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Crystal Accumulation in the Hanford Waste Treatment Plant High Level Waste Melter: Summary of 2018 and 2019 Experiments

K. M. Fox
M. D. Fowley
August 2019
SRNL-STI-2019-00264, Revision 0
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Crystal Accumulation in the Hanford Waste Treatment Plant High Level Waste Melter: Summary of 2018 and 2019 Experiments

K. M. Fox
M. D. Fowley

August 2019
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EXECUTIVE SUMMARY

This report describes the results of experiments completed in 2018 and 2019 with the full-scale, room temperature WTP HLW melter riser system. Observations from the experiments are provided and discussed, and recommendations are made for future testing.

Lowering the air lance in the riser was found to have little impact on the layer of settled particles. A sample of the settled particles at the bottom of the riser was collected after approximately 15 pouring and idling cycles. No discernable differences in the volumetric particle size distribution were measured between the original, unused magnetite particles and the settled material collected from the bottom of the riser.

Density-matched, polymer spheres were added to the system to serve as reference points, or tracers, to monitor fluid motion. The spheres were observed to follow the flow of the silicone oil and magnetite particles during the pouring and idle cycles. Image analysis software could be used to determine position data as a function of time for each of the tracers during idling of the system, and the data could then be used to validate numerical simulations of particle flow and accumulation within the full-scale melter.

A method was developed for direct addition of magnetite particles to the bottom of the riser to determine whether measurable differences in pouring parameters would occur with a larger blockage. A visual comparison of the appearance of the settled layer at the bottom of the riser before and immediately after pouring revealed few observable differences. Pouring cycle data showed that the air flow rate and pressure needed to maintain the targeted pour rate overlapped within the uncertainty of the measurements. Calculations showed that a more significant blockage would be needed before measurable differences in pouring parameters occurred. Observation of the settled layer at the bottom of the throat revealed that this material did not become resuspended during pouring, as it had in previous tests. This may imply that, for the full-scale WTP HLW melter, the ability to resuspend settled particles will be reduced as the thickness of the settled layers is increased.
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<table>
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<th>Abbreviation</th>
<th>Description</th>
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</thead>
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<tr>
<td>DOE</td>
<td>U.S. Department of Energy</td>
</tr>
<tr>
<td>HLW</td>
<td>High-Level Waste</td>
</tr>
<tr>
<td>LAW</td>
<td>Low-Activity Waste</td>
</tr>
<tr>
<td>ORP</td>
<td>Office of River Protection</td>
</tr>
<tr>
<td>SRNL</td>
<td>Savannah River National Laboratory</td>
</tr>
<tr>
<td>$T_{1%}$</td>
<td>Glass temperature where the melt is in equilibrium with one volume percent of crystals</td>
</tr>
<tr>
<td>vol %</td>
<td>volume percent</td>
</tr>
<tr>
<td>WTP</td>
<td>Hanford Tank Waste Treatment and Immobilization Plant</td>
</tr>
</tbody>
</table>
1.0 Introduction
The U.S. Department of Energy (DOE) Office of River Protection (ORP) is building the Tank Waste Treatment and Immobilization Plant (WTP) at the Hanford Site in Washington to remediate 56 million gallons of radioactive waste that is being temporarily stored in 177 underground tanks. Radioactive waste will be separated into high-level waste (HLW) and low-activity waste (LAW) fractions that will be vitrified in stable borosilicate glass with Joule-heated, ceramic refractory lined melters. Efforts are being made to increase the loading of Hanford tank wastes in glass while maintaining an adequate ability to meet process, regulatory, and product quality requirements.

Glass formulation and melter testing data have suggested that significant increases in waste loading in HLW and LAW glasses are possible over current system planning estimates. Belsher and Meinert identified five constraints that were most influential on the estimated Hanford HLW glass volumes, and by extension, most restricting to waste loading. One of those constraints was the limit of no more than one volume percent spinel crystals in the melt at a temperature of 950 °C.

Historically, crystallization constraints are placed in process control systems to prevent premature or catastrophic failure of the melter through bulk devitrification (also described as volume crystallization) or crystal accumulation and, thus, to mitigate negative impacts of crystals as glass is produced. The baseline method of controlling crystallization in the WTP HLW melter uses a model that predicts the temperature, $T_{1\%}$, at which the equilibrium fraction of spinel crystals in the melt is 1 volume percent (vol %). An alternative crystal-tolerant glass approach may allow higher waste loading for WTP processing while maintaining a chemically durable glass product. Some crystalline phases, such as spinel, do not impact the durability of the waste form but may accumulate in the melter or riser and restrict or prevent its operation. However, prediction of spinel precipitation and accumulation could potentially allow for formulating higher waste loading, durable glasses if an alternative strategy for operating and idling a melter with some amount of tolerable crystals can be developed and implemented.

Actual melter operation is likely to involve situations where accumulation of spinel crystals can occur. Methods of recovering from such an event will make the crystal-tolerant approach more robust, and allow for continued use of a melter in the event of excessive crystal accumulation.

To better understand crystal settling, accumulation, and resuspension in critical areas of the WTP HLW melter, a full-scale, room temperature test system has been designed and constructed. The road map for development of crystal-tolerant HLW glasses noted that an accumulation of crystals in the melter riser could prevent discharge of the molten glass into canisters, especially when considering frequent and periodic idling. Therefore, the test system focuses on the throat and riser of the WTP HLW melter. The system uses transparent materials to allow for the observation of particle behavior under a variety of process conditions. The system will be used to develop and demonstrate potential methods for recovery in the event of an unacceptable amount of crystal accumulation.

A series of experiments was completed with the room temperature system in fiscal year 2016. Accumulation of particles was observed at the bottom of the riser and along the bottom of the throat after each experiment. Measurements of the accumulated layer thicknesses showed that the settled particles at the bottom of the riser did not vary in thickness during pouring cycles or idle periods. Some of the settled particles at the bottom of the throat were re-suspended during subsequent pouring cycles, and settled back to approximately the same thickness after each idle period. The cause of the consistency of the accumulated layer thicknesses was not yet clear, but was hypothesized to be related to particle flow back to the feed tank.

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a Jantzen and Brown provide a brief review of the potential, negative effects of crystallization within a melter.
Additional experiments reinforced the observation of particle flow along a considerable portion of the throat during idle periods.

More recently, the accumulation and resuspension of particles in the riser was shown to be repeatable over the short term, mainly because the change in thickness of the accumulated layer was negligible in each pouring and idling cycle. A longer term view over multiple experiments showed a gradual increase in thickness of the accumulated layer. There was a slight increase in the thickness of the accumulated layer in the riser as the experiments progressed. The degree of resuspension of particles in the throat during a pouring cycle (when the data were normalized to account for flow rate, pouring time, and oil viscosity) was reduced as the experiments progressed. This was hypothesized to be due to settling or compacting of the accumulated layer over time. Lowering the air lance in the riser redistributed the accumulated particles at the bottom of the riser, but did not appear to resuspend the particles.

This report describes the results of experiments completed in 2018 and 2019 with the full-scale, room temperature WTP HLW melter riser system. Observations from the experiments are provided and discussed, and recommendations are made for future testing.

2.0 Quality Assurance

This work was performed following a Task Technical and Quality Assurance Plan and an experimental plan. 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. Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiments L0008-00162 and C3489-00079.

3.0 Experimental Runs

3.1 Test Conditions and System Operation

The design, materials selection, and operation of the full-scale, room temperature WTP HLW melter riser test system have been described in an earlier report. The following sets of experiments are the subject of this report:

- The first set of experiments was run to determine whether lowering the height of the air lance in the riser could be an effective means of removing accumulated particles.
- The second set of experiments evaluated the particle size distribution of the accumulated material to determine whether there was a difference relative to that of the original particles placed into the system.
- The third set of experiments was a proof-of-concept demonstration to show that fluid and particle flows could be monitored via the addition of tracer materials and image analysis.
- The fourth set of experiments was run to determine whether intentionally increasing the thickness of the settled particle layer would lead to measurable differences in pouring parameters.

For most tests, a particle loading of 0.1 vol % in the fluid was selected and used to maintain visibility within the system. A nominal pouring rate of 3.18 lpm (0.84 gpm) was selected and used as the baseline for testing. This pouring rate is the volume of glass planned to be poured per unit time in the actual melter. The rate was calculated using a nominal WTP HLW melter pour rate of 520 lbs of glass in a period of 29 minutes (8.13 kg/min), and an arbitrary glass density of 2.56 g/cm³. A reduced, or low pouring rate was also

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[^Note]: Note that the production rate of 4 MT/day given in Reference 13 is higher than the design capacity production rate of 3 MT/day given in the HLW Waste Form Qualification Report. The higher production rate, and therefore higher pouring rate, was used in this study and considered to be an upper bound.
selected to reflect potential changes in the operation of the WTP HLW melter that might impact particle re-suspension. The low pouring rate was set at approximately 2/3 of the high rate (2.3 lpm or 0.60 gpm).

The ambient temperature in the laboratory varied from day to day, resulting in changes to the viscosity of the silicone oil. Qualitatively, this did not appear to impact particle settling behavior among the experiments. The temperature of the fluid during each experiment was recorded to allow for calculation of the viscosity of the silicone oil.

During idle periods, a continuous purge (approximately 0.2 scfh) of the air lance in the riser was maintained to better simulate planned operation of the WTP HLW melter. The idle purge was observed to produce a complex particle and fluid flow pattern in the riser during idle periods. The feed tank agitator was left running during idle periods to simulate flow in the melter induced by bubblers and thermal gradients.

Changes in thickness of the accumulated particle layers during pouring and idle periods were measured using still images from the video recordings following the methods described in a previous report. A compilation of run parameters and accumulated layer thickness measurements for all of the experiments completed with the system is included for reference as Appendix A. The following sections describe, in chronological order, the details of each set of experiments covered by this report.

3.2 Air Lance Height Testing

Previous testing demonstrated that lowering the height of the air lance in the riser to a level below that of the baseline design redistributed the particles that had settled at the bottom of the riser, but did not effectively resuspend the particles during a pouring cycle. Additional experiments were run to further observe the effects of changing the height of the air lance in the riser, as this is anticipated to be a modification that could be implemented if needed at WTP. Note that the settled layer from previous experiments was not disturbed prior to the start of the additional experiments (idle time of about 180 days).

Three pouring and idling cycles were completed with the air lance returned to its prototypic height of 3.25 inches above the bottom of the riser. A nominal pouring rate of 0.84 gpm was used for all tests in the series. The settled layer at the bottom of the riser did not return to its previous height or appearance after the air lance was raised and the system was run. There was little visible change to the settled layer after the first cycle, as shown in Figure 3-1. The two additional pouring and idling cycles again resulted in little visible change to the settled layer. Measurements of the settled layer thickness are provided in Table 3-1. Differences are within the precision of the measurements.
Figure 3-1. Appearance of the Settled Layer at the Bottom of the Riser (in rectangle) After Operation with the Air Lance at 1.5 inches Above the Bottom (a), and After Operation with the Air Lance at 3.25 inches Above the Bottom (b)
### Table 3-1. Average Measured Thickness of the Settled Layer at the Bottom of the Riser During the Air Lance Height Test Series

<table>
<thead>
<tr>
<th>Pour Test Number</th>
<th>Air Lance Height Above Bottom of Riser (inches)</th>
<th>Average Thickness of Settled Layer at Bottom of Riser (pixels)</th>
</tr>
</thead>
<tbody>
<tr>
<td>21</td>
<td>3.25</td>
<td>38.0</td>
</tr>
<tr>
<td>22</td>
<td>3.25</td>
<td>37.0</td>
</tr>
<tr>
<td>23</td>
<td>3.25</td>
<td>38.7</td>
</tr>
<tr>
<td>24</td>
<td>1.5</td>
<td>40.3</td>
</tr>
<tr>
<td>25</td>
<td>1.5</td>
<td>38.7</td>
</tr>
<tr>
<td>26</td>
<td>1.5</td>
<td>40.7</td>
</tr>
<tr>
<td>27</td>
<td>1.5</td>
<td>40.0</td>
</tr>
<tr>
<td>28</td>
<td>3.25</td>
<td>42.3</td>
</tr>
</tbody>
</table>

The air lance was then lowered to 1.5 inches above the bottom of the riser and four pouring and idling cycles were run. Minor redistribution of the settled particles was observed after the first cycle with the air lance lowered, as shown in Figure 3-2. The subsequent pouring and idling cycles produced no visible changes in the settled layer. The particles did not appear to be effectively resuspended during pouring. Measurements of the settled layer thickness for these runs are included in Table 3-1. Differences are within the precision of the measurements.

A higher resolution camera was used to monitor the settled layer at the bottom of the riser during the last pouring and idling cycle with the air lance at 1.5 inches above the bottom of the riser. Images taken before and after the pouring and idling cycle are shown in Figure 3-3. Again, there appears to be little or no visible change in the settled layer thickness or distribution.
Figure 3-2. Appearance of the Settled Layer at the Bottom of the Riser (in rectangle) Before (a) and After (b) Operation with the Air Lance Lowered to 1.5 inches Above the Bottom
Figure 3-3. Higher Resolution Images of the Settled Layer at the Bottom of the Riser Before (a) and After (b) Operation with the Air Lance Lowered to 1.5 inches Above the Bottom
A final pouring and idling cycle was run with the air lance returned to the nominal height of 3.25 inches above the bottom of the riser. The results were consistent in that there was only a minor redistribution of the settled layer of particles, as shown in Figure 3-4. Measurement of the settled layer thickness is included in Table 3-1.

This series of tests demonstrated that lowering the air lance has minimal impact on the layer of settled particles; e.g., shear stresses were insufficient to entrain the settled solids. Some degree of redistribution of the settled bed occurred, but little if any resuspension was achieved. It is possible that lowering the air lance further would be more effective in resuspending the settled particles; however, this would have to be balanced with the risk of refractory erosion. In the actual melter, settled spinel crystals may interact with each other and the surrounding refractory, increasing the difficulty of resuspension.
Figure 3-4. Appearance of the Settled Layer at the Bottom of the Riser (in rectangle) Before (a) and After (b) Operation with the Air Lance Raised to 3.25 inches Above the Bottom
3.3 Particle Size Distribution Measurements

A sample of the settled particles at the bottom of the riser was collected after approximately 15 pouring and idling cycles had been completed with the system.\textsuperscript{a} Isopar-L\textsuperscript{b} was used to dilute the sample, since the silicone oil would have been too viscous for the instrument used to measure particle size distribution. Isopar-L was added to the magnetite and silicone oil at a ratio of about 3:1 by volume. This reduced the viscosity to an acceptable range based on visual observation.

The particle size distributions of the sample from the bottom of the riser, and a sample of unused magnetite particles prepared as described in an earlier report,\textsuperscript{7} were measured using a laser diffraction analyzer (Microtrac S3000, Montgomeryville, PA). The results of the analyses are plotted in Figure 3-5. The data show no discernable differences in the volumetric particle size distribution between the original, unused magnetite particles and the settled material collected from the bottom of the riser.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{particle_size_distributions.png}
\caption{Particle Size Distributions of the Original Magnetite Particles and the Particles Collected from the Bottom of the Riser}
\end{figure}

3.4 Fluid Flow Monitoring

The use of transparent materials in constructing the test system allows for direct observation of particle motion during pouring cycles and idle periods. Potential quantification of this motion requires a method for tracking via real time and time-lapse video recordings. The silicone oil is transparent and the magnetite particles are relatively small. Therefore, additional particles were added to the system to serve as reference points, or tracers, to monitor fluid motion. Two types of commercially available spherical particles were selected and added to the system for proof of concept testing. White polypropylene spheres\textsuperscript{c} with diameters of 2400 – 2500 µm and a density of 0.9 g/cm\textsuperscript{3} were used in the first test. Green polyethylene microspheres\textsuperscript{d} with a diameters of 850 – 1000 µm and a density of 0.98 g/cm\textsuperscript{3} were used in the second test.

\textsuperscript{a} The sample was pulled in October 2017, prior to the testing described in this report.
\textsuperscript{b} CAS No. 64742-48-9
\textsuperscript{c} Part number PPS-WHT-09 2.45±0.055µm-100, Cospheric LLC, Santa Barbara, California
\textsuperscript{d} Part number GPMS-0.98 850-1000µm – 10 g, Cospheric LLC, Santa Barbara, California
For each of the two tests, the tracer spheres were added gradually by hand to the top of the feed tank over a period of about 30 minutes. The feed tank agitator and recycle pump were started and operated during the addition period, and continued to operate for at least another 30 minutes to uniformly distribute the spheres throughout the tank. After the additions, a short pouring cycle was started. Pouring was terminated once the tracer spheres were observed to have passed into the riser and throat. Time-lapse recording was started at the completion of the pouring cycles and continued for about 24 hours. Motion of the tracer spheres was observed by reviewing the time-lapse recordings.

The tracer spheres were selected from a commercial vendor based on their size (to provide visibility in video recordings), color (to provide contrast in video recordings), and density (to match that of the silicone oil). The first test utilized spheres that had the advantages of being relatively large and white, to provide for good visibility in video recordings, but were less dense relative to the silicone oil (0.9 g/cm³ vs. ~0.975 g/cm³). Frames from the start and finish of the time-lapse video recording are shown in Figure 3-6. The frame from the start of the time-lapse recording (the start of the idle period) shows three of the white spheres in the throat, highlighted by red circles. The spheres had all floated to the top of the throat after about 8 hours of the idle period, as shown in Figure 3-6. The density difference between these spheres and the silicone oil, the size of the spheres, and the viscosity of the silicone oil resulted in a rise velocity that was too great such that these spheres were not suitable as tracers.

Figure 3-7 shows similar frames of the time-lapse video recording after the addition of the green spheres. The green spheres are smaller and their color provides less contrast in the video recordings, but their density is closer to that of the silicone oil (0.98 g/cm³ vs. ~0.975 g/cm³). The green spheres were observed to follow the flow of the silicone oil and magnetite particles during the idle period. They remained well dispersed within the system after about 8 hours of the idle period, as shown in Figure 3-7.

Although it was not attempted as part of this proof-of-concept test, image analysis software could be used to determine position data as a function of time for each of the tracers during idling of the system. Measurements during a pouring cycle would likely be difficult given the highly dynamic nature of the system while pouring. Position vs. time data could then be used to validate numerical simulations of particle flow and accumulation within the full-scale melter. A limitation of this approach is that position data are currently provided in only two dimensions. Additional cameras would be needed to determine complete three-dimensional locations of the tracers.
Figure 3-6. Photos of white tracers in the throat immediately after a pouring cycle (top) and after 8 hours of idling (bottom). Tracers are highlighted by red circles and ovals.
Figure 3-7. Photos of green tracers in the throat immediately after a pouring cycle (top) and after 8 hours of idling (bottom). Tracers are highlighted by red circles.

3.5 Increase in Settled Layer Thickness
The thickness of the settled particle layer at the bottom of the riser was intentionally built up to determine whether measurable differences in pouring parameters could be observed, as well as to monitor redistribution or resuspension of the settled particles.
To prepare for this test, a method was developed for direct addition of magnetite particles to the bottom of the riser. Unused magnetite particles, prepared as described in an earlier report, were blended with enough silicone oil to produce a pourable mixture. A transparent tube with an outside diameter of 0.75 inches and an inside diameter of 0.5 inches was inserted from the top of the riser down to approximately 2 inches above the bottom of the riser. The mixture of additional magnetite particles and silicone oil was poured into the top of the tube and allowed to settle to the bottom of the riser. The mass of magnetite particles added via this method is shown in Table 3-2.

Table 3-2. Masses of Magnetite Particles Added to the Riser to Increase Settled Layer Thickness

<table>
<thead>
<tr>
<th>Addition Number</th>
<th>Mass of Magnetite Particles added (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>118.9</td>
</tr>
<tr>
<td>2</td>
<td>29.3</td>
</tr>
<tr>
<td>3</td>
<td>37.7</td>
</tr>
<tr>
<td>4</td>
<td>29.7</td>
</tr>
<tr>
<td>5</td>
<td>40.5</td>
</tr>
<tr>
<td>6</td>
<td>30.3</td>
</tr>
</tbody>
</table>

During Addition Number 1, visual observations during the settling of the added particles revealed that a significant quantity of the particles flowed past the layer at the bottom of the riser and down into the throat. To reduce this effect, a narrower transparent tube (0.5 inch outside diameter and 0.375 inch inside diameter) was used for subsequent additions. The tube was placed further into the riser, such that the bottom of the tube was approximately 1 inch above the bottom of the riser, on the side opposite the throat. The mass of particles added in each addition was also reduced in an attempt to minimize any inertia effect and subsequent flow into the throat. Further particle additions using this configuration were observed to minimize the flow into the throat. The masses of the additional particle additions are listed in Table 3-2. The total mass of magnetite particles added increased the particle loading in the overall system from about 0.10 vol % to about 0.15 vol %. Images of the bottom of the riser before and after the series of particle additions are shown in Figure 3-8. The increase in thickness of the settled layer is obvious, with the layer being thicker on the left side of the image where the transparent tube was located.
A pouring and idling cycle was run after the particle additions were completed. A visual comparison of the settled layer at the bottom of the riser before and immediately after pouring revealed little change (Figure 3-9), although the particles suspended in the oil immediately after pouring obscure visibility (Figure 3-9b). A second view of the settled layer before and immediately after pouring is given in Figure 3-10. The spacing between the white lines is the same in each image in this figure. Again, there is little visible change in the appearance of the settled layer, with the exception of some build-up on left side of image. This build-up is no longer visible after about 24 hours of idling (Figure 3-11, where the spacing between the white lines is the same in each image), which may indicate that the build-up was an artifact of flow during pouring rather than a redistribution of the particles.
Figure 3-9. Appearance of the Intentional Buildup of the Settled Layer
Before (a) and immediately after (b) Pouring; Top of Settled Layer is Highlighted
Figure 3-10. Settled Layer Thickness Before (a) and Immediately After Pouring (b)
Figure 3-11. Photos of the Built Up Layer Before the Pouring and Idling Cycle (a) and After About 24 Hours of Idling (b)
Pouring cycle data for the test with intentionally built up solids were reviewed and compared with those from earlier tests to identify any measurable differences. Data for Test 31, where the layer of settled solids was intentionally built up, are compared in Table 3-3 with data from other recent tests where the air lance height was set to the nominal 3.25 inches above the bottom of the riser. As shown in the table, the air flow rate and pressure needed to maintain the targeted pour rate overlap within the uncertainty of the measurements. The viscosity of the silicone oil was somewhat higher for Test 31 due to the laboratory being cooler relative to the previous tests. This does not appear to have had a significant effect on the pouring parameters. Overall, increasing the thickness of the settled layer does not appear to have impacted the pouring parameters measured in this experiment.

Table 3-3. Measured Operation Parameters for Pouring Cycles with Air Lance at Nominal Height

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Average Pour Rate (lbs/min)</th>
<th>Average Air Flow Rate (slpm)</th>
<th>Average Air Pressure (psig)</th>
<th>Average Silicone Oil Temperature (°C)</th>
<th>Calculated Silicone Oil Viscosity (cP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>31*</td>
<td>6.84 +/- 0.27</td>
<td>35.2 +/- 2.2</td>
<td>21.92 +/- 1.44</td>
<td>22.1</td>
<td>5866</td>
</tr>
<tr>
<td>28</td>
<td>6.76 +/- 0.26</td>
<td>34.5 +/- 1.6</td>
<td>21.60 +/- 1.63</td>
<td>23.7</td>
<td>5700</td>
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<tr>
<td>23</td>
<td>6.71 +/- 0.12</td>
<td>34.0 +/- 0.1</td>
<td>21.62 +/- 1.67</td>
<td>24.5</td>
<td>5608</td>
</tr>
<tr>
<td>22</td>
<td>6.60 +/- 1.60</td>
<td>33.7 +/- 2.1</td>
<td>21.60 +/- 1.54</td>
<td>23.8</td>
<td>5688</td>
</tr>
<tr>
<td>21</td>
<td>6.86 +/- 0.43</td>
<td>32.9 +/- 3.9</td>
<td>21.61 +/- 1.48</td>
<td>24.1</td>
<td>5654</td>
</tr>
</tbody>
</table>

*Test 31 was run after intentionally increasing the thickness of the settled layer at the bottom of the riser.

Video recordings of the pouring cycle after building up the settled layer at the bottom of the riser were further reviewed to identify any differences relative to previous tests. Observation of the settled layer at the bottom of the throat revealed that this material did not appear to become resuspended during pouring, as it had in previous tests. An example of particle resuspension in this area during Test 28 is shown in Figure 3-12. The white lines are spaced equally in each photo. Note that the area covered by the settled solids is smaller after the pouring cycle (Figure 3-12b). Figure 3-13 shows similar photos before and after pouring when the layer of settled solids at the bottom of the riser had been intentionally built up. Note that no resuspension of the settled layer in the throat is visible. This may imply that, for the full-scale WTP HLW melter, the ability to resuspend settled particles will be reduced as the thickness of the settled layers is increased.

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*a The +/- uncertainty values are the standard deviation of the data over the duration of the steady-state portion of the test.
Figure 3-12. Photos of the Settled Layer Along the Bottom of the Throat Before (a) and After (b) an Earlier Pouring Cycle
Figure 3-13. Photos of the Settled Layer Along the Bottom of the Throat After Intentionally Increasing the Thickness of the Settled Layer Before (a) and After (b) an Earlier Pouring Cycle
A hydraulic calculation was performed to determine the pressure drop through the throat as settling particles continue to build up during periods of low or no flow conditions (Figure 3-14). The calculation assumed that the particles settle uniformly throughout the length of the throat, and ignored the elevation difference between the inlet and outlet of the throat. During flow conditions, the settled particles were assumed not to be resuspended and the particles in suspension were assumed not to settle. The particles in the fluid were assumed not to impact the hydraulic calculation, given the low solids fraction in the system.

![Figure 3-14. Cross-sectional Area of Throat Filling with Solids (Shown as y)](image)

For a flow rate of 3.18 L/min (0.84 gpm), throat length of 54 cm (21.2 inches), and an assumed glass density of 2.5 g/cm³, the pressure drop was calculated for various fluid viscosity values representing molten glass at various temperatures: 50, 500, and 1700 Poise. The results of the hydraulic calculations are plotted in Figure 3-15. In all cases, the flow was found to be laminar. As the viscosity of the fluid (glass) increases, the pressure drop increases and the solids fill height where the pressure drop starts to sharply increase occurs earlier. The results show that the amount of blockage would have to be higher than what was tested with the room temperature system in order to create measurable changes in the airflows needed for pouring. The examples provided in Figure 3-15, coupled with air lance operating data, could potentially be used to assess whether the throat of the actual melter is beginning to experience significant blockage.
4.0 **Summary**

The sets of experiments described in this report had four sets of objectives:

- Determine whether lowering the height of the air lance in the riser can be an effective means of removing accumulated particles
- Evaluate the size distribution of the accumulated particles
- Demonstrate that fluid and particle flows can be monitored via the addition of tracer materials
- Determine whether intentionally increasing the thickness of the settled particle layer leads to measurable differences in pouring parameters

Minor redistribution of the settled particles was observed after the first cycle with the air lance lowered. The subsequent pouring and idling cycles produced no visible changes in the settled layer. The particles did not appear to be effectively resuspended during pouring, demonstrating that lowering the air lance from 3.25 inches to 1.5 inches above the bottom of the riser has little impact on the layer of settled particles. This indicates that the air lift did not generate sufficient stresses or velocities to resuspend the settled solids. It is possible that lowering the air lance further would be more effective in resuspending the settled particles; however, this would have to be balanced with the risk of refractory erosion.

A sample of the settled particles at the bottom of the riser was collected after approximately 15 pouring and idling cycles had been completed with the system, for comparison with the particle size distribution of the original, unused magnetite particles. Isopar-L was used to dilute the sample, since the silicone oil would have been too viscous for the instrument used to measure particle size distribution. The resulting data showed no discernable differences in the volumetric particle size distribution between the original, unused magnetite particles and the settled material collected from the bottom of the riser.

The use of transparent materials in constructing the test system allows for direct observation of particle and fluid motion, although the silicone oil is transparent and the magnetite particles are relatively small.
Density-matched, polymer spheres were added to the system to serve as reference points, or tracers, to monitor fluid motion. The spheres were observed to follow the flow of the silicone oil and magnetite particles during the pouring and idle cycles. Image analysis software could be used to determine position data as a function of time for each of the tracers during idling of the system. Measurements during a pouring cycle would likely be difficult given the highly dynamic nature of the system while pouring. Position vs. time data could then be used to validate numerical simulations of particle flow and accumulation within the full-scale melter. Additional cameras are needed to determine complete three-dimensional locations of the tracers.

A method was developed for direct addition of additional magnetite particles to the bottom of the riser to determine whether measurable differences in pouring parameters would occur with a larger blockage. A pouring and idling cycle was run after the particle additions were completed. A visual comparison of the appearance of the settled layer at the bottom of the riser before and immediately after pouring revealed little change. Pouring cycle data showed that the air flow rate and pressure needed to maintain the targeted pour rate overlapped within the uncertainty of the measurements. Overall, intentionally increasing the thickness of the settled layer did not appear to impact the pouring parameters measured in this experiment. Calculations showed that a more significant blockage would be needed before measurable differences in pouring parameters occurred. Observation of the settled layer at the bottom of the throat revealed that this material did not appear to become resuspended during pouring, as it had in previous tests. This may imply that, for the full-scale WTP HLW melter, the ability to resuspend settled particles as the settled layer thickens is minimal since the condition of flow is laminar throughout the reduced flow area. Future testing should consider how throat reduction can be determined using the feedback from operation of the air lift pouring system. The ability to assess throat reduction would provide the facility with a means to determine whether melter replacement will be required. Testing should be coupled with flow calculations and simulations, a means to sufficiently control pressure drop in the test system, and use of various viscosity fluids at room temperature.
5.0 References


Appendix A  Compilation of Run Parameters and Accumulated Layer Thickness Measurements
## Purge Air

**High rate with lance at 1.5". Feed agitator on overnight.

**Comments**

Randolph2 is small randolph. Used Randolph1 for make-up.

**CAPT-1005-021016**  N/A  37  35  6.8  0.  84  16.7  17.56  106.60  3.25  25 Hz  50  20  28 N/A New make-up tank agitator motor with VFD (speed in Hz).

Randolph2 is small randolph. Used Randolph1 for make-up.

**CAPT-1320-042816**  96  25  10 - - 25.4  60.  07  131.63  3.25  25 ? ? 25 ? N/A

**1/28/2016**  **CAPT-1015-012816**  N/A  38  38  7.4  0.91  22.5  9.26  67.77  3.25  60  55  30  30  30 N/A New make-up tank agitator motor.

Particle loading increased to 0.15 vol%.

**CAPT-1500-120815**  Pour test 2  12/9/2015  CA  P T-1450-120915  N/A  70  10  3.4  0.4  22.3  3.25  0  0  ? 30 N/A ?

Pour test 4  12/17/2015

Pour test 7  1/7/2016  **CAP T-0927-010716**  N/A  45  45  7.3  0.89  20  3.25  0  0  50  0  30 N/A

**Test Date**  **DAS Filename**

- Oil Only
- Randolph2
- Wilden

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<th>Speed (%)</th>
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<td>45</td>
<td>7.3</td>
<td>0.89</td>
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**High rate with lance at 3.25". Running with tracers, white spheres.**

**High rate with lance at 3.25". Running with tracers, green spheres.**

**High rate with lance at 3.25". Running with tracers, white & green spheres.**

**High rate with lance at 3.25". Running with tracers, black, white & green spheres.**
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<th>Date</th>
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<th>Start time</th>
<th>Finish time</th>
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<td>1:15:15</td>
<td>1:15:15</td>
<td>0.84 gpm</td>
</tr>
</tbody>
</table>

**Notes:**
- Flow rate (cP) is measured in centipoise (cP).
- Time (minutes) refers to the duration of the experiment.
- Change (%): indicates the percentage change in the measured parameter.
Distribution:
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