

# Engineering Scale Demonstration of a Prospective Cast Stone Process

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### **EXECUTIVE SUMMARY**

The primary disposition path of Low Activity Waste (LAW) at the DOE Hanford Site is vitrification. A cementitious waste form is one of the alternatives being considered for the supplemental immobilization of the LAW that will not be treated by the primary vitrification facility. Washington River Protection Solutions (WRPS) has been directed to generate and collect data on cementitious or pozzolanic waste forms such as Cast Stone.

The goal of this project for WRPS was to obtain data on the performance of the Cast Stone waste form for immobilizing LAW. WRPS will incorporate the resulting data into a data package and Environmental Risk Assessment for review by an independent body and ORP to support the selection of a supplemental treatment technology to immobilize approximately 50-60% of the LAW.

This report documents an engineering-scale demonstration with non-radioactive simulants that was performed at SRNL using the Scaled Continuous Processing Facility (SCPF) to fill an 8.5 ft container with simulated Cast Stone grout. The Cast Stone formulation was chosen from the previous screening tests. Legacy salt solution from previous Hanford salt waste testing was adjusted to correspond to the average composition generated from the Hanford Tank Waste Operation Simulator (HTWOS). The dry blend materials, ordinary portland cement (OPC), Class F fly ash, and ground granulated blast furnace slag (GGBFS or BFS), were obtained from Lafarge North America in Pasco, WA. Over three days, the SCPF was used to fill a 1600 gallon container, staged outside the facility, with simulated Cast Stone grout. The container, staged outside the building approximately 60 ft from the SCPF, was instrumented with x-, y-, and z-axis thermocouples to monitor curing temperature. The container was also fitted with two formed core sampling vials. For the operation, the targeted grout production rate was 1.5 gpm. This required a salt solution flow rate of approximately 1 gpm and a premix feed rate of approximately 580 lb/h. During the final day of operation, the dry feed rate was increased to evaluate the ability of the system to handle increased throughput. Although non-steady state operational periods created free surface liquids, no bleed water was observed either before or after operations. The final surface slope at a fill height of 39.5 inches was 1-1.5 inches across the 8.5 foot diameter container, highest at the final fill point and lowest diametrically opposed to the fill point. During processing, grout was collected in cylindrical containers from both the mixer discharge and the discharge into the container. These samples were stored in a humid environment either in a closed box proximal to the container or inside the laboratory. Additional samples collected at these sampling points were analyzed for rheological properties and density. Both the rheological properties (plastic viscosity and yield strength) and density were consistent with previous and later SCPF runs.

After approximately four months, the top was removed from the container to expose the surface and assess the monolith. Cores were collected to probe the surface and provide initial evaluation of the container. The container was covered with a tarp to protect it from the elements as curing continued. After an additional two months (six total), the container was cored from surface to bottom in eight locations. The analysis of the cores will be detailed in a future report.

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## LIST OF ABBREVIATIONS

| BET          | Brunauer-Emmett-Teller                            |
|--------------|---|
| DAS          | Data Acquisition System                           |
| DOE/ORP      | Department of Energy – Office of River Protection |
| GGBFS or BFS | Ground granulated blast furnace slag              |
| gpm          | Gallons per minute                                |
| HTWOS        | Hanford Tank Waste Operation Simulator            |
| LAW          | Low Activity Waste                                |
| OPC          | Ordinary portland cement                          |
| SCPF         | Scaled Continuous Processing Facility             |
| SRNL         | Savannah River National Laboratory                |
| W/DM         | Water to dry materials ratio                      |
| WRPS         | Washington River Protection Solutions             |

#### **1.0 Introduction**

The Department of Energy – Office of River Protection (DOE/ORP) has directed Washington River Protection Solutions (WRPS) to generate and collect data on cementitious or pozzolanic waste forms such as Cast Stone. A cementitious waste form is one of the alternatives being considered for Supplemental Immobilization of Hanford Low Activity Waste (LAW), along with vitrification, bulk vitrification, and fluidized bed steam reforming.

The goal of this project for WRPS was to obtain data on the performance of the Cast Stone waste form for immobilizing LAW. WRPS will incorporate the resulting data into a data package and Environmental Risk Assessment for review by an independent body and ORP to support the selection of a supplemental treatment technology to immobilize approximately 50-60% of the LAW.

In support of goal, a testing program was developed to obtain additional information on the Cast Stone option for immobilizing the LAW.<sup>1</sup> Screening tests to examine expected ranges in waste composition, waste concentration, dry materials sources, and free water (in the waste liquid)-to-dry blend mix ratios are currently being performed.<sup>2</sup>

As part of the data package, an engineering-scale demonstration with non-radioactive simulants was performed. The Scaled Continuous Processing Facility (SCPF) at Savannah River National Laboratory (SRNL) was used to fill a container with simulated Cast Stone grout to display the ability to operate a process to immobilize a simulated LAW salt solution in a cementitious waste form.

#### **2.0 Experimental Procedure**

To describe the activities performed as part of the Engineering Scale Demonstration, heretofore referred to as the ES Demo, a Task Technical and Quality Assurance Plan was issued.<sup>3</sup> This plan identifies the approach for the dry blends, salt solution, container, processing strategy, and maintenance of the filled container.

#### 2.1 Dry Blend Materials

Ordinary portland cement (OPC), ground granulated blast furnace slag (BFS), and Class F fly ash were obtained from Lafarge North America in Pasco Washington, a source that would be readily available at the Hanford Site. The dry materials and ratios are the same as in the Screening Matrix tests in Reference 2. The dry materials were characterized using compositional analysis, X-ray diffraction analysis, particle size distribution, and Brunauer–Emmett–Teller (BET) surface area measurements. The properties were compared to the dry materials used in the screening tests, Reference 2. The dry materials were weighed and blended using the baseline Cast Stone concentrations, Table 2-1, and staged in bins.

| Dry Blend<br>Component | (wt %) | Targeted Mass<br>(g) | Acceptable Mass<br>(g) |
|------------------------|--------|----------------------|------------------------|
| OPC                    | 8      | 560                  | 555-565                |
| BFS                    | 47     | 3290                 | 3270-3310              |
| Fly ash                | 45     | 3150                 | 3130-3170              |

Table 2-1. Dry Blend Mixture for the ES Demo

#### 2.2 Salt Solution Waste Simulant

The salt solution waste simulant was prepared using salt simulants remaining from the fractional crystallization pilot scale test.<sup>4</sup> Select totes of legacy salt solution were analyzed and blended in a single tank. The blended salt solution was trimmed to approximate the targeted composition of the 7.8 M Na Overall Average from the bench scale testing.<sup>5</sup> Table 2-2 shows the composition of the main components of the targeted salt solution and the composition of the blended totes after trim chemicals were added. The components that were targeted for approximation were aluminum, sodium, and nitrate + nitrite. The

differences between the measured values for each of the analytes was within the analytical error associated with each of the components.

| Salt Solution   | Concentration<br>(mg/L)     |                  |  |  |
|-----------------|-----------------------------|------------------|--|--|
| Component       | Targeted<br>Overall Average | Blended/adjusted |  |  |
| Al              | 12.9                        | 11.4             |  |  |
| Cr              | 1.74                        | 1.58             |  |  |
| K               | 2.00                        | 6.36             |  |  |
| Na              | 179                         | 169              |  |  |
| Na (Mol/L)      | 7.8                         | 7.3              |  |  |
| PO <sub>4</sub> | 7.29                        | 2.18             |  |  |
| $SO_4$          | 12.8                        | 9.41             |  |  |
| Cl              | 2.33                        | 4.61             |  |  |
| NO <sub>2</sub> | 40.5                        | 33.4             |  |  |
| NO <sub>3</sub> | 157                         | 193              |  |  |
| Density         | 1.346                       | 1.362            |  |  |
| Wt% solids      | 38.4                        | 40.5             |  |  |

 Table 2-2. Targeted and Measured Salt Solution Compositions.

#### 2.3 Waste Form Container

The receptacle for the Cast Stone waste form is a 1600 gallon, 102 inch diameter, polyethylene container, Figure 2-1. The container was staged outside of the facility, approximately 60 ft from the SCPF. The container was fitted with three grout entry points, an array of thermocouples spanning the diameter and expected fill height of the interior, and two refurbished emplaced core vials recovered from previous testing, Figure 2-2.<sup>6</sup> Temperature data were recorded for 14 days after processing.



Figure 2-1. Plastic-Mart 1600VTS container used as Cast Stone receptacle.



#### Figure 2-2. Pour points, thermocouple tree and emplaced core vial locations annotated on container.

#### 2.4 <u>Scaled Continuous Processing Facility</u>

The SCPF, Figure 2-3, uses a gravimetric feeder to supply the dry materials to a Readco-Kurimoto 2-inch continuous processor. The mixer uses co-rotating, twin shafts configured with intermeshing paddles. Clearance between intermeshing paddles and between the paddles and the barrels of approximately oneeighth inch produces a self-wiping action that minimized material buildup. The mixer motor is variable speed and controlled through an integral touch-screen panel. A variable speed gear pump delivers the liquid feed (water or salt solution) to the grout mixer. The salt solution flow was measured using a mass flow meter rated for 0-17 gpm. Fresh grout is discharged from the mixer into the grout pump hopper fitted with an agitator. A peristaltic grout pump transfers grout from the grout hopper through tubing to the grout receipt container in Figure 2-2. Pressure, temperature and flow instrumentation were installed in the process to monitor test conditions. Instrument outputs were recorded electronically on a Data Acquisition System (DAS) with the exception of the dry material feeder operating conditions, which were recorded manually.

#### 2.5 Sampling

During processing, fresh grout was sampled at two locations: at the discharge of the grout mixer and at the grout tank. A sampling chute above the grout hopper allowed sampling of fresh grout from the discharge of the mixer and a diverter valve at the container enabled sampling without discontinuing flow to the container. The density of freshly prepared grout was measured with weight per gallon sample cups (Gardco) using a simplified ASTM method.<sup>7</sup> A flow curve was processed for samples from both locations using a Haake VT550 rotoviscometer. The VT550 was used to obtain a flow curve (shear stress versus shear rate data) using a concentric geometry bob and cup. The data were analyzed using a Bingham Plastic rheological model, providing yield stress and plastic viscosity values. Additional sample material was collected in 2 in x 4 in cylinders, placed in zip top bags with moistened towels, and stored either in a lidded box near the grout container, or indoors behind the mixer.



Figure 2-3. Components of the scaled continuous processing facility.

#### 2.6 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

#### **3.0 Results and Discussion**

The dry materials and salt solution used were characterized. Calculations were performed to determine processing parameters such as dry feed rate, salt solution feed rate, and processing duration.

#### 3.1 Dry Blend Materials

The compositions of the dry blend components were similar to those of the dry blend components used in the screening tests and were within the industry standards,<sup>8,9,10</sup> Table 3-1. The X-ray diffraction patterns, particle size distributions, and surface area measurements by the BET method were similar to the properties of the dry blend materials used in the Screening Matrix tests, Reference 2. Figure 3-1 is the X-ray diffraction patterns for the dry blend components in this study shown with the materials used in the Screening Matrix tests for comparison. The shape of the amorphous part of the pattern and the identity of the major phases are the features of interest as the intensity scales (y-axis) on the plots are not the same. Figure 3-2 is the overlay of the particle size distributions for the three materials. Although there are some variations, both the general shapes of the curves and the particle size ranges are similar between the lots of material. The similarity in the surface area of the dry materials is shown in Table 3-2. The differences

in the properties of the dry materials that can be gleaned from these tables illustrate the inherent variability associated with materials used in the concrete industry.

|                                | Cemen              | t (wt%)            | BFS (              | (wt%)   | Fly Ash            | n (wt%) |
|--------------------------------|--------------------|--------------------|--------------------|---------|--------------------|---------|
| Component                      | Screening<br>Study | ES Demo            | Screening<br>Study | ES Demo | Screening<br>Study | ES Demo |
| Al <sub>2</sub> O <sub>3</sub> | 4.83               | 4.79               | 12.71              | 12.91   | 16.26              | 19.42   |
| CaO                            | 63.14              | 55.67 <sup>*</sup> | 42.56              | 41.73   | 13.00              | 14.63   |
| Fe <sub>2</sub> O <sub>3</sub> | 3.55               | 3.42               | 0.59               | 0.72    | 5.93               | 4.73    |
| K <sub>2</sub> O               | 0.32               | 0.41               | 0.34               | 0.85    | 1.49               | 1.63    |
| MgO                            | 0.67               | 0.80               | 4.17               | 5.16    | 4.40               | 4.24    |
| MnO <sub>2</sub>               | 0.32               | 0.09               | 0.24               | 0.20    | 0.07               | 0.13    |
| SiO <sub>2</sub>               | 20.57              | 20.11              | 32.96              | 31.27   | 51.79              | 47.12   |
| TiO <sub>2</sub>               | 0.23               | 0.26               | 0.36               | 0.42    | 0.75               | 0.74    |
| $SO_4$                         | 4.86               | 3.05               | 4.92               | 3.43    | 1.02               | 0.77    |
| Total                          | 98.49              | 28.14              | 98.85              | 96.69   | 94.71              | 93.41   |

## Table 3-1. Composition of the Dry Blend Materials used in the Screening Matrix Tests and the ES Demo.

<sup>\*</sup>Calcium analysis is flawed as the value is lower than expected and the total oxides are lower than typical.



Figure 3-1. Comparison of the X-Ray Diffraction Patterns of the Dry Materials Used for the ES Demo and the Screening Matrix Test.



Figure 3-2. Comparison of the Particle Size Distribution Plots for the Dry Materials Used for the ES Demo and the Screening Matrix Test.

| Table 3-2. Comparison of the Surface Area by BET for the Dry Materials Used | d for the ES | Demo |
|---|--------------|------|
| and the Screening Matrix Test.  |              |      |

| Dry Material Component | ES Demo (m <sup>2</sup> /g) | Screening Matrix (m <sup>2</sup> /g) |
|------------------------|-----------------------------|--------------------------------------|
| Cement                 | 1.56                        | 1.57                                 |
| BFS                    | 2.55                        | 3.03                                 |
| Fly Ash                | 1.08                        | 0.79                                 |

#### 3.2 Salt Solution Waste Simulant

The components that were desirable to approximate in the ES Demo were aluminum, sodium, and nitrate + nitrite. Table 3-3 is the analytical results of the components that were targeted to be approximated in the ES Demo salt solution simulant. The statistical analysis in Reference 2 demonstrated that there was no significant differences in Cast Stone properties across salt solutions with greater variability that the targeted and measured salt solutions in this study.

## Table 3-3. Difference in Components that were Targeted for Approximation in ES Demo Salt Solution.

|                 | Conce           | Difference      |                |
|-----------------|-----------------|-----------------|----------------|
| Salt Solution   | ( <b>n</b>      |                 |                |
| Component       | Targeted        | Blended/Trimmed | Target-trimmed |
|                 | Overall Average | Totes           | target         |
| Al              | 12.9            | 11.4            | 13%            |
| Na              | 179             | 169             | 5.6%           |
| Na (Mol/L)      | 7.8             | 7.3             |                |
| $NO_2$          | 40.5            | 33.4            | -              |
| NO <sub>3</sub> | 157             | 193             | -              |
| $NO_2 + NO_3$   | 197.5           | 226.4           | 15%            |

#### 3.3 Scaled Continuous Processing Facility

The initial operating parameter for the dry materials was 595 lb/h. The salt solution feed was set at 0.895 gpm to maintain a water to dry materials ratio near 0.60. The W/DM of 0.60 was identified in Reference 3 as the controlling parameter. The dry feed rate was selected to meet the processing rate to fill the grout container over the allotted time frame, with the assumption that there was minimal stoppage time. It is a practical goal of processing to maintain a constant dry feed rate unless that has been identified as a variable parameter. The salt solution feed rate is adjusted based on the water content of the salt solution and the dry feed rate to obtain the targeted W/DM. However, the salt solution flow meter was not able to control the flow rate precisely enough to rely on the salt solution feed values. Although the measured values are reported with three significant figures for calculation purposes, the practical flow rate is 1 gpm.

$$\frac{W}{DM} = \frac{\text{mass of water in salt solution}}{\text{mass of dry materials blend}}$$
(1)

Where the mass of the water in the salt solution is:

$$\frac{1.362 \text{ g salt solution}}{1 \text{ ml salt solution}} \times \frac{1 \text{ lb}}{453.592 \text{ g}} \times \frac{3785 \text{ ml}}{1 \text{ gal}} \times \frac{0.595 \text{ lb water}}{1 \text{ lb salt solution}} = \frac{6.76 \text{ lb water}}{1 \text{ gal salt solution}};$$

Where the lb water/lb salt solution is from Table 2-2 and

$$\frac{W}{DM} = \frac{\frac{6.76 \text{ lb water}}{1 \text{ gal salt solution}} \times \frac{0.895 \text{ gal salt solution}}{1 \text{ min}}}{\frac{595 \text{ lb dry materials blend}}{1 \text{ h}} \times \frac{1 \text{ hr}}{60 \text{ min}}} = 0.61$$
(2)

These conditions resulted in a calculated grout density of  $107.2 \text{ lb/ft}^3$  (1.72 g/ml), determined from the mass flow meter in the grout line, and a grout processing rate of 1.36 gpm into the container as calculated by the DAS. Small adjustments were made to the salt solution flow to maintain a constant W/DM. The SCPF produced grout for approximately 3.9 h, resulting in ~11.25 inches of grout in the container, Figure 3-3.



Figure 3-3. Grout container fill height (in inches) after 3.9 hours of processing.

Processing was halted to preclude the onset of filling the lower emplaced core vial so the vial could be filled in a single lift. In Reference 6, vials were filled under various processing conditions. For this testing, the goal of filling the emplaced core vials was to produce a typical sample. Figure 3-4 shows the grout surface at the conclusion of the first lift. The self-leveling ability of the grout slurry was evident at a fill rate of ~26 min/in. Although not necessary, a self-leveling slurry improves operational flexibility by increasing the surface area serviced by a pour point.



Figure 3-4. Grout surface at the end of the first lift.

The second lift was performed the following day using similar operating parameters as the initial lift: 595 lb/hr and 0.925 gpm, for a targeted W/DM of 0.63. The uncertainty of the salt solution flow meter lead to variability in the W/DM calculation. This in turn increased the precision in the control of the salt solution feed rate and the resulting W/DM. These conditions resulted in a calculated grout density of 105.2 lb/ft<sup>3</sup> (1.69 g/ml) and a grout processing rate of 1.40 gpm into the container. Small adjustments were made to the salt solution flow to maintain a constant W/DM. The SCPF produced grout for approximately 6.1 h resulting in ~14.25 inches of grout added to the container. The average salt solution flow rate for the second day of processing was 0.910 gpm and the average grout processing rate was 1.38 gpm. During processing, the lower emplaced core was filled, Figure 3-5. The emplaced core sampler fills via an overflow weir. Details of the design, fill, and extraction of the emplaced cores are provided in Reference 6. Figure 3-6 shows the pour stream entering the container. Air entrained in the grout slurry rises to the surface; however, not all of the air is released. During processing, a film of excess salt solution was observed on the surface of the pour, Figure 3-7. The salt solution flow rate was subsequently reduced to 0.895 gpm – the flow rate used during the first lift. All of the excess salt solution had been incorporated into the grout slurry prior to the completion of the lift.



Figure 3-5. Filling of lower emplaced core vial.



Figure 3-6. Grout slurry pour stream.



Figure 3-7. Excess salt solution accumulating on grout surface.

The third and final lift was performed using similar operating parameters as the initial lift: 595 lb/h and 0.905 gpm, for a targeted W/DM of 0.61. These conditions resulted in a calculated grout density of 104.8 lb/ft<sup>3</sup> (1.68 g/ml) and a grout processing rate of 1.39 gpm into the container. The SCPF produced grout for approximately 2 h. To evaluate the ability to produce grout at a faster rate, the dry materials feed rate was increased to 608 lb/h. Issues with the ability to precisely measure the salt solution flow rate resulted in the salt solution feed rate reduced to 0.871 gpm. The W/DM shifted to 0.57, the calculated grout density averaged 105.3 lb/ft<sup>3</sup> (1.69 g/ml) and the grout processing rate was 1.37 gpm into the container. The SCPF was operated using these parameters for ~1.6 h. The SCPF processed the increased dry feed rate without issue and the decision was made to further increase the dry feed rate. The dry materials feed rate was increased to 625 lb/h with the salt solution flow rate increased to 0.895 gpm. The resulting W/DM was 0.58, the calculated grout density averaged 105.7 lb/ft<sup>3</sup> (1.69 g/ml) and a grout processing rate of 1.38 gpm into the container. The SCPF was operated using these parameters for  $\sim 2.4$  h. The third lift raised the final height of the container level to 39.5 inches. Processing concluded when the available blended dry materials supply was exhausted. Figure 3-8 is the final grout surface after the third lift. All but three of the uppermost thermocouples were covered due to the slope associated with the final days processing. The maximum difference in height across the 8.5 ft container was 1.5 inches based on the measuring tape shown in Figure 2-2 and the corresponding tape (not shown) on the opposite side of the container.



Figure 3-8. Final grout surface after third lift.

#### 3.4 Sampling

During processing, grout slurry samples were periodically collected from the mixer exit and at the container. Samples were evaluated for density and rheological properties. Additional samples collected in 2 in x 4 in cylinders were stored for future analysis. It was observed during handling of the collected samples that grout slurries obtained directly from the mixer exit contained soft agglomerates of dry materials that had not yet been fully incorporated into the slurry. The inhomogeneity of the samples obtained from the mixer may have exacerbated the variability in density measurements for samples collected at the mixer exit. Conversely, after a residence time of approximately 60 s in the grout mixer hopper and transfer through 60 ft of tubing, samples collected from the container appeared homogeneous. The observed transition from inhomogeneous to homogeneous grout slurry was also noted in previous testing.<sup>11</sup>

| Sample I | Date/Time | Sample Location  | Density |
|----------|-----------|------------------|---------|
| Date     | Time      | Mixer/ container | (g/ml)  |
| 10/07/13 | 11:30     | Mixer            | 1.59    |
| 10/07/13 | 11:45     | container        | 1.80    |
| 10/07/13 | 13:50     | Mixer            | 1.65    |
| 10/07/13 | 14:10     | container        | 1.72    |
| 10/08/13 | 9:40      | Mixer            | 1.72    |
| 10/08/13 | 10:05     | container        | 1.80    |
| 10/08/13 | 13:10     | Mixer            | 1.75    |
| 10/08/13 | 13:40     | container        | 1.78    |
| 10/09/13 | 10:35     | Mixer            | 1.65    |
| 10/09/13 | 10:55     | container        | 1.74    |
| 10/09/13 | 12:30     | Mixer            | 1.73    |
| 10/09/13 | 12:55     | container        | 1.77    |
| 10/09/13 | 14:15     | Lab Prepared     | 1.79    |

Table 3-4. Density of Samples Collected from the SCPF at Various Locations.

Table 3-5 is the plastic viscosity values and Bingham plastic yield stress values calculated from the down curve of the rheology flow curve. Specific rheology testing in Reference 11 notes the thixotropy associated with grout slurry samples collected at the mixer and shortly after the mixer. Only the down curve was considered for analysis to provide a more direct comparison and eliminate the heterogeneity associated with the less sheared samples. Samples collected from the grout container experienced additional shear in the grout mixer tank, grout pump, and transfer hose. All down curves were analyzed as Bingham Plastic fluids. The variation in the measured plastic viscosities and yield strengths were likely a result of the salt solution flow control. Overall, the plastic viscosities and yield strengths were similar to those obtained in the testing in Reference 11. due to the uncertainties in controlling liquid flow, comparison of the mixer and tank rheological properties for a sample point cannot be properly addressed.

| Sample Date/Time |      | Sample<br>Location  | Down Curve                   |   |                | Fitted                      |
|------------------|------|---------------------|------------------------------|---|----------------|-----------------------------|
| Date             | Time | Mixer/<br>container | Plastic<br>Viscosity<br>(cP) | Bingham<br>Plastic Yield<br>Stress (Pa) | R <sup>2</sup> | Range<br>(s <sup>-1</sup> ) |
| 100713           | 1130 | Mixer               | 126.9                        | 3.22                                    | 0.9855         | 10-300                      |
| 100713           | 1145 | container           | 131.8                        | 7.21                                    | 0.9968         | 10-300                      |
| 100713           | 1350 | Mixer               | 103.5                        | 3.40                                    | 0.9988         | 10-300                      |
| 100713           | 1410 | container           | 105.0                        | 7.26                                    | 0.9933         | 10-300                      |
| 100813           | 940  | Mixer               | 88.5                         | 2.37                                    | 0.9990         | 10-300                      |
| 100813           | 1005 | container           | 103.5                        | 3.17                                    | 0.9990         | 10-300                      |
| 100813           | 1310 | Mixer               | 81.2                         | 1.47                                    | 0.9991         | 10-300                      |
| 100813           | 1340 | container           | 84.8                         | 1.94                                    | 0.9989         | 10-300                      |
| 100913           | 1035 | Mixer               | 92.3                         | 1.85                                    | 0.9991         | 10-300                      |
| 100913           | 1055 | container           | 91.3                         | 2.40                                    | 0.9986         | 10-300                      |
| 100913           | 1230 | Mixer               | 91.7                         | 2.91                                    | 0.9990         | 10-300                      |
| 100913           | 1255 | container           | 111.7                        | 4.23                                    | 0.9984         | 10-300                      |

Table 3-5. Rheological Properties of the Down Curve of the Samples Collected.

#### 3.5 Waste Form Container

After completion of the processing, the waste form container was monitored for temperature for 14 days. Thermocouple trees with three thermocouples (at bottom of container, at  $\sim 20$  inches from the bottom, and at ~39 inches from the bottom—one inch below the expected surface) were installed in the center and at equidistant radial positions. Figure 3-9 shows the temperature plot of each thermocouple tree. The top leftmost plot is the center tree and each row of plots is a radial arm from the center. The purpose of this figure is to demonstrate the symmetry of the temperature in the grout container rather than the specific temperatures. The maximum temperature, 69 °C, was reached at the mid-height thermocouples in the installations located 12 inches from the center. This temperature was reached 119 h after pouring was completed and the maximum temperature was maintained for ~10 h before dropping below 69 °C. Figure 3-10 is a representation of the potentially different temperature zones present in the grout container as a result of the temperature profiles in Figure 3-9 and the operating strategy of the SCPF. Since the temperature profile in the cylindrical grout container was symmetrical, a radial figure was used for clarity. The cold joints represent the top surface of the previous day of operation that is exposed to the atmosphere. The size of the smaller zones were artificially increased to facility the identity of cores for testing. Coring of the container occurred in April 2014 and the results of the coring and analysis will be presented in another report.



Figure 3-9. Temperature profiles across the radius of the container over 14 days.

|                       |                              | Exposed Zone (4") |                    |      |  |  |  |  |  |
|-----------------------|------------------------------|-------------------|--------------------|------|--|--|--|--|--|
|                       | Day 3 (14")                  |                   |                    |      |  |  |  |  |  |
|                       | ↓<br>T                       | Cold Joint        |                    |      |  |  |  |  |  |
| Exterior wall of tank | Max T < 50 °C<br>Day 2 (14") | Max T ~ 60 °C     | Centerline of tank | 39.5 |  |  |  |  |  |
| t                     | 1                            | Cold Joint        |                    |      |  |  |  |  |  |
|                       | Day 1 (11")                  |                   |                    |      |  |  |  |  |  |

Figure 3-10. Graphic representation of the radial temperature zones resulting from the Engineering Scale Demonstration.

#### 4.0 Conclusions

The SRNL SCPF was successfully operated over three days to fill a 1600 gallon polyethylene container with simulated Cast Stone. Cast Stone grout was processed at a nominal rate of 1.3 gpm for over 16 h. The grout produced was self-leveling across an 8 ft diameter with a maximum height differential across the diameter of 1.5 in from the pour entry point to a diametrically opposed point on the grout container. In-process adjustments of the salt solution flow were able to incorporate and further deter the formation of excess salt solution on the surface. In-process adjustments of the dry materials feed demonstrated the ability to process at faster rates than planned.

#### **5.0 Recommendations**

In this testing, the grout transfer line was run directly from the grout pump to the container. Testing performed in Reference 11 indicated that the rheological properties of the grout slurry benefited from additional processing time prior to entering the container. Additional testing to evaluate the benefit of either a larger mixer hopper or longer grout line to better incorporate the dry materials into the slurry should be performed. The increased processing time improves the incorporation of soft agglomerate of dry feed into the mix and reduces the thixotropic behavior of the grout slurry.

The SCPF is configured to support the Savannah River Site Saltstone Facility. Unit operations such as the mixer grout hopper and grout pump should be evaluated with the SCPF for use at a potential Hanford Cast Stone facility to provide additional input into the design basis if Cast Stone is selected as the supplementary treatment process for Hanford LAW. This data could also be used as design input for the treatment of Hanford Secondary Waste associated with the Waste Treatment Plant. The current Hanford LAW process is anticipated to treat approximately 15 gpm of LAW salt solution. Additional testing should also be performed to determine the maximum processing rates that could be achieved with the 2-inch continuous processor.

#### **6.0 References**

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- <sup>10</sup> ASTM Standard C618-08a, 2008, "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete," ASTM International, West Conshohocken, PA, 2008.
- <sup>11</sup> M. D. Fowley, Cozzi, A. D., Fox, K.M., and Hansen, E.K., "Saltstone Studies Using the Scaled Continuous Processing Facility," SRNL-STI-2014-00406, September 2014.

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