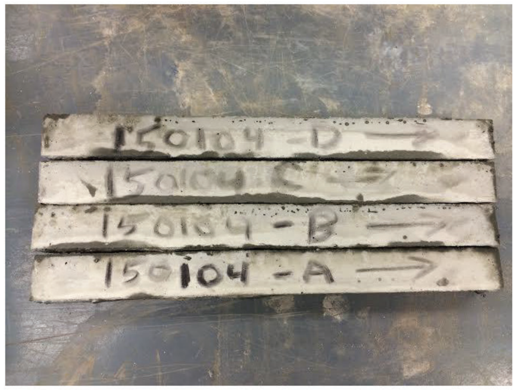


Aggregate 140040 Post Autoclave

Aggregate 150094 Post Autoclave



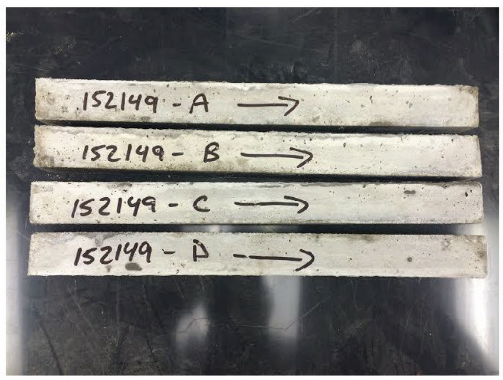
Aggregate 150098 Post Autoclave



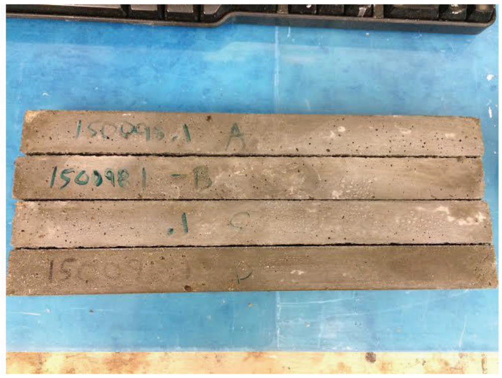
Aggregate 150104 Pre-Autoclave

Aggregate 150104 Post Autoclave

Aggregate 152149 Post-Autoclave



Aggregate 152149 Pre-Autoclave



Aggregate 150098.1 Post Autoclave

A.3 Laboratory 3 Final Report – Clemson University

Battelle Memorial Institute

Round Robin Testing of Alkali-Silica Reactivity of Aggregates Using Autoclaving Techniques

Scientific Services Program
TCN 15-032

Under Prime Contract / Solicitation No. W911NF-11-D-001

FINAL REPORT

Submitted By:

Prasada Rao Rangaraju, Ph.D., PE
Hassan Dezfouli Rashidian

Glenn Department of Civil Engineering
College of Engineering, Computing and
Clemson University
Clemson, South Carolina USA

February 21, 2017

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EXECUTIVE SUMMARY

Existing test methods to evaluate alkali-silica reactivity of aggregates such as Accelerated Mortar Bar Test (ASTM C1260), concrete prism test methods (ASTM C1293) and Miniature Concrete Prism Test (AASHTO TP110) take considerably long time to assess the potential reactivity of aggregates. While these tests methods provide a more reliable basis for selection of aggregates and/or of ASR mitigation measures, the results from these tests are of little value in situations that require rapid assessment, particularly in contingency construction operations that support the rapid and reliable fielding of weapon systems in military installations. While significant research has been conducted in the past in developing rapid test methods such as autoclave test methods, there has not been a concerted effort in standardizing one method or studying the variability in the test results to develop a degree of reliability. Recent efforts by US Army Corps of Engineers, Engineering Research and Development Center (ERDC) have selected an autoclave method that appears to show promise in rapid identification of aggregate reactivity. The research effort presented in this report is part of a larger study being conducted by ERDC to evaluate the variability in the autoclave test results, induced by operators and the testing equipment, using a large number of well characterized aggregates and a portland cement.

This report summarizes the findings from autoclave testing conducted at Clemson University on a set of 20 aggregates that were carefully characterized for their mineralogy and alkali-silica reactivity using the standard ASTM C 1260 test method. Parallel testing with same set of materials were conducted at University of Alabama, Tuscaloosa, University of Texas at Austin and Construction Technology Labs (CTL) and ERDC. Findings from the work conducted at Clemson showed that results from autoclave testing yielded a good correlation between the results from the two test methods for most aggregates, although some aggregates appear to deviate. Similarly, the percent loss in dynamic modulus of elasticity of test specimens correlated well with the magnitude of expansion observed in the autoclave test method. Further studies are needed to identify specific influence of autoclave testing conditions on the degree and nature of reactivity in aggregates having specific mineralogies to improve reliability of this test.

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CHAPTER 1 – INTRODUCTION

Alkali-silica reaction (ASR) in concrete was first recognized by Stanton in the late 1930s as a source of concrete deterioration [Stanton, 1940]. ASR is a chemical reaction between alkali hydroxides present in the pore solution of concrete and reactive forms of silica (SiO_2) found in certain aggregates (e.g.: siliceous limestone, quartzite, greywacke, granitic-gneiss, rhyolite, etc.). The alkali hydroxides are generally derived from alkali sulfates present in cement, although other sources of alkalis that can be either internal and/or external to the matrix of concrete such as aggregates, supplementary cementitious materials, chemical admixtures and deicing chemicals can also contribute to the alkali loading in concrete. The result of ASR is the formation of a hydrous gel, often referred to as ASR gel. Formation of the ASR gel alone in itself may not be as deleterious to concrete as when the ASR gel absorbs moisture and shows a tendency to swell. Depending on the viscosity of the ASR gel and the microstructure of the surrounding concrete, the swelling of the ASR gel can result in significant tensile stresses to generate in the matrix of concrete causing cracking and consequent deterioration in the integrity of the structure.

Soon after the initial discovery of ASR distress in concrete by Stanton, several test methods have been developed to help identify reactive nature of the aggregates, including Stanton himself [Stanton, 1943]. The early test methods included mortar bar test, which was later standardized as ASTM C227 test method, quick chemical test (ASTM C289), gel pat test and others. Subsequently many other variants of these test methods were developed, that either reduced the duration of the test methods or addressed the reliability of the results from the tests. Vast majority of literature pertaining to alkali-silica reaction and the associated test methods and its mitigation has been published in the proceedings of the 14 international conferences that were held on alkali-aggregate reactions in concrete, with the most recent conference being the one held at Sao Paolo, Brazil in 2016.

Among the test methods that have gained industry acceptance the Accelerated Mortar Bar Test (AMBT) (e.g., ASTM C1260, CSA A23.2–25A, RILEM TC191-ARP-

AAR2), originally proposed by Oberholster and Davis in 1986 has widely been adopted as an accelerated test method for evaluating alkali-silica reactivity of aggregate [Oberholster and Davis, 1986]. On the other hand, the Concrete Prism Test (CPT) (e.g., ASTM C1293, CSA A23.2–14A, RILEM TC191-ARP-AAR3) is recognized as the most reliable test procedure which requires at least one or two years for results depending upon the purpose of the test. The long duration required in the concrete prism test renders this method impractical for use in routine testing and evaluation of aggregate materials. The limitations of AMBT and CPT test methods have spurred research in development of new test procedures that are rapid and reliable in evaluating aggregate reactivity and efficacy of ASR mitigation measures. More recently, a Miniature Concrete Prism Test (MCPT, AASHTO TP110) has been developed to assess the aggregate reactivity in a more reliable manner than AMBT but in much shorter duration than CPT of only 8 weeks [Latifee and Rangaraju, 2015].

While these tests methods provide different degrees of reliability in selection of aggregates and/or of ASR mitigation measures, the results from these tests are of little value in situations where rapid assessment of aggregates is required particularly in contingency construction operations that support military installations. However, even MCPT takes 56 days to assess the aggregate reactivity.

To address the need of rapid assessment of aggregate reactivity, several autoclave based test methods have been investigated in the past [Tamura et al., 1989; Nishibayashi et al., 1991; Fournier et al., 1991; Giannani and Folliard, 2013]. However, the reliability of these methods has not been studied in a systematic manner. The research effort presented in this report is part of an effort by the US Army Corps of Engineers, Engineering Research and Development Center (ERDC) to conduct a controlled study on a selected number of aggregates to evaluate the variability in the test results from a defined autoclave-based ASR test method. This report summarizes the findings from this testing conducted at Clemson University on a set of 20 aggregates that were carefully characterized for their mineralogy and alkali-silica reactivity using the standard ASTM C 1260 test method. Parallel testing with same set of materials were conducted at

University of Alabama, Tuscaloosa, University of Texas at Austin and Construction Technology Labs (CTL) and ERDC.

CHAPTER 2 – LITERATURE REVIEW

A detailed review of several different autoclave-based test methods for assessing alkali-reactivity of aggregates was provided by Grattan-Bellew [Grattan-Bellew, 1997] and Thomas et al. [Thomas et al., 2006]. A summary of this review is presented in this report to serve as a source of information and provide a context to the work conducted in this research study.

Generally, autoclave test methods involve testing mortar specimens that are prepared by using high alkali cement and aggregate that is in question [Tamura et al., 1989; Nishibayashi et al., 1991; Fournier et al., 1991; Tang et al. 1994], although the use of concrete prisms has also been recently explored [Giannani and Folliard, 2013]. In these test methods the alkali content ($Na_2O_{eq.}$) of the mortar/concrete mixture is boosted, by addition of reagent grade alkali hydroxides to the mix water to supplement the alkali contribution from the cement, to achieve a total alkali loading ranging from 1.5% to 3.5% $Na_2O_{eq.}$ by weight of cement. The purpose of boosting the alkali levels in test specimens to such high levels is to accelerate the reaction and reduce the test duration needed to characterize aggregate reactivity. Also, the use of an autoclave allows the test specimens to be subjected to a combination of high temperature and pressure, which can accelerate the alkali-silica reaction in test specimens. A survey of literature on different autoclave test methods showed that typical peak temperature employed ranged between 110°C and 140°C and pressure ranged between 0.15 MPa and 0.20 MPa [Tamura et al., 1989; Nishibayashi et al., 1991; Fournier et al., 1991; Giannani and Folliard, 2013; Tang et al. 1994]. The test duration employed in the typical autoclave test methods ranged between 4 and 6 hours, that includes the ramping times between ambient temperature and the peak temperature both at the beginning and the conclusion of the tests. In one particular study, 24 hour autoclave test duration was found suitable, although larger concrete prisms were used as test specimens rather than mortar bars which are typically employed [Giannani and Folliard, 2013]. The change in the length of the test specimens resulting from the ASR induced in the autoclave test is used as a basis to characterize the reactivity of the aggregates. Analysis of expansion results from autoclave-based testing for ASR was based on threshold expansion levels, typically ranging between 0.10% and 0.15% for mortar bar test specimens.

Among the differences across the different autoclave test methods proposed in literature, dimensions of the test specimens, total alkali content of the mixture, exposure conditions in terms of the magnitude and duration of the temperature and pressure in the autoclave are the principal variables [Tamura et al., 1989; Nishibayashi et al., 1991; Fournier et al., 1991; Tang et al. 1994]. Also, virtually all of the autoclave test methods are conducted immediately after curing the mortar bars in the fog room for 24 hours or 48 hours. The autoclave test method standardized by French as AFNOR P18-588 employ an additional 4 hours of curing the mortar bars in steam curing after an initial 24 hours curing in fog room [Grattan-Bellew, 1997]. Thus, the entire duration needed for testing the aggregate in any of the autoclave test method is typically no more than 2 or 3 days.

While autoclave test methods do provide a rapid assessment tool, there is concern about the validity of the test results when compared with results from the more established methods such as the ASTM C1260 and ASTM C1293 test methods. Xiaofang reported that expansion observed in autoclave test method for mortar bars containing coarse grained calcitic and dolomitic aggregates (with grain size > 1.25 mm) was due to the differential expansion between *a* and the *c* crystallographic axes of the mineral rather than due to alkali-silica reaction in the aggregate [Xiaofang, 1996]. Fournier and Berube suggest from their research that the mortar bar expansions observed in test methods that use elevated temperature such as autoclave test methods may be due to swelling of the clay minerals, particularly when evaluating non-reactive limestones [Berube and Fournier, 1992].

One of the concerns with the autoclave test method is the variability in the test results. Literature on variability in test results from selected autoclave test methods has shown that the coefficient of variation (COV) can range between 15% and 30% for the Chinese autoclave method and between 12% and 16% for the GBRC method proposed by Tamura et al [Corneille, A. and Bollotte, B, 1994]. Other autoclave test methods have not been evaluated rigorously.

The purpose of this research effort is to participate in a collaborative effort with 4 other participants – ERDC, University of Alabama, University of Texas, and Construction Technology Labs, Inc. (CTL) to study the variability of an autoclave test method using a given set of materials that include a Type I/II portland cement and twenty aggregates of different mineralogy and reactivity. The collective results from this database would then be used to develop guidance on limits on variability in the autoclave test results.

CHAPTER 3 – EXPERIMENTAL PROGRAM

3.1 SCOPE OF THE STUDY

The scope of this project is to evaluate the alkali-silica reactivity of 20 different aggregates that were provided by ERDC using the specified autoclave test method. The expansion behavior of the mortar bars prepared with selected aggregates in the autoclave test would then be compared with the expansion behavior in the accelerated mortar bar test method (ASTM C1260 test). In addition, the mortar bar expansion values from the autoclave test method were also compared with the measured percent loss in dynamic modulus of elasticity values to ascertain the degree of damage induced by ASR distress. For this purpose the dynamic modulus of elasticity of test specimens were measured before and after conducting the autoclave test. Also, the visual condition of the test specimens was documented before and after the autoclave test to examine and assess the extent of damage.

3.2 MATERIALS AND TEST METHODS

Materials

For preparing mortar mixtures, ASTM C150 Type I-II Portland cement from Holcim (US) Inc. from their Ste. Genevieve plant was used. The alkali content of the cement was reported as 0.54% $Na_2O_{eq.}$ mill certificate, however upon independent testing by ERDC, the alkali content of the cement was found to be only 0.524% $Na_2O_{eq.}$ The cement had a Blaine specific surface area of 386 m^2/kg and a clinker composition of 61%, 7%, 6% and 10% of C_3S, C_2S, C_3A and C_4AF, respectively, along with a sulfate content of 3.6%. A mill certificate showing the oxide composition and other physical properties of the cement is attached in Appendix A. A reagent grade sodium hydroxide was used to boost alkali level of the mixtures. The aggregates that were used in this project are listed in Table 1 along with their code number assigned by ERDC for this project. Information pertaining to the mineralogy of aggregate used in the study was provided by ERDC and is included in this report as Appendix B.

Table 1. List of the aggregates used in this project

Number	Code	Name	Aggregate Type
1	120016	Rogers Gordonville	Coarse
2	120021	Cookeville	Coarse
3	120032	Rogers Liberty	Coarse
4	120102	China Lake	Coarse
5	130002	Gold Hill, NC	Coarse
6	130006	Las Placitas, NM	Coarse
7	130037	Jobe El Paso, TX	Coarse
8	130038	Adairsville Atlanta, GA	Fine
9	130041	Green Brothers, MS	Fine
10	140037	Green Bros	Fine
11	140040	Calera	Coarse
12	140085	Nevada	Fine
13	150047	Wabasha	Fine
14	150094	Vulcan Lithia, GA	Fine
15	150096	Dell Rapids	Fine
16	150098	Spratt, Canada	Coarse
17	150098.1	Sudbury, Canada	Coarse
18	150100	Geneva	Coarse
19	150104	Yardville Great Eastern	Fine
20	152149	Arkadelphia	Fine

Preparation of Materials

In this project, aggregates and cement provided by ERDC were used as-received with no further preparations. Some of these aggregates were fine aggregates while others were coarse aggregates that were crushed to meet aggregate gradation requirements per ASTM C1260 test. Graded aggregate from each source were received in sealed plastic bags and weighed 1320 grams, along with a sealed bag of portland cement containing 587 g of Type I-II portland cement. The materials supplied were sufficient to cast a set of four 1" x 1" x 11.25" mortar bars for each of the aggregate sources. In preparing the mortars, 276 ml of tap water was used for each set. In this study, the alkali content of the mortar mixture was fixed at 3.5% $Na_2O_{eq.}$ by weight of the cement. Considering that the alkali content of the cement was only 0.524% $Na_2O_{eq.}$, the alkali content of the mixture was further boosted to 3.5% $Na_2O_{eq.}$by weight of cement, by addition of 22.54 g of reagent grade NaOH to the mix water. Figure 1 shows a picture of the molds and the typical materials used in preparing the mortar mixtures. The mixture proportions of the ingredients used in preparation of mortar mixtures for this project are presented in Table 2.

Figure 1. Sample Molds and As-Received Aggregates and Cement Materials for the Project

Table 2. Mixture Proportions

Material	Aggregates	Cement	Water	NaOH
Weight (g)	1320	587	276	22.54

Mixing Procedure

For the purpose of mixing mortars, a Horbart-N50A mortar mixer was used. The first step of the procedure was to dissolve the required amount of NaOH pellets in 276 ml of water and make a NaOH solution. Then, the cement and the solution were introduced in to the mixer and mixed for 30 seconds at the low speed. At this point, aggregate was added slowly (30 seconds) to the mixture and it was mixed for an additional 30 seconds at the medium speed. The mixture was then allowed to rest for 90 seconds followed by mixing at a medium speed for 60 seconds to complete the mixing sequence.

Molding and Curing

After the mixing process, mortar was placed in two sets of a 2-gang prism mold to yield mortar bars of 1" x 1" x 11.25" dimensions with gage studs at the ends for length-change measurements.. Mixtures were placed and vibrated in two layers to form a cohesive prism, and after labeling, they were placed in a standard curing room. After 48 h, specimens were taken out from the curing room and their molds were removed. Finally, aggregate serial number and the specimen's code number were written on the surface of each specimen. At this point, zero reading was taken and recorded for each of specimens. Figure 2, shows a picture of the specimens after the casting (left) and after demolding and labeling (right).

Curing in Autoclave, Length-Change and Dynamic Modulus of Elasticity Measurements

In this study, a BOEKEL cement autoclave (Model 25515016) was used (shown in Figure 3) for conditioning the mortar bars to accelerate ASR. Before the zero-day length change readings were taken, the mortar bars were tested using a Grindo-Sonic MK5 unit for their initial dynamic modulus of elasticity measurement per ASTM E1876-15. At this point, the zero-day length reading of the mortar bars was taken using a length comparator, conforming to ASTM C 157 specification, and recorded along with the length measurement on the invar reference bar. After the zero-day length reading was taken, the specimens were placed in the autoclave along with 500 ml of water.

Figure 2. Specimens just after the molding (left), and specimens after 48 h of curing (right)

Figure 3. BOEKEL Autoclave Used in this Project

The autoclave needed approximately 30 minutes to ramp up from ambient temperature to 130°C and a pressure of 35 psi, the conditioning parameters set for this research study. After reaching the desired stable temperature and pressure, the mortar bars were conditioned in the autoclave for duration of 5 hours to promote ASR. The autoclave would then be turned off and allowed to cool to 90°C and then samples would be removed and placed in a preheated water bath maintained at 80°C. Temperature of the water (with specimens immersed in it) would be reduced by addition of cold water to ambient temperature over a span of 15 minutes. Finally the samples would be removed from the water, dried with a towel and then their length change would be measured. At this time, the final measurement on the dynamic modulus of elasticity was taken to determine the percent loss in the stiffness of the test specimens.

CHAPTER 4 – RESULTS AND DISCUSSION

4.1 AUTOCLAVE EXPANSION TEST RESULTS

The autoclave expansion results of each mixture are presented in Figure 4 and the numerical data along with the standard deviation and the coefficient of variation are shown in Table 3. These results have been sorted by the expansion levels of the individual aggregates, starting with the mixture having the lowest expansion to the mixture having the highest expansion.

Table 3. Mortar bar expansion results from autoclave test

Number	Code	Name	Expansion (%)	STD (%)	COV (%)
1	120021	Cookeville, TN	0.062	0.002	3.1%
2	150094	Vulcan Lithia, GA	0.062	0.002	3.5%
3	130038	Adairsville Atlanta, GA	0.073	0.002	3.0%
4	150104	Yardville Great Eastern	0.083	0.005	5.7%
5	120102	China Lake	0.105	0.002	2.1%
6	150047	Wabasha	0.167	0.006	3.7%
7	120032	Rogers Liberty	0.177	0.009	5.2%
8	150096	Dell Rapids	0.189	0.004	2.0%
9	150100	Geneva	0.226	0.009	3.8%
10	140037	Green Bros	0.249	0.015	6.1%
11	140085	Nevada	0.251	0.008	3.3%
12	130041	Green Brothers, MS	0.279	0.021	7.6%
13	150098.1	Sudbury, Canada	0.295	0.009	3.2%
14	140040	Calera	0.324	0.013	3.9%
15	152149	Arkadelphia	0.329	0.012	3.6%
16	150098	Spratt, Canada	0.335	0.008	2.4%
17	120016	Rogers Gordonville	0.395	0.017	4.3%
18	130037	Jobe, El Paso, TX	0.433	0.003	0.7%
19	130002	Gold Hill, NC	0.536	0.005	0.9%
20	130006	Las Placitas, NM	0.759	0.005	0.7%

The results from these tests showed that the lowest and the highest ASR-expansion occurred in mixtures containing Cookeville aggregate (ID 120021) and the Las Placitas aggregate (ID

130006). These results were calculated based on average expansion of four mortar bars for each of the mixtures. The standard deviation and the coefficient of variation (COV) of the mixtures are reported in Table 3. Figure 5 shows the COV of expansion results of the aggregates in the same order as presented in Figure 4.

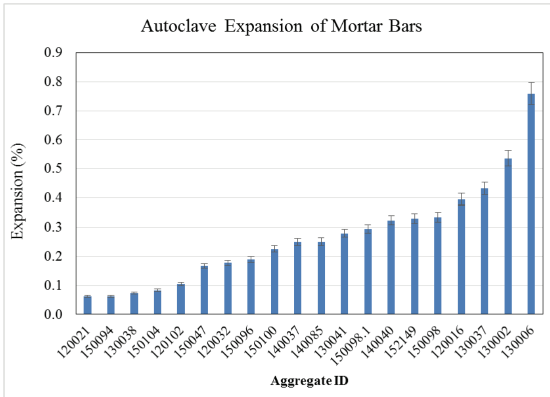


Figure 4. Expansion results of mortar bars made with different type of aggregates

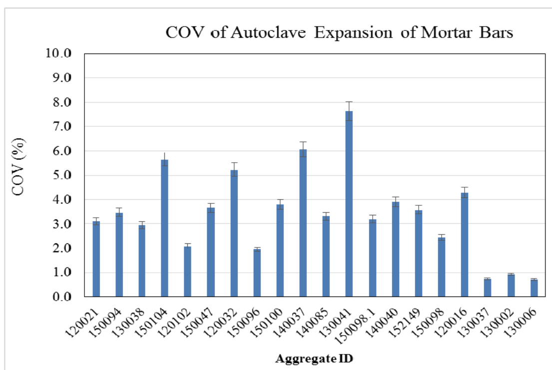


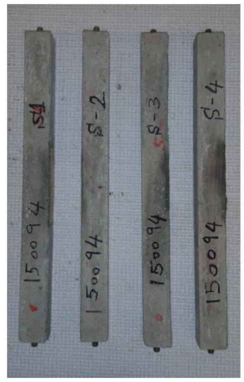
Figure 5. COV of autoclave expansion results of mortar bars made with different type of aggregates

From these results, it can be seen that the COV of autoclave expansion results for any given aggregate source range between 0.7% to 7.6%, which is lower or comparable to acceptable COV level from other typical test methods employed to evaluate mortar and concrete properties. Comparing the trends between the results shown in Figures 4 and 5, there does not appear to be any particular trend that can be observed between the COV of autoclave results and the magnitude of expansion in the autoclave test; however, it is noteworthy that the COV is lowest in three mixtures containing the three most reactive aggregates based on the autoclave expansion values.

4.2 PHOTOGRAPHIC DOCUMENTATION OF DAMAGE IN MORTAR BAR SPECIMENS

To supplement the expansion data, visual observations were made on the mortar bars to document any signs of cracking by taking photographs soon after the final length-change reading was taken on the mortar bar specimens. Figures 6, 7, 8 and 9 show photographs of mortar bar specimens after being subjected to the autoclave test. The number listed next to the aggregate source in these Figures is the average expansion value of the mortar bars in the autoclave test. Figures 6 and 7 show mortar bars with expansion level below 0.28%, while Figures 8 and 9 show mortar bars that experienced expansion levels above 0.28%.

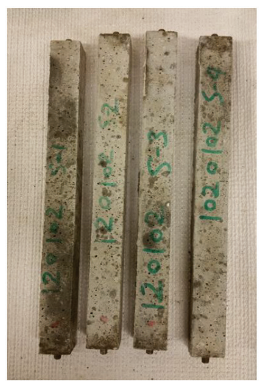
Cookeville – 0.062%



Vulcan Lithia – 0.062%

Adairsville Atlanta – 0.073%

Yardville Great Eastern – 0.083%



China Lak – 0.105%

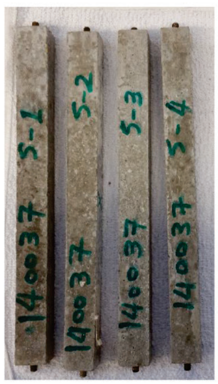
Wabasha – 0.167%

Figure 6 – Photographs of Mortar Bars Subjected to Autoclave Test (exp < 0.28%)

Rogers Liberty – 0.177%

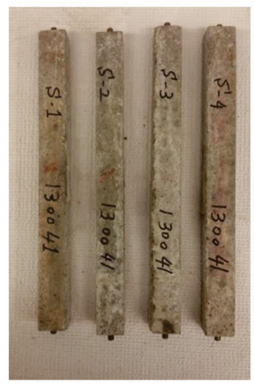
Dell Rapids – 0.189%

Geneva – 0.226%



Green Bros – 0.249%

Nevada – 0.251%

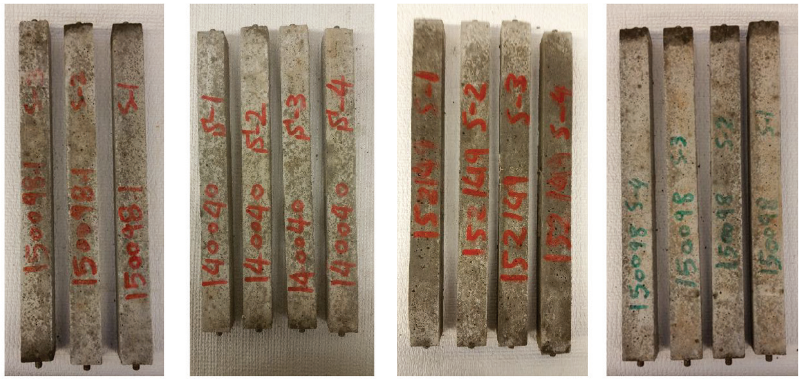


Green Brothers – 0.279%

Figure 7 – Photographs of Mortar Bars Subjected to Autoclave Test (exp < 0.28%)

With vast majority of aggregates, even though a significant level of expansion was registered in the mortar bars (up to 0.28% expansion), there were no signs of visual evidence in mortar bars to suggest extensive cracking. For mixtures with higher expansion (> 0.28%), however, minor cracking could be observed on the surface of the mortar bars. Photographs of mortar bars made with four different aggregates that resulted in expansion between 0.29% and 0.34% are shown in

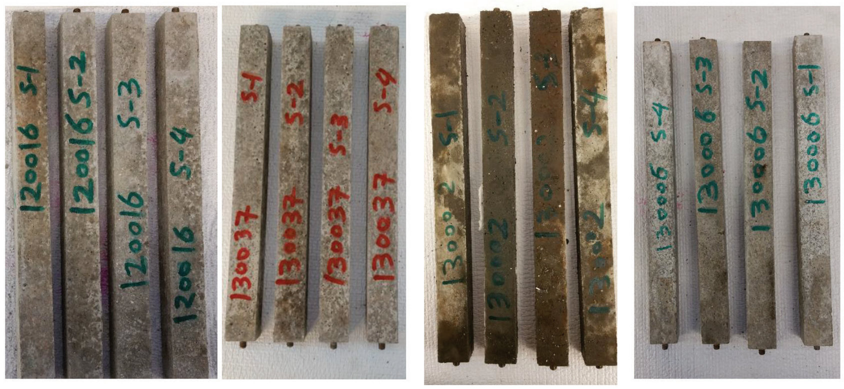
Figure 8. In addition, photographs of mortar bars that showed expansion between 0.40% and 0.76% are shown in Figure 9. These two sets of mortar bars exhibited some signs of cracking on the surface although the evidence was not compelling in some cases. Although, the purpose of this investigation was not to investigate the microstructural characteristics or changes that result in the expansion and cracking of the mortar bars, it is important to study these aspects in order to better explain behavior of different aggregates in the autoclave test method and the manner in which they manifest the ASR distress.



Sudbury- 0.295% Calera – 0.324% Arkadelphia – 0.329% Spratt – 0.335

Some cracks on Sudbury specimens Crack seen on Calera specimens

Figure 8. Photographs of Mortar Bars Subjected to Autoclave Testing (exp > 0.29% & < 0.40) View of the mortar bars made with Sudbury, Calera, Arkadelphia and Spratt (top). Cracks seen on Sudbury and Calera specimens (bottom)



Rogers Gordonville - 0.395% | Jobe El Paso – 0.433% | Gold Hill – 0.536% | Las Placitas– 0.795

Some cracks on Gold Hill specimens | Crack seen on Las Placitas specimen

Figure 9. Photographs of Mortar Bars Subjected to Autoclave Testing (exp > 0.40% & < 0.70) View of the mortar bars made with Rogers Gordonville, Jobe El Paso, Gold Hill and Las Placitas (top). Cracks seen on Gold Hill and Las Placitas specimens (bottom)

4.3 DYNAMIC MODULUS OF ELASTICITY

The loss in dynamic modulus of elasticity is related to the cumulative damage in test specimens such as mortars and concretes and is often used as a measure to characterize the damage induced by freeze-thaw cycles in saturated concrete test specimens in ASTM C666 test procedure. Considering that ASR damage will likely show a similar behavior, the percent loss in dynamic modulus of elasticity was monitored on test specimens subjected to the autoclave test. Table 4 shows the initial and the final dynamic modulus of elasticity of mortar bars to show the influence of ASR on loss of stiffness in mortar bars caused by the ASR induced in the autoclave heat treatment. Figure 10 shows the correlation between the autoclave expansion and the loss in dynamic modulus of elasticity of the mortar bars. Based on the correlation between the autoclave expansion and the loss in dynamic modulus of elasticity shown in Figure 11, it is apparent that there is a general trend in higher loss in modulus of elasticity with increasing levels of expansion observed in the autoclave test, although the degree of correlation is only 0.54.

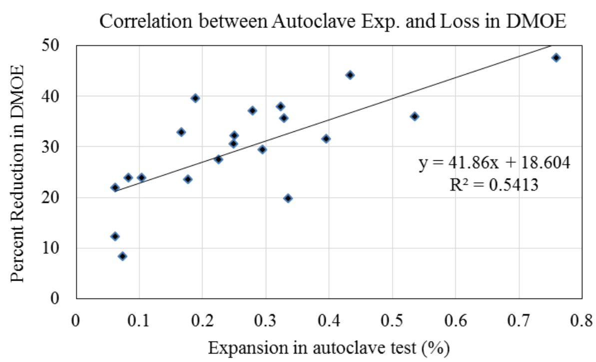


Figure 10. Correlation Between Autoclave Expansion and Loss in Dynamic Modulus of Elasticity

Table 4. Change in Dynamic Modulus of Elasticity of Mortar Bars Subjected to Autoclave Test

Sample #	Code	Name	Average DME Before Test, GPa	Average DME After Test, GPa	% loss in DME
1	130038	Adairsville Atlanta, GA	22.7	20.8	8%
2	120021	Cookeville	19.2	16.8	12%
3	150098	Spratt, Canada	19.1	15.3	20%
4	150094	Vulcan Lithia, GA	19.7	15.4	22%
5	120032	Rogers Liberty	21.2	16.2	24%
6	120102	China Lake	20.4	15.5	24%
7	150104	Yardville Great Eastern	20.6	15.7	24%
8	150100	Geneva	20.6	15.0	27%
9	150098.1	Sudbury, Canada	19.5	13.8	29%
10	140037	Green Bros	20.7	14.4	31%
11	120016	Rogers Gordonville	21.9	15.0	32%
12	140085	Nevada	17.2	11.7	32%
13	150047	Wabasha	25.1	16.9	33%
14	152149	Arkadelphia	21.0	13.5	36%
15	130002	Gold Hill, NC	20.4	13.1	36%
16	130041	Green Brothers, MS	23.1	14.5	37%
17	140040	Calera	20.3	12.6	38%
18	150096	Dell Rapids	20.0	12.1	40%
19	130037	Jobe, El Paso, TX	20.4	11.4	44%
20	130006	Las Placitas, NM	19.6	10.3	48%

4.4 CORRELATION AUTOCLAVE AND AMBT RESULTS

The expansion results of all the aggregates from the autoclave test method were compared with the results from the ASTM C1260 test in order to study any trends that exist between the outcomes from the two tests and the possible validation of autoclave test method as a suitable and a rapid alternative to the ASTM C1260 test method. Table 5 presents the expansion of mortar bars subjected to autoclave test method along with the 14-day and 28-day expansion results from the ASTM C1260 test. A bar chart and a scatter plot, showing the correlations between the results from these two methods, are shown in Figures 11a and 11b, respectively. Based on the trends observed in these figures and the data shown in Table 5, it can be concluded broadly that the expansion results from autoclave test show a similar trend to that of 14-day and 28-day expansion results from the ASTM C1260 test. However, specific aggregates show marked deviation from the trend. For instance, Yardville Great Eastern aggregate and Las Placitas aggregates showed significantly lower expansion in autoclave test compared to either 14-day or 28-day expansion result in the ASTM C1260 test. However, Rogers and Gold Hill aggregates show greater expansion in autoclave test compared to either the 14-day or the 28-day expansion reading from the ASTM C1260 test. The reasons for these deviations in the expansion behavior of certain aggregates may stem from the physical, chemical and the mineralogical nature of the reactive components in these aggregates.

Table 5. Expansion results of aggregates in ASTM C1260 and Autoclave Test

Code	Name	ASTM C1260 14-day Expansion, %	ASTM C1260 28-day Expansion, %	Autoclave Expansion, %
120032	Rogers Liberty	0.10	0.16	0.18
130038	Adairsville Atlanta, GA	0.08	0.11	0.07
150104	Yardville Great Eastern	0.19	0.35	0.08
130041	Green Brothers, MS	0.23	0.32	0.28
130037	Jobe El Paso, TX	0.41	0.61	0.43
130002	Gold Hill, NC	0.33	0.48	0.54
130006	Las Placitas, NM	1.02	1.26	0.76

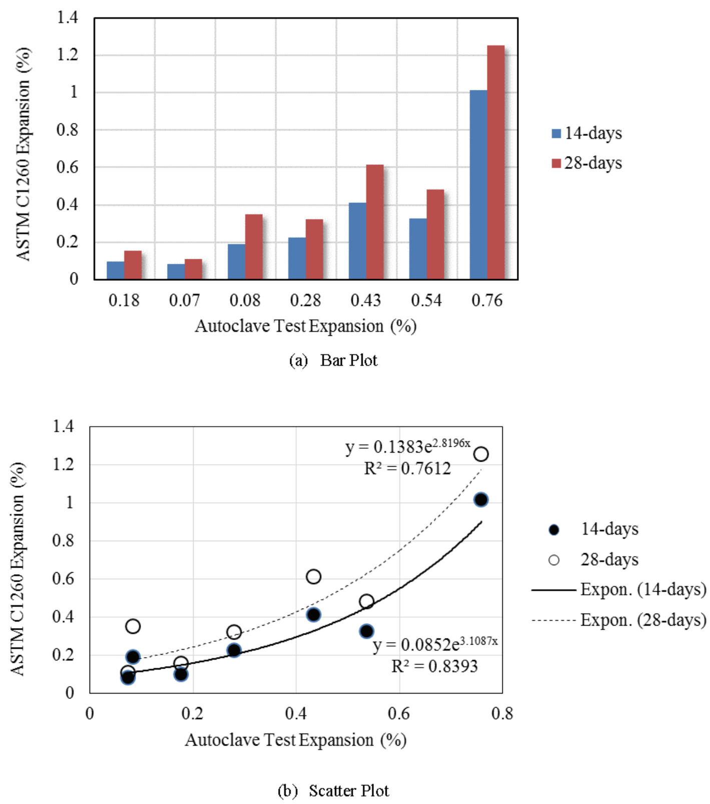


Figure 11. Correlation between expansion results from AMBT (14-days and 28-days) and Autoclave tests

4.5. CORRELATION BETWEEN MINERALOGY OF AGGREGATES AND AUTOCLAVE EXPANSION

The mineralogy of aggregates evaluated in this study was provided by ERDC and a tabulated list of minerals reported for each aggregate is presented in Table 5, along with the autoclave expansion results. Although, a correlation between the mineralogy of aggregates and the autoclave expansion observed in test specimens would provide for a basis for further analyzing the expansion behavior of the test specimens, the presence of a diversity of minerals and a lack of quantifiable amount of reactive silica present in these aggregates makes this task difficult. However, using these results, an attempt to correlate the quantities of primary minerals present in majority of the aggregates (calcite, dolomite, quartz and albite) and the mortar bar expansion observed in the autoclave test is shown in Figure 12. Although no significant correlation can be observed between the expansion results and the amount of the selected minerals, a more careful analysis of the reactive silica present in the aggregate may provide a meaningful basis for such a correlation in future.

Table 6. Petrography results of the tested aggregate

Code	Name	Calcite	dolomite	quartz	Anorthite	orthoclase	Muscovite	Albite	Clinochlore	Expansion %
120021	Cookeville	84.7	9.8	5.5						0.062
150094	Vulcan Lithia, GA			19.4	31			22.9		0.062
130038	Adairsville Atlanta, GA		99							0.073
152149	Arkadelphia			92.4				6.4		0.083
120102	China Lake			17.6	48.1	6.6		9.2		0.105
140085	Nevada	27.1		27.3	25.8	8.9			1.2	0.167
120032	Rogers Liberty	73.1	20.1	6.8						0.177
150098	Spratt, Canada	76.7	13.7	9.6						0.189
150104	Yardville Great Eastern			99.5						0.226
140037	Green Bros			94						0.249
140040	Calera	61.5	35.6	2.9						0.251
130041	Green Brothers, MS									0.279
150047	Wabasha	3.1	4.3	38.8		26	5.2	10.8	8.7	0.295
150096	Dell Rapids			95.3						0.324
150098.1	Sudbury, Canada		7.5	34.3			13.9	16.3	26.2	0.329
150100	Geneva		11.8	73.7		14.2				0.335
120016	Rogers Gordonville	5.7	74.8	17.3						0.395
130037	Jobe El Paso, TX	17		18			2	5		0.433
130002	Gold Hill, NC	1		18			39	9	31	0.536
130006	Las Placitas, NM	5		27		10	17	38		0.759

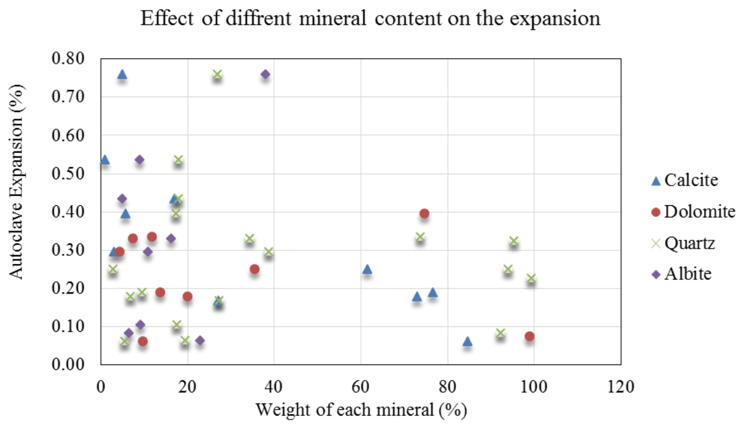


Figure 12. Correlation between selected mineral and the Autoclave expansion of mortar bars

CHAPTER 5 - CONCLUSIONS

Based on the autoclave testing conducted on 20 different aggregates in this study along with the measurement of change in the dynamic modulus of elasticity before and after the autoclave test, and the 14-day and 28-day expansion data from the accelerated mortar bar tests provided by ERDC, the following conclusions are drawn:

- Autoclave testing procedure as adopted in this study does provide a rapid alternative to the existing standard test procedures to evaluate alkali-silica reactivity of aggregates. The entire testing can be conducted within 3 days, compared to 2 weeks for AMBT, 8 weeks for MCPT and 52 weeks for CPT.
- The expansion results from the autoclave test were generally found to be greater than the expansion results of the same mixture tested in ASTM C1260 test method at 14-days, with exception of a couple of aggregates. Although the linear correlation between the results from the two tests is not ideal, the correlation between the expansion results from the autoclave test and the ASTM C1260 was found to be good using the best fitting exponential curve with a R^2 value of 0.76.
- The COV of results in the autoclave test was found to be low for all the aggregates evaluated in this study, ranging between 0.7% and 7.6%. However, the COV was not found to show any particular trend with increasing levels of autoclave expansion.
- Significant loss in the dynamic modulus of elasticity was observed in test specimens that exhibited high levels of expansion in the autoclave test, indicating the accumulated damage due to ASR distress caused in the test. However, the correlation between the autoclave expansion and the percent loss in dynamic modulus of elasticity of test specimens showed only a modest correlation of R^2 value of 0.54 using a linear fit.

APPENDIX A

Version 6.40

Holcim

Material Certification Report

Material:	Portland Cement	Test Period:	01-Feb-2015
Type:	I-II	To:	28-Feb-2015

Certification

This Holcim cement meets the specifications of ASTM C150 for Type I-II cement, and complies with AASHTO M85 specifications for Type I-II cement.

General Information

Supplier:	Holcim (US) Inc.	Source Location:	Ste. Genevieve Plant
Address:	2942 US Highway 61 Bloomsdale, MO 63627		2942 US Highway 61 Bloomsdale, MO 63627
Telephone:	636-524-8155	Contact:	Erin Watson
Date Issued:	13-Mar-2015		

The following information is based on average test data during the test period.
The data is typical of cement shipped by Holcim; individual shipments may vary.

Tests Data on ASTM Standard Requirements

Chemical

Item	Limit[A]	Result
SiO_2 (%)	-	19.7
Al_2O_3 (%)	6.0 max	4.4
Fe_2O_3 (%)	6.0 max	3.3
CaO (%)	-	64.1
MgO (%)	6.0 max	2.3
SO_3 (%)	3.0 max[B]	3.6
Loss on Ignition (%)	3.0 max	2.6
Insoluble Residue (%)	0.75 max	0.45
CO_2 (%)	-	1.3
Limestone (%)	5.0 max	3.2
$CaCO_3$ in Limestone (%)	70 min	89
Inorganic Processing Addition (%)	5.0 max	0.0
Potential Phase Compositions[C]:		
C_3S (%)	-	61
C_2S (%)	-	7
C_3A (%)	8 max	6
C_4AF (%)	-	10
$C_3S + 4.75C_3A$ (%)	-	89.0

Physical

Item	Limit[A]	Result
Air Content (%)	12 max	7
Blaine Fineness (m^2/kg)	260 min	386
Autoclave Expansion (%) (C151)	0.80 max	0.05
Compressive Strength MPa (psi):		
3 days	12.0 (1740) min	29.4 (4270)
7 days	19.0 (2760) min	36.4 (5290)
Initial Vicat (minutes)	45-375	87
Mortar Bar Expansion (%) (C1038)	-	0.011

Tests Data on ASTM Optional Requirements

Chemical

Item	Limit[A]	Result
Equivalent Alkalies (%)	0.60 max	0.54

Physical

Item	Limit[A]	Result
False Set (%)	50 min	89

Notes

[A] Dashes in the limit / result columns mean Not Applicable.
[B] It is permissible to exceed the specification limit provided that ASTM C1038 Mortar Bar Expansion does not exceed 0.020 % at 14 days.
[C] Adjusted per Annex A1.6 of ASTM C150 and AASHTO M85.
[D] Test result represents most recent value and is provided for information only. Analysis of Heat of Hydration has been carried out by CTLGroup, Skokie, IL.
Equavalent Alkalies (%) Minimum = 0.51, Maximum = 0.57
This data may have been reported on previous mill certificates.

Additional Data

Inorganic Processing Addition Data

Item	Result[A]
Type	-
Amount (%)	-
SiO_2 (%)	-
Al_2O_3 (%)	-
Fe_2O_3 (%)	-
CaO (%)	-
SO_3 (%)	-

Base Cement Phase Composition

Item	Result
C_3S (%)	63
C_2S (%)	8
C_3A (%)	6
C_4AF (%)	10

By , Quality Manager

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APPENDIX B:- RAW DATA

Length before placing in Autoclave:

		reference	s-1	s-2	s-3	s-4
140040	Calera	0.0309	0.3245	0.3202	0.3014	0.3306
130041	Green Brothers, MS	0.0314	0.1719	0.2841	0.3557	0.2933
150104	Yardville Great Eastern	0.0309	0.335	0.331	0.3491	0.2924
150094	Vulcan Lithia, GA	0.0314	0.2563	0.2483	0.2725	0.2653
152149	Arkadelphia	0.0521	0.2897	0.3049	0.3857	0.3561
130006	Las Placitas, NM	0.0286	0.2216	0.1708	0.2903	0.3146
120102	China Lake	0.0297	0.315	0.336	0.3354	0.3974
130038	Adairsville Atlanta, GA	0.0297	0.1589	0.2979	0.2884	0.242
140037	Green Bros	0.0288	0.27984	0.313	0.1762	0.3283
120016	Rogers Gordonville	0.0288	0.1463	0.2754	0.2662	0.3012
120021	Cookeville	0.0269	0.1514	0.2762	0.2592	0.2611
130002	Gold Hill, NC	0.0269	0.2378	0.3037	0.3125	0.2637
150098	Spratt, Canada	0.0263	0.3365	0.3113	0.3279	0.2954
150098.1	Sudbury, Canada	0.0263	0.3401	0.3414	0.3556	-
150096	Dell Rapids	0.0539	0.3279	0.2907	0.357	0.3637
150100	Geneva	0.0539	0.2102	0.3004	0.3637	0.2704
140085	Nevada	0.0521	0.3042	0.3424	0.3303	0.304
150047	Wabasha	0.0614	0.2918	0.1951	0.3138	0.3267
120032	Rogers Liberty	0.0614	0.3731	0.3362	0.3282	0.3847
130037	Jobe El Paso, TX	0.0278	0.3065	0.2345	0.3102	0.3094

Length after testing in Autoclave:

		reference	s-1	s-2	s-3	s-4
140040	Calera	0.0309	0.3586	0.3534	0.3327	0.3617
130041	Green Brothers, MS	0.0314	0.2001	0.315	0.3806	0.321
150104	Yardville Great Eastern	0.0309	0.3426	0.3395	0.3574	0.3013
150094	Vulcan Lithia, GA	0.0314	0.2629	0.2544	0.2786	0.2714
152149	Arkadelphia	0.0521	0.3235	0.3358	0.4191	0.3897
130006	Las Placitas, NM	0.0286	0.2972	0.2463	0.3671	0.3902
120102	China Lake	0.0297	0.3254	0.3468	0.3458	0.4076
130038	Adairsville Atlanta, GA	0.0297	0.1662	0.3053	0.296	0.249
140037	Green Bros	0.0288	0.3042	0.3405	0.2003	0.352
120016	Rogers Gordonville	0.0288	0.1878	0.3163	0.3039	0.3392
120021	Cookeville	0.0264	0.157	0.2822	0.2647	0.2667
130002	Gold Hill, NC	0.0264	0.2908	0.3565	0.3664	0.3163
150098	Spratt, Canada	0.0269	0.3695	0.3465	0.3623	0.3291
150098.1	Sudbury, Canada	0.0269	0.3715	0.3706	0.3853	-
150096	Dell Rapids	0.0532	0.3455	0.3092	0.3755	0.382
150100	Geneva	0.0532	0.232	0.3234	0.3843	0.2925
140085	Nevada	0.0521	0.3295	0.3656	0.3558	0.3288
150047	Wabasha	0.0614	0.308	0.2112	0.3314	0.3437
120032	Rogers Liberty	0.0614	0.3918	0.3543	0.3461	0.4009
130037	Jobe El Paso, TX	0.0277	0.3495	0.2779	0.353	0.353

Autoclave Expansion Test Results:

Code	Name	reference	EXPANSION% s-1	s-2	s-3	s-4	avg
140040	Calera	0.0309	0.341	0.332	0.313	0.311	0.324
130041	Green Brothers, MS	0.0314	0.282	0.309	0.249	0.277	0.279
150104	Yardville Great Eastern	0.0309	0.076	0.085	0.083	0.089	0.083
150094	Vulcan Lithia, GA	0.0314	0.066	0.061	0.061	0.061	0.062
152149	Arkadelphia	0.0521	0.338	0.309	0.334	0.336	0.329
130006	Las Placitas, NM	0.0286	0.756	0.755	0.768	0.756	0.759
120102	China Lake	0.0297	0.104	0.108	0.104	0.102	0.105
130038	Adairsville Atlanta, GA	0.0297	0.073	0.074	0.076	0.07	0.073
140037	Green Bros	0.0288	0.2436	0.275	0.241	0.237	0.249
120016	Rogers Gordonville	0.0288	0.415	0.409	0.377	0.38	0.395
120021	Cookeville	0.0264	0.061	0.065	0.06	0.061	0.062
130002	Gold Hill, NC	0.0264	0.535	0.533	0.544	0.531	0.536
150098	Spratt, Canada	0.0269	0.324	0.346	0.338	0.331	0.335
150098.1	Sudbury, Canada	0.0269	0.308	0.286	0.291	-	0.295
150096	Dell Rapids	0.0532	0.183	0.192	0.192	0.19	0.189
150100	Geneva	0.0532	0.225	0.237	0.213	0.228	0.226
140085	Nevada	0.0521	0.253	0.239	0.262	0.248	0.251
150047	Wabasha	0.0614	0.162	0.161	0.176	0.17	0.167
120032	Rogers Liberty	0.0614	0.187	0.181	0.179	0.162	0.177
130037	Jobe El Paso, TX	0.0277	0.431	0.435	0.429	0.437	0.433

Change in dynamic module of elasticity (GPa)

Sample #	Code	Name	avg before test	avg after test	% loss
1	130038	Adairsville Atlanta, GA	22.7	20.8	8%
2	120021	Cookeville	19.2	16.8	12%
3	150098	Spratt, Canada	19.1	15.3	20%
4	150094	Vulcan Lithia, GA	19.7	15.4	22%
5	120032	Rogers Liberty	21.2	16.2	24%
6	120102	China Lake	20.4	15.5	24%
7	150104	Yardville Great Eastern	20.6	15.7	24%
8	150100	Geneva	20.6	15.0	27%
9	150098.1	Sudbury, Canada	19.5	13.8	29%
10	140037	Green Bros	20.7	14.4	31%
11	120016	Rogers Gordonville	21.9	15.0	32%
12	140085	Nevada	17.2	11.7	32%
13	150047	Wabasha	25.1	16.9	33%
14	152149	Arkadelphia	21.0	13.5	36%
15	130002	Gold Hill, NC	20.4	13.1	36%
16	130041	Green Brothers, MS	23.1	14.5	37%
17	140040	Calera	20.3	12.6	38%
18	150096	Dell Rapids	20.0	12.1	40%
19	130037	Jobe El Paso, TX	20.4	11.4	44%
20	130006	Las Placitas, NM	19.6	10.3	48%

REFERENCES

Berube, M.A. and Fournier, B. "Accelerated Test Methods for Alkali-Aggregate Reactivity" In CANMET-ACI International Symposium on Advances in Concrete Technology, Athens, Greece, 1992, p. 42.

Corneille, A. and Bollotte, B. "Results of a Round Robin Test Program for the Validation of the Test Methods in the French Recommendations for the Prevention of AAR Damage in Concrete", ACI-SP 145-38, pp. 725-740.

Fournier, B., Berube, M.A. and Bergeron, G. "A Rapid Autoclave Mortar Bar Method to Determine the Potential Alkali-Silica Reactivity of St. Lawrence Lowlands Carbonate Aggregates (Quebec, Canada)", Cement, Concrete and Aggregates, Vol. 13, No. 1, 1991, pp. 58-71.

Giannini, Eric R., and Folliard, Kevin J., A Rapid Test to Determine Alkali-Silica Reactivity of Aggregates Using Autoclaved Concrete Prisms, SN3235, Portland Cement Association, Skokie, Illinois, USA, 2013, 21 pages.

Grattan-Bellew, P.E. "A critical review of ultra-accelerated tests for alkali-silica reactivity" Cement and Concrete Composites, Vol. 19, No. 5-6, pp. 403-414, 1997.

Hobbs, D.W. 1988. Alkali Silica Reaction in Concrete, Thomas Telford.

Latifee, E.R. and Rangaraju, P.R. "Miniature Concrete Prism Test – Rapid Test Method for Evaluating Alkali Silica Reactivity of Aggregates" *ASCE Journal of Materials in Civil Engineering*, Vol. 27, No. 7, 2014

Nishibayashi, S., Kuroda, T., and Inoue, S. 1996. Expansion characteristics of AAR in concrete by autoclave method. Proceedings of the 10th international conference on alkali-
aggregate reaction in concrete , Melbourne, Australia: CSIRO, 370–376.

Oberholster, R.E. and Davies, G. (1986). "An accelerated method for testing the potential alkali reactivity of siliceous aggregates." Cement and Concrete Research 16(2): 181 - 189.

Stanton, T.E. 1940. "Expansion of concrete through reaction between cement and aggregate." Proceedings of the American Society of Civil Engineers, 66(10): 1781-1811.

Stanton, T.E. 1943. Studies to develop an accelerated test procedure for the determination of adversely reactive cement-aggregate combinations. ASTM Proceedings 43, 875-905.

Tamura, H., Takahashi, T., and Ohashi, M. 1989. A test method on rapid identification of the future susceptibility of alkali-aggregate reaction in fresh concrete (fresh-con GBRC rapid method). Proceedings of the 8th international conference on alkali-aggregate reaction in concrete , Japan, London: Elsevier Applied Science, 351–356.

Tang, MS, Lan, XF and Han, SF. 1994. Autoclave method for identification of alkali-reactive carbonate rocks. Cement and concrete and composites, 16(3): 163–167

Thomas, M.D.A, Fournier, B., Folliard, K. Idekar, J. and Shehata, M. "Test methods for evaluating preventive measures for controlling expansion due to alkali–silica reaction in concrete", Cement and Concrete Research, Vol. 36, No. 10, 2006, pp. 1842-1856.

Xiaofang, M.U. "Studies on Alkali Carbonate Reactions", Ph.D. Dissertation, Nanjing University of Chemical Technology, 1996.

A.4 Laboratory 4 Final Report – CTL Group

Round Robin Testing of Alkali-Silica Reactivity of Aggregates using Autoclaving Techniques

SSP TCN 15-032
Under Prime Contract/Solicitation No. W911NF-11-D-0001

Principal Point-of-Contact:
Anthony Bentivegna, PhD, PE
Construction Technology Laboratories, Inc. (d/b/a CTLGroup)
5400 Old Orchard Road
Skokie, IL 60077
847-972-3092
ABentivegna@CTLGroup.com

Submittal Date:
July 13, 2016

CTLGroup
www.CTLGroup.com

INTRODUCTION

As required by SSP TCN 15-032, this document serves as the final report for the round robin testing of alkali-silica reactivity (ASR) of aggregates using autoclaving techniques.

The objective of this work was to provide support for rapid test methods for evaluating the potential for ASR and contribute to the intra- and inter-laboratory variability in results. The results of the testing will be used to transition the developed test method for use in contingency construction operations that support the rapid and reliable fielding of weapons systems.

SCOPE

As authorized, CTLGroup participated in the round-robin testing for evaluating ASR of aggregates using an autoclave. Multiple laboratories were instructed to follow a predefined set of procedures for casting and measuring samples exposed to a high temperature and pressure autoclave environment. Due to the current disconnect between rapid testing and its correlation to true field performance, the primary purpose of this project was to evaluate the repeatability of results within the same laboratory as well as results collected from different laboratories. This final report outlines the testing methods, summary of results, and discussion of the results towards future applications.

METHODS

CTLGroup received aggregates from 20 different sources for testing. Upon arrival, all samples were inventoried and logged into a Laboratory Information Management System (LIMS) for sample management and tracking. The received samples were documented and analyzed according to the provided procedures supplied. The initial preparation and casting of the mortar specimens for the aggregates closely assimilated to ASTM C1260[1]. A fully automated paste/mortar mixer was used to create all test specimens, as shown in Figure 1.

The fully automated paste/mortar mixer was programmed to follow the ASTM C305[2] mixing timeline and processes. Deionized water (DI) (276 g) was first added to the mixing bowl. In addition to the water, NaOH solution was added to the water mixture in order to produce a final

[1] ASTM C1260, "Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)," ASTM International, DOI: 10.1520/C1260-14.

[2] ASTM C305, "Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency," ASTM International, DOI: 10.1520/C0305-14.

total alkali loading of 3.5% by weight of the cement. After which, the cement (587 g of a Type I-II portland cement (Holcim – St. Genevieve) with Na_2O_{eq} of 0.54%) was added to the mixing water/NaOH solution and subsequently mixed for 30 seconds on low mixing speed. The tested aggregate (1320g) that had been previously proportioned and prepared as per ASTM C1260 was added to the mixing bowl over a 30 second period on low mixing speed. The mixer then transitioned to a medium speed and mixed for an additional 30 second period. After which, the mixer was stopped and mortar that had aligned itself on the walls of the mixing bowl was scrapped towards the mixer's blades over a 15 second period followed by a 90 second rest period. The mixing program then continued for 60 second at a medium mixing speed.

After the completion of the mixing process four (4) 1x1x11.25 inch stainless steel mortar bar molds that had been sealed with wax to minimize moisture loss at steel-to-steel interfaces were filled and tamped in two separate lifts within a 2 minutes and 15 seconds period by the technician. The mortar sample process delineated itself from ASTM C1260 procedures by extending the curing time prior to demolding to 48 hours at a 23°C holding temperature.

The samples were then removed from the moist curing environment and demolded. The specimens were then labeled and pictured (Before Image) according to the proper aggregate ID and sample number. The initial measurements of the samples were recorded at room temperature (23°C) utilizing a length change comparator.

Samples were then carefully set into a stainless steel mesh holding apparatus to hold the mortar bars in an up-right position. The holding apparatus was then placed into the autoclave (Yamato SQ510C). The apparatus employed for holding the mortar bars in an up-right position and the autoclave used for this testing program are shown in Figure 2.

The autoclave ran on an automated pre-programmed pressure and temperature system. The program directed the autoclave to ramp to a temperature and pressure of 130°C and 25 psi. Once stabilized the temperature and pressure was maintained and continued for a 5 hour period. After the dwell period, the autoclave was cooled to approximate room temperature (23°C). After which, the technician removed the samples placing them in a matching water bath temperature subsequently cooling the samples to assure they reached 23°C within a 15 minute period. Once the sample temperature met the room temperature the specimens were measured using the length comparator. Photographs were then taken of the specimens, and any observed damage was documented.

RESULTS

The results of the autoclave expansion testing is summarized in Table 1 and the individual data sheets are attached to this report.

DISCUSSION

The work outlined in this report has been performed to provide analytical support of a rapid test method for the evaluation of potential alkali-silica reactive (ASR) aggregates through the utilization of an autoclave testing chamber. This section summarizes the observations regarding the testing procedure as well as the results.

TESTING PROCEDURE

One of the inherent benefits of the proposed autoclave testing procedure is how closely it assimilates to the widely utilized ASTM C1260, with respect to casting and sample preparation procedures. Laboratories that have previously tested aggregates through ASTM C1260 will be able to rely on past experience making for an easy transition to utilizing the autoclave testing apparatus. The test also provides significantly more rapid results by providing final expansion data in just 3 days after casting and 8 hours after the initial comparative measurement. The proposed method also eliminates issues associated with procuring and measuring specimens directly out of the hazardous 1N NaOH 80°C bath. The autoclave testing procedure thereby significantly decreases the user's potential for injury in comparison to the 14-day AMBT testing period in addition to limiting thermal expansive/shrinkage changes derived from the sample's transition from solution to comparator stand.

VARIABILITY BETWEEN SPECIMENS

The autoclave expansion results are plotted in ascending order (i.e. increasing expansion (%) from left to right) with the coefficient of variation (%) between each bar for each mixture overlaid for each mixture in Figure 3

From Figure 3, there are six (6) aggregate sources with average autoclave expansions below 0.10%, and 14 aggregate sources with average expansions greater than 0.10%. From ASTM C1260 and work completed by Rogers[3], the average coefficient of variation for testing

[3] Rogers, C.A., "Multi-laboratory Study of the Accelerated Mortar Bar Test (ASTM Test Method C1260) for Alkali-Silica Reaction," Cement, Concrete, and Aggregates, Vol 21, 1999, pp. 185–194.

conducted in the same laboratory should be within 2.94% for specimens that expanded more than 0.10%. For all the autoclave expansion tests, eleven mixtures had a coefficient of variation which exceeded the 2.94% precision allowance; of those mixtures, eight had an average expansion greater than 0.10% and three had an expansion less than 0.10%. Lower expanding aggregates had a higher coefficient of variation than those with more expansive aggregates, which is expected based off the equation for the calculation of the coefficient. Due to the minimal amount of measurements and the highly aggressive nature of this test it was important to assure that the samples from the same mix maintained similar expansive trends relative to one another.

COMPARISON TO STANDARDIZED TESTING (ASTM C1260 AND C1293)

Graphical comparisons of the autoclave expansion results to ASTM C1260 and ASTM C1293[4] are shown in Figure 4 and 5, respectively.

In Figures 4 and 5, the orange-dashed line indicates a theoretical perfect correlation between the autoclave expansion and ASTM C1260 and ASTM C1293 results, respectively. Therefore, if a pair of results were to fall directly on this line it would indicate a direct correlation between the two test methods. Realistically, it is highly unlikely that any results would directly correlate between the two test methods, the next best trends are for all the results to fall in the lower left and upper right quadrants. These results indicate both test methods are able to identify aggregates which both pass or both fail the two test methods plotted on the graph. If results are plotted in the upper left quadrant (<0.04% x-axis and >0.1% y-axis) and the lower right quadrants (>0.04% x-axis and <0.1% y-axis) would indicate a discrepancy between the two test methods with either a false-positive or a false-negative.

From Figure 4, ASTM C1260 expansion measurements are typically larger (13 out of the 19 comparable aggregates) than the measurements obtained through the autoclave test method, which suggested that the autoclave method is less aggressive than ASTM C1260. Generally, there is a stronger correlation (15 out of the 19 comparable aggregates) of results from both tests which would indicate an agreement with either a pass-pass or fail-fail results. However,

[4] ASTM C1293, "Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction," ASTM International, DOI: 10.1520/C1293-08BR15.

there are four aggregates which produced results with discrepancies between the two test methods.

In Figure 5, ASTM C1293 data shows that the least reactive aggregate that passes ASTM C1293 (Rodgers Liberty 120032 with 0.0077%) fails according to the autoclave test and passes and/or agrees according to 14 day ASTM C1260 test. The next least reactive aggregate that fails ASTM C1293, (Cookeville – 120021 with 0.1227%) passes with respect to both the autoclave technique and ASTM C1260 14-day testing and fails ASTM C1293. However, the autoclave technique saw nearly 60% more expansion than ASTM C1260 at 14-day, which constitutes an improvement for testing highly reactive aggregates. The increase in expansion observed denotes the benefit of testing aggregates with minimal amounts of quartz with the autoclave technique. The decreased expansion observed with aggregate 120021 with ASTM C1260 is due to the excessive amounts of alkalis available in the soak solution (1N NaOH) compared to the low quartz content previously mentioned.

ADDITIONAL OBSERVATIONS

In addition to the expansion observations, the following inherent benefits of the proposed autoclave testing practices were noted:

- Mixing and casting procedures are analogous with ASTM C1260,
- Decreased testing duration aids in accelerating results for contingency construction,
- Measurement and evaluation of samples at room temperature minimizes thermal associated with ASTM C1260, and
- Specimen exposure to DI water instead of NaOH solution decreases the aggressiveness of the test as well as deters the activation of otherwise dormant siliceous aggregates while simultaneously decreasing worker interaction with hazardous chemicals

SUMMARY

As authorized, CTLGroup participated in the round robin testing for evaluating alkali-silica reactivity of aggregates through the autoclave. From the aggregates provided, fourteen (14) of the aggregates had expansion values which exceeded the threshold for innocuous behavior, and six (6) aggregates were below this threshold.

The proposed test method is easily adaptable for most laboratories as the procedures closely mimic those of existing standardized test methods. In addition, other benefits of the test include decreased testing duration and a reduction in potential safety concerns for technicians through reduced chemicals and high temperature interactions.

There was some concern with the repeatability of the test results within the same set of mortar bars from the same mixture. Calculated coefficients of variation exceeded acceptable levels published in ASTM C1260 and by existing literature.

CTLGroup recommends completing additional testing with both fast and slow reacting aggregates, incorporation of SCMs, and concrete samples to evaluate the robustness and accuracy with respect to field performance. Other variables to be considered for testing are to investigate lowering the total alkali content of the test, and/or testing to eliminate the pessimism effect by testing with 90% non-reactive and 10% reactive aggregate.

TABLE 1: SUMMARY OF ASR AUTOCLAVE EXPANSION RESULTS

Aggregate Serial No.	Aggregate Source	Calculated Autoclave Expansion, %:					
		A	B	C	D	Average	Coefficient of Variation
120016	Rogers Gordonsville, TN	0.432	0.437	0.391	0.404	0.416	5.3
120021	Cookeville, TN	0.052	0.055	0.052	0.054	0.053	2.8
120032	Rogers Liberty, TN	0.223	0.217	0.217	0.221	0.220	1.4
120102	China Lake, CA	0.067	0.067	0.068	0.060	0.066	5.6
130002	Gold Hills, NC	0.431	0.455	0.446	0.479	0.453	4.4
130006	Las Placitas, NM	0.741	0.749	0.734	0.735	0.740	0.9
130037	Jobe, TX	0.495	0.511	0.505	0.499	0.503	1.4
130038	Adairsville, GA	0.046	0.045	0.044	0.044	0.045	2.1
130041	Green Brothers, MS	0.272	0.273	0.267	0.283	0.274	2.4
140037	Green Brothers #2, MS	0.253	0.246	0.260	0.277	0.259	5.1
140040	Calera, AL	0.301	0.290	0.308	0.314	0.303	3.4
140085	Nevada Test Site	0.055	0.056	0.046	0.057	0.054	9.5
150047	Wabasha, MN	0.177	0.173	0.175	0.175	0.175	0.9
150094	Vulcan Atlanta	0.066	0.068	0.064	0.065	0.066	2.6
150096	LG Everest	0.171	0.180	0.173	0.178	0.176	2.4
150098	Spratt, ONT	0.356	0.344	0.335	0.330	0.341	3.3
150098.1	Sudbury, ONT	0.348	0.332	0.320	0.313	0.328	4.7
150100	Geneva	0.157	0.146	0.157	0.159	0.155	3.8
150104	Clayton, NJ - Yardville	0.058	0.057	0.052	0.054	0.055	5.0
152149	Arkadelphia	0.144	0.132	0.174	0.192	0.161	17.1

FIGURE 1: FULLY AUTOMATED PASTE/MORTAR MIXER

FIGURE 2: (LEFT) STAND FOR HOLDING THE SAMPLES AND (RIGHT) AUTOCLAVE AND MORTAR SAMPLES IN SITU

Battelle Memorial Institute: SSP TCN 15-032
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CTLGroup Project No.: 400113

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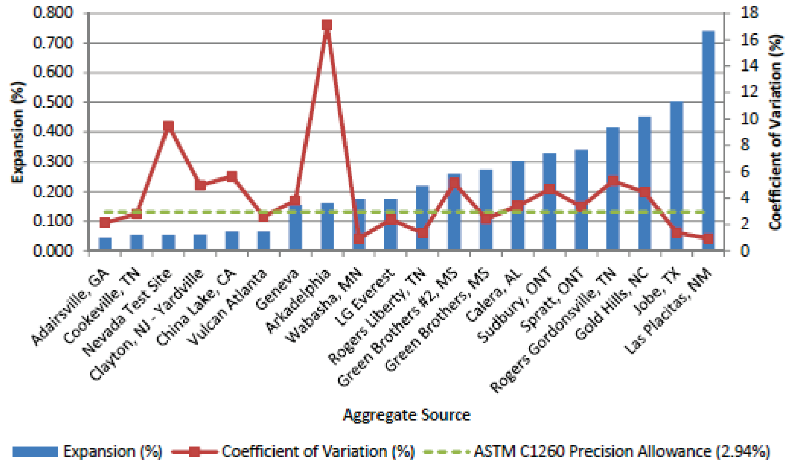

FIGURE 3: AVERAGE AUTOCLAVE EXPANSION OVERLAID WITH THE COEFFICIENT OF VARIATION BETWEEN EACH SAMPLE

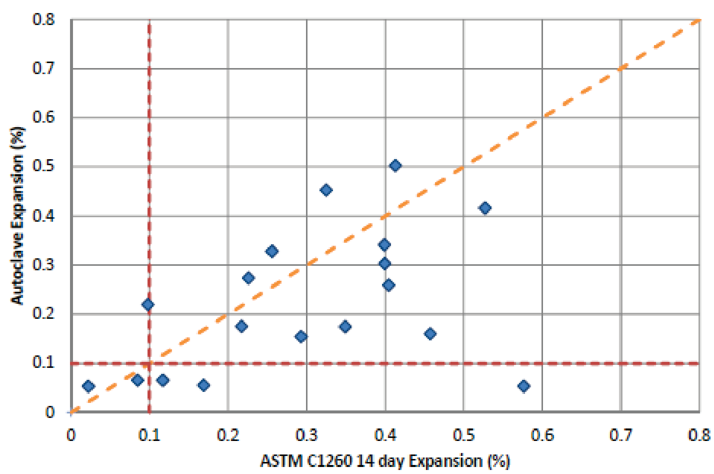

FIGURE 4: AUTOCLAVE EXPANSION RESULTS COMPARED TO ASTM C1260

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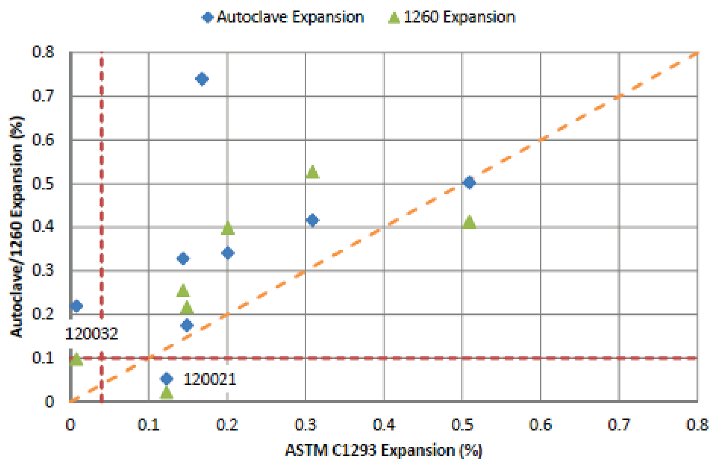

FIGURE 5: AUTOCLAVE AND ASTM C1260 EXPANSION RESULTS COMPARED TO ASTM C1293 EXPANSION

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Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	120016
Aggregate Source:	Rogers Gordonsville, TN
Aggregate Type:	Coarse
CTLGroup ID:	4090310
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 13, 2016
Test Date:	January 15, 2016

120016 Rogers Gordonsville, TN

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0094	0.0016	-0.0394	-0.0220	---
Final Comparator Length, in.:	0.0338	0.0453	-0.0003	0.0184	---
Calculated Autoclave Expansion, %:	**0.432**	**0.437**	**0.391**	**0.404**	**0.416**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

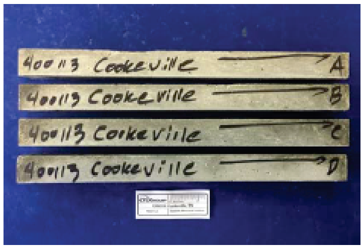
ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	120021
Aggregate Source:	Cookeville, TN
Aggregate Type:	Coarse
CTLGroup ID:	4090316
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 12, 2016
Test Date:	January 14, 2016

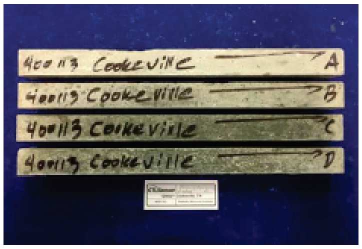
120021 Cookeville, TN

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0016	-0.0220	-0.0498	0.0124	---
Final Comparator Length, in.:	0.0068	-0.0165	-0.0446	0.0178	---
Calculated Autoclave Expansion, %:	**0.052**	**0.055**	**0.052**	**0.054**	**0.053**

Before Testing



After Testing



Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
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Client:	Battelle Memorial Institute	CTLGroup Proj. No.:	400113
Project:	Battelle Memorial Institute	CTLGroup Proj. Mgr.:	A. Bentivegna
		Technicians:	VS, WD
Contact:	Todd Weidner	Approved:	J. Gajda
Date Received:	October 23, 2015	Report Date:	July 13, 2016

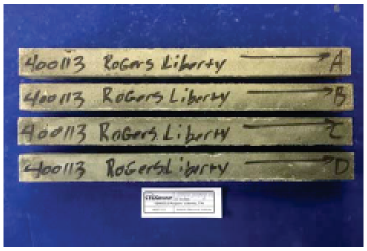
ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	120032
Aggregate Source:	Rogers Liberty, TN
Aggregate Type:	Coarse
CTLGroup ID:	4090319
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 12, 2016
Test Date:	January 14, 2016

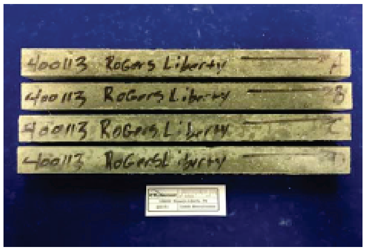
120032 Rogers Liberty, TN

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0337	-0.0042	-0.0047	0.0128	---
Final Comparator Length, in.:	-0.0114	0.0175	0.0170	0.0349	---
Calculated Autoclave Expansion, %:	**0.223**	**0.217**	**0.217**	**0.221**	**0.220**

Before Testing



After Testing



Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGROUP

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	120102
Aggregate Source:	China Lake, CA
Aggregate Type:	Coarse
CTLGroup ID:	4090306
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 13, 2016
Test Date:	January 15, 2016

120102 China Lake, CA

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0085	0.0007	0.0097	-0.0012	---
Final Comparator Length, in.:	-0.0018	0.0074	0.0165	0.0048	---
Calculated Autoclave Expansion, %:	**0.067**	**0.067**	**0.068**	**0.060**	**0.066**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
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Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

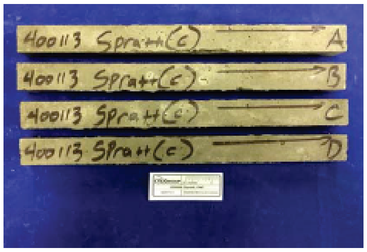
Sample Identification	
Client Aggregate Serial No.:	150098
Aggregate Source:	Spratt, ONT
Aggregate Type:	Coarse
CTLGroup ID:	4090317
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 14, 2015
Test Date:	December 16, 2015

150098 Spratt, ONT

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0001	-0.0040	-0.0147	-0.0054	---
Final Comparator Length, in.:	0.0355	0.0304	0.0188	0.0276	---
Calculated Autoclave Expansion, %:	**0.356**	**0.344**	**0.335**	**0.330**	**0.341**

Before Testing

After Testing



Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	130002
Aggregate Source:	Gold Hills, NC
Aggregate Type:	Coarse
CTLGroup ID:	4090313
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 14, 2015
Test Date:	December 16, 2015

130002 Gold Hills, NC

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0099	-0.0009	0.0435	-0.0015	---
Final Comparator Length, in.:	0.0530	0.0446	0.0881	0.0464	---
Calculated Autoclave Expansion, %:	**0.431**	**0.455**	**0.446**	**0.479**	**0.453**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	130006
Aggregate Source:	Las Placitas, NM
Aggregate Type:	Coarse
CTLGroup ID:	4090307
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 16, 2015
Test Date:	December 18, 2015

130006 Las Placitas, NM

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0057	0.0076	0.0050	0.0067	---
Final Comparator Length, in.:	0.0798	0.0825	0.0784	0.0802	---
Calculated Autoclave Expansion, %:	**0.741**	**0.749**	**0.734**	**0.735**	**0.740**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGROUP

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

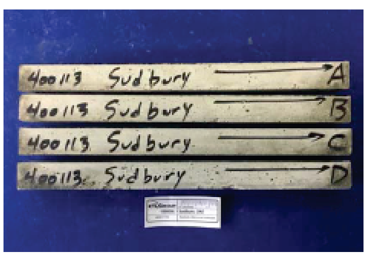
ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	150098.1
Aggregate Source:	Sudbury, ONT
Aggregate Type:	Coarse
CTLGroup ID:	4090314
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 12, 2016
Test Date:	January 14, 2016

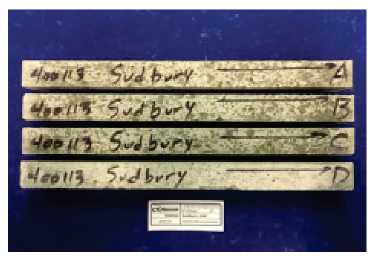
150098.1 Sudbury, ONT

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0302	-0.0044	-0.0156	-0.0030	---
Final Comparator Length, in.:	0.0046	0.0288	0.0164	0.0283	---
Calculated Autoclave Expansion, %:	**0.348**	**0.332**	**0.320**	**0.313**	**0.328**

Before Testing



After Testing



Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGROUP

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	130037
Aggregate Source:	Jobe, TX
Aggregate Type:	Coarse
CTLGroup ID:	4090312
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	February 8, 2016
Test Date:	February 10, 2016

130037 Jobe, TX

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0402	-0.0129	0.1132	0.0739	---
Final Comparator Length, in.:	0.0897	0.0382	0.1637	0.1238	---
Calculated Autoclave Expansion, %:	**0.495**	**0.511**	**0.505**	**0.499**	**0.503**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	130038
Aggregate Source:	Adairsville, GA
Aggregate Type:	Coarse
CTLGroup ID:	4090309
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 12, 2016
Test Date:	January 14, 2016

130038 Adairsville, GA

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0116	-0.0240	-0.0413	0.0141	---
Final Comparator Length, in.:	-0.0070	-0.0195	-0.0369	0.0185	---
Calculated Autoclave Expansion, %:	**0.046**	**0.045**	**0.044**	**0.044**	**0.045**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	130041
Aggregate Source:	Green Brothers, MS
Aggregate Type:	Fine
CTLGroup ID:	4090308
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 16, 2015
Test Date:	December 18, 2015

130041 Green Brothers, MS

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0370	0.0372	-0.0298	-0.0313	---
Final Comparator Length, in.:	-0.0098	0.0645	-0.0031	-0.0030	---
Calculated Autoclave Expansion, %:	**0.272**	**0.273**	**0.267**	**0.283**	**0.274**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	Battelle Memorial Institute	CTLGroup Proj. No.:	400113
Project:	Battelle Memorial Institute	CTLGroup Proj. Mgr.:	A. Bentivegna
		Technicians:	VS, WD
Contact:	Todd Weidner	Approved:	J. Gajda
Date Received:	October 23, 2015	Report Date:	July 13, 2016

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	140037
Aggregate Source:	Green Brothers #2, MS
Aggregate Type:	Fine
CTLGroup ID:	4090301
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	January 13, 2016
Test Date:	January 15, 2016

140037 Green Brothers #2, MS

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0006	0.0074	-0.0628	-0.0310	---
Final Comparator Length, in.:	0.0247	0.0320	-0.0368	-0.0033	---
Calculated Autoclave Expansion, %:	**0.253**	**0.246**	**0.260**	**0.277**	**0.259**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

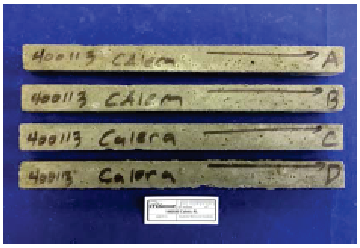
Sample Identification	
Client Aggregate Serial No.:	140040
Aggregate Source:	Calera, AL
Aggregate Type:	Fine
CTLGroup ID:	4090304
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 15, 2015
Test Date:	December 17, 2015

140040 Calera, AL

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0109	0.0107	-0.0056	0.0007	---
Final Comparator Length, in.:	0.0410	0.0397	0.0252	0.0321	---
Calculated Autoclave Expansion, %:	**0.301**	**0.290**	**0.308**	**0.314**	**0.303**

Before Testing

After Testing



Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	140085
Aggregate Source:	Nevada Test Site
Aggregate Type:	Fine
CTLGroup ID:	4090315
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 14, 2015
Test Date:	December 16, 2015

140085 Nevada Test Site

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0011	0.0097	-0.0131	-0.0067	---
Final Comparator Length, in.:	0.0066	0.0153	-0.0085	-0.0010	---
Calculated Autoclave Expansion, %:	**0.055**	**0.056**	**0.046**	**0.057**	**0.054**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification

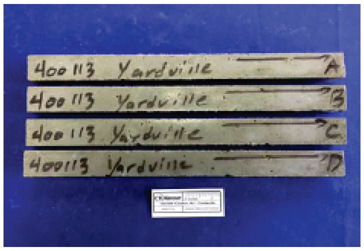
Client Aggregate Serial No.:	150104
Aggregate Source:	Clayton, NJ - Yardville
Aggregate Type:	Fine
CTLGroup ID:	4090302
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 15, 2015
Test Date:	December 17, 2015

150104 Clayton, NJ - Yardville

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0173	0.0268	0.0173	0.0171	---
Final Comparator Length, in.:	0.0231	0.0325	0.0225	0.0225	---
Calculated Autoclave Expansion, %:	**0.058**	**0.057**	**0.052**	**0.054**	**0.055**

Before Testing

After Testing



Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	150047
Aggregate Source:	Wabasha, MN
Aggregate Type:	Fine
CTLGroup ID:	4090318
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 14, 2015
Test Date:	December 16, 2015

150047 Wabasha, MN

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0151	-0.0143	0.0153	-0.0142	---
Final Comparator Length, in.:	0.0026	0.0030	0.0328	0.0033	---
Calculated Autoclave Expansion, %:	**0.177**	**0.173**	**0.175**	**0.175**	**0.175**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**October 23, 2015**	Report Date:	**July 13, 2016**

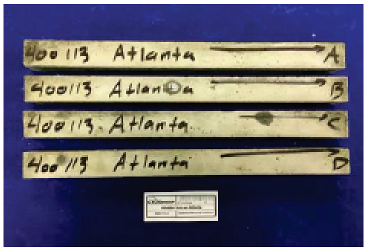
ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	150094
Aggregate Source:	Vulcan Atlanta
Aggregate Type:	Fine
CTLGroup ID:	4090303
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 15, 2015
Test Date:	December 17, 2015

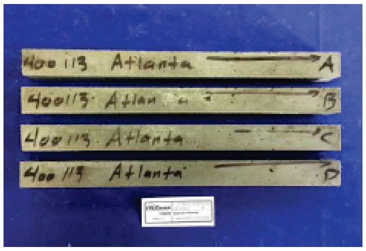
150094 Vulcan Atlanta

	A	B	C	D	Average
Initial Comparator Length, in.:	-0.0076	0.0746	0.0610	0.0708	---
Final Comparator Length, in.:	-0.0010	0.0814	0.0674	0.0773	---
Calculated Autoclave Expansion, %:	**0.066**	**0.068**	**0.064**	**0.065**	**0.066**

Before Testing



After Testing



Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	Battelle Memorial Institute	CTLGroup Proj. No.:	400113
Project:	Battelle Memorial Institute	CTLGroup Proj. Mgr.:	A. Bentivegna
		Technicians:	VS, WD
Contact:	Todd Weidner	Approved:	J. Gajda
Date Received:	October 23, 2015	Report Date:	July 13, 2016

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	152149
Aggregate Source:	Arkadelphia
Aggregate Type:	Fine
CTLGroup ID:	4090305
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	December 16, 2015
Test Date:	December 18, 2015

152149 Arkadelphia

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0622	0.0334	-0.0694	-0.0579	---
Final Comparator Length, in.:	0.0766	0.0466	-0.0520	-0.0387	---
Calculated Autoclave Expansion, %:	**0.144**	**0.132**	**0.174**	**0.192**	**0.161**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**April 6, 2016**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	150096
Aggregate Source:	LG Everest
Aggregate Type:	Coarse
CTLGroup ID:	4194802
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	April 19, 2016
Test Date:	April 21, 2016

150096 LG Everest

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0278	0.0370	-0.0126	0.0152	---
Final Comparator Length, in.:	0.0449	0.0550	0.0047	0.0330	---
Calculated Autoclave Expansion, %:	**0.171**	**0.180**	**0.173**	**0.178**	**0.176**

Before Testing

After Testing

Notes:

1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

CTLGroup

Client:	**Battelle Memorial Institute**	CTLGroup Proj. No.:	**400113**
Project:	**Battelle Memorial Institute**	CTLGroup Proj. Mgr.:	**A. Bentivegna**
		Technicians:	**VS, WD**
Contact:	**Todd Weidner**	Approved:	**J. Gajda**
Date Received:	**April 6, 2016**	Report Date:	**July 13, 2016**

ASR Autoclave Expansion with Mortar Bars

Sample Identification	
Client Aggregate Serial No.:	150100
Aggregate Source:	Geneva
Aggregate Type:	Coarse
CTLGroup ID:	4194803
Cement Type:	Type I/II
Cement Source:	Holcim, St. Genevieve
CTLGroup ID:	4090311
Cast Date:	April 19, 2016
Test Date:	April 21, 2016

150100 Geneva

	A	B	C	D	Average
Initial Comparator Length, in.:	0.0893	0.0336	-0.0153	0.0567	---
Final Comparator Length, in.:	0.1050	0.0482	0.0004	0.0726	---
Calculated Autoclave Expansion, %:	**0.157**	**0.146**	**0.157**	**0.159**	**0.155**

Before Testing

After Testing

Notes:
1. Test specimens are 1x1x11.25-in. mortar bars.
2. Aggregate arrived at CTLGroup pre-washed, sieved, and weighed for batching.
3. NaOH was added to the water to achieve a total alkali loading in the mortar of 3.5%.
4. Test specimens batched, cured and tested per your direction.
5. An automated autoclave was used for testing.
6. A positive value denotes expansion; a negative value denotes shrinkage.
7. This report may not be reproduced except in its entirety.

Appendix B: Petrographic Analysis of Aggregates

B.1 Vulcan-Cookville Coarse Aggregate Sample (CMB No. 120010)

The Vulcan-Cookeville coarse aggregate sample (CMB 120010) was classified as a dense limestone. The color of the particles varied from grayish orange pink (5YR 7/2), pale brown (5YR 5/2), very pale green (10G 8/2) to medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B1). The particles ranged from angular to sub-angular in shape with some quartz inclusions. Aggregates were mostly dense with a small percentage being vuggy or fractured. Analysis of the XRD patterns (Figure B2) indicated that the predominant phase in the material was calcite. The weight percent data obtained by whole pattern fitting using Jade 2010 software is shown in Figure B3. Calcite ($CaCO_3$) (66.4%), dolomite ($CaMg(CO_3)_2$) (30.6%), quartz (SiO_2) (2.5%), and trace amounts of chlorite ($(Mg,Fe^{2+})_5Al_2Si_3O_{10}(OH)_8$) and feldspar ($NaAlSi_3O_8$) were identified for this sample. The quartz within the sample had an index of refraction that was equal to or greater than 1.544 of the immersion oil, indicating no chalcedony present in the sample.

Figure B1. Vulcan-Cookeville Coarse Aggregate Sample (CMB 120010).

Figure B2. X-ray pattern for Vulcan-Cookeville Coarse Aggregate Sample (CMB 120010).

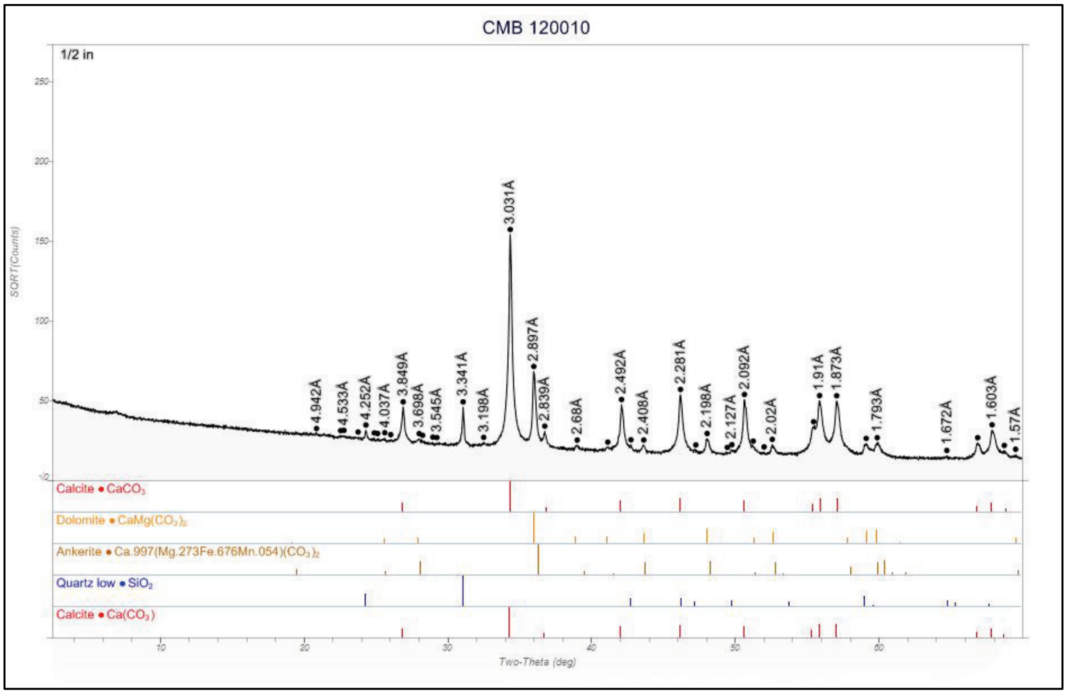
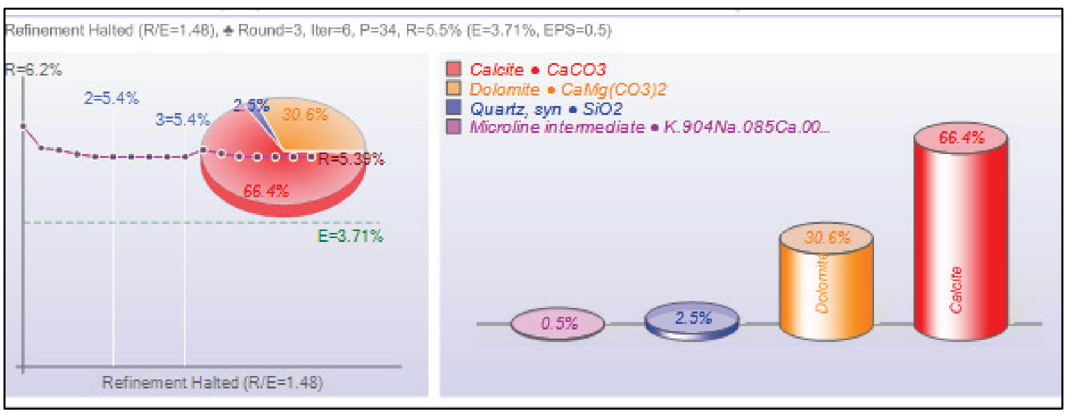


Figure B3. Whole pattern fit with mineral percentages for Vulcan-Cookeville Coarse Aggregate Sample (CMB 120010).



B.2 Rogers-Gordonville Coarse Aggregate Sample (CMB No. 120016)

The Rogers-Gordonville coarse aggregate sample (CMB 120016) was classified as a dense dolostone. The color of the particles varied from very pale orange (10YR 8/2), medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B4). The particles ranged from angular to sub-angular in shape with some quartz and sphalerite inclusions. The aggregates were mostly dense with a small percentage being vuggy or fractured. Analysis of the XRD pattern (Figure B5) indicated that the predominant phase in the material was dolomite. The weight percent data obtained by whole pattern fitting using Jade 2010 software is shown in Figure B6. Dolomite ($CaMg(CO_3)_2$) (74.8%), quartz (SiO_2) (17.3%), calcite ($CaCO_3$) (5.7%), and microcline ($KAlSi_3O_8$) (2.1%) were identified for this sample. The quartz within the sample had an index of refraction that was equal to or greater than 1.544 of the immersion oil, indicating no chalcedony present in the sample.

Figure B4. Rogers-Gordonville Coarse Aggregate Sample (CMB No. 120016).

Figure B5. X-ray pattern for Rogers-Gordonville Coarse Aggregate Sample (CMB No. 120016).

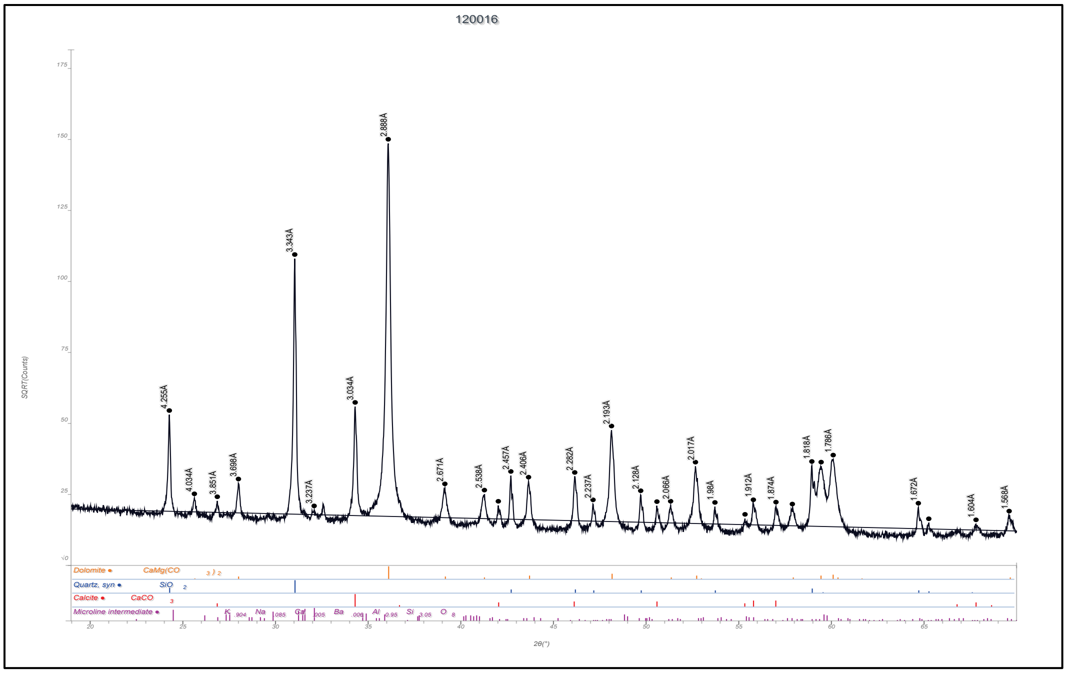
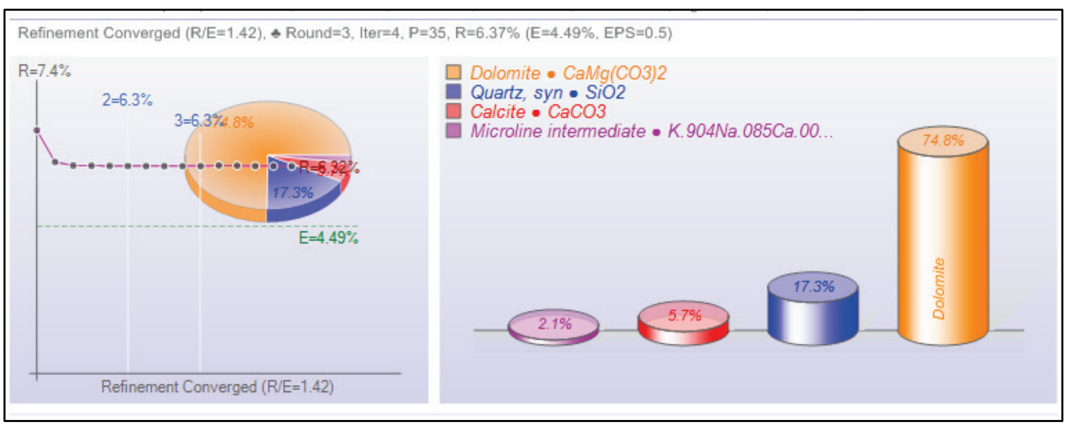


Figure B6. Whole pattern fit with mineral percentages Rogers-Gordonville Coarse Aggregate Sample (CMB No. 120016).



B.3 Cookville Coarse Aggregate Sample (CMB No. 120021)

The Cookeville coarse aggregate sample (CMB 120021) was classified as an ooilitic to dense limestone. The color of the particles varied from light greenish gray (5Y 8/1), medium gray (N5), medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B7). The particles ranged from angular to sub-angular in shape with some quartz inclusions. The aggregates were mostly dense with a small percentage being vuggy or fractured. Analysis of the XRD patterns (Figure B8) indicated that the predominant phase in the material was calcite. The weight percent data obtained by whole pattern fitting using Jade 2010 software is shown in Figure B9. Calcite ($CaCO_3$) (84.7%), dolomite ($CaMg(CO_3)_2$) (9.8%), and quartz (SiO_2) (5.5%) were identified for this sample. The quartz within the sample had an index of refraction that was equal to or greater than 1.544 of the immersion oil, indicating no chalcedony present in the sample.

Figure B7. Cookville Coarse Aggregate Sample (CMB No. 120021).

Figure B8. X-ray pattern for Cookville Coarse Aggregate Sample (CMB No. 120021).

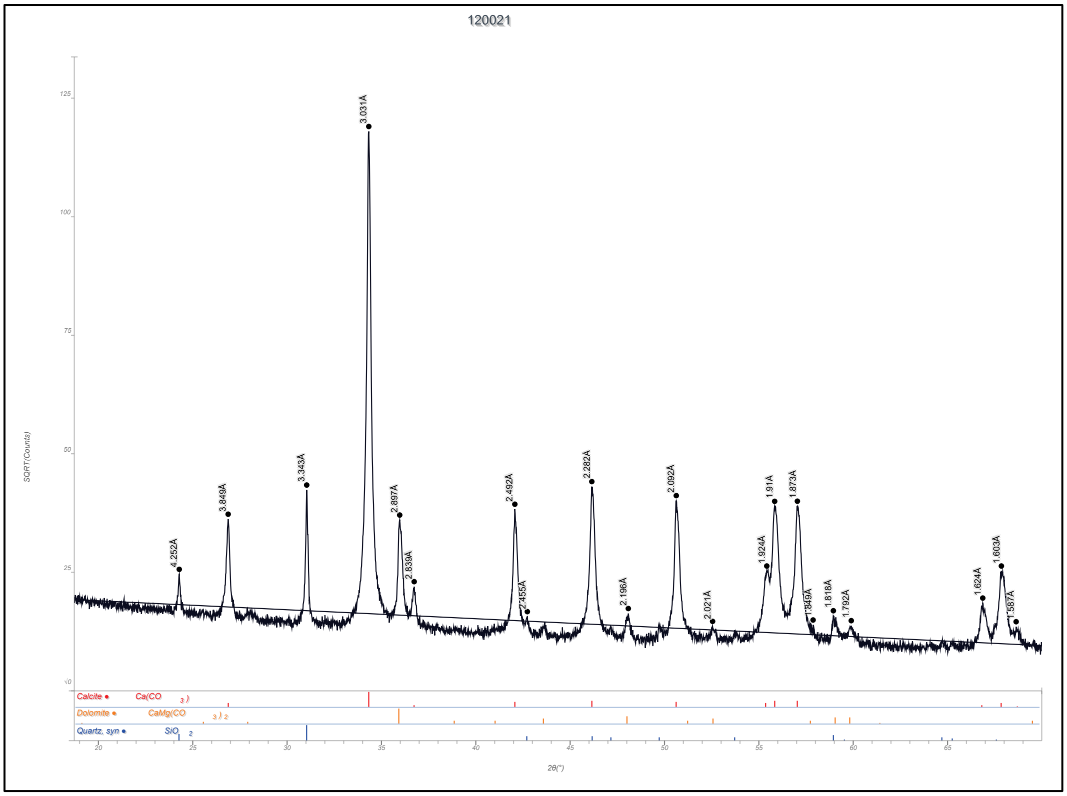
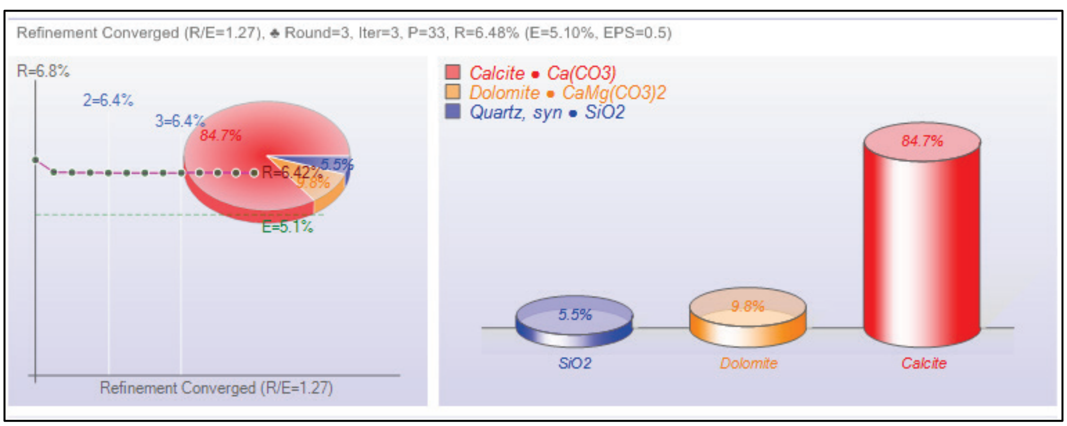


Figure B9. Whole pattern fit with mineral percentages Cookville Coarse Aggregate Sample (CMB No. 120021).



B.4 Rogers-Liberty Coarse Aggregate Sample (CMB No. 120032)

The Rogers-Liberty coarse aggregate sample (CMB No. 120032) was classified as a dense limestone. The color of the particles varied from medium dark gray (N4), medium gray (N5), medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B10). The particles ranged from angular to sub-angular in shape with some quartz inclusions. The aggregates were mostly dense with a small percentage being vuggy or fractured. Analysis of the XRD patterns (Figure B11) indicated that the predominant phase in the material was calcite. The weight percent data obtained by whole pattern fitting using Jade 2010 software is shown in Figure B12. Calcite ($Ca(CO_3)$) (73.1%), dolomite ($CaMg(CO_3)_2$) (20.1%), quartz (SiO_2) (6.8%), with trace amounts of albite ($NaAlSi_3O_8$) were identified for this sample. The quartz within the sample had an index of refraction that was equal to or greater than 1.544 of the immersion oil, indicating no chalcedony present in the sample.

Figure B10. Rogers-Liberty Coarse Aggregate Sample (CMB No. 120032).

Figure B11. X-ray pattern for Rogers-Liberty Coarse Aggregate Sample (CMB No. 120032).

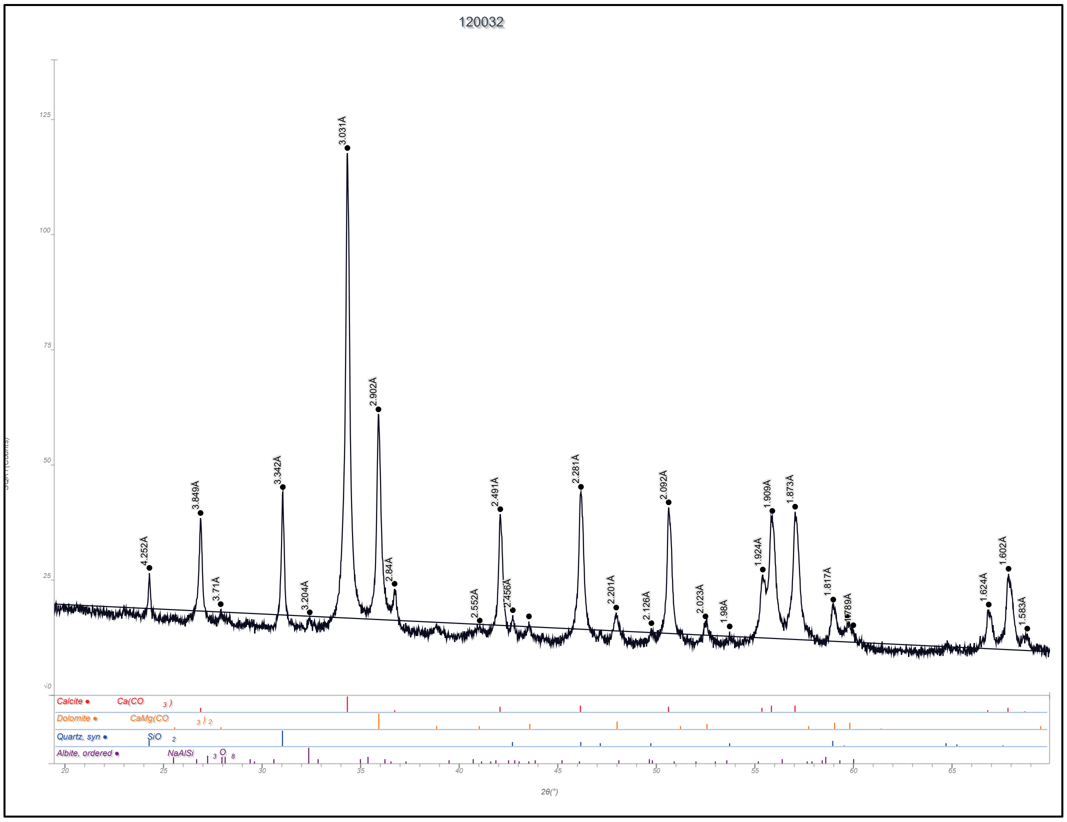
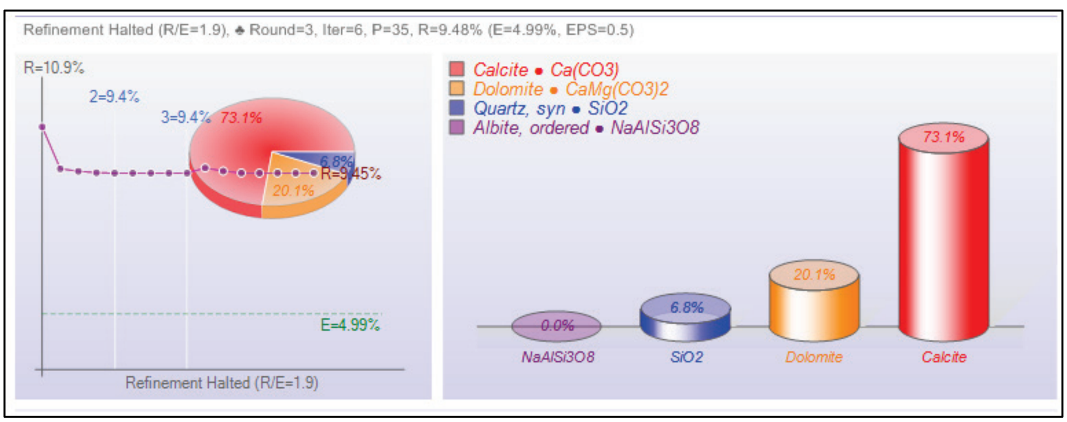


Figure B12. Whole pattern fit with mineral percentages Rogers-Liberty Coarse Aggregate Sample (CMB No. 120032).



B.5 China Lake Coarse Aggregate Sample (CMB No. 120102)

The China Lake coarse aggregate sample (CMB No. 120102) was classified as an intermediate to felsic igneous rock. The color of the particles varied from moderate orange pink (10R 7/4), very pale orange (10YR 8/2), grayish orange (10YR 7/4), pale yellowish orange (10YR 8/6), yellowish gray (5Y 8/1), pinkish gray (5YR 8/1), medium dark gray (N4), medium gray (N5), medium light gray (N6), to white (N9) in color (Figure B13). The particles ranged from angular to sub-angular in shape. The aggregates were mostly dense with a small percentage being vuggy or fractured. Analysis of the XRD patterns (Figure B14) indicated that the predominant phase in the material was a calcium-rich feldspar. The weight percent data obtained by whole pattern fitting using Jade 2010 software is shown in Figure B15. Anorthite (48.4%), quartz (SiO_2) (17.6%), magnesio-hornblende (9.3%), albite (9.2%), orthoclase (6.6%), biotite (5.0%), clinopyroxene (2.5%), and trace amounts of talc (0.8%), clinochlore (0.3%), and cristobalite (0.3%) were identified for this sample.

Figure B13. China Lake Coarse Aggregate Sample (CMB No. 120102).

Figure B14. X-ray pattern for China Lake Coarse Aggregate Sample (CMB No. 120102).

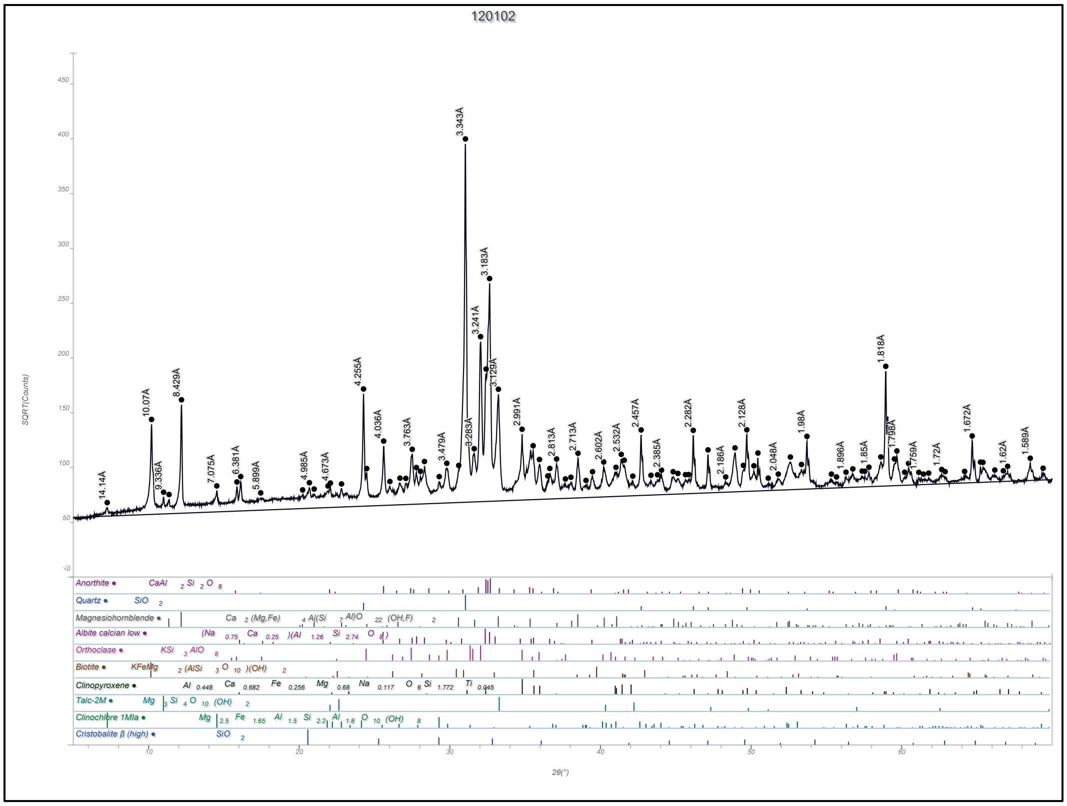
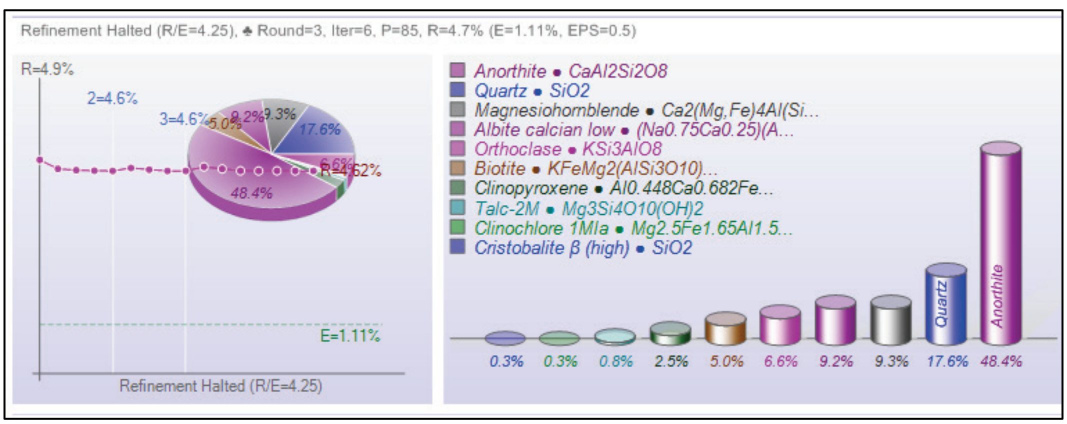


Figure B15. Whole pattern fit with mineral percentages China Lake Coarse Aggregate Sample (CMB No. 120102).



B.6 Spratt Coarse Aggregate Sample (CMB No. 130001)

The Spratt coarse aggregate sample (CMB No. 130001) was classified as a dense limestone. The color of the particles varied from medium dark gray (N4), medium gray (N5), medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B16). The particles ranged from angular to sub-angular in shape with some quartz inclusions.

Analysis of the XRD patterns (Figure B17) indicated that the predominant phase in the material was calcite. The weight percent data obtained by whole pattern fitting using Jade 2010 software are given in Figure B18. Calcite ($CaCO_3$) (82.5%), dolomite ($CaMg(CO_3)_2$) (12.1%), and quartz (SiO_2) (5.3%) were identified for this sample.

Figure B16. Spratt Coarse Aggregate Sample (CMB No. 130001).

Figure B17. X-ray pattern for Spratt Coarse Aggregate Sample (CMB No. 130001).

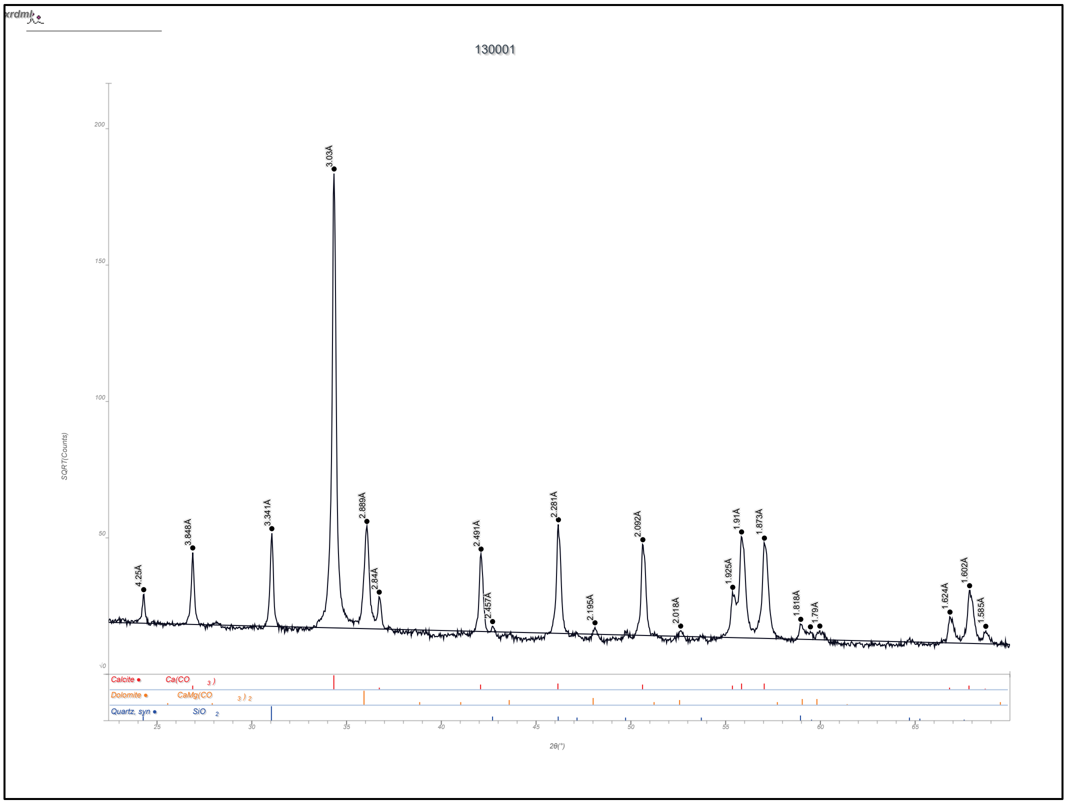
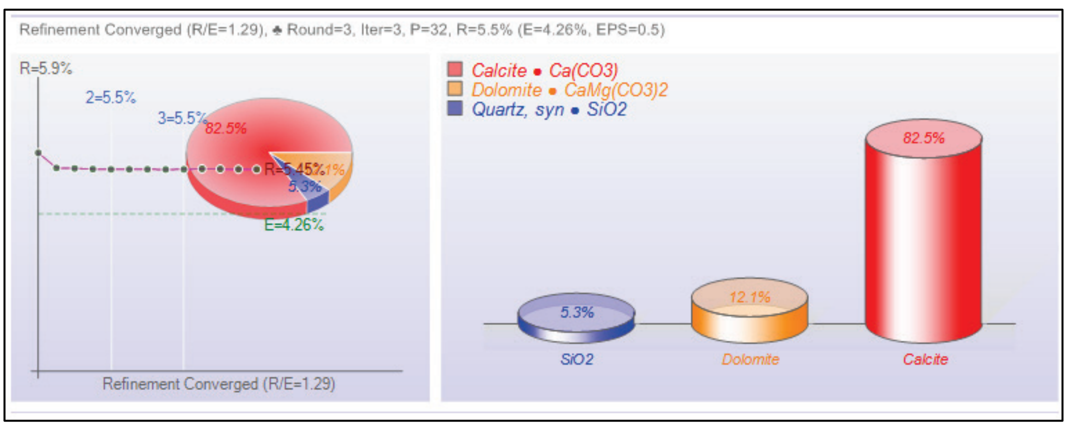


Figure B18. Whole pattern fit with mineral percentages Spratt Coarse Aggregate Sample (CMB No. 130001).



B.7 NC Rhyolite Coarse Aggregate Sample (CMB No. 130002)

The NC rhyolite coarse aggregate sample (CMB No. 130002) was classified as metapelite green schist. The color of the particles varied from grayish blue (5B 5/6), very light gray (N8), medium light gray (N6), medium gray (N5) to medium dark gray (N3) in color, with dark yellowish orange (10YR 6/6) staining (Figure B19). Analysis of the XRD patterns (Figure B20) indicated that the predominant phase in the material was muscovite. Figure B21 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (39%), chlorite/clinochlore ($((Mg, Fe)_3(Si,Al)_4O_{10}$-$(OH)_2 \cdot (Mg,Fe)_3(OH)_6)$(31%), quartz ($SiO_2$) (18%), albite ($Na(AlSi_3O_8)$) (9%), orthoclase ($KAlSi_3O_8$) (1%), and trace amounts of cristobalite (SiO_2) were identified for this sample.

Figure B19. NC Rhyolite Coarse Aggregate Sample (CMB No. 130002).

Figure B20. X-ray pattern for NC Rhyolite Coarse Aggregate Sample (CMB No. 130002).

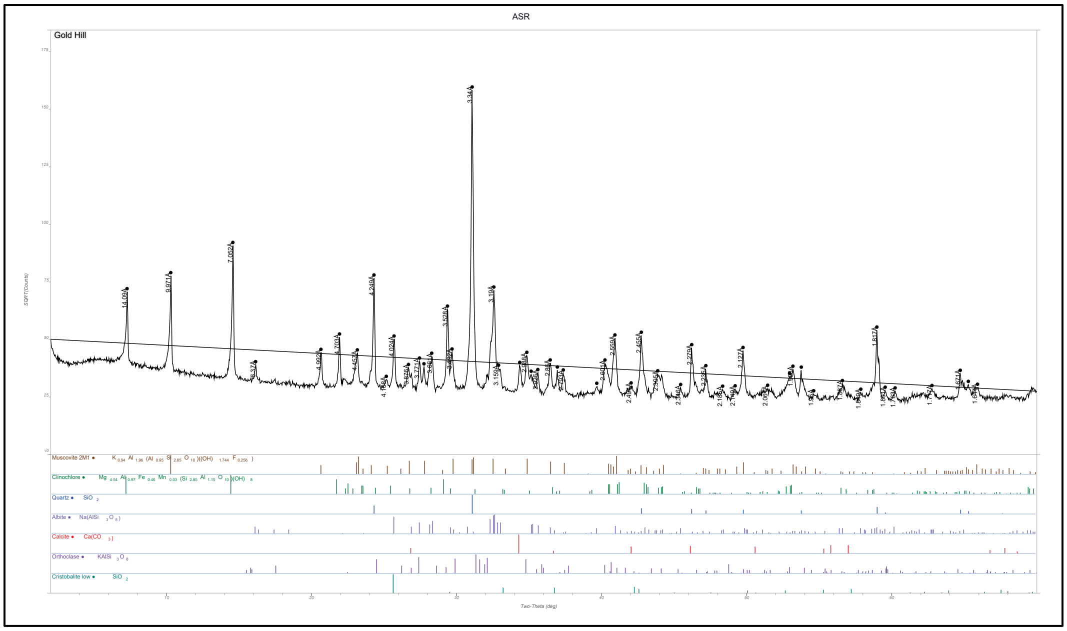
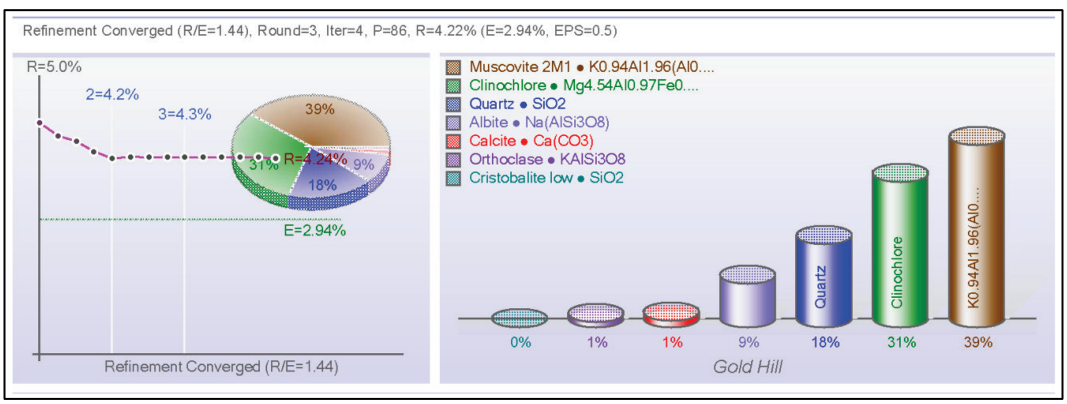


Figure B21. Whole pattern fit with mineral percentages NC Rhyolite Coarse Aggregate Sample (CMB No. 130002).



B.8 Las Placitas Coarse Aggregate Sample (CMB No. 130006)

The Las Placitas coarse aggregate sample (CMB No. 130006) was classified as a quartzite and granite mixture. The color of the particles varied from very pale orange (10YR 8/2), pale yellowish orange (10YR 8/6), grayish orange (10YR 7/4), light brown (5YR 6/4), greenish gray (SG 6/1), grayish green (5GY 6/1), dark greenish gray (5GY 4/1, 5G 4/1), very light gray (N8), light brown (5YR 6/4) to moderate brown (5YR 4/4) in color (Figure B22).The aggregates were angular to well-rounded, mostly dense with a small fraction being vuggy (vesicular). Analysis of the XRD patterns (Figure B23) indicated that the predominant phase in the material was sodium feldspar. Figure B24 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Albite ($Na(AlSi_3O_8)$) (38%), quartz (SiO_2) (27%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (17%), orthoclase ($KAlSi_3O_8$) (10%), calcite ($CaCO_3$) (5%), cristobalite ($SiO_2$) (2%), actinolite ($Ca_2(Mg,Fe^{2+})_5Si_8O_{22}(OH)_2$ (1%), and trace amounts of and chlorite/clinochlore ($(Mg, Fe)_3(Si,Al)_4O_{10}$-$(OH)_2 \cdot (Mg,Fe)_3(OH)_6$) and hematite ($Fe_2O_3$) were identified for this sample.

Figure B22. Las Placitas Coarse Aggregate Sample (CMB No. 130006).

Figure B23. X-ray pattern for Las Placitas Coarse Aggregate Sample (CMB No. 130006).

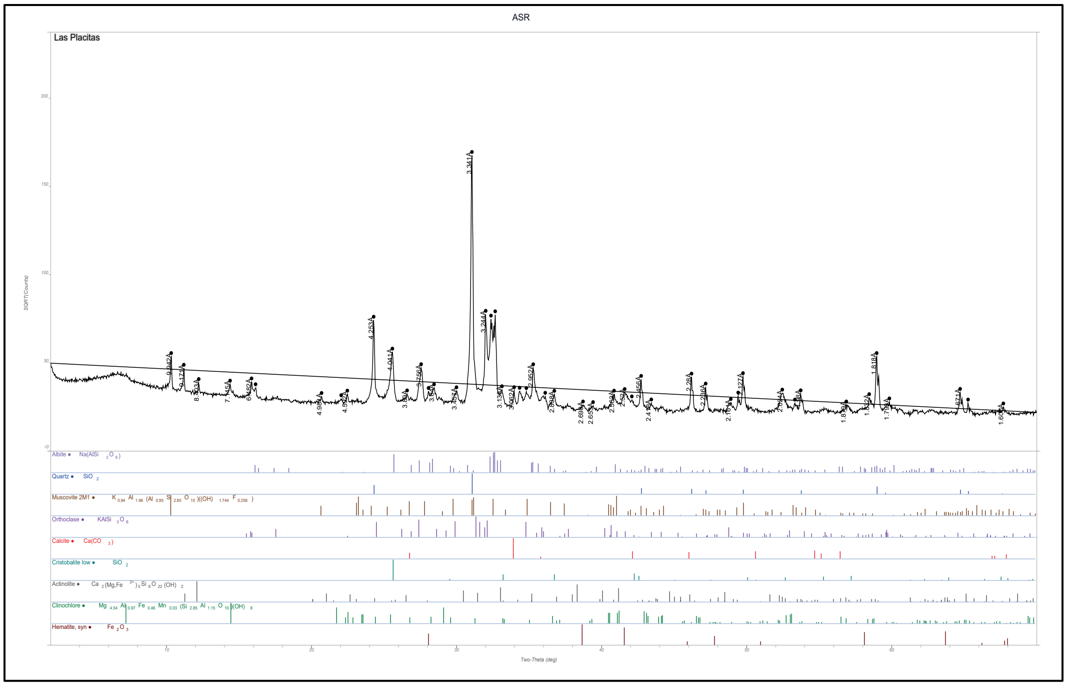
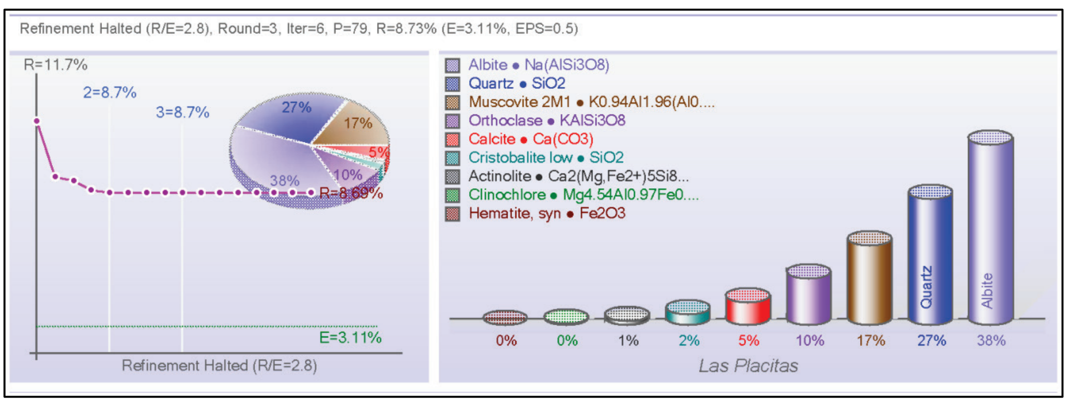


Figure B24. Whole pattern fit with mineral percentages Las Placitas Coarse Aggregate Sample (CMB No. 130006).



B.9 Sudbury Coarse Aggregate Sample (CMB No. 130030)

The Sudbury coarse aggregate sample (CMB No. 130030) was classified as a green schist, quartzite and granite mixture. The color of the particles varied from moderate orange pink (5YR 8/4), pale yellowish orange (10YR 8/6), very pale orange (10YR 8/2), grayish orange (10YR 7/4), light brown (5YR 5/6), dark yellowish brown (10YR 4/2), grayish blue (5B 5/6), light brownish gray (5YR 6/1), greenish gray (5G 6/1), grayish green (5GY 6/1), yellowish gray (5Y 8/1), medium bluish gray (5B 5/1), dark greenish gray (5GY 4/1, 5G 4/1), white (N9), very light gray (N8), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4) in color (Figure B25). Analysis of the XRD patterns (Figure B26) indicated that the predominant phase in the material was muscovite. Figure B27 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (28%), albite ($Na(AlSi_3O_8)$) (24%), chlorite/clinochlore ($(Mg, Fe)_3(Si,Al)_4(O_{10}\text{-}OH)_2 \cdot (Mg,Fe)_3(OH)_6$) (20%), quartz ($SiO_2$) (20%), orthoclase ($KAlSi_3O_8$) (5%), ferropargasite ($NaCa_2(Fe,Mg)Al(Si_6Al_2)O_{22}(OH,Cl)_2$) (4%), and trace amounts of cristobalite (SiO_2) and calcite ($CaCO_3$) were identified for this sample.

Figure B25. Sudbury Coarse Aggregate Sample (CMB No. 130030).

Figure B26. X-ray pattern for Sudbury Coarse Aggregate Sample (CMB No. 130030).

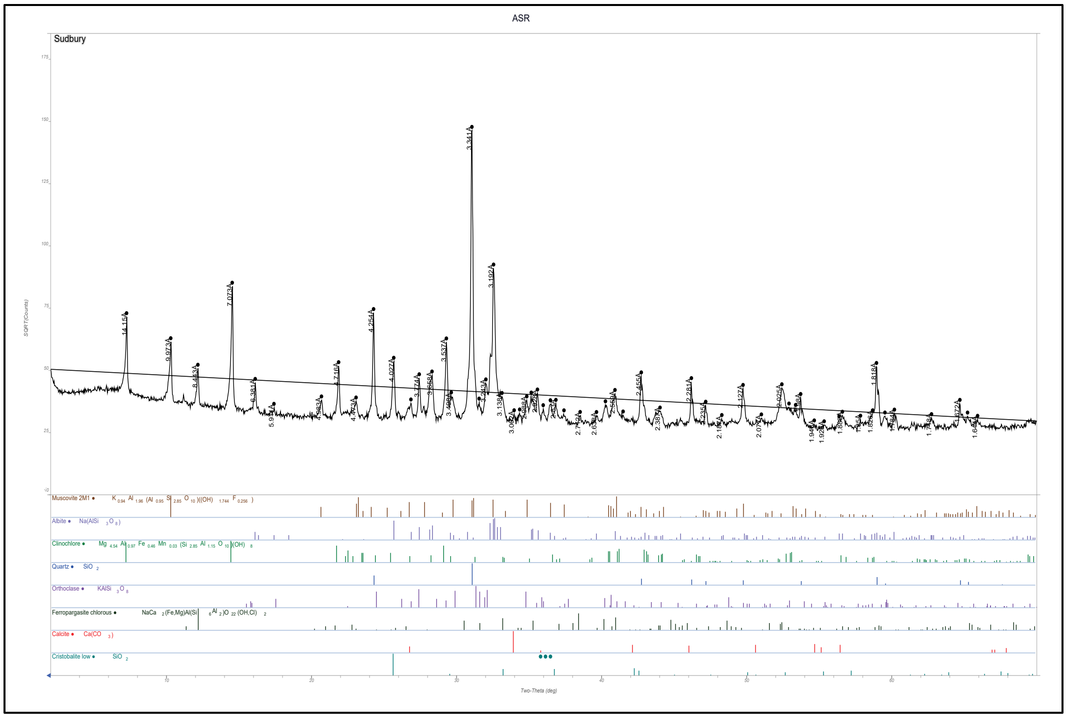
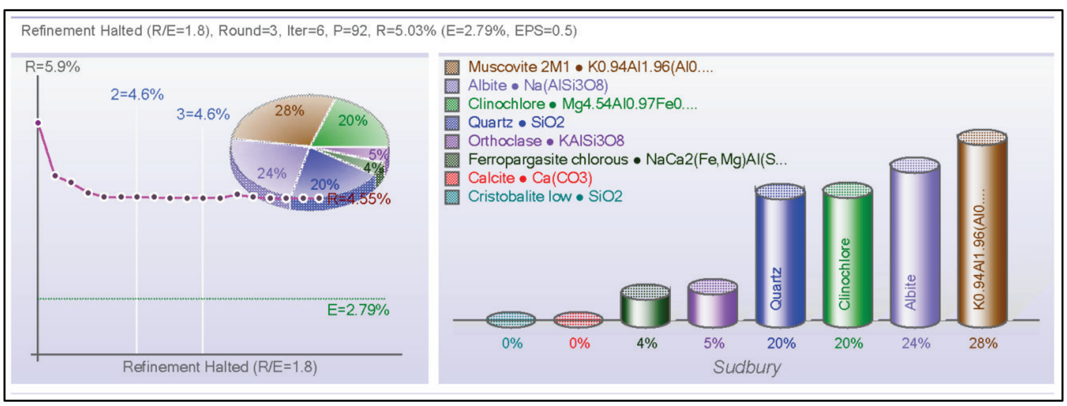


Figure B27. Whole pattern fit with mineral percentages Sudbury Coarse Aggregate Sample (CMB No. 130030).



B.10 Jobe Coarse Aggregate Sample (CMB No. 130037)

The Jobe coarse aggregate sample (CMB No. 130037) was classified as complex alluvial gravel with granite/rhyolite, basalt, welded tuff, chert and amber. The color of the particles varied from pale red (5R 6/2), dark red (5R 3/4), grayish red (10R 4/2), moderate reddish brown (10R 4/6), grayish orange pink (10R 8/2), grayish blue (5B 5/6), white (N9), very light gray (N8), medium light gray (N6), medium gray (N5) to medium dark gray (N3) in color (Figure B28). Analysis of the XRD patterns (Figure B29) indicated that the predominant phase in the material was microcline. Figure B30 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Microcline ((K,Na,Ca,Ba)$AlSi_3O_8$) (56%), quartz (SiO_2) (18%), calcite ($CaCO_3$) (17%), albite (Na($AlSi_3O_8$)) (5%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (2%), cristobalite (SiO2) (1%), and hematite (Fe_2O_3) (1%) were identified for this sample.

Figure B28. Jobe Coarse Aggregate Sample (CMB No. 130037).

Figure B29. X-ray pattern for Jobe Coarse Aggregate Sample (CMB No. 130037).

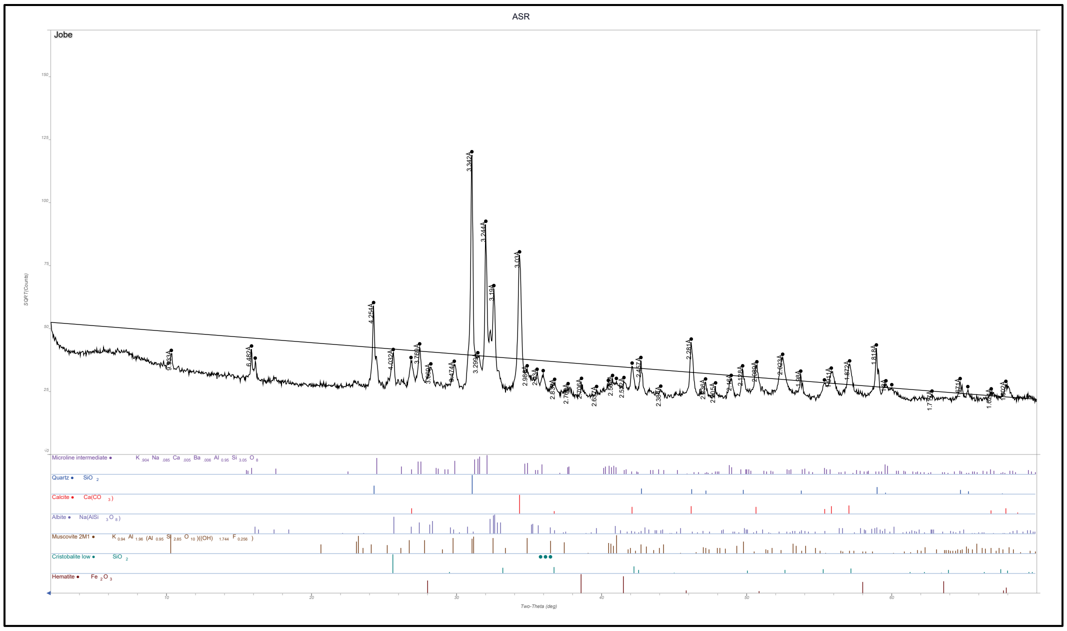
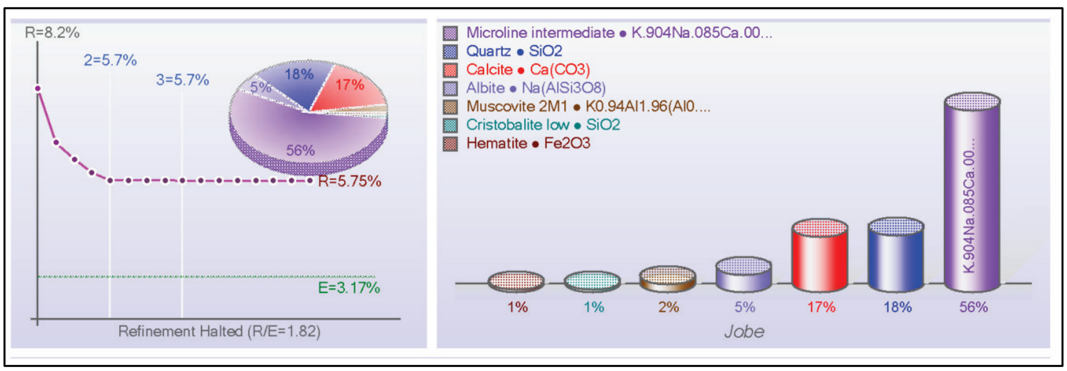


Figure B30. Whole pattern fit with mineral percentages Jobe Coarse Aggregate Sample (CMB No. 130037).



B.11 Adairsville Coarse Aggregate Sample (CMB No. 130038)

The Adairsville coarse aggregate sample (CMB No. 130038) was classified as a dolostone. The color of the particles varied from grayish blue (5B 5/6), very light gray (N8), medium light gray (N6), medium gray (N5) to medium dark gray (N3) in color (Figure B31). Analysis of the XRD patterns (Figure B32) indicated that the predominant phase in the material was dolomite. Figure B33 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($Ca,Mg(CO_3)_2$) (99%), with trace amounts of quartz (SiO_2), ankerite ($Ca(Fe^{+2}Mg)(CO_3)_2$), feldspar ($KAlSi_3O_8/(Ca,Na)(Si,Al)_4O_8$) were identified for this sample.

Figure B31. Adairsville Coarse Aggregate Sample (CMB No. 130038).

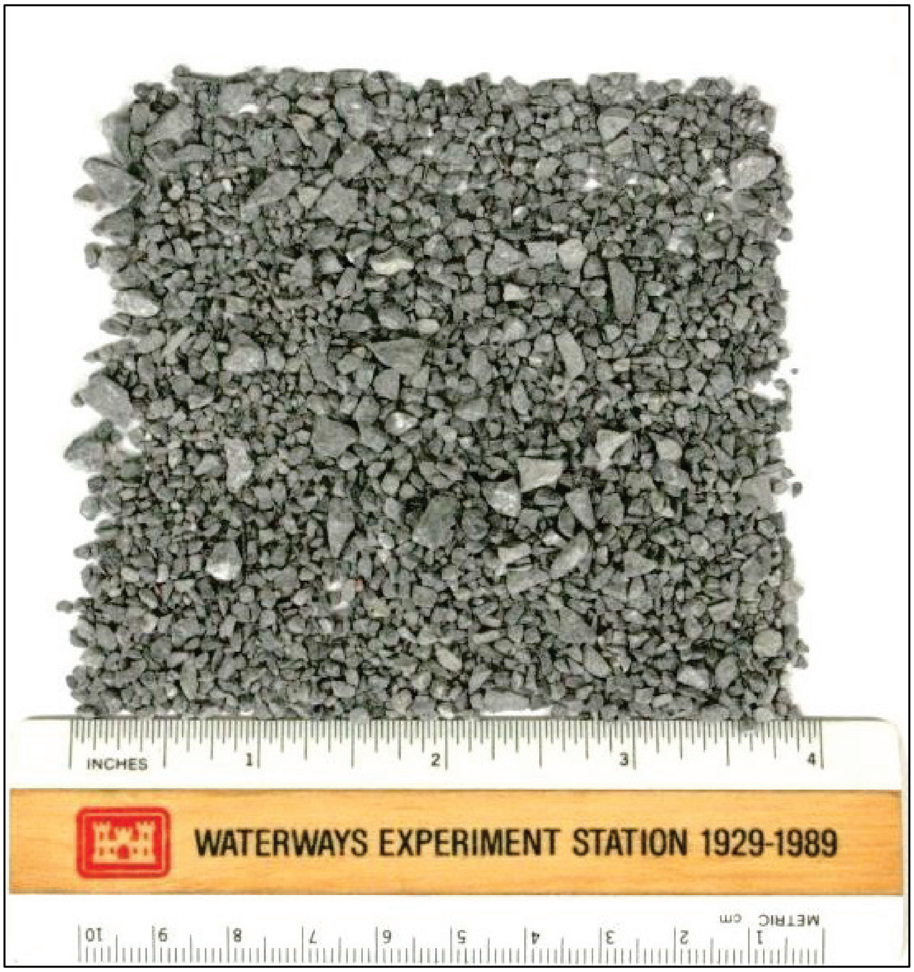


Figure B32. X-ray pattern for Adairsville Coarse Aggregate Sample (CMB No. 130038).

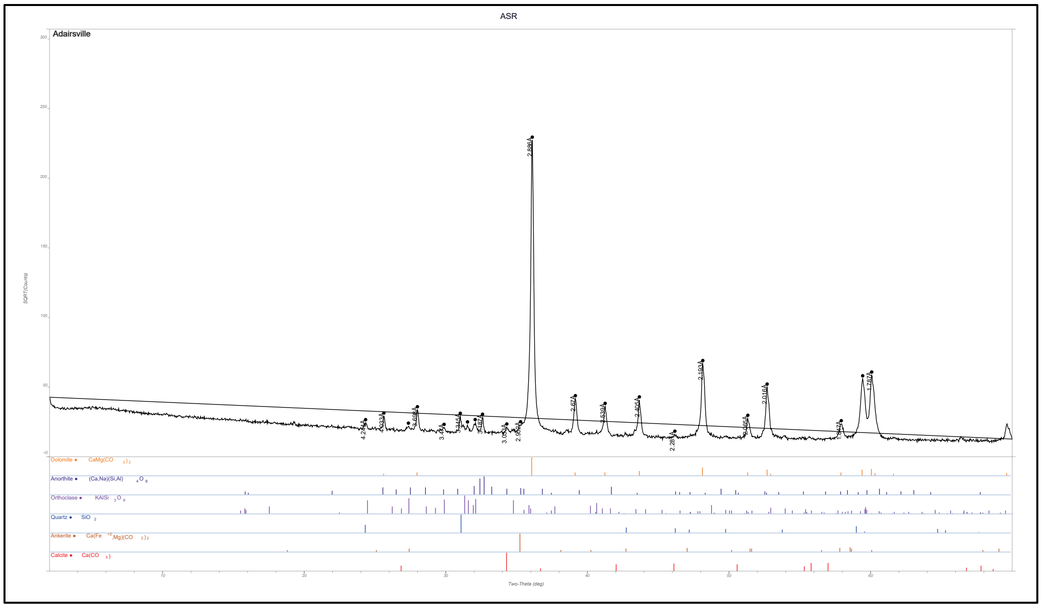
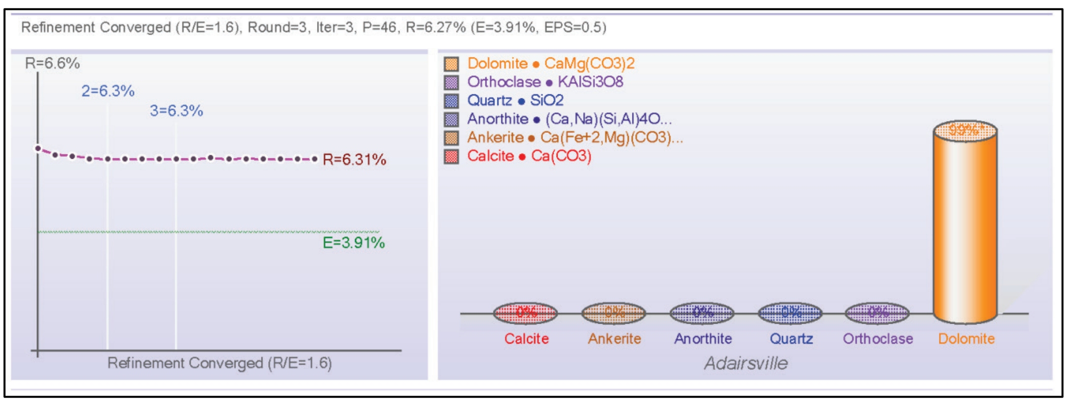


Figure B33. Whole pattern fit with mineral percentages Adairsville Coarse Aggregate Sample (CMB No. 130038).



B.12 Beltane Opal Coarse Aggregate Sample (CMB No. 130039)

The Beltane opal coarse aggregate sample (CMB No. 130039) was classified as an opal. The color of the particles varied from white (N9), pinkish gray (5YR 8/1), very pale orange (10YR 8/2), moderate orange pink (5Yr 8/4) to pale yellowish orange (10YR 8/6) in color (Figure B34). Analysis of the XRD patterns (Figure B35) indicated that the predominant phase in the material was tridymite. Figure B36 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Tridymite (SiO_2) (39%), cristobalite α (SiO_2) (28%), quartz (SiO_2) (20%), cristobalite β (SiO_2) (8%), and kaolinite ($Al_2Si_2O_5OH_4$) (5%) were identified for this sample.

Figure B34. Beltane Opal Coarse Aggregate Sample (CMB No. 130039).

Figure B35. X-ray pattern for Beltane Opal Coarse Aggregate Sample (CMB No. 130039).

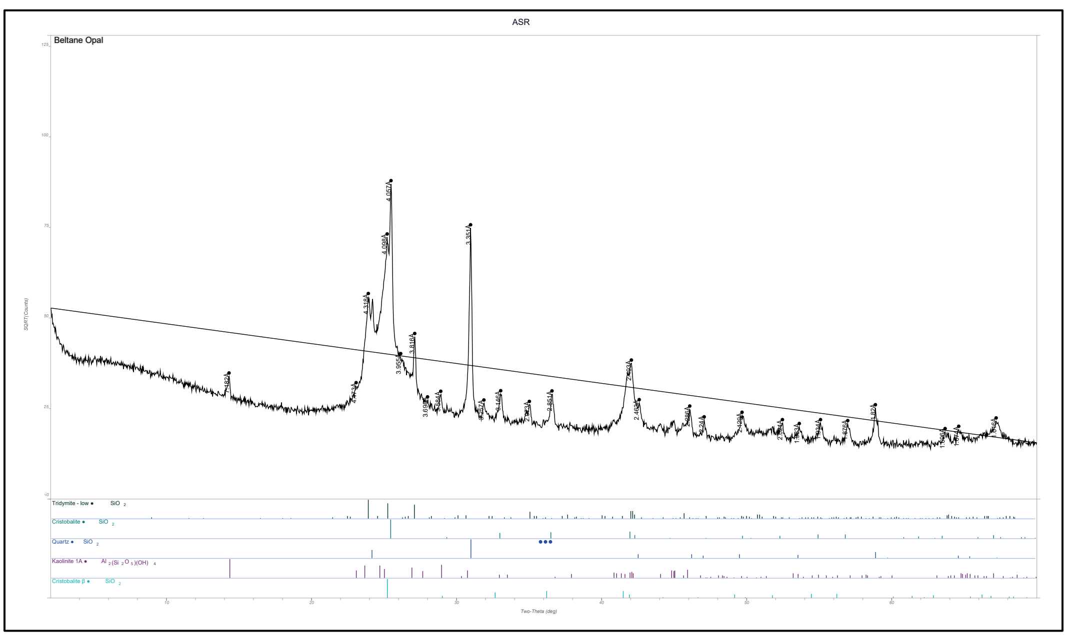
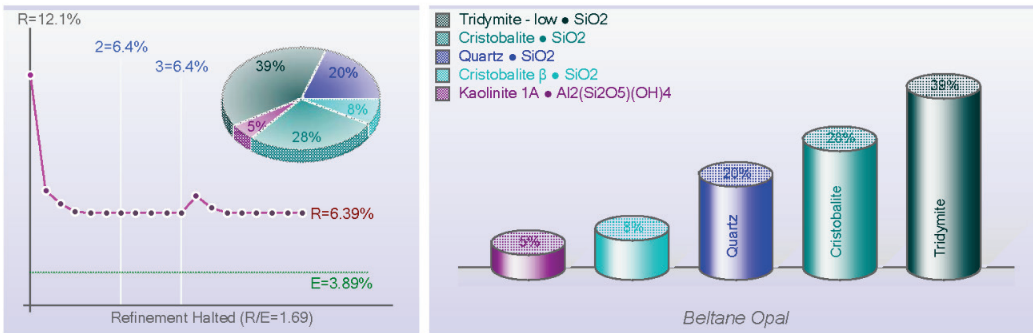


Figure B36. Whole pattern fit with mineral percentages Beltane Opal Coarse Aggregate Sample (CMB No. 130039).

Phase ID (5)	Chemical Formula	PDF-#	NA	NR	NP	Wt% (esd)	RIR
Quartz	SiO_2	01-074-1811	---	12	14	20 (1)	3.06
Tridymite - low	SiO_2	01-088-1535	---	140	16	39 (2)	1.59
Kaolinite 1A	$Al_2(Si_2O_5)(OH)_4$	01-078-1996	---	77	13	5 (1)	1.12
Cristobalite, α, syn	SiO_2	01-089-3434	---	23	14	28 (1)	4.90
Cristobalite β, syn	SiO_2	01-082-1405	---	23	14	8 (1)	4.77

XRF(Wt%): SiO2=97.1%, Al2O3=2.1%

Refinement Halted (R/E=1.69), Round=3, Iter=7, P=64, R=6.58% (E=3.89%, EPS=0.5)



B.13 Calera Coarse Aggregate Sample (CMB No. 130040)

The Calera coarse aggregate sample (CMB No. 130040) was classified as a dolostone. The color of the particles varied from grayish blue (5B 5/6), light bluish gray (10B 5/1), very light gray (N8), medium light gray (N6) to medium gray (N5) in color (Figure B37). Analysis of the XRD patterns (Figure B38) indicated that the predominant phase in the material was dolomite. Figure B39 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($Ca,Mg(CO_3)_2$) (73%), calcite ($CaCO_3$) (26%), and quartz (SiO_2) (1%) with trace amounts of anorthite ($CaAl_2Si_3O_8$) were identified for this sample.

Figure B37. Calera Coarse Aggregate Sample (CMB No. 130040).

Figure B38. X-ray pattern for Calera Coarse Aggregate Sample (CMB No. 130040).

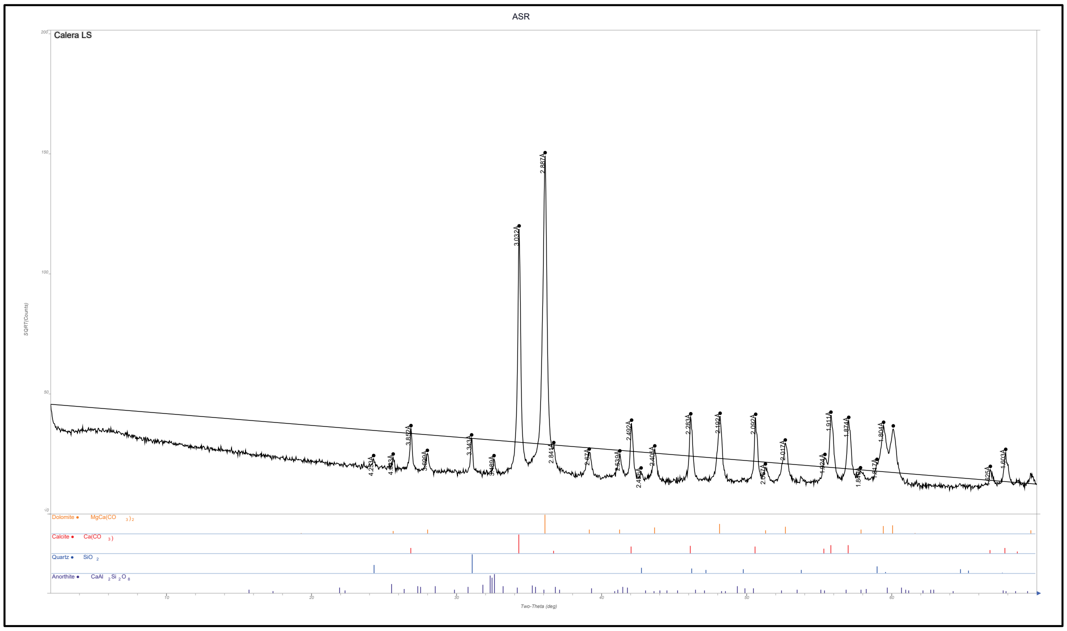
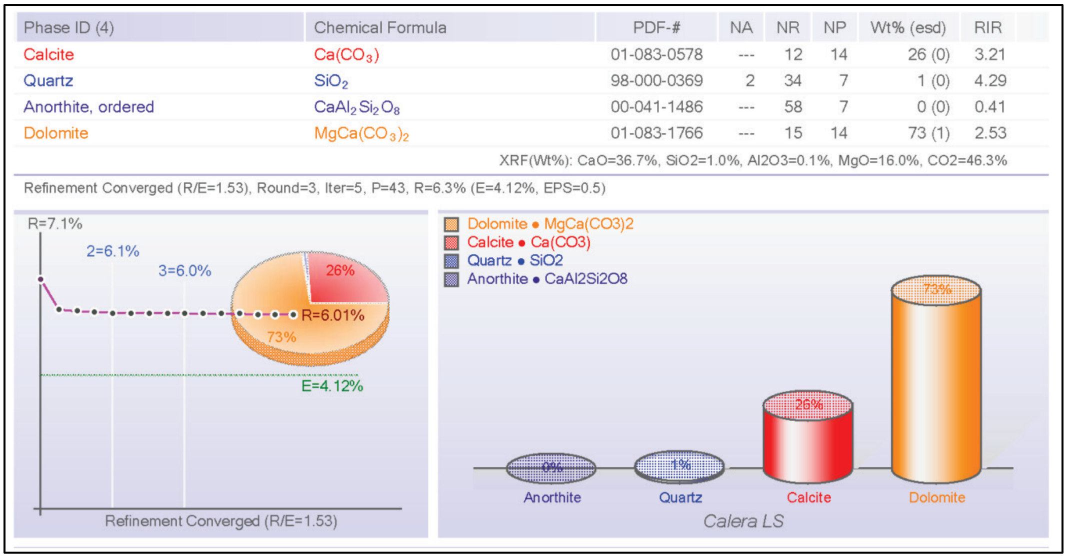


Figure B39. Whole pattern fit with mineral percentages Calera Coarse Aggregate Sample (CMB No. 130040).



Phase ID (4)	Chemical Formula	PDF-#	NA	NR	NP	Wt% (esd)	RIR
Calcite	$Ca(CO_3)$	01-083-0578	---	12	14	26 (0)	3.21
Quartz	SiO_2	98-000-0369	2	34	7	1 (0)	4.29
Anorthite, ordered	$CaAl_2Si_2O_8$	00-041-1486	---	58	7	0 (0)	0.41
Dolomite	$MgCa(CO_3)_2$	01-083-1766	---	15	14	73 (1)	2.53

XRF(Wt%): CaO=36.7%, SiO2=1.0%, Al2O3=0.1%, MgO=16.0%, CO2=46.3%

Refinement Converged (R/E=1.53), Round=3, Iter=5, P=43, R=6.3% (E=4.12%, EPS=0.5)

B.14 Green Brothers Fine Aggregate Sample (CMB No. 130041)

The Green Brothers fine aggregate sample (CMB No. 130041) was classified as quartz gravel. The color of the particles varied from moderate red (5R 4/6), moderate reddish brown (10R 4/6), pale yellowish orange (10YR 8/6), moderate orange pink (5YR 8/4), light brown (5YR 5/6), moderate yellowish brown (10YR 5/4), moderate brown (5YR 3/4) and grayish brown (5YR 3/4) in color (Figure B40). Analysis of the XRD patterns (Figure B41) indicated that the predominant phase in the material was quartz. Figure B42 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (94%), orthoclase ($KAlSi_3O_8$) (5%), and iron (Fe) (1%) were identified for this sample.

Figure B40. Green Brothers Fine Aggregate Sample (CMB No. 130041).

Figure B41. X-ray pattern for Green Brothers Fine Aggregate Sample (CMB No. 130041).

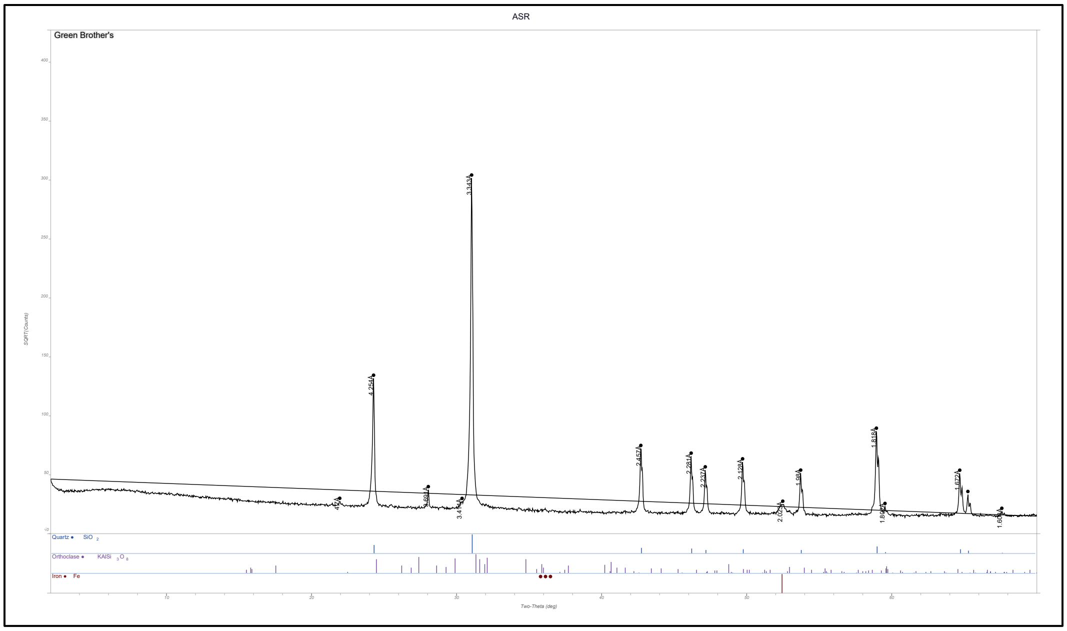
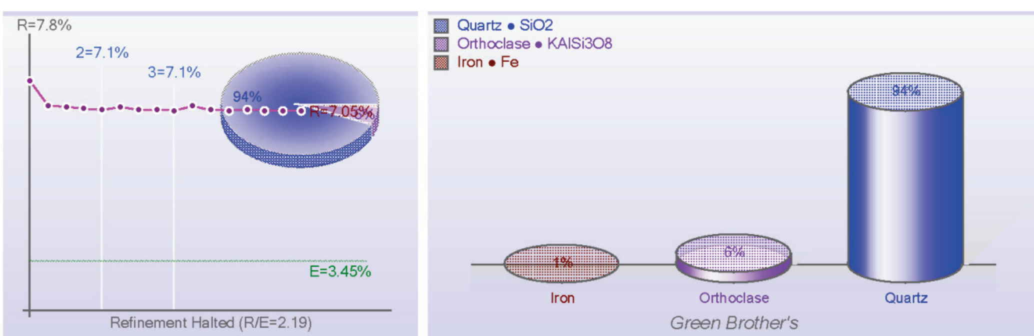


Figure B42. Whole pattern fit with mineral percentages Green Brothers Fine Aggregate Sample (CMB No. 130041).

Phase ID (3)	Chemical Formula	PDF-#	NA	NR	NP	Wt% (esd)	RIR
Quartz	SiO_2	98-000-0369	2	34	14	94 (1)	4.29
Orthoclase	$KAlSi_3O_8$	98-000-0338	9	95	11	6 (1)	0.69
Iron	Fe	01-089-7194	---	1	4	1 (0)	10.77

XRF(Wt%): Fe2O3=0.9%, K2O=1.0%, SiO2=97.3%, Al2O3=1.1%

Refinement Halted (R/E=2.19), Round=3, Iter=6, P=36, R=7.54% (E=3.45%, EPS=0.5)



B.15 Calera Coarse Aggregate Sample (CMB No. 130040 - No. 67)

The Calera coarse aggregate sample (CMB No. 130030 – No. 67) was classified as a dolostone. The color of the particles varied from grayish blue (5B 5/6), light bluish gray (10B 5/1), very light gray (N8), medium light gray (N6) to medium gray (N5) in color (Figure B43). Analysis of the XRD patterns (Figure B44) indicated that the predominant phase in the material was dolomite. Figure B45 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($CaMg(CO_3)_2$) (60.8%), calcite ($CaCO_3$) (36.3%), and quartz (SiO_2) (2.9%) were identified in this sample.

Figure B43. Calera Coarse Aggregate Sample (CMB No. 130040 - No. 67).

Figure B44. X-ray pattern for Calera Coarse Aggregate Sample (CMB No. 130040 - No. 67).

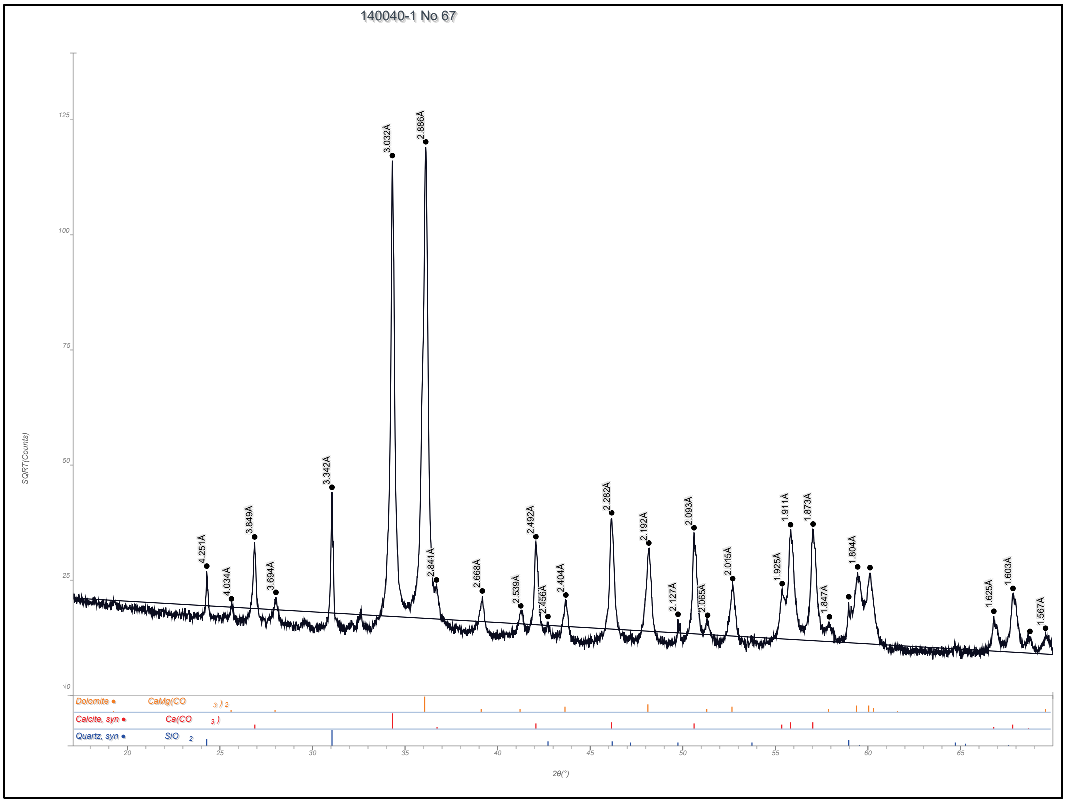
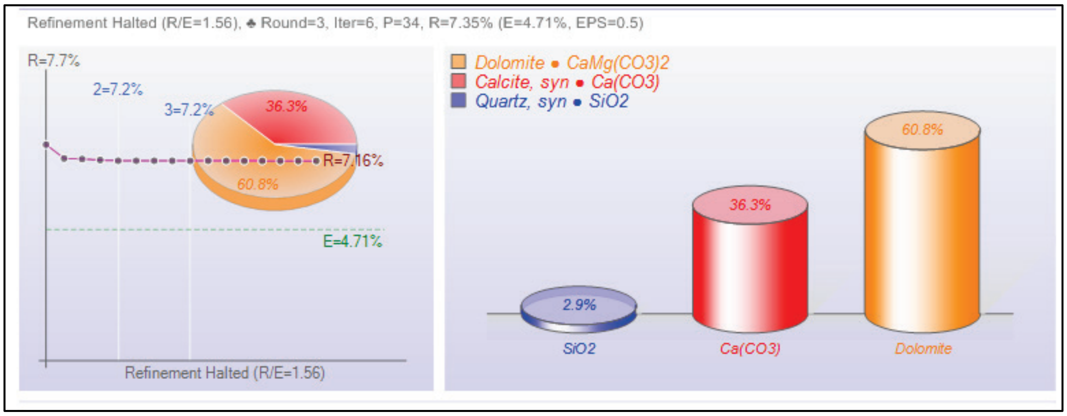


Figure B45. Whole pattern fit with mineral percentages Calera Coarse Aggregate Sample (CMB No. 130040 - No. 67).



B.16 Calera Coarse Aggregate Sample (CMB No. 130040)

The Calera coarse aggregate sample (CMB No. 130040) was classified as a dolostone. The color of the particles varied from grayish blue (5B 5/6), light bluish gray (10B 5/1), very light gray (N8), medium light gray (N6) to medium gray (N5) in color (Figure B46). Analysis of the XRD patterns (Figure B47) indicated that the predominant phase in the material was dolomite. Figure B48 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Calcite ($CaCO_3$) (61.5%), dolomite ($CaMg(CO_3)_2$) (35.6%), and quartz (SiO_2) (2.9%) were identified in this sample.

Figure B46. Calera Coarse Aggregate Sample (CMB No. 130040).

Figure B47. X-ray pattern for Calera Coarse Aggregate Sample (CMB No. 130040).

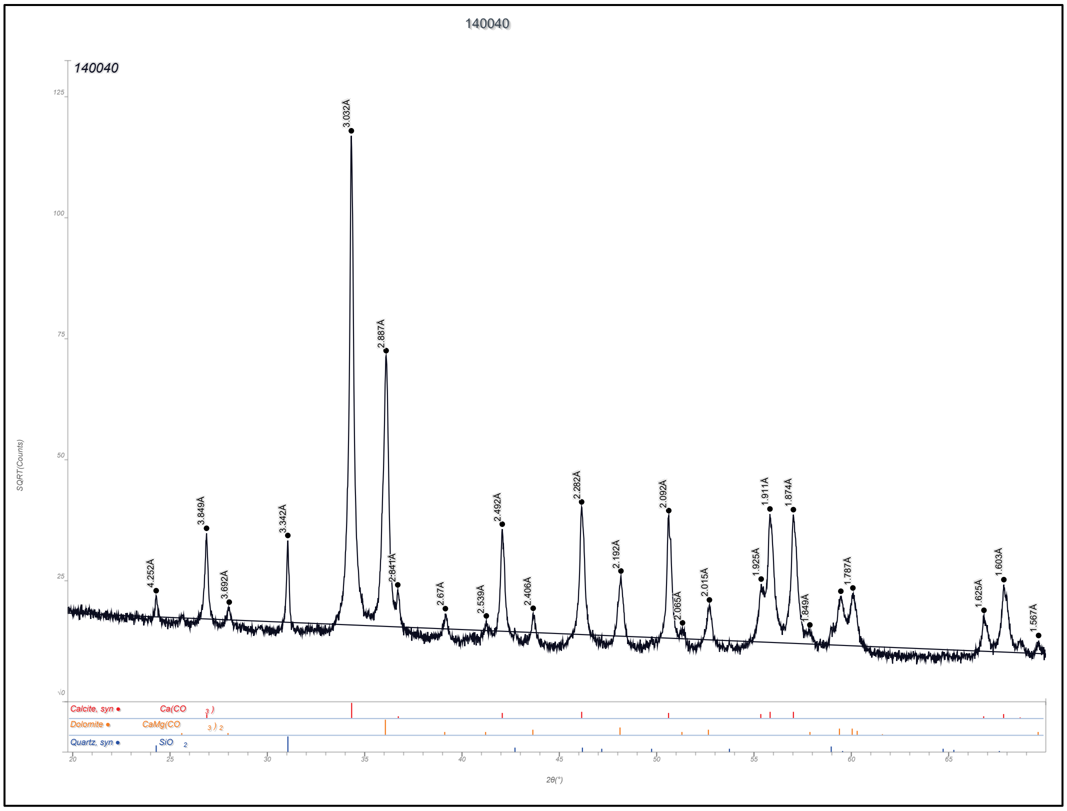
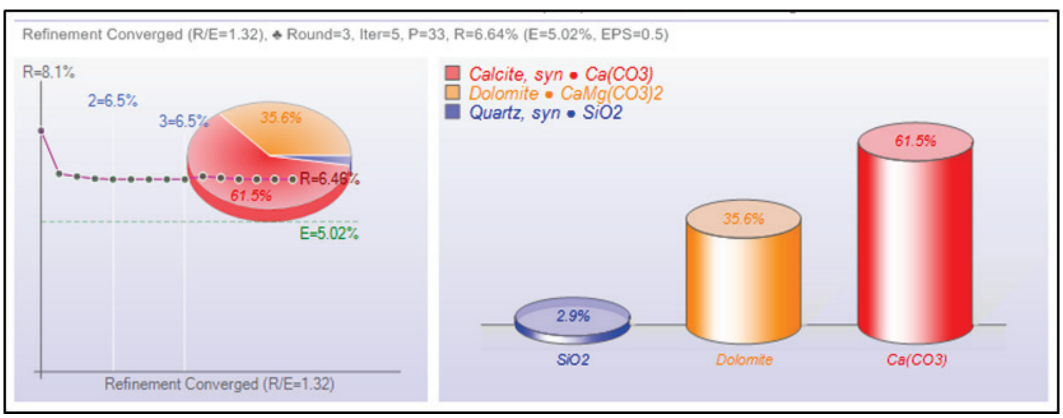


Figure B48. Whole pattern fit with mineral percentages Calera Coarse Aggregate Sample (CMB No. 130040).



B.17 Green Brothers Fine Aggregate Sample (CMB No. 140037)

The Green Brothers fine aggregate sample (CMB No. 140037) was classified as quartz gravel. The color of the particles varied from moderate red (5R 4/6), moderate reddish brown (10R 4/6), pale yellowish orange (10YR 8/6), moderate orange pink (5YR 8/4), light brown (5YR 5/6), moderate yellowish brown (10YR 5/4), moderate brown (5YR 3/4) and grayish brown (5YR 3/4) in color (Figure B49). Analysis of the XRD patterns (Figure B50) indicated that the predominant phase in the material was quartz. Figure B51 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (94.2%), microcline ($KAlSi_3O_8$) (5.7%), and trace amounts of cristobolite (SiO_2) were identified for this sample.

Figure B49. Green Brothers Fine Aggregate Sample (CMB No. 140037).

Figure B50. X-ray pattern for Green Brothers Fine Aggregate Sample (CMB No. 140037).

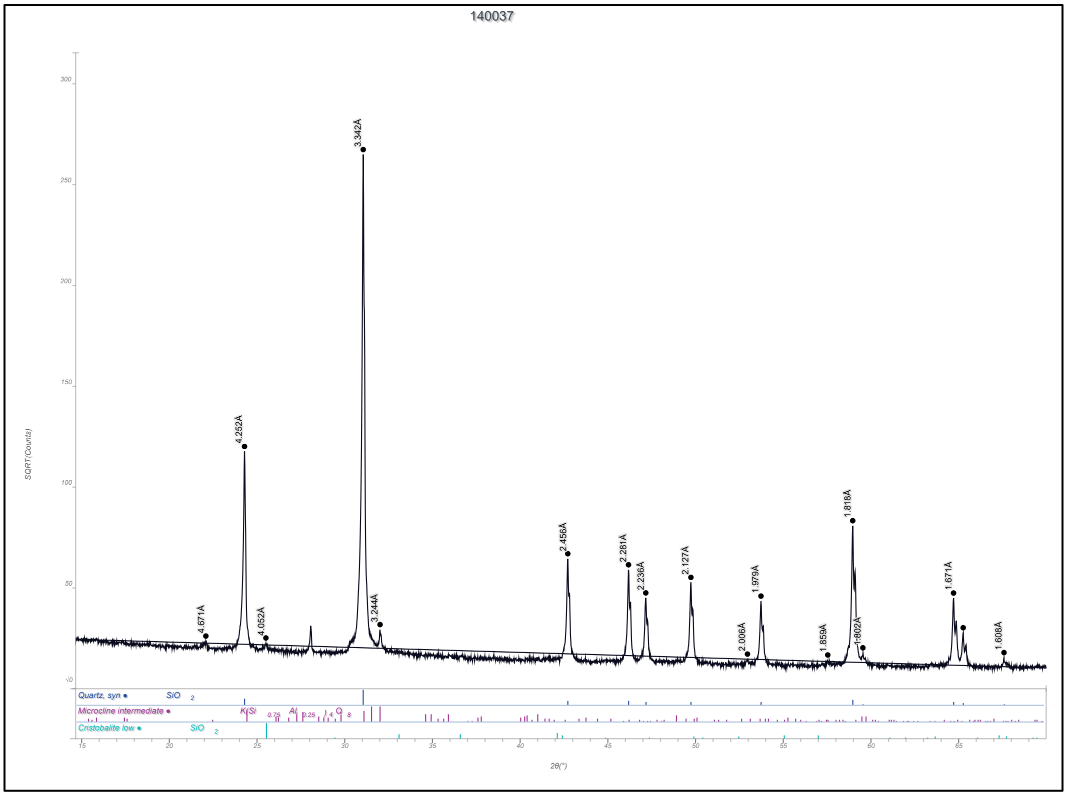
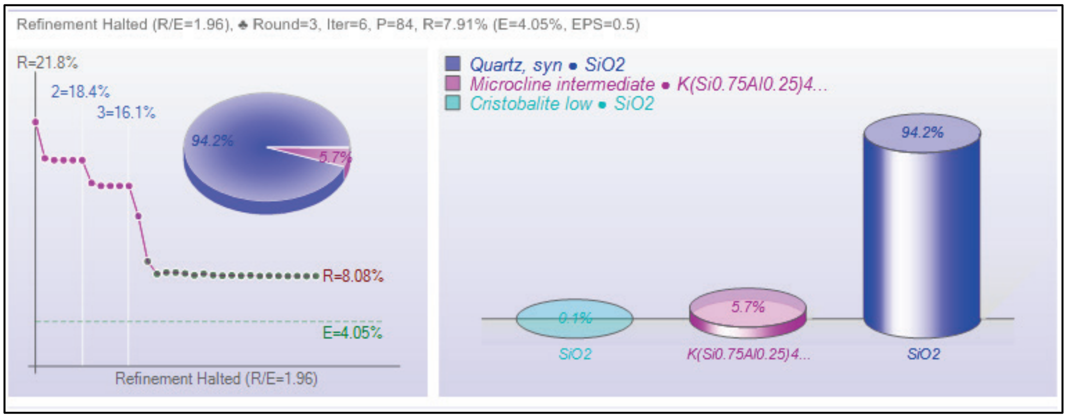


Figure B51. Whole pattern fit with mineral percentages Green Brothers Fine Aggregate Sample (CMB No. 140037).



B.18 Nevada Coarse Aggregate Sample (CMB No. 140084)

The Nevada coarse aggregate sample (CMB No. 140084) was classified as a complex gravel with fine conglomerate to arkose aggregates. The color of the particles varied from white (N9), very light gray (N8), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4), grayish pink (5R 8/2), grayish orange pink (10R 8/2), moderate orange pink (10R 7/4), light red (5R 6/6), pale reddish brown (10R 5/4), grayish red (10R 4/2), dark reddish brown (10R 3/4), very pale orange (10YR 8/2), grayish orange (10YR 7/4), dark yellowish orange (10YR 6/6), moderate yellowish brown (10YR 5/4) in color (Figure B52). Analysis of the XRD patterns (Figure B53) indicated that the predominant phase in the material was quartz. Figure B54 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (56.0%), microcline ($KAlSi_3O_8$) (20.6%), calcite ($CaCO_3$) (10.9%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (10.0%), cristobalite (SiO_2) (1.7%) and trace amounts of albite (Na(Al3O8)) and iron (Fe) were identified for this sample.

Figure B52. Nevada Coarse Aggregate Sample (CMB No. 140084).

Figure B53. X-ray pattern for Nevada Coarse Aggregate Sample (CMB No. 140084).

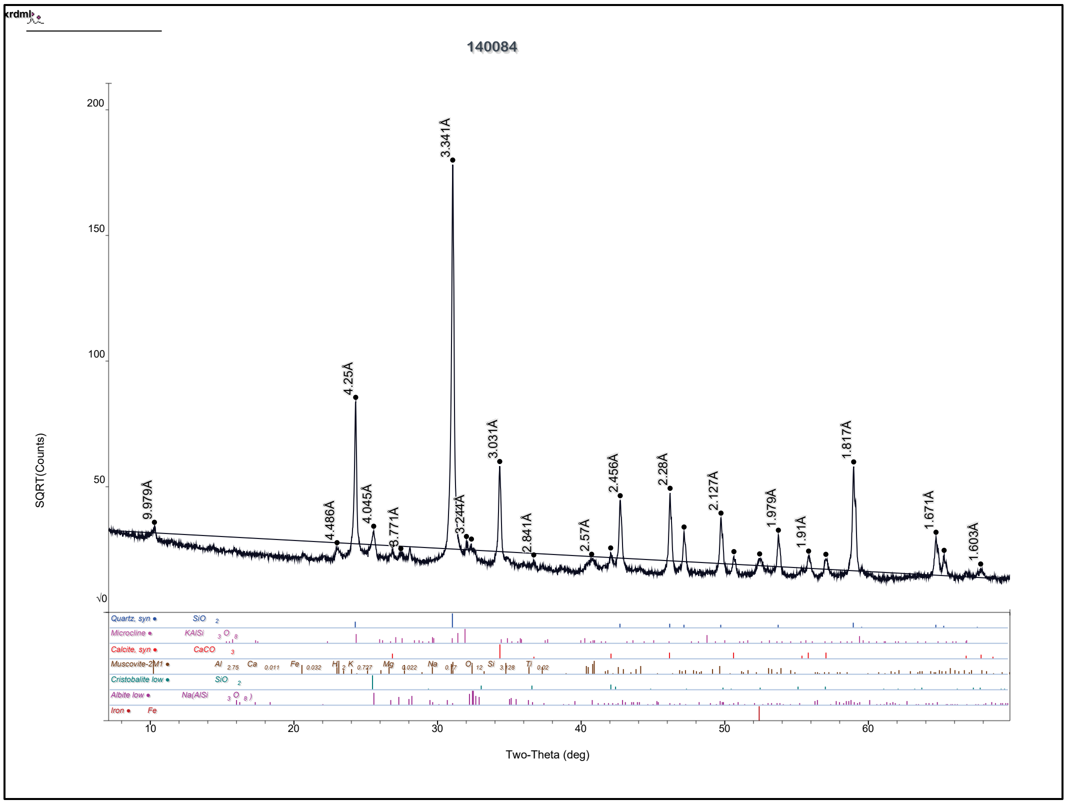
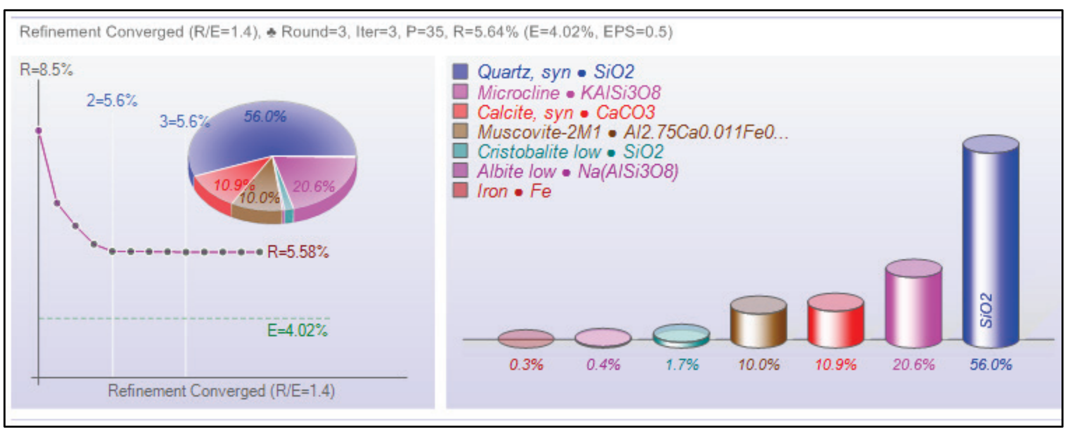


Figure B54. Whole pattern fit with mineral percentages Nevada Coarse Aggregate Sample (CMB No. 140084).



B.19 Nevada Fine Aggregate Sample (CMB No. 140085)

The Nevada fine aggregate sample (CMB No. 140085) was classified as a complex gravel. The color of the particles varied from white (N9), very light gray (N8), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4), grayish pink (5R 8/2), grayish orange pink (10R 8/2), moderate orange pink (10R 7/4), light red (5R 6/6), pale reddish brown (10R 5/4), grayish red (10R 4/2), dark reddish brown (10R 3/4), very pale orange (10YR 8/2), grayish orange (10YR 7/4), dark yellowish orange (10YR 6/6), moderate yellowish brown (10YR 5/4) in color (Figure B55). Analysis of the XRD patterns (Figure B56) indicated that the predominant phase in the material was quartz. Figure B57 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (27.3%), calcite ($CaCO_3$) (27.1%), anorthite ($CaAl_2Si_3O_8$) (25.8%), orthoclase ($KAlSi_3O_8$) (8.9%), cristobalite (SiO_2) (6.5%), stilbite ($Na_{1.6}Ca_4(Al_9Si_{27}O_{72}(H_2O)_{31.52})$ (2.4%), chlorite/clinochlore ($(Mg, Fe)_3(Si,Al)_4O_{10}\text{-}(OH)_2 \cdot (Mg,Fe)_3(OH)_6$) (1.2%), and biotite ($K_{0.78}Na_{0.22}Mg_{1.63}Fe_{0.85}Ti_{0.33}Al_{1.55}Si_{2.84}O_{11}(OH)$) (0.9%) were identified for this sample.

Figure B55. Nevada Fine Aggregate Sample (CMB No. 140085).

Figure B56. X-ray pattern for Nevada Fine Aggregate Sample (CMB No. 140085).

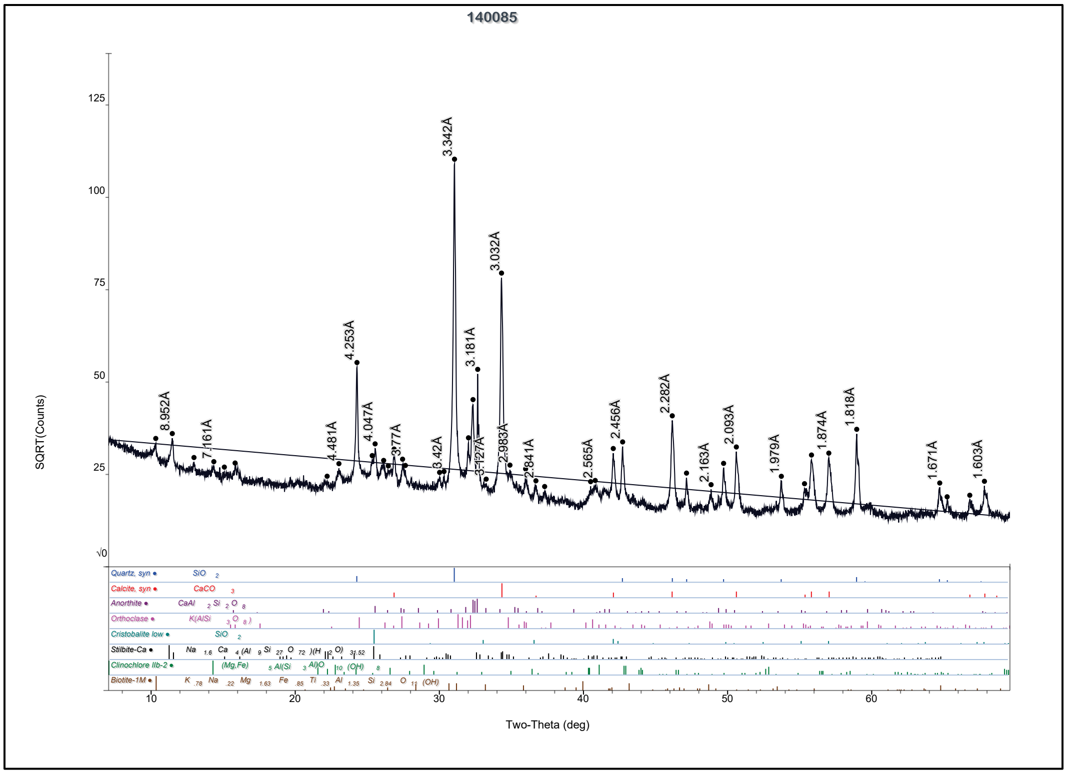
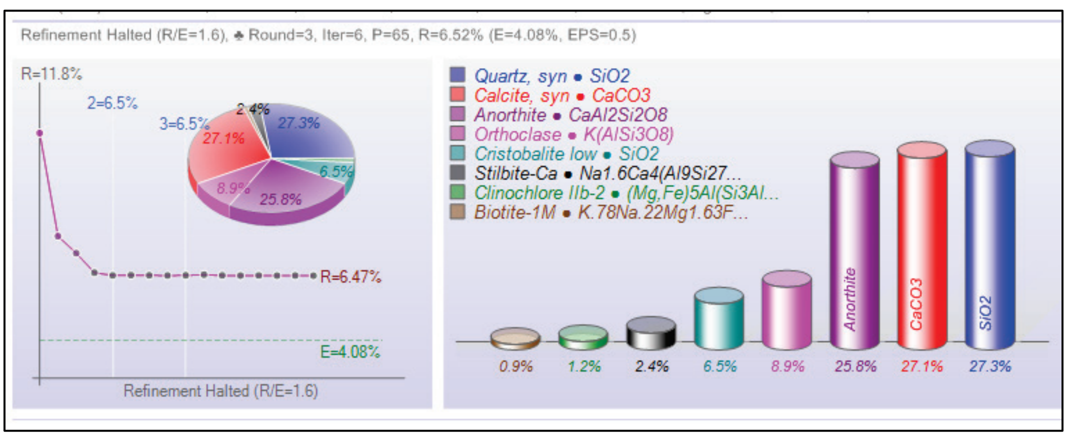


Figure B57. Whole pattern fit with mineral percentages Nevada Fine Aggregate Sample (CMB No. 140085).



B.20 Wabasha, MN, Coarse Aggregate Sample (CMB No. 150046)

The Wabasha, MN, coarse aggregate sample (CMB No. 150046) was classified a complex gravel with intermediate to felsic composition aggregates. The color of the particles varied from moderate orange pink (5YR 8/4), very pale orange (10YR 8/2), grayish orange (10YR 7/4), moderate red (5R 4/6), grayish brown (5Y 3/2), dusky yellowish brown

(10YR 2/2), grayish orange pink (10R 8/2), moderate orange pink (10R 7/4), moderate reddish orange (10R 6/6), grayish red (10R 4/2), pale reddish brown (10R 5/4), moderate reddish brown (10R 4/6), dark reddish brown (10R 3/4), very dusky red (10R 2/2), moderate brown (5YR 3/4), light bluish gray (5B 7/1), medium bluish gray (5B 5/1), yellowish gray (5Y 8/1), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4), dark gray (N3) in color (Figure B58). Analysis of the XRD patterns (Figure B59) indicated that the predominant phase in the material was quartz. Figure B60 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (25.4%), chlorite/clinochlore ($(Mg, Fe)_3(Si,Al)_4O_{10}(OH)_2 \cdot (Mg,Fe)_3(OH)_6$) (18.5%), albite ($Na(Al_3O_8)$) (15.2%) orthoclase ($KAlSi_3O_8$) (12.3%), actinolite ($Ca_2(Mg,Fe^{2+})_5Si_8O_{22}(OH)_2$) (11.3%), dolomite ($CaMg(CO_3)_2$) (8.3%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (6.7%) and calcite ($CaCO_3$) (2.4%) were identified for this sample.

Figure B58. Wabasha, MN, Coarse Aggregate Sample (CMB No. 150046).

Figure B59. X-ray pattern for Wabasha, MN, Coarse Aggregate Sample (CMB No. 150046).

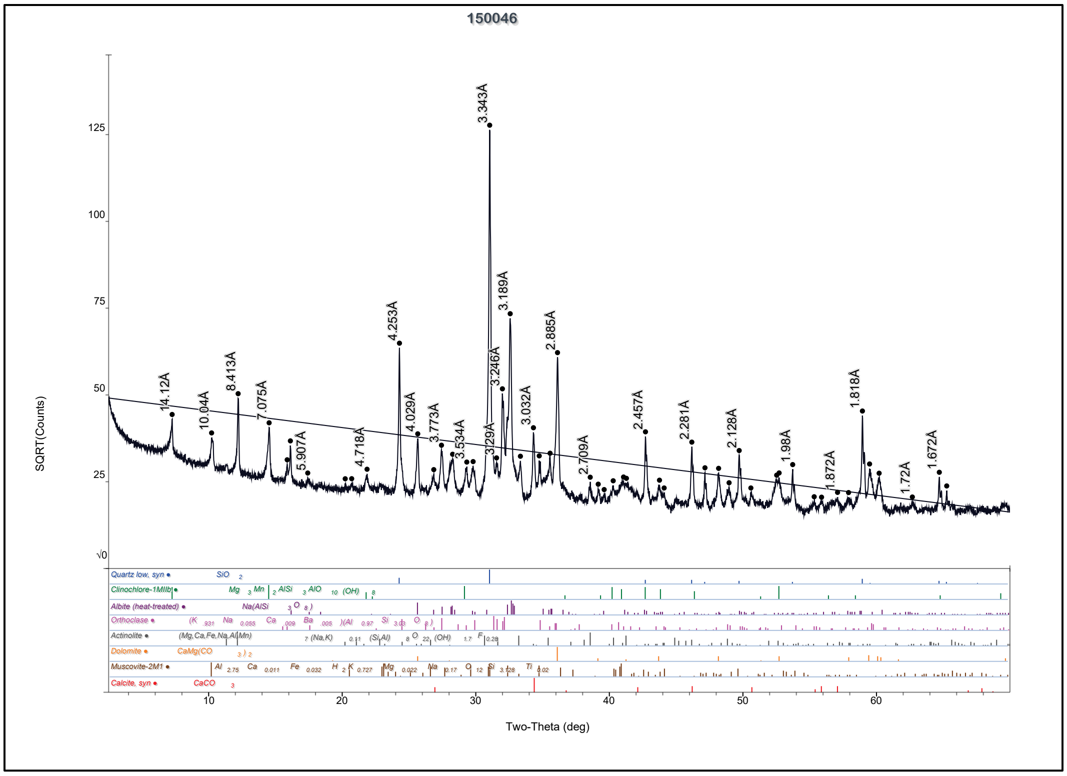
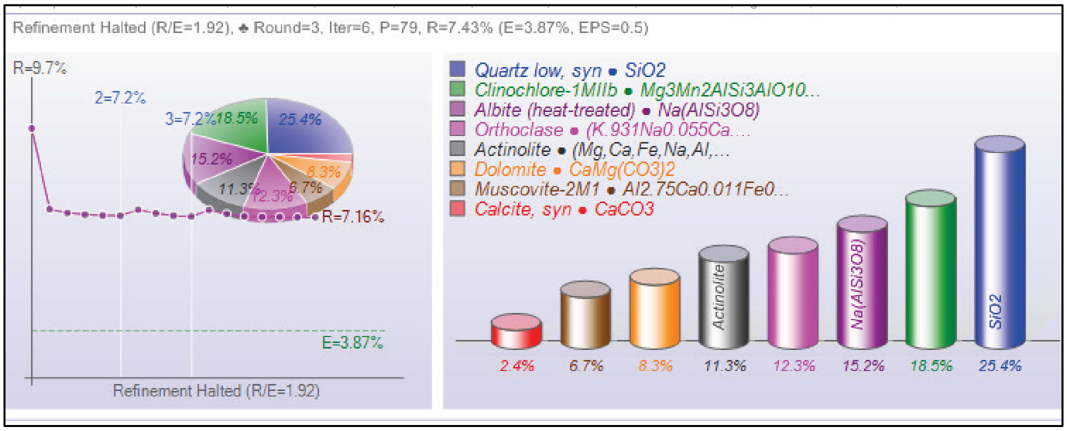


Figure B60. Whole pattern fit with mineral percentages Wabasha, MN, Coarse Aggregate Sample (CMB No. 150046).



B.21 Wabasha, MN, Fine Aggregate Sample (CMB No. 150047)

Wabasha, MN, fine aggregate sample (CMB No. 150047) was classified a complex gravel with intermediate to felsic composition aggregates. The color of the particles varied from moderate orange pink (5YR 8/4), very pale orange (10YR 8/2), grayish orange (10YR 7/4), moderate red (5R 4/6), grayish brown (5Y 3/2), dusky yellowish brown (10YR 2/2), grayish orange pink (10R 8/2), moderate orange pink (10R 7/4), moderate reddish orange (10R 6/6), grayish red (10R 4/2), pale reddish brown (10R 5/4), moderate reddish brown (10R 4/6), dark reddish brown (10R 3/4), very dusky red (10R 2/2), moderate brown (5YR 3/4), light bluish gray (5B 7/1), medium bluish gray (5B 5/1), yellowish gray (5Y 8/1), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4), dark gray (N3) , in color (Figure B61). Analysis of the XRD patterns (Figure B62) indicated that the predominant phase in the material was quartz. Figure B63 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (38.8%), orthoclase ($KAlSi_3O_8$) (26.0%), albite ($Na(Al_3O_8)$) (10.8%), clinochlore ($(Mg,Fe)_3(Si,Al)_4O_{10}(OH)_2\cdot(Mg,Fe)_3(OH)_6$) (8.7%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (5.2%), dolomite ($Ca,Mg(CO_3)_2$) (4.3%), actinolite ($Ca_2(Mg,Fe_2+)5Si_8O_{22}(OH)_2$) (3.2%), and calcite ($CaCO_3$) (3.1%) were identified for this sample.

Figure B61. Wabasha, MN, Fine Aggregate Sample (CMB No. 150047).

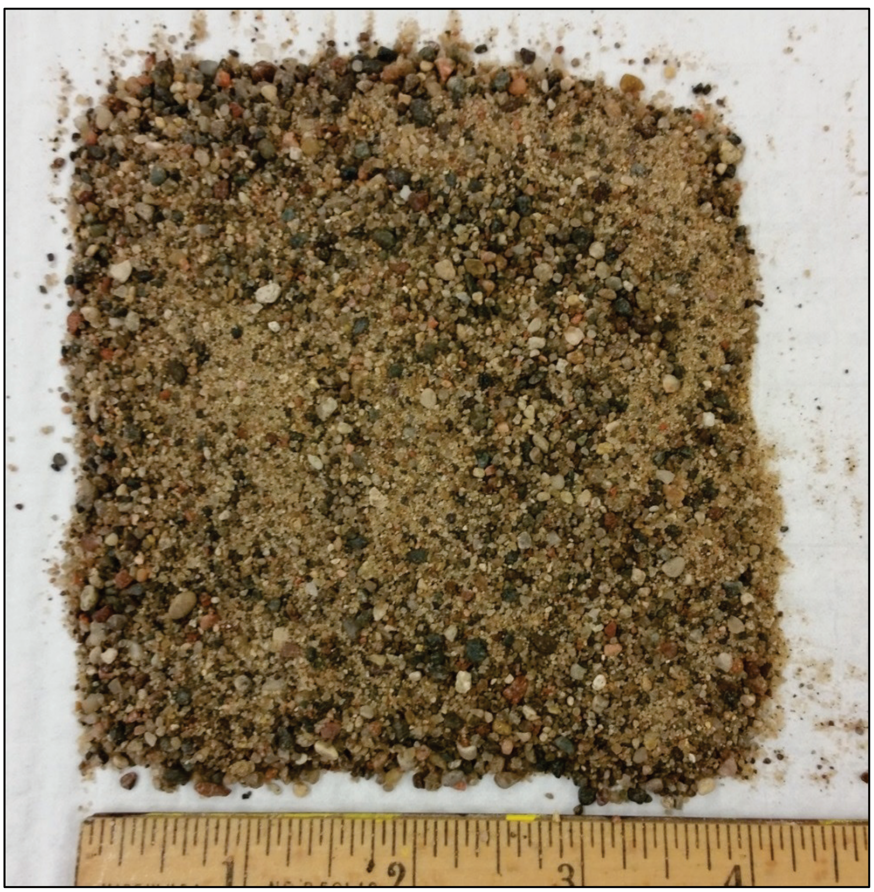


Figure B62. X-ray pattern for Wabasha, MN, Fine Aggregate Sample (CMB No. 150047).

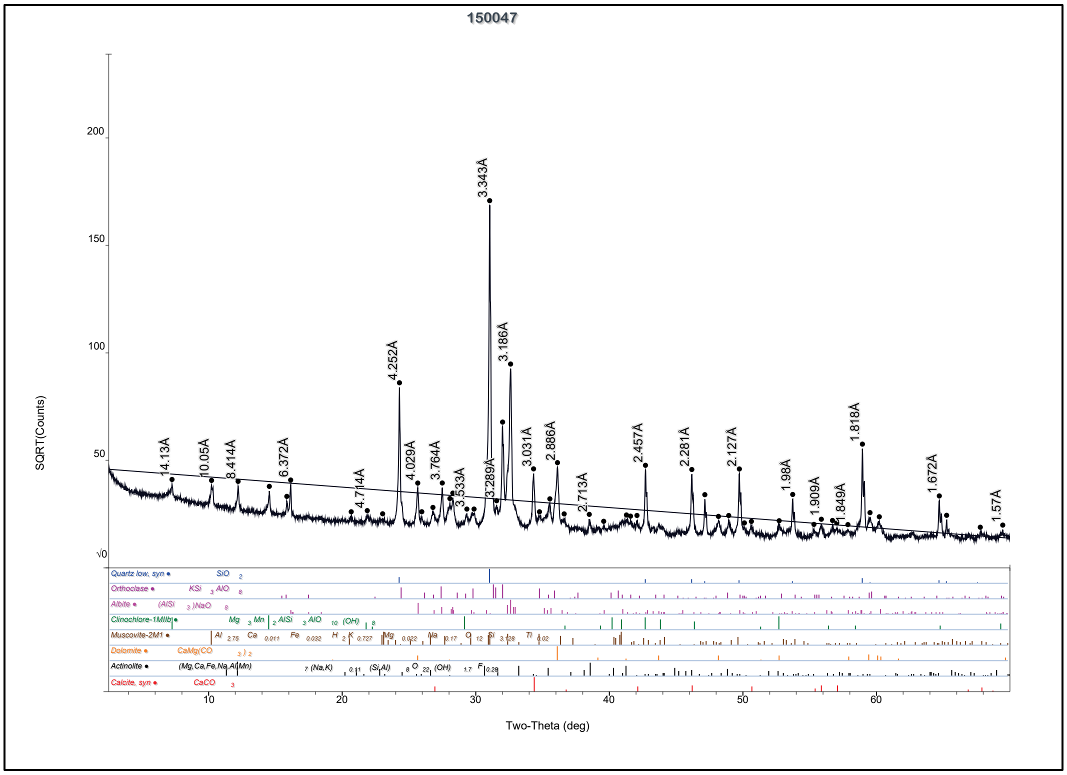
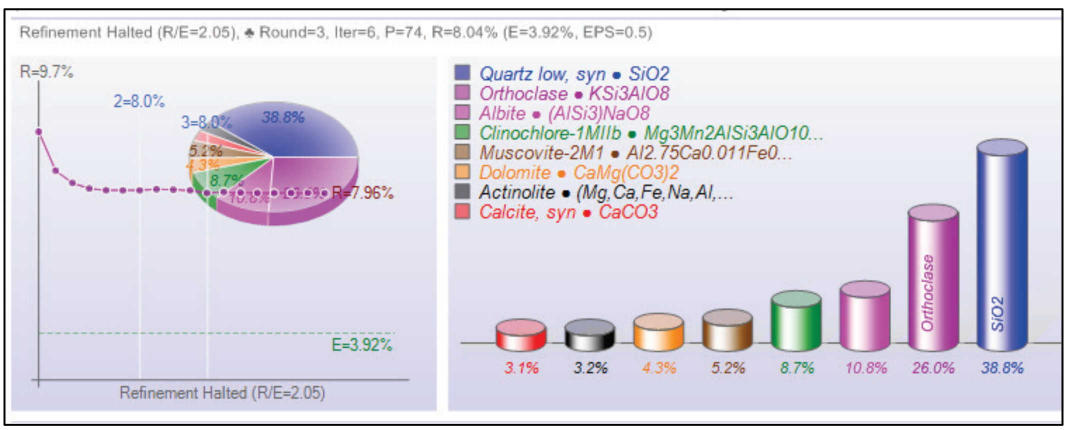


Figure B63. Whole pattern fit with mineral percentages Wabasha, MN, Fine Aggregate Sample (CMB No. 150047).



B.22 Adairsville, Atlanta, GA, Fine Aggregate Sample (CMB No. 150093)

The Adairsville, Atlanta, GA, fine aggregate sample (CMB No. 150093) was classified as a dolostone. The color of the particles varied from white (N9), very light gray (N8), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4), dark gray (N3) in color (Figure B64). Analysis of the XRD patterns (Figure B65) indicated that the predominant phase in the material was dolomite. Figure B66 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($Ca,Mg(CO_3)_2$) (98.9%), orthoclase ($KAlSi_3O_8$) (0.7%), quartz (SiO_2) (0.4%), and calcite ($CaCO_3$) (0.1%) were identified for this sample.

Figure B64. Adairsville, Atlanta, GA, Fine Aggregate Sample (CMB No. 150093).

Figure B65. X-ray pattern for Adairsville, Atlanta, GA, Fine Aggregate Sample (CMB No. 150093).

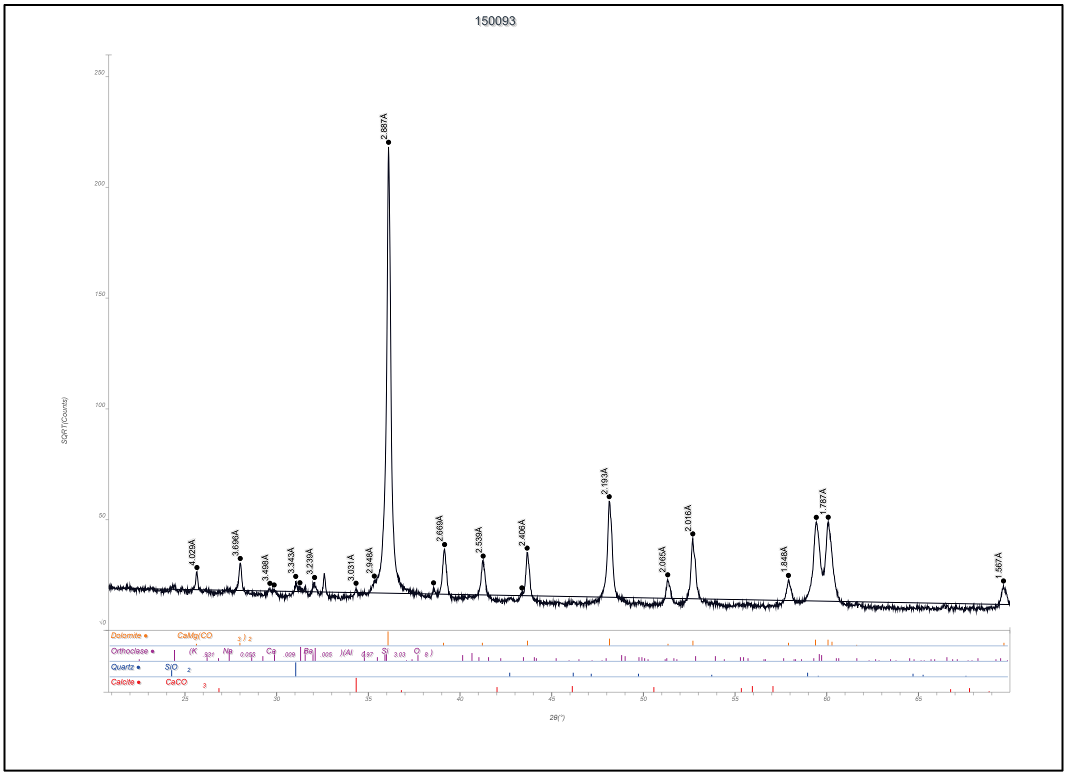
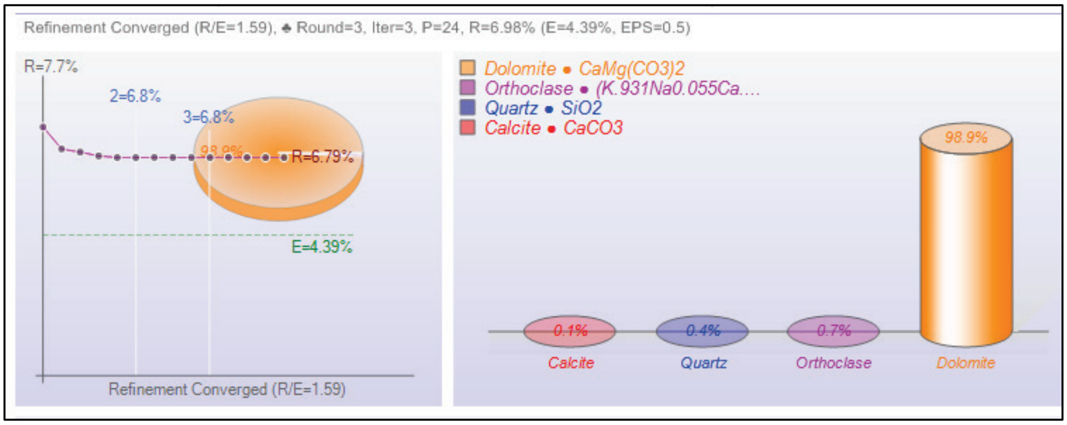


Figure B66. Whole pattern fit with mineral percentages Adairsville, Atlanta, GA, Fine Aggregate Sample (CMB No. 150093).



B.23 Vulcan, Atlanta, GA, Fine Aggregate Sample (CMB No. 150094)

The Vulcan, Atlanta, GA, fine aggregate sample (CMB No. 150094) was classified as a feldspar-rich gravel. The color of the particles varied from pale yellowish brown (10YR 6/2), dark yellowish orange (10YR 6/6), pale yellowish orange (10YR 8/6), yellowish gray (5Y 8/1), white (N9), very light gray (N8), grayish black (N2) and black (N1) in color (Figure B67).

Analysis of the XRD patterns (Figure B68) indicated that the predominant phase in the material was quartz. Figure B69 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Anorthite ($CaAl_2Si_3O_8$) (31.0%), albite ($Na(Al_3O_8)$) (22.9%), quartz (SiO_2) (19.4%), microcline ($KAlSi_3O_8$) (17.9%), biotite ($KFeMg_2(Al_3O_{10})(OH)_2$) (8.5%), and coesite (SiO2) (0.2%) were identified for this sample.

Figure B67. Vulcan, Atlanta, GA, Fine Aggregate Sample (CMB No. 150094).

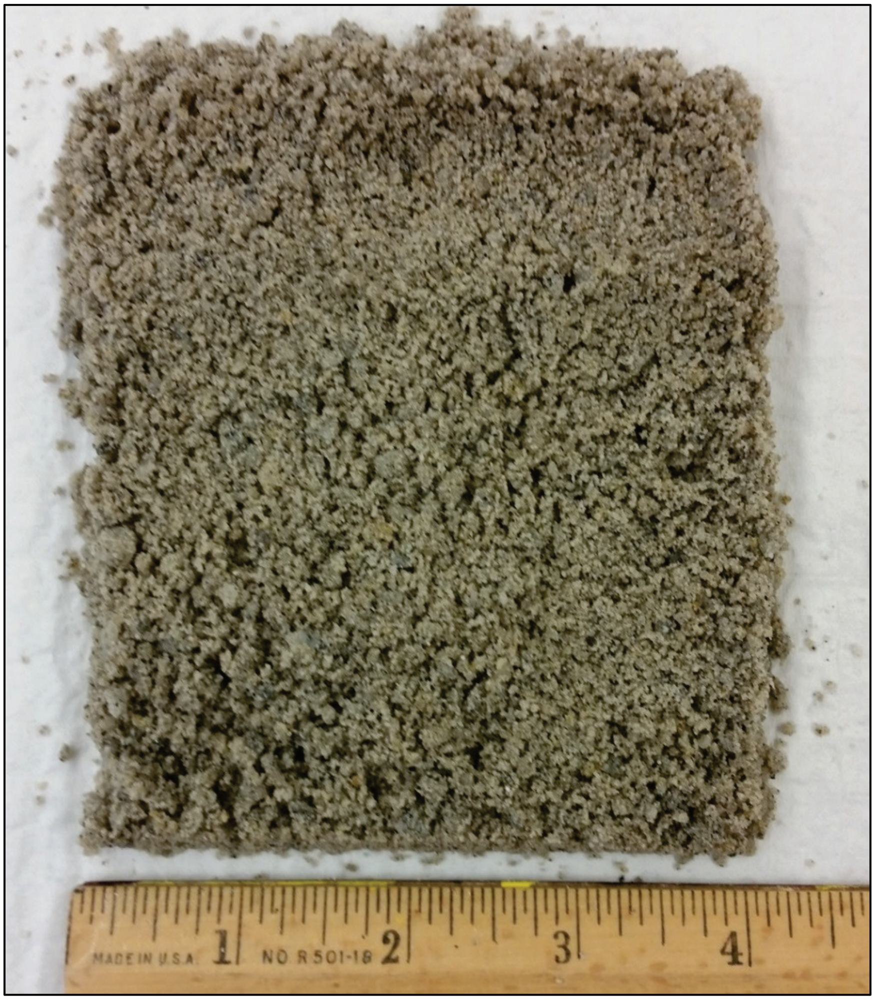


Figure B68. X-ray pattern for Vulcan, Atlanta, GA, Fine Aggregate Sample (CMB No. 150094).

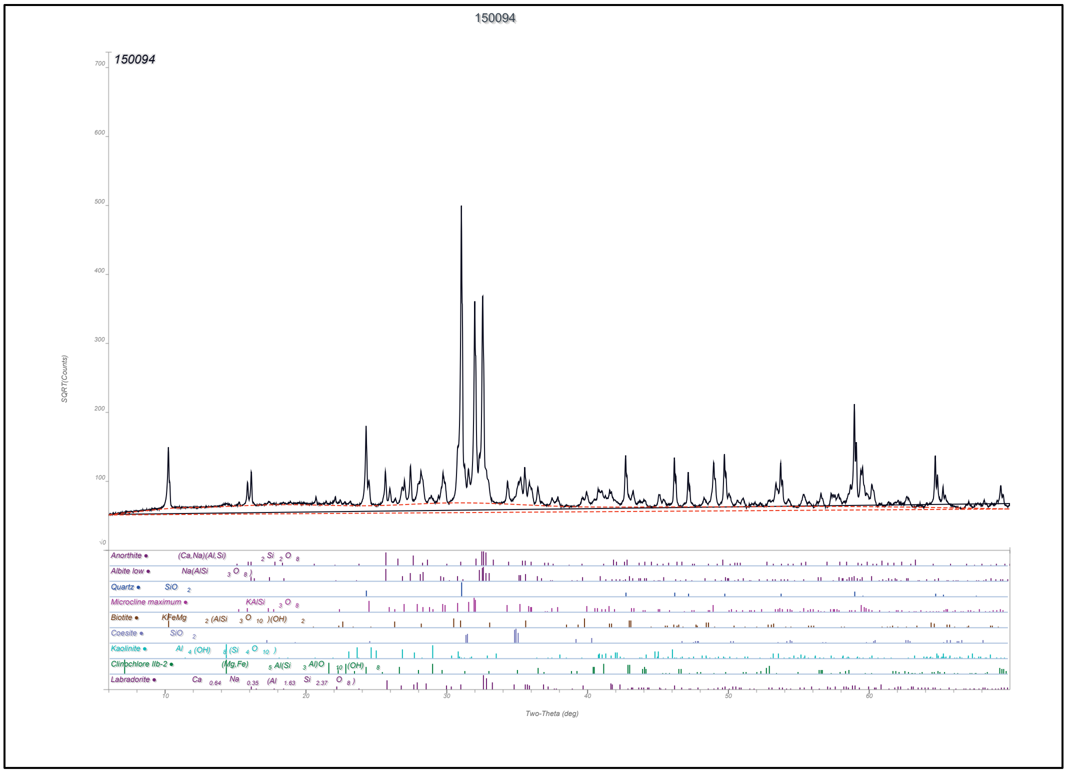
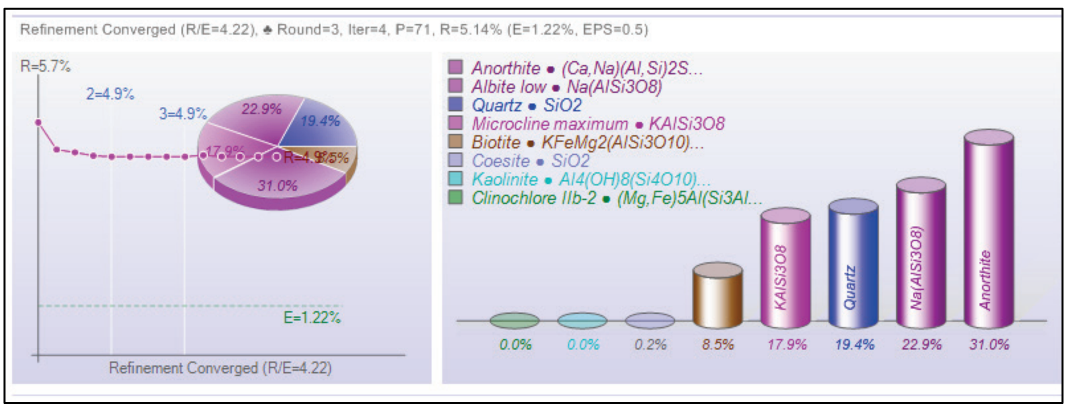


Figure B69. Whole pattern fit with mineral percentages Vulcan, Atlanta, GA, Fine Aggregate Sample (CMB No. 150094).



B.24 Vulcan, Atlanta, GA, Coarse Aggregate Sample (CMB No. 150095)

The Vulcan, Atlanta, GA, coarse aggregate sample (CMB No. 150095) was classified as intermediate to felsic crushed aggregate. The color of the particles varied from moderate reddish orange (10R 6/6), moderate orange pink (5YR 8/4), very pale orange (10YR 8/2), pale yellowish orange (10YR 8/6), dark yellowish orange (10YR 6/6), white (N9), medium dark gray (N4) and dark gray (N3) in color (Figure B70). Analysis of the XRD patterns (Figure B71) indicated that the predominant phase in the material was anorthite. Figure B72 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Anorthite ($CaAl_2Si_3O_8$) (44.8%), microcline ($KAlSi_3O_8$) (27.9%), quartz (SiO_2) (20.1%), and biotite ($KFeMg_2(Al_3O_{10})(OH)_2$) (7.2%) were identified for this sample.

Figure B70. Vulcan, Atlanta, GA, Coarse Aggregate Sample (CMB No. 150095).

Figure B71. X-ray pattern for Vulcan, Atlanta, GA, Coarse Aggregate Sample (CMB No. 150095).

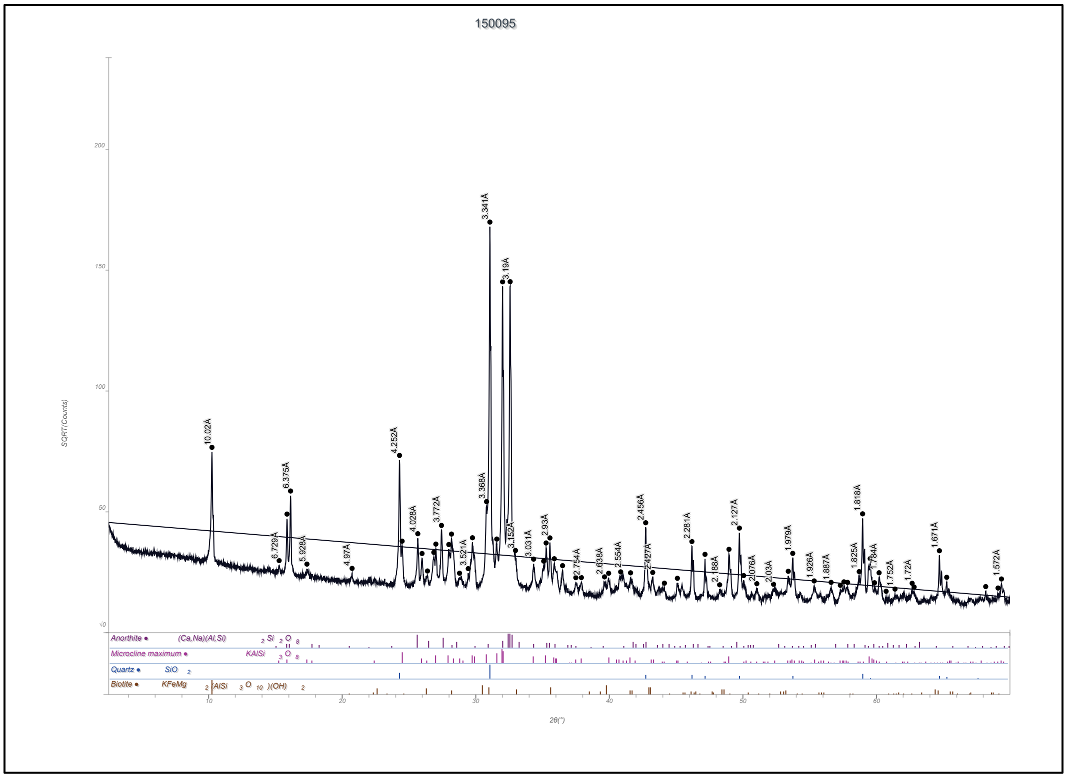
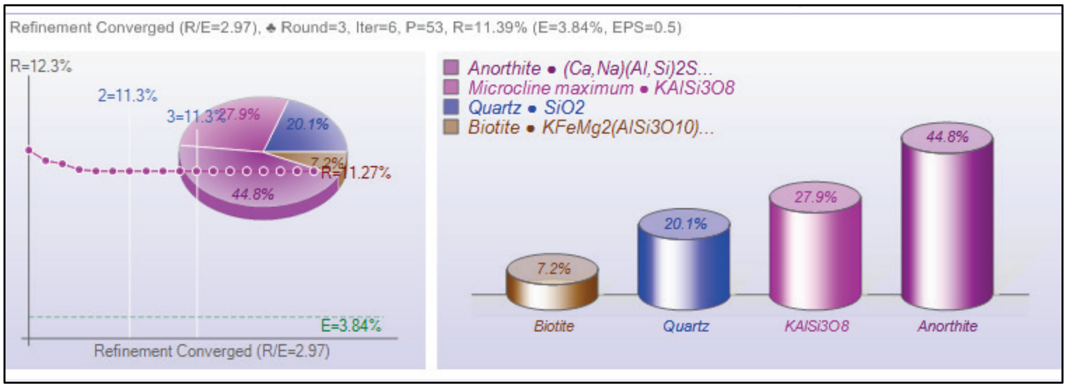


Figure B72. Whole pattern fit with mineral percentages Vulcan, Atlanta, GA, Coarse Aggregate Sample (CMB No. 150095).



B.25 LG Everist Dell Rapids, SD, Fine Aggregate Sample (CMB No. 150096)

The LG Everist Dell Rapids, SD, fine aggregate sample (CMB No. 150096) was classified as quartz sand. The color of the particles varied from grayish pink (5R 8/2), pale red (5R 6/2), pale red (10R 6/2), moderate red (5R 5/4), grayish red (10R 4/2), pale reddish brown (10R 5/4), and grayish red (10R 4/2) in color (Figure B73). Analysis of the XRD patterns (Figure B74) indicated that the predominant phase in the material was quartz. Figure B75 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (95.3%), kaolinite ($Al_2Si_2O_5OH_4$) (4.5%), and trace amounts of cristobalite (SiO_2) and tridymite (SiO_2) were identified for this sample.

Figure B73. LG Everist Dell Rapids, SD, Fine Aggregate Sample (CMB No. 150096).

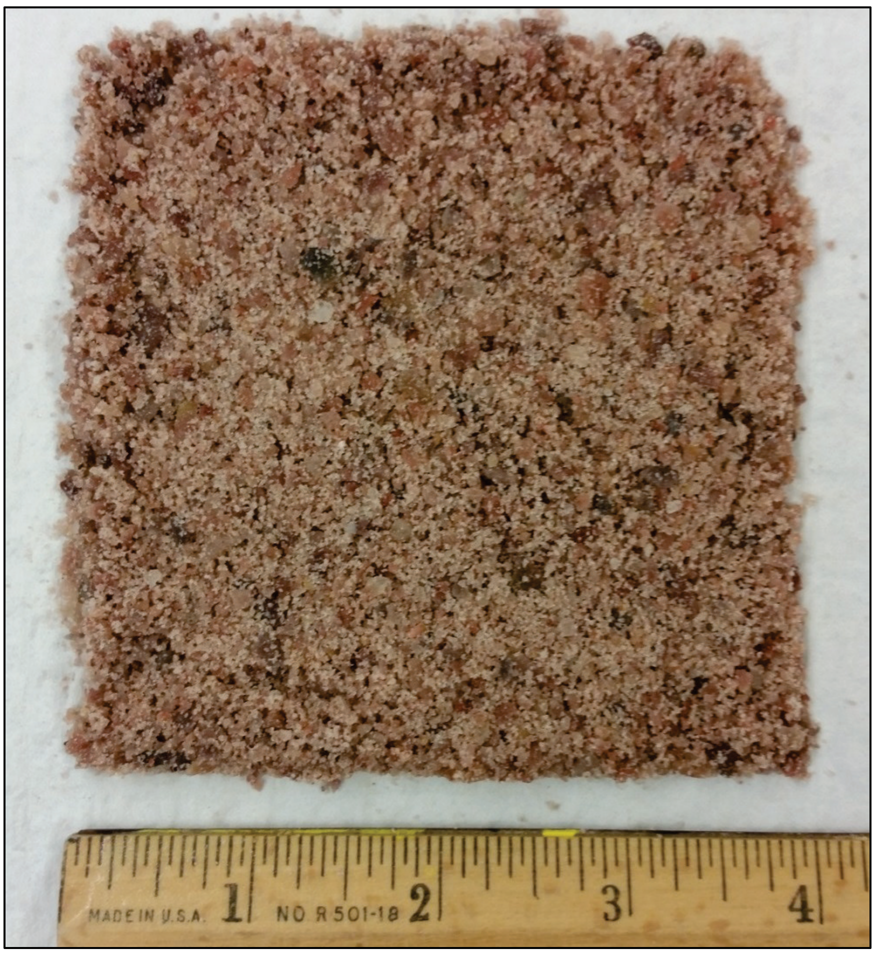


Figure B74. X-ray pattern for LG Everist Dell Rapids, SD, Fine Aggregate Sample (CMB No. 150096).

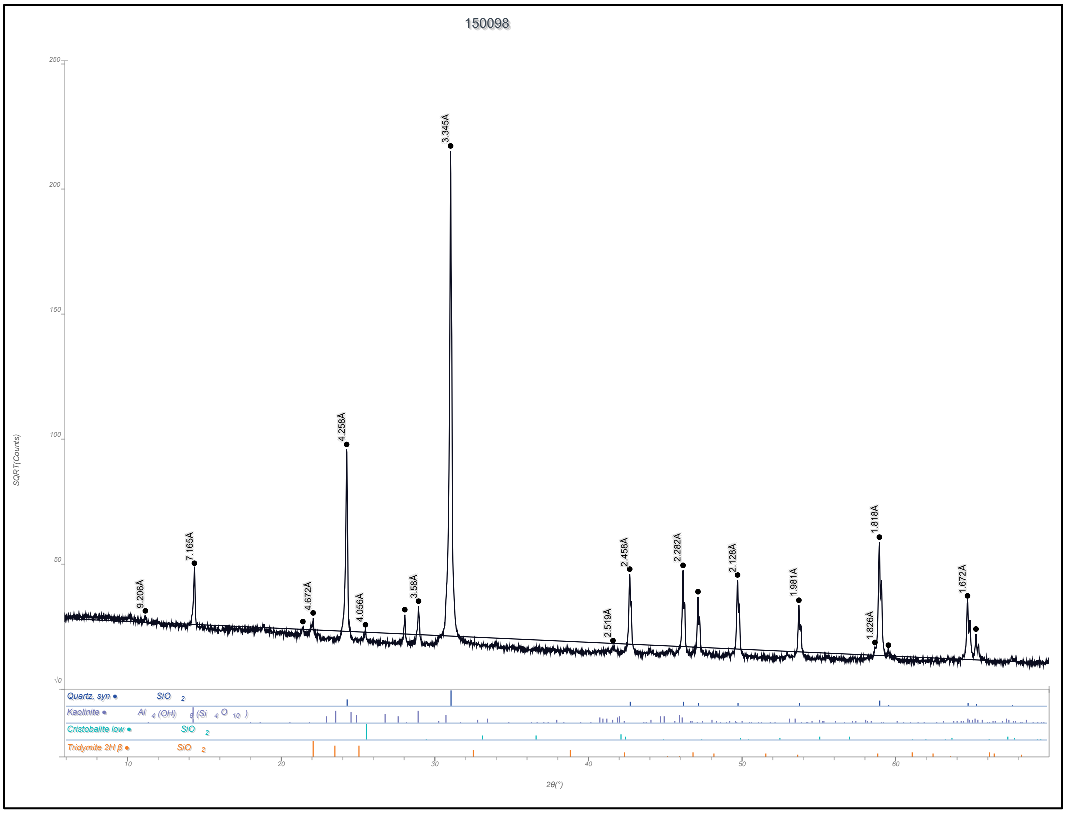
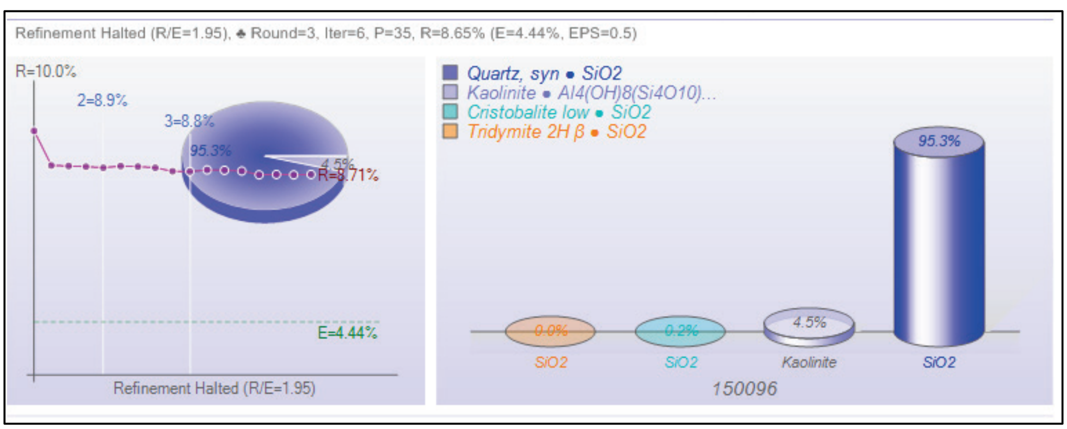


Figure B75. Whole pattern fit with mineral percentages LG Everist Dell Rapids, SD, Fine Aggregate Sample (CMB No. 150096).



B.26 Knife River, Cheyenne, WY, Fine Aggregate Sample (CMB No. 150097)

Knife River, Cheyenne, WY, fine aggregate sample (CMB No. 150097) was classified a feldspar-rich sand. The color of the particles varied from moderate pink (5R 7/4), moderate orange pink (10R 7/4), moderate reddish orange (10R 6/6), moderate reddish brown (10R 4/6), pale reddish brown (10R 5/4), very pale orange (10YR 8/2), pale yellowish orange (10YR 8/6), dark yellowish orange (10YR 6/6), light brown (5YR 5/6), pale olive (10Y 6/2), dark greenish gray (5GY 4/1), white (N9), very light gray (N8), dark gray (N3), and grayish black (N2) in color (Figure B76). Analysis of the XRD patterns (Figure B77) indicated that the predominant phase in the material was anorthite. Figure B78 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Anorthite ($CaAl_2Si_3O_8$) (27.1%), microcline ($KAlSi_3O_8$) (24.9%), albite ($Na(Al_3O_8)$) (24.8%), Quartz (SiO_2) (19.1%), biotite ($KFeMg_2(Al_3O_{10})(OH)_2$) (3.5%), and a trace amount of cordierite ($Mg_2Al_4Si_5O_{18}$) were identified for this sample.

Figure B76. Knife River, Cheyenne, WY, Fine Aggregate Sample (CMB No. 150097).

Figure B77. X-ray pattern for Knife River, Cheyenne, WY, Fine Aggregate Sample (CMB No. 150097).

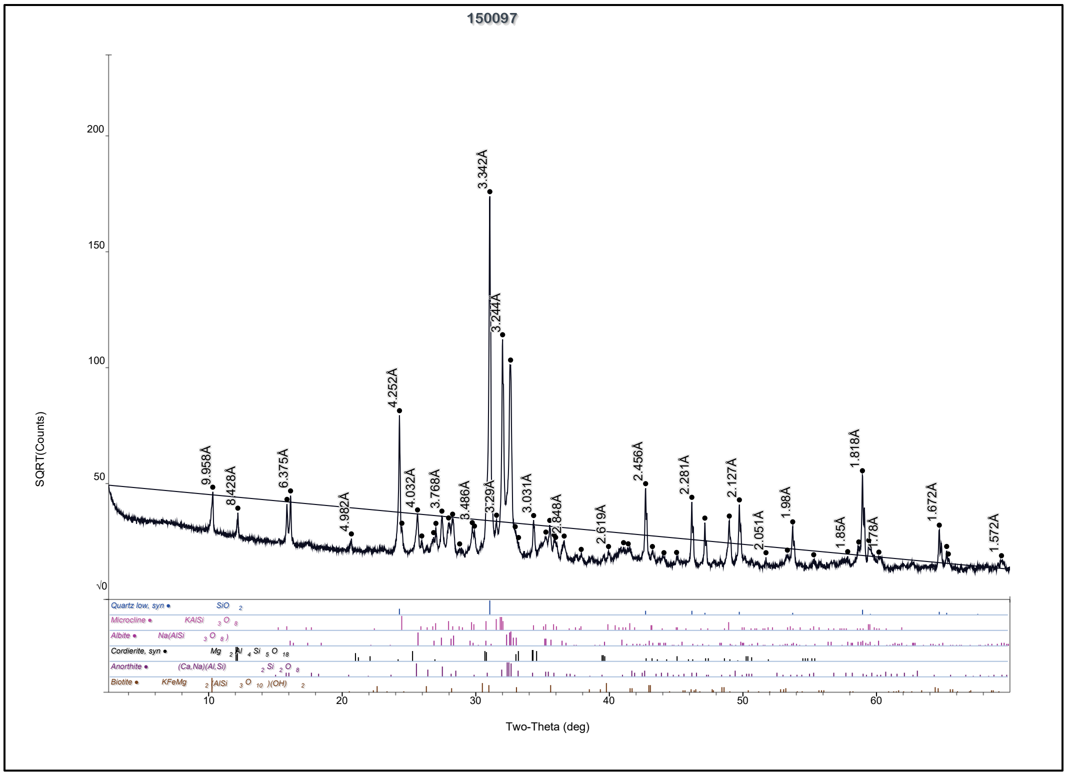
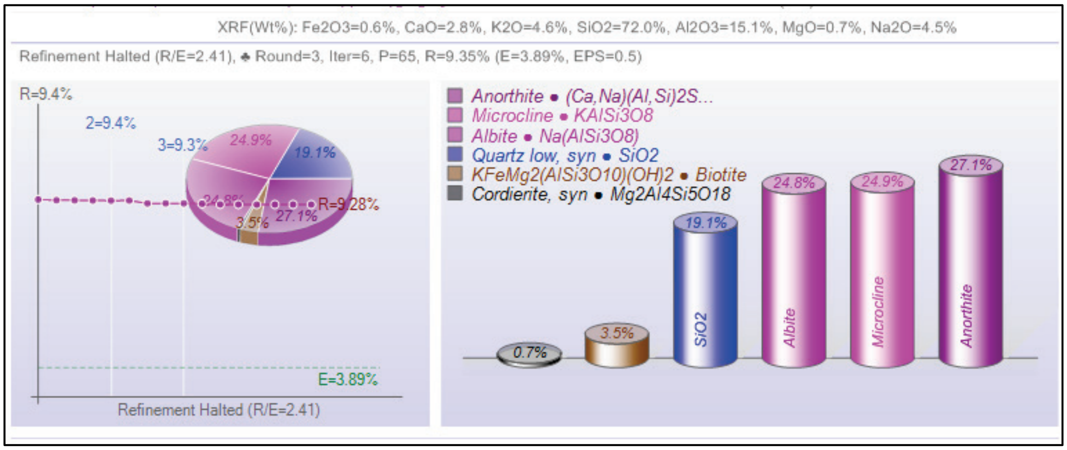


Figure B78. Whole pattern fit with mineral percentages Knife River, Cheyenne, WY, Fine Aggregate Sample (CMB No. 150097).



B.27 Spratt-Canada Coarse Aggregate Sample (CMB No. 150098)

The Spratt-Canada coarse aggregate sample (CMB No. 150098) was classified as a dense limestone. The color of the particles varied from medium dark gray (N4), medium gray (N5), medium light gray (N6), light gray (N7), very light gray (N8) to white (N9) in color (Figure B79). The particles ranged from angular to sub-angular in shape with some quartz inclusions. Analysis of the XRD patterns (Figure B80) indicated that the predominant phase in the material was calcite. The weight percent data obtained by whole pattern fitting using Jade 2010 software are given in Figure B81. Calcite ($CaCO_3$) (76.7%), dolomite ($CaMg(CO_3)_2$) (13.7%), and quartz (SiO2) (9.6%) were identified for this sample.

Figure B79. Spratt-Canada Coarse Aggregate Sample (CMB No. 150098).

Figure B80. X-ray pattern for Spratt-Canada Coarse Aggregate Sample (CMB No. 150098).

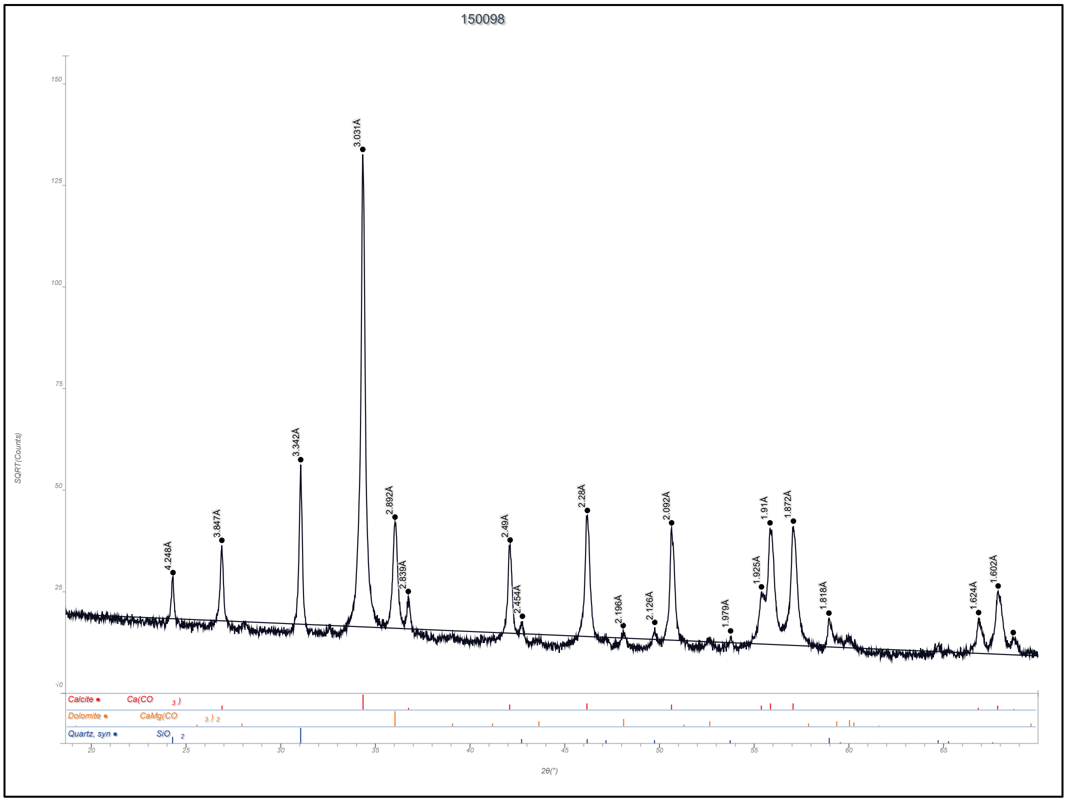
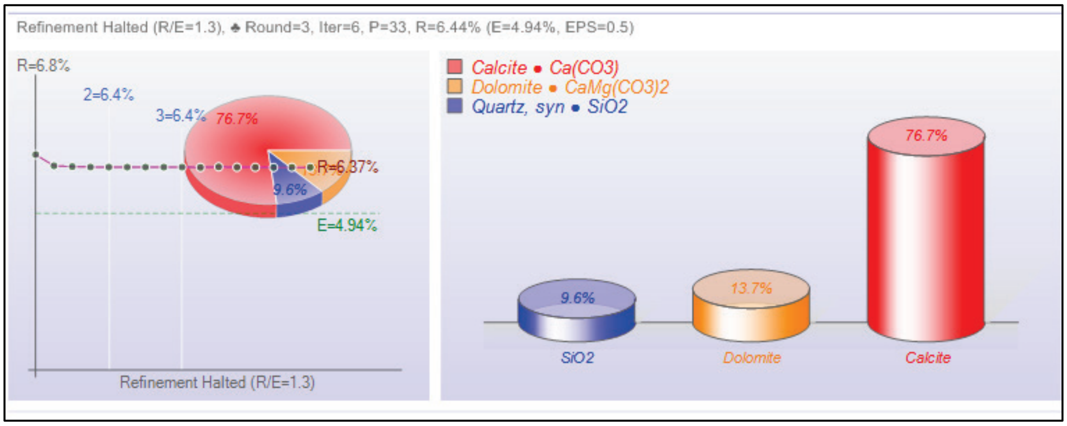


Figure B81. Whole pattern fit with mineral percentages Spratt-Canada Coarse Aggregate Sample (CMB No. 150098).



B.28 Sudbury-Canada Coarse Aggregate Sample (CMB No. 150098.1)

The Sudbury-Canada coarse aggregate sample (CMB No. 150098.1) was classified as a green schist, quartzite, and granite mixture. The color of the particles varied from moderate orange pink (5YR 8/4), pale yellowish orange (10YR 8/6), very pale orange (10YR 8/2), grayish orange (10YR 7/4), light brown (5YR 5/6), dark yellowish brown (10YR 4/2), grayish blue (5B 5/6), light brownish gray (5YR 6/1), greenish gray (5G 6/1), grayish green (5GY 6/1), yellowish gray (5Y 8/1), medium bluish gray (5B 5/1), dark greenish gray (5GY 4/1, 5G 4/1), white (N9), very light gray (N8), light gray (N7), medium light gray (N6), medium gray (N5), medium dark gray (N4) in color (Figure B82). Analysis of the XRD patterns (Figure B83) indicated that the predominant phase in the material was quartz. Figure B84 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (34.3%), chlorite/clinochlore ($(Mg, Fe)_3(Si,Al)_4O_{10}$-$(OH)_2 \cdot (Mg,Fe)_3(OH)_6$) (26.2%), albite ($Na(AlSi_3O_8)$) (16.3%), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) (13.9%), anorthite ($CaAl_2Si_3O_8$) (7.5%), and fluor-riebeckite ($Al_{0.35}Ca_{0.01}FFe_{4.37}HK_{0.29}Li_{0.33}Mn_{0.18}Na_{2.03}O_{23}Si_{7.75}$) were identified for this sample.

Figure B82. Sudbury-Canada Coarse Aggregate Sample (CMB No. 150098.1).

Figure B83. X-ray pattern for Sudbury-Canada Coarse Aggregate Sample (CMB No. 150098.1).

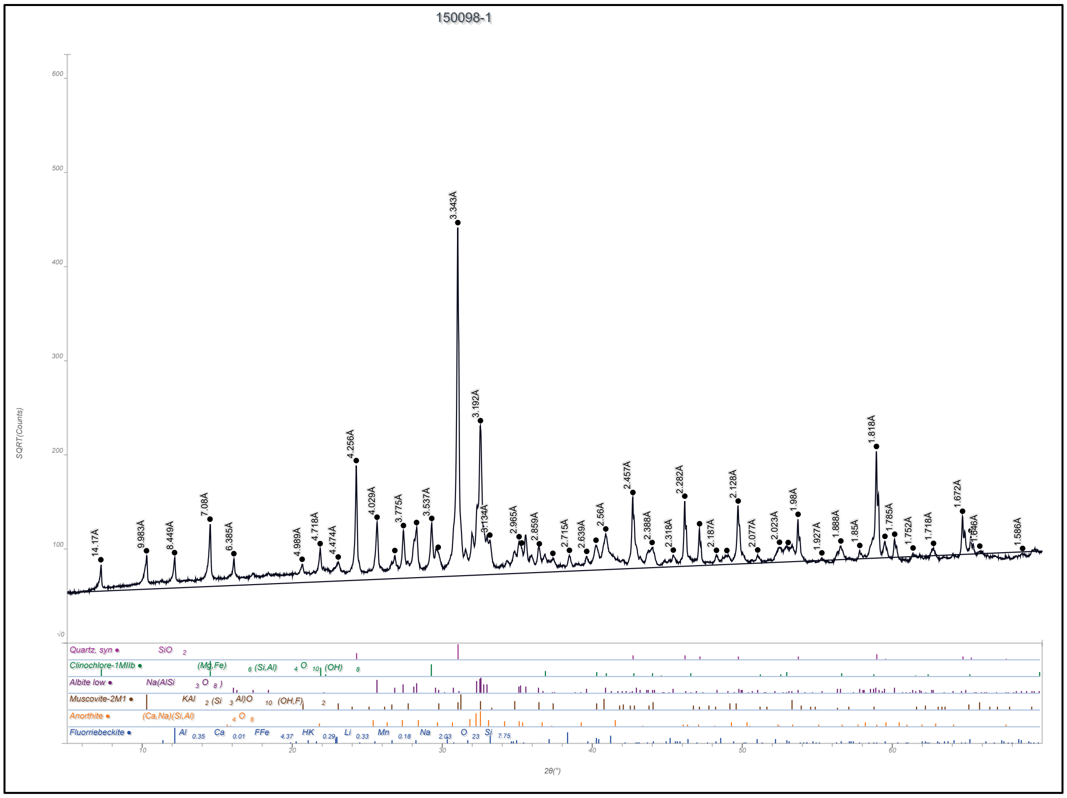
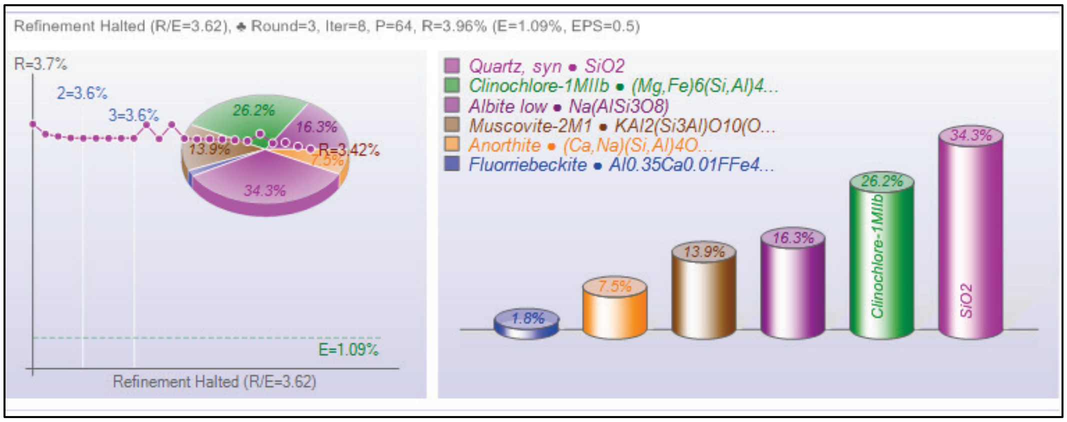


Figure B84. Whole pattern fit with mineral percentages Sudbury-Canada Coarse Aggregate Sample (CMB No. 150098.1).



B.29 Geneva, Utah, Fine Aggregate Sample (CMB No. 150099)

The Geneva, Utah, fine aggregate sample (CMB No. 150099) was classified as quartz sand. The color of the particles varied from very pale orange (10YR 8/2), pale yellowish brown (10YR 6/2), pale yellowish orange (10YR 8/6), grayish orange pink (5YR 7/2), moderate brown (5YR 4/4), white (N9), very light gray (N8) and dark gray (N3) in color (Figure B85). Analysis of the XRD patterns (Figure B86) indicated that the predominant phase in the material was quartz. Figure B87 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (70.1%), dolomite ($CaMg(CO_3)_2$) (23.6%), orthoclase ($KAlSi_3O_8$) (5.6%), and trace amounts of tridymite (SiO_2), cristobalite (SiO_2), and calcite ($CaCO_3$) were identified for this sample.

Figure B85. Geneva, Utah, Fine Aggregate Sample (CMB No. 150099).

Figure B86. X-ray pattern for Geneva, Utah, Fine Aggregate Sample (CMB No. 150099).

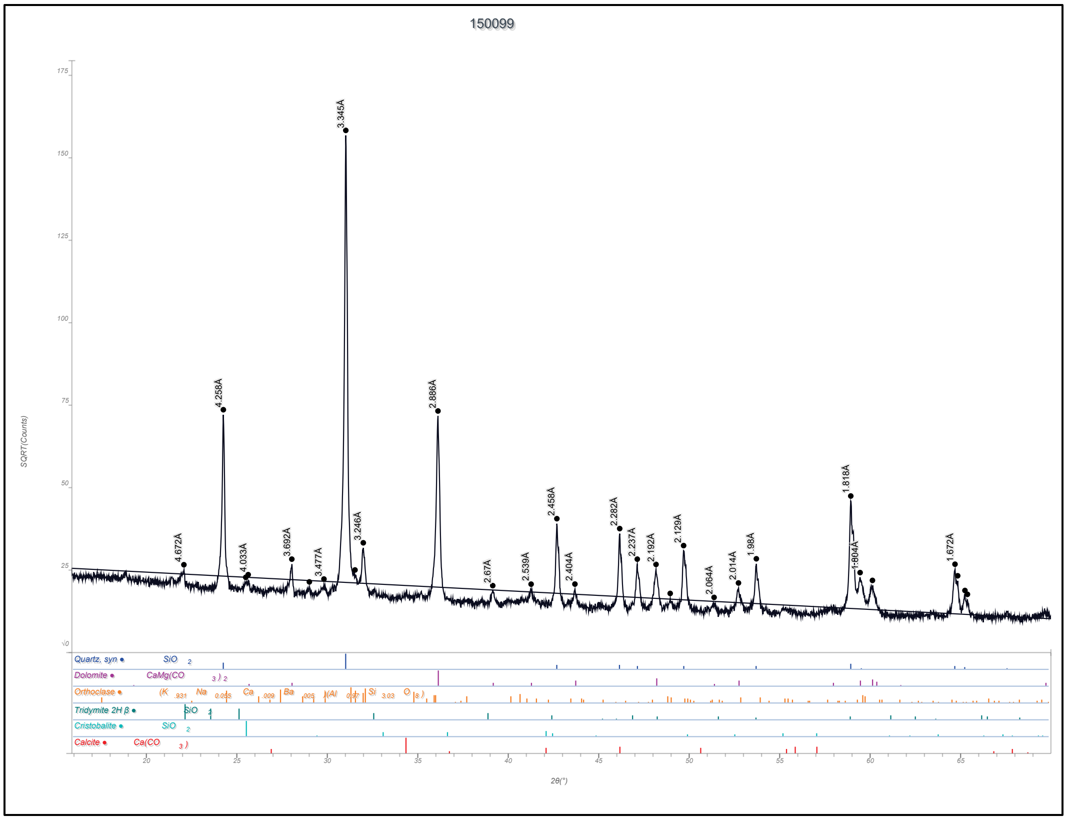
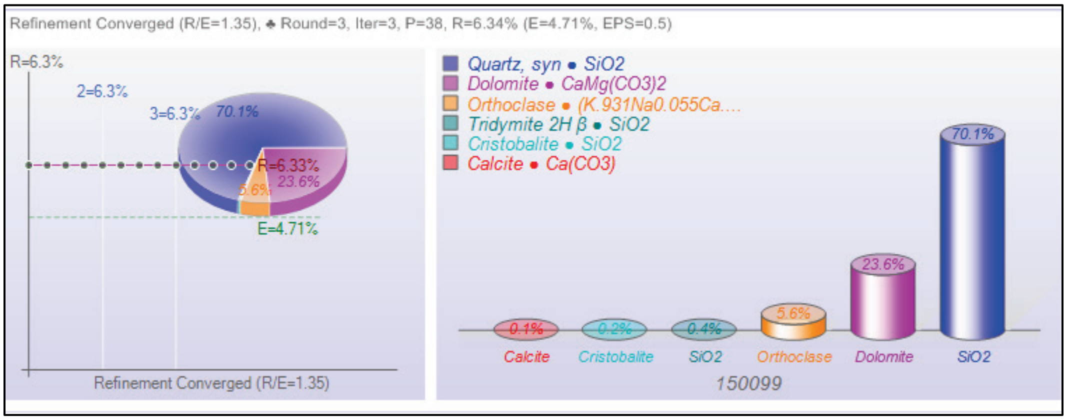


Figure B87. Whole pattern fit with mineral percentages Geneva, Utah, Fine Aggregate Sample (CMB No. 150099).



B.30 Geneva, Utah, Coarse Aggregate Sample (CMB No. 150100)

The Geneva, Utah, coarse aggregate sample (CMB No. 150100) was classified as quartz sandstone. The color of the particles varied from very pale orange (10YR 8/2), pale yellowish brown (10YR 6/2), pale yellowish orange (10YR 8/6), grayish orange pink (5YR 7/2), moderate brown (5YR 4/4), white (N9), very light gray (N8) and dark gray (N3) in color (Figure B88). Analysis of the XRD patterns (Figure B89) indicated that the predominant phase in the material was quartz. Figure B90 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (73.7%), orthoclase ($KAlSi_3O_8$) (14.2%), dolomite ($CaMg(CO_3)_2$) (11.8%), and trace amounts of tridymite (SiO_2) and calcite ($CaCO_3$) were identified for this sample.

Figure B88. Geneva, Utah, Coarse Aggregate Sample (CMB No. 150100).

Figure B89. X-ray pattern for Geneva, Utah, Coarse Aggregate Sample (CMB No. 150100).

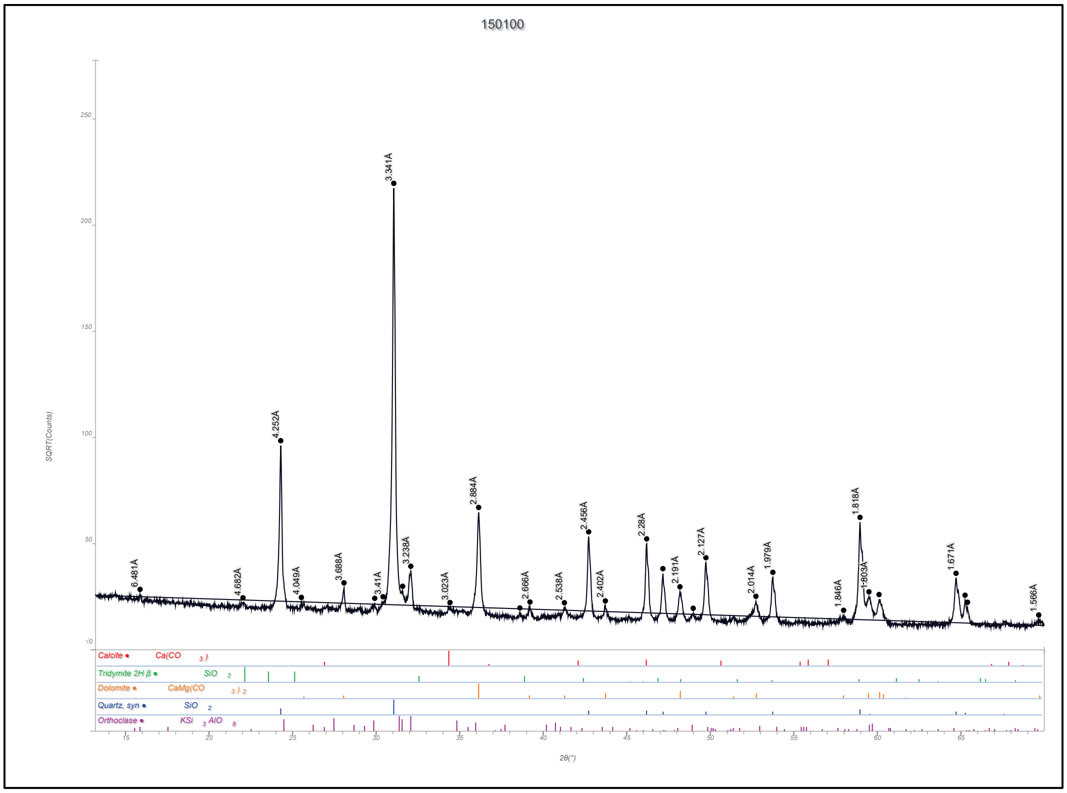
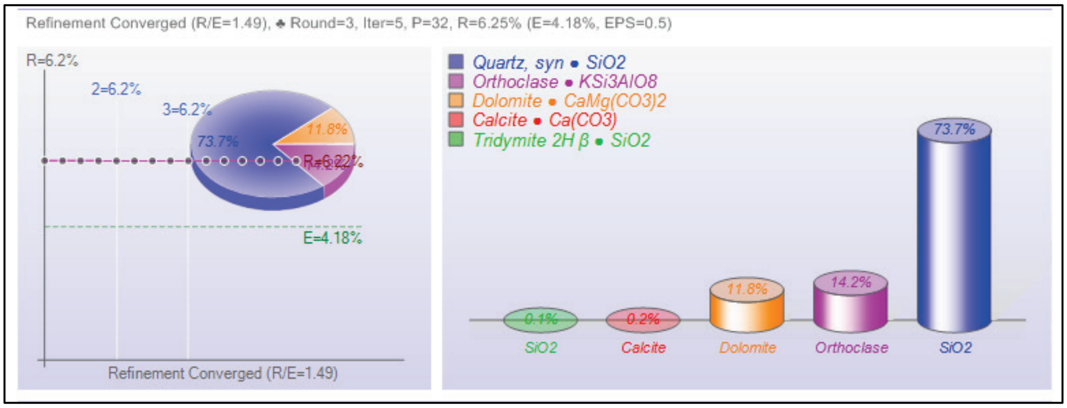


Figure B90. Whole pattern fit with mineral percentages Geneva, Utah, Coarse Aggregate Sample (CMB No. 150100).



B.31 Geneva, Utah, Fine Aggregate Sample (CMB No. 1500101)

The Geneva, Utah, fine aggregate sample (CMB No. 1500101) was classified as quartz-rich sand. The color of the particles varied from moderate red (5R 5/4), dark reddish brown (10R 3/4), very pale orange (10YR 8/2), moderate orange pink (5YR 8/4), grayish orange (10YR 7/4), dark yellowish orange (10YR 6/6), pinkish gray (5YR 8/1), yellowish gray (5Y 8/1), white (N9), very light gray (N8), light gray (N7), medium light gray (N6), and dark gray (N3) in color (Figure B91). Analysis of the XRD patterns (Figure B92) indicated that the predominant phase in the material was quartz. Figure B93 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (77.1%), albite ($Na(Al_3O_8)$) (9.5%), dolomite (CaMg(CO3)2) (7.4%), orthoclase ($KAlSi_3O_8$) (5.2%), and trace amounts of calcite ($CaCO_3$) (0.8%) and muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) were identified for this sample.

Figure B91. Geneva, Utah, Fine Aggregate Sample (CMB No. 1500101).

Figure B92. X-ray pattern for Geneva, Utah, Fine Aggregate Sample (CMB No. 1500101).

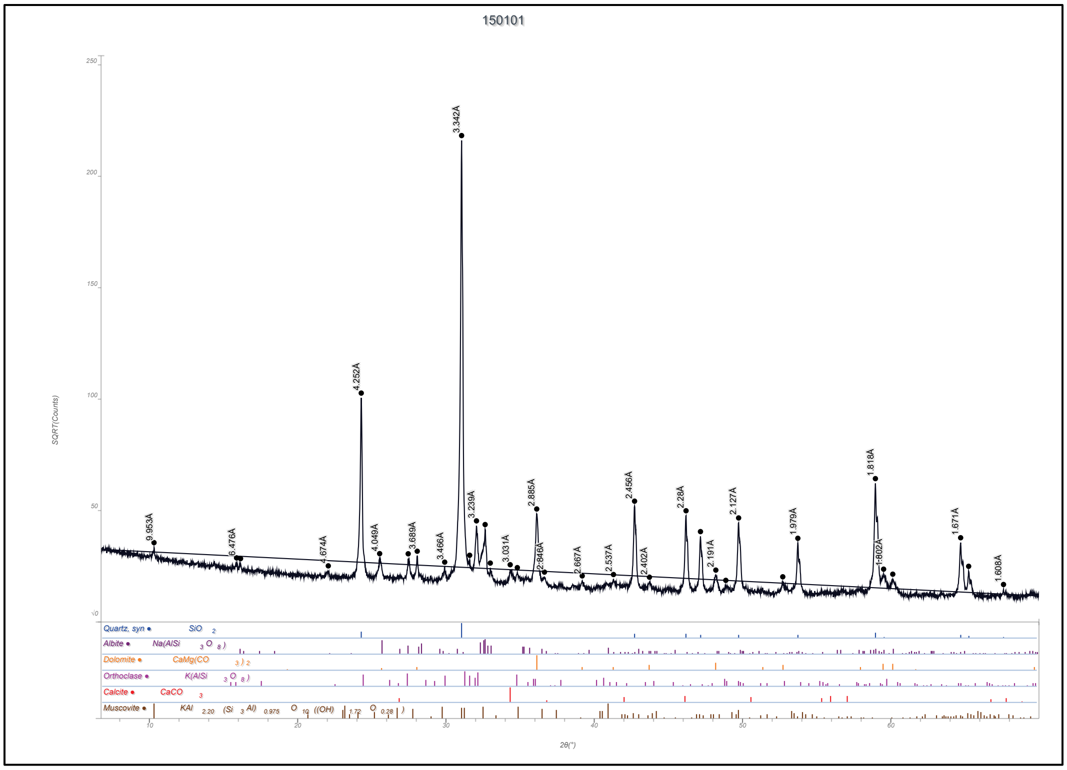
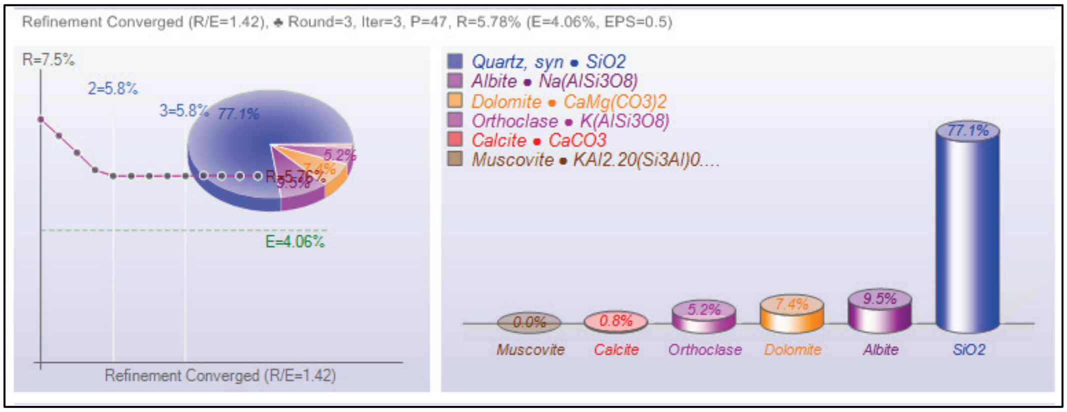


Figure B93. Whole pattern fit with mineral percentages Geneva, Utah, Fine Aggregate Sample (CMB No. 1500101).



B.32 Geneva, Utah, Fine Aggregate Sample (CMB No. 150102)

The Geneva, Utah, fine aggregate sample (CMB No. 1500102) was classified as quartz-rich sand. The color of the particles varied from moderate red (5R 5/4), dark reddish brown (10R 3/4), very pale orange (10YR 8/2), moderate orange pink (5YR 8/4), grayish orange (10YR 7/4), dark yellowish orange (10YR 6/6), pinkish gray (5YR 8/1), yellowish gray (5Y 8/1), light brownish gray (5YR 6/1), white (N9), very light gray (N8), light gray (N7), medium light gray (N6), and dark gray (N3) in color (Figure B94). Analysis of the XRD patterns (Figure B95) indicated that the predominant phase in the material was dolomite. Figure B96 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($CaMg(CO_3)_2$) (39.6%), quartz (SiO_2) (31.6%), calcite ($CaCO_3$) (23.6%), albite ($Na(Al_3O_8)$) (3.9%), and trace amounts of orthoclase ($KAlSi_3O_8$) and muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) were identified for this sample.

Figure B94. Geneva, Utah, Fine Aggregate Sample (CMB No. 150102).

Figure B95. X-ray pattern for Geneva, Utah, Fine Aggregate Sample (CMB No. 150102).

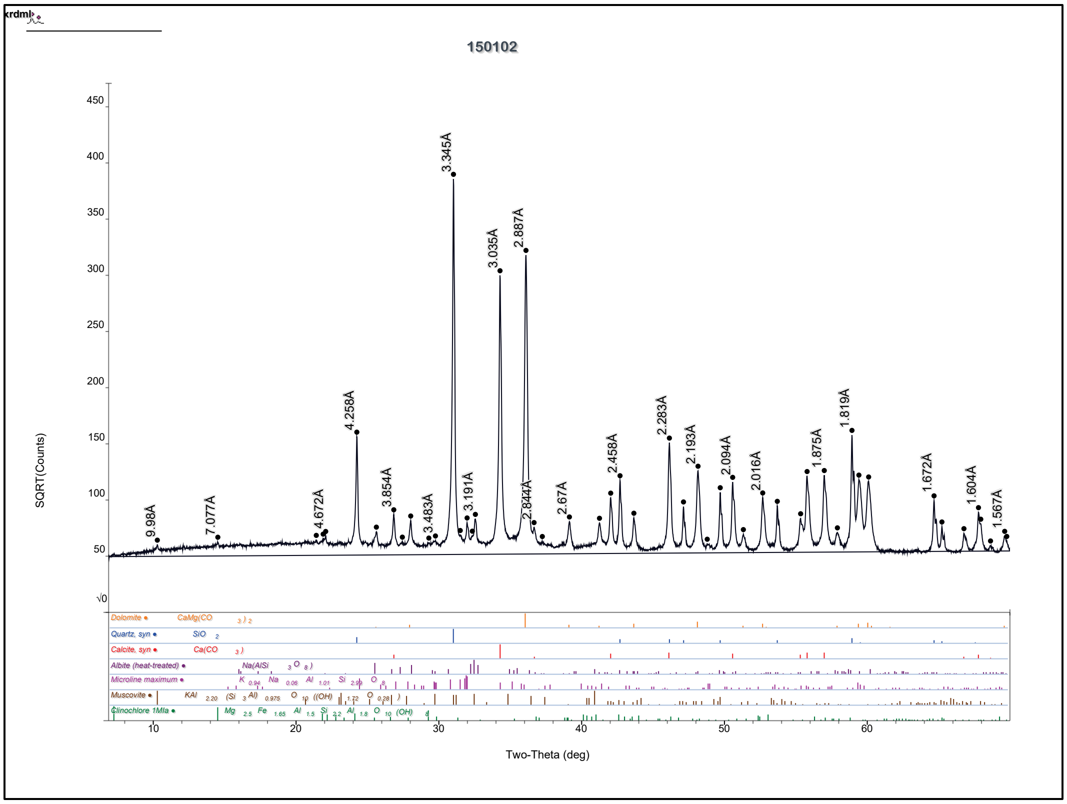
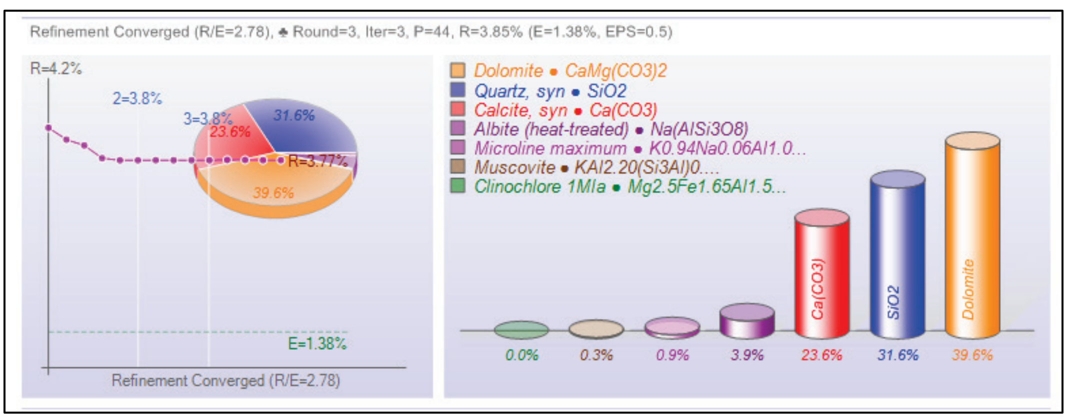


Figure B96. Whole pattern fit with mineral percentages Geneva, Utah, Fine Aggregate Sample (CMB No. 150102).



B.33 Geneva, Utah, Coarse Aggregate Sample (CMB No. 150103)

The Geneva, Utah, coarse aggregate sample (CMB No. 150103) was classified as a complex gravel. The color of the particles varied from grayish pink (5R 8/2), moderate red (5R 5/4), pale red (10R 6/2), pale reddish brown (10R 5/4), very pale orange (10YR 8/2), pale yellowish orange (10YR 8/6), medium bluish gray (5B 5/1), white (N9), very light gray (N8), light gray (N7), medium light gray (N6), and dark gray (N3) in color (Figure B97). Analysis of the XRD patterns (Figure B98) indicated that the predominant phase in the material was dolomite. Figure B99 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Dolomite ($CaMg(CO_3)_2$) (50.1%), quartz (SiO_2) (28.9%), calcite ($CaCO_3$) (19.5%), and trace amounts of albite ($Na(Al_3O_8)$), orthoclase ($KAlSi_3O_8$), and muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) were identified for this sample.

Figure B97. Geneva, Utah, Coarse Aggregate Sample (CMB No. 150103).

Figure B98. X-ray pattern for Geneva, Utah, Coarse Aggregate Sample (CMB No. 150103).

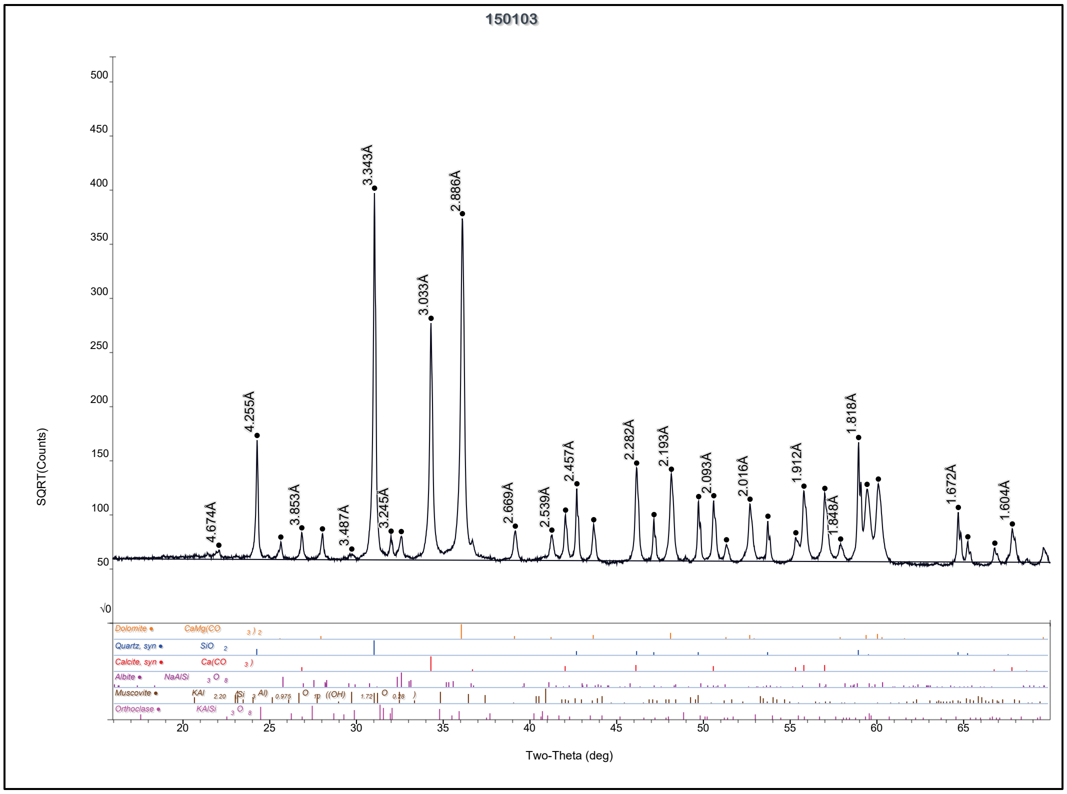
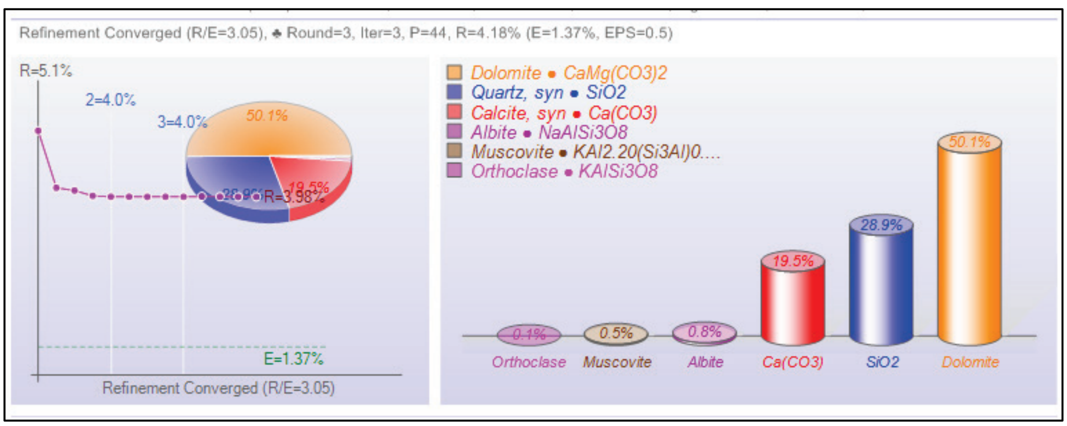


Figure B99. Whole pattern fit with mineral percentages Geneva, Utah, Coarse Aggregate Sample (CMB No. 150103).



B.34 Clayton/Yardville Fine Aggregate Sample (CMB No. 150104)

The Clayton/Yardville fine aggregate sample (CMB No. 150104) was classified as quartz sand. The color of the particles varied from moderate red (5R 4/6), moderate reddish brown (10R 4/6), pale yellowish orange (10YR 8/6), very pale orange (10YR 8/2), light brown (5YR 5/6), yellowish gray (5Y 8/1), white (N9) and very light gray (N8) in color (Figure B100). Analysis of the XRD patterns (Figure B101) indicated that the predominant phase in the material was quartz. Figure B102 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (99.5%), tridymite (SiO_2) (0.2%), and cristobalite (SiO_2) (0.2%) were identified for this sample.

Figure B100. Clayton/Yardville Fine Aggregate Sample (CMB No. 150104).

Figure B101. X-ray pattern for Clayton/Yardville Fine Aggregate Sample (CMB No. 150104).

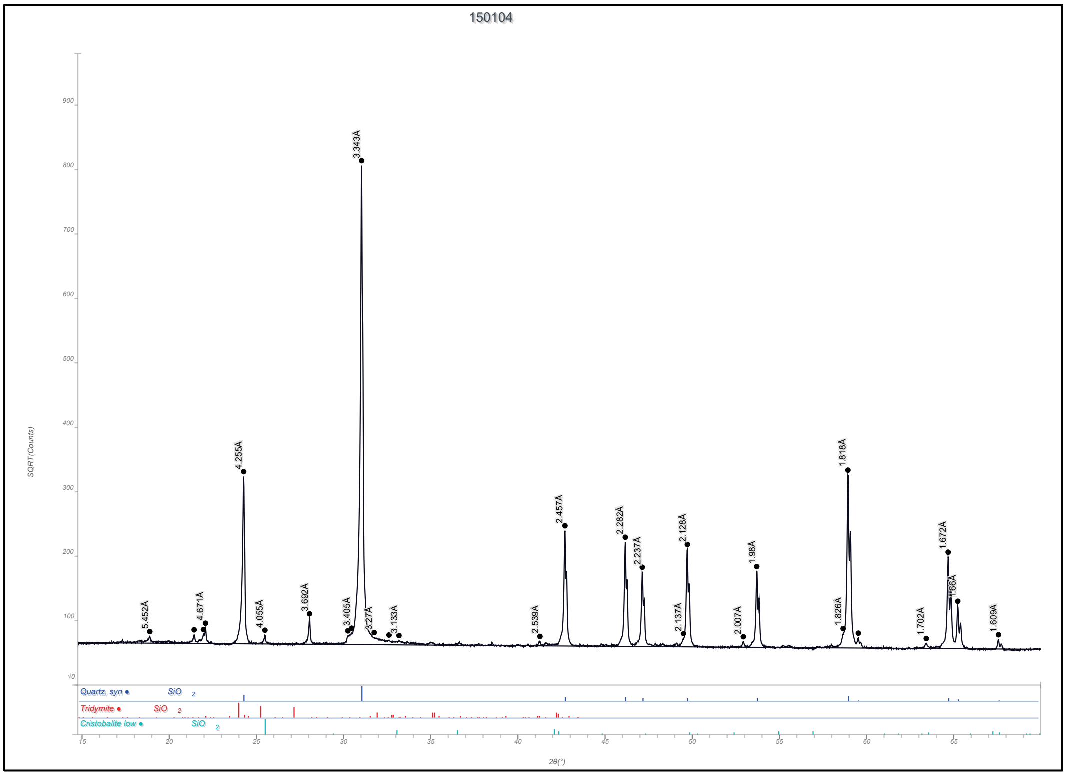
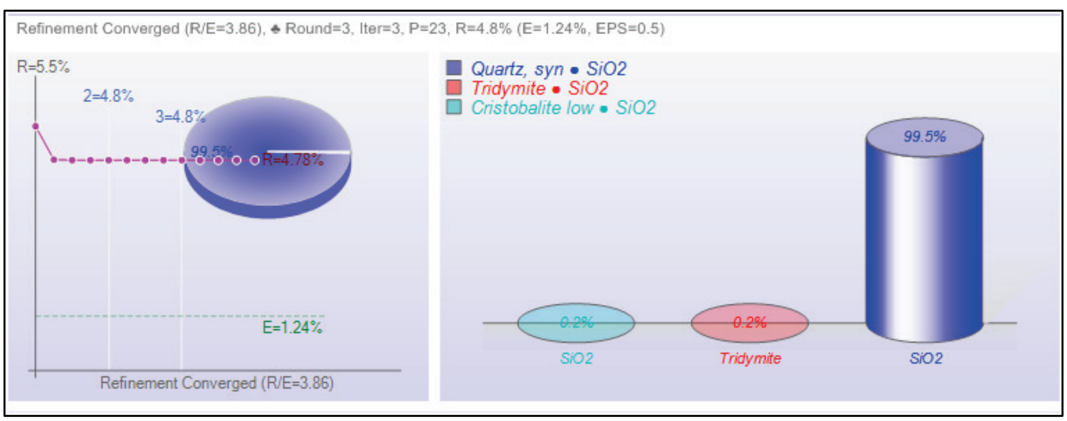


Figure B102. Whole pattern fit with mineral percentages Clayton/Yardville Fine Aggregate Sample (CMB No. 150104).



B.35 Arkadelphia Fine Aggregate Sample (CMB No. 152149)

The Arkadelphia fine aggregate sample (CMB No. 152149) was classified as quartz sand. The color of the particles varied from pale reddish brown (10R 5/4), grayish red (10R 4/2), moderate orange pink (5YR 8/4), grayish orange pink (10R 8/2), pale yellowish orange (10YR 8/6), pale yellowish brown (10YR 6/2), dark yellowish orange (10YR 6/6), moderate brown (5YR 4/4), pale brown (5YR 5/2), light brown (5YR 6/4), yellowish gray (5Y 7/2), medium bluish gray (5B 5/1), dark greenish gray (5G 4/1), yellowish gray (5Y 8/1), white (N9), medium dark gray (N4), and dark gray (N3) in color (Figure B103). Analysis of the XRD patterns (Figure B104) indicated that the predominant phase in the material was quartz. Figure B105 contains the weight percent data obtained by whole pattern fitting using Jade 2010 software. Quartz (SiO_2) (92.4%), albite ($Na(Al_3O_8)$) (6.4%), dolomite ($Ca,Mg(CO_3)_2$) (0.9%), calcite ($CaCO_3$) (0.2%), and cristobalite (SiO2) (0.1%) were identified for this sample.

Figure B103. Arkadelphia Fine Aggregate Sample (CMB No. 152149).

Figure B104. X-ray pattern for Arkadelphia Fine Aggregate Sample (CMB No. 152149).

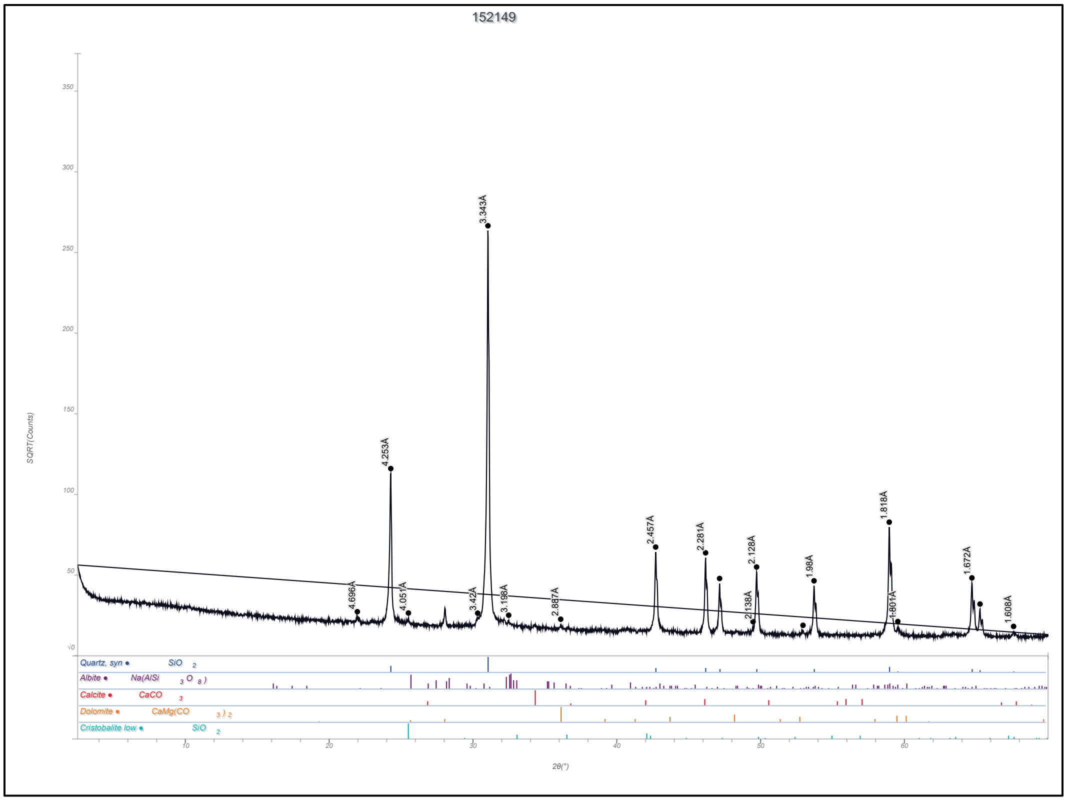
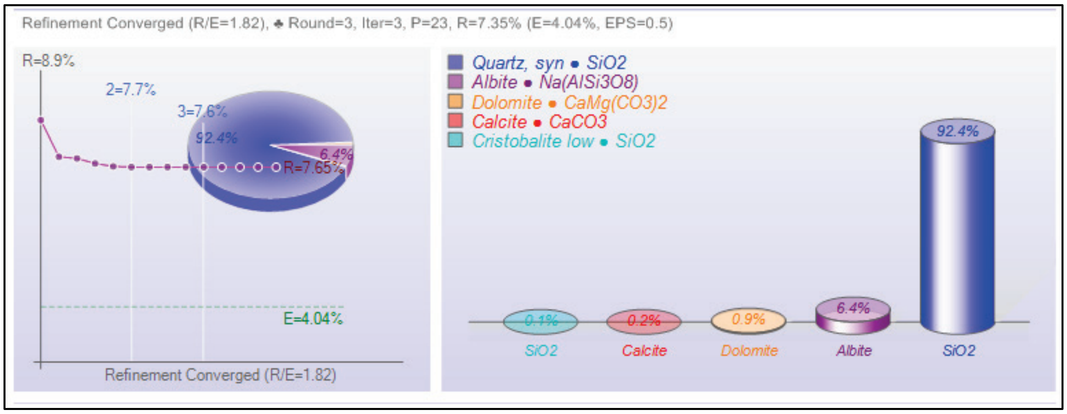


Figure B105. Whole pattern fit with mineral percentages Arkadelphia Fine Aggregate Sample (CMB No. 152149).



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14. ABSTRACT
This study focused on development and evaluation of a novel autoclaving procedure for the rapid evaluation of concrete aggregates for alkali-silica reaction susceptibility. The use of high pressure and temperature auto-claving methods was selected to speed evaluation timeframes from months to a few days. A total of 30 aggregates of varying mineralogy and reactivity were evaluated as part of the study that was divided into two phases. The first phase of research focused on development and optimiza-tion of the autoclaving procedures, including temperature, alkali loading, and test duration. Results from Phase 1 were used to identify optimal testing parameters that were then used during a Phase 2 round robin testing study, which included internal laboratory testing as well as four external laboratories. Strong correlation was observed between autoclave expansion results and the results of ASTM C1260 and C1293 standard test methods. In addition, within- and multi-laboratory variability were with-in acceptable bounds provided by ASTM standards. Overall, the research indicated that autoclaving is a viable option for rapid evaluation of aggre-gates using testing procedures that can be completed in approximately three days.

15. SUBJECT TERMS

ASR	Concrete – Testing	Durability
Alkali-silica reaction	Autoclave	Alkali-aggregate reactions
	Testing	Aggregates (building materials)
	Concrete – expansion and contraction	

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