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# Slurry Sampling in a Radioactive Waste Vitrification Facility (U)

by
J. L. Steimke
Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

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# Slurry Sampling in a Radioactive Waste Vitrification Facility

John L. Steimke, Westinghouse Savannah River Co., Aiken SC 29808

## Abstract

The U.S. Department of Energy plans to vitrify millions of liters of high level radioactive waste at the Savannah River Site (SRS) in South Carolina. A slurry of radioactive sludge, water containing dissolved salts and glass frit will be processed in 40,000 liter tanks and pumped to a glass melting furnace where the water will be vaporized and organic compounds decomposed. The remaining molten radioactive glass will be cast in canisters and eventually placed in long term storage. Two problems were observed during testing of the vitrification process using a non-radioactive slurry simulant. First, the stream of slurry pumped to the glass melter contained less glass frit than the slurry in the feed tank from which it originated. Second, the method of collecting small samples of slurry from the tanks to monitor the weight ratio of glass frit to sludge was found to produce biased samples. In response, test facilities that duplicated the hardware for the two situations were built and tested. The cause for the first sampling problem was that the slurry stream to the melter was drawn off vertically upward as a small side stream from a larger pipe. Because of inertial and gravitational effects the relatively dense glass frit was depleted in the side stream. Inertial effects also caused the second sampling problem. There was a projection into a slurry flow in a pipe at the point of drawoff which caused an enrichment of glass frit. In addition, the sampling velocity was insufficient. Hardware changes were developed that solved both problems. The changes have been installed at the Vitrification Facility.

## Literature survey

Nasr-El-Din and coworkers wrote a series of papers on tests of sampling using sand and water slurries. Nasr-El-Din, et al. [1984] described testing of a sharp-tipped isokinetic probe positioned in the middle of a pipe, facing into the flow. They found that the most accurate sampling of the sand concentration in the pipe occurred when the liquid velocity drawn into the probe matched the local velocity in the pipe. Drawing liquid into the probe more slowly resulted in a higher concentration of sand being drawn through the probe and vice versa. Nasr-El-Din, et al. [1985] described testing of side wall samplers consisting of holes of different sizes in the wall of a vertical pipe having upward flow. Four conclusions can be drawn from their data. First, their side wall sampler always drew in a lower concentration of sand than was flowing in the pipe. Second, sampling was more accurate for fine sand (0.165 mm) than coarse sand (0.72 mm), other conditions remaining the same. Third, sampling was more accurate when slurry velocities at the sampling hole were equal to the slurry velocities in the pipe than when sampling velocities were significantly lower. Fourth, the size of the sampling hole was important because sampling was more accurate for an 8 mm hole than for a 3 mm hole, the velocities remaining the same. They also performed tests with a projection into the main flow stream immediately downstream of the hole for the side wall sampler. The projection caused the side stream to be significantly enriched in sand for sampling velocities less than 40% of the main stream velocity. Sampling became more accurate as the sampling velocity increased. Nasr-El-Din, et al. [1989] described testing of slurry flow through a T-junction where the approach flow was horizontal. They found that the sand concentration in the branch of the junction was less than the supply sand concentration when the branch pointed up and greater when the branch pointed down. This will be referred to as the gravity effect.

## Test Facilities and Procedure

Two test facilities were built for this work. The first was the Melter Feed Facility [Steimke, 1994a] which duplicated the hardware that is used to pump slurry from the slurry storage tank to the glass melter in the Vitrification Facility. It was later rebuilt to form the Sampling Valve Facility [Steimke, 1995] which duplicated the hardware used to collect samples from the slurry storage tank. Figure 1 is a schematic of the Melter Feed Facility which consists of two hydraulic loops: the recirculation loop and the feed loop. Slurry consisted of the simulated waste sludge, glass frit (0.15 mm), and water. The slurry had the rheology of a Bingham plastic. Depending on the amount of water in the slurry the consistency and yield stress varied from 10 cp and 70 dynes/cm$^2$ to 35 cp and 240 dynes/cm$^2$. The slurry was placed in the mechanically agitated reservoir. Slurry was drawn at about 300 L/min through two centrifugal pumps located above the reservoir, around the 13 meter long 2" (51 mm) recirculation loop, through a magnetic flowmeter, a 25 mm diameter flow restrictor, and

back to the reservoir. A vacuum pump was used to pull the slurry up to prime the pumps. Once slurry had been pulled into the pumps, the vacuum line was valved off and one or both pumps started.

The flow restrictor in the recirculation loop generated the pressure which drove about one percent of the slurry flowing in the recirculation line through a strainer and into the 12 meter long 12.7 mm I.D. feed line. A funnel located at the end of the feed line where the actual glass melter would have been collected the slurry for return to the slurry reservoir. The feed strainer was located in the top of a horizontal section of the recirculation line and consisted of 26 holes of 2.1 mm diameter bored through the top surface of the pipe. The pump speed on the recirculation loop was adjusted to set the slurry flow in the feed line which was measured using a magnetic flow meter.

The Sampling Valve Facility, Figure 2, used the tank, pumps and agitator from the Melter Feed Facility. Slurry from the Melter Feed Test and a somewhat different type of slurry were used at different times during testing. Approximately 150 L/min of slurry were drawn through pumps, through a magnetic flowmeter and around a recirculation loop. A 12.7 mm flow restrictor positioned just before the slurry flowed back into the bottom of the tank served to increase the pressure in the recirculation loop.

A transfer line (15.7 mm I.D.) was attached at a 45° angle to a vertical section of recirculation line which drew off a flow of slurry in the range from 15 L/min to 38 L/min. The sampling valve was positioned 12 meters downstream in the transfer line using a standard 3/4" Swagelok ® tee. The sampling valve is shown in Figure 3 and was manufactured by Hinds International, Inc. The valve had a plunger which resembled a valve on an internal combustion engine. Normally the outer surface of the plunger was nearly flush with the inside of the 3/4" (19.1 mm) tubing and no flow passed through the valve as shown, labeled original valve closed . When a sample was desired the plunger was pushed forward 6.4 mm so that it projected into the flow stream in the 3/4" tubing. Slurry flowed past the plunger, in and out of the sample vial, out the overflow line and back to the slurry reservoir. To collect a sample the valve was held open for a period of time, either 7 and 30 seconds, then closed.

To run a test, steady state hydraulic and temperature conditions were established. For the Melter Feed Facility slurry samples were collected from the reservoir and the end of the melter feed line. For the Sampling Valve Facility, samples were collected from the reservoir, the sample vials and the overflow from the sample vials. Reservoir slurry samples were collected with a Composite Liquid Waste Sampler (coliwasa) [de Vera, 1990], which is a four foot long glass tube with a valve at the lower end. It was inserted into the reservoir with the valve open. The valve was closed and the coliwasa withdrawn. The contents were drained into a flask. Careful density measurements were made of the slurry samples. Flows, pressures and temperatures were recorded on a data acquisition system.

## Relationship Between Density, Li/Fe and Elemental Composition of Slurry

It was necessary to determine how accurately the slurry collected by the melter feed line or the slurry collected by the sampling valve matched the slurry in the slurry reservoir. This was determined by comparing densities of slurry samples collected at the same time from the slurry reservoir and either flowing through the Melter Feed Line or flowing through the sampling valve. The method takes advantage of the fact that the glass frit is twice as dense as the rest of the slurry. This comparison was only valid when no dilution of slurry or significant evaporation of water from slurry occurred between the collection of samples to be compared. In addition to the density measurements, elemental chemical analyses were performed for some of the samples to determine the relationship between changes in density and changes in the proportion of glass frit to sludge. Lithium (Li) was the tracer for glass frit and iron (Fe) was the tracer for sludge. In principle, the accuracy of sampling could have been tracked using only elemental analyses but that method would have been too slow and expensive. Figure 4 shows the relationship between changes in density and changes in Li/Fe. Each point plotted was computed from the results of ten elemental analyses. A change of 1% in density is proportional to a change of 9% in Li/Fe. It was concluded that measurement of changes in density was a fast, reliable method to infer changes in Li/Fe for these experiments.

## Melter Feed Facility Tests

The Melter Feed Facility was run with the original side wall feed strainer hardware, for feed flows of approximately 0.9 L/min, 1.7 L/min and 3.4 L/min, three different slurry dilution factors and slurry temperatures of 30°C or 40°C. Ratios of the feed line and reservoir densities are plotted in Figure 5 versus velocity ratio, defined as the slurry velocity in the strainer holes divided by the slurry velocity in the recirculation line. A density ratio of one indicates perfect sampling. The areas of the pipe and the strainer were 88.6 $mm^2$ and 1905 $mm^2$, respectively. With the original 26 hole feed strainer the density ratio was always less than one, referred to as frit depletion, but increased with increasing velocity ratio. The right hand ordinate shows the inferred ratios of Li/Fe for the slurry in the feed line and the reservoir, based on the changes in Li/Fe being nine times as large as the changes in density. The observed percentage differences in Li/Fe were significant in the vitrification process.

The data of Nasr-El-Din et al., [1985] for side wall sampling in the absence of a protrusion and a sand diameter of 0.165 mm are also plotted in Figure 5, although the ordinate for those data is the ratio of sand concentrations in the side stream and the main stream. The trends in the Nasr-El-Din study and the Melter Feed test were the same although the slurries and internal dimensions were different.

Based on the literature survey it was anticipated that the observed frit depletion was occurring at the feed strainer. There were two possible mechanisms. The first mechanism was the gravity effect mentioned by Nasr-El-Din, et al. [1989]. In the present study relatively dense glass frit had to turn upward to enter the strainer. The first mechanism was checked by first rotating the original strainer 90° so that the frit was drawn off horizontally, then rotating the strainer an additional 90° so that the frit was drawn off downward. These two changes had only a small effect on the accuracy of slurry sampling. Therefore, it was concluded that the gravity effect was not the primary effect.

The second mechanism, the inertial effect, was also investigated by Nasr-El-Din, et al. [1985]. In the present study the relatively dense frit moving along the recirculation pipe had to make a sharp 90° turn into 26 small holes to enter the strainer. The inertial effect was investigated by replacing the 26 small holes with one larger hole having the same area which was expected to improve sampling. Also, the single hole was drilled at a 45° angle to the recirculation line so that the frit had to turn less to enter the hole. This change greatly decreased frit depletion and indicated that inertia was the dominant effect. However, frit depletion was not eliminated. This is consistent with the Nasr-El-Din paper on side wall sampling [1985] in which some sand depletion was always observed for side wall sampling. In an effort to enrich the frit content and eliminate frit depletion the face of the strainer was rotated to partially face the oncoming slurry. Three thin bars oriented in the direction of flow were placed across the opening to reject lumps in the slurry. Different face angles were tested. The most accurate sampling was obtained using a strainer design having a face angle of 30° with the wall of the pipe as shown in Figure 6. Slurry density ratios for the modified strainer design are plotted in Figure 5. The average density ratio improved to 1.002, which indicated good sampling through the feed strainer. The inferred ratio for Li/Fe for the feed line and reservoir was 1.018 which satisfied the requirements of the vitrification process. There was an additional benefit with the modified strainer. The modified strainer showed no evidence of plugging, whereas five of the 26 holes of the original strainer plugged during testing.

## Sampling Valve Facility Tests

The Sampling Valve Facility was first run using the original sampling valve. The test matrix included two types of slurry, three slurry dilution factors, transfer flows of 15 L/min, 23 L/min, 30 L/min and 38 L/min and vial flows ranging from 1.5 L/min to 9 L/min. Most of the runs were done at 30°C. Slurry overflowing from the sampling valve was collected. Density was accurately measured for slurry samples.

The ratio of the densities of the slurry at the valve overflow and the slurry reservoir is plotted in Figure 7 as a function of velocity ratio, defined as the slurry velocity in the sampling valve divided by the velocity in the transfer line. The velocity in the valve was based on the area around the rod of the valve, 163 $mm^2$. The velocity in the transfer line was based on the flow area of the transfer line, 194.2 $mm^2$. Also plotted were data from Nasr-El-Din [1985] for side wall sampling just upstream of a protrusion in the flow. The Nasr-El-Din sand was larger, 0.325 mm, than the glass frit in the present study, 0.15 mm, and the Nasr-El-Din liquid phase was water, rather than the more viscous simulated sludge in the present study. Therefore, the sampling errors were larger for the Nasr-El-Din study. The two sets of data were plotted using different vertical coordinate axes to allow a qualitative comparison of trends. No quantitative comparisons are made. For both data sets the sampled stream was enriched in solids for velocity ratios less than 0.4. Sampling accuracy generally improved as the velocity ratio increased. The accuracy of sampling for Nasr-El-Din was good for velocity ratios above 0.7. The largest velocity ratio tested in the present study was 0.55. The largest density ratio observed in the present study was 1.023. This corresponds to a Li/Fe ratio for the overflow and reservoir of 1.21 which was unacceptable for the vitrification process.

Tests of the original sampler showed that the slurry drawn through the sampling valve was enriched in frit. This agreed with the trend of the Nasr-El-Din results for side wall sampling with a projection. Therefore, it was decided to modify the sampler to accomplish two objectives, namely, to remove the projection into the transfer line and to increase the velocity ratio without the necessity of increasing the volumetric flow of slurry drawn through the sampler. The modification was to lengthen the side arm of the tee in Figure 3 by 6.4 mm. Therefore, when the plunger was in the closed position its flat face was recessed from the inner diameter of the transfer line by 6.4 mm. To open the valve the plunger was moved forward by 6.4 mm, flush with the inner diameter. Thus the projection into the flow was removed. However, this changed the area and also velocity for sampling. Sampled slurry had to flow though an annulus around the head of the plunger, an area of 49 $mm^2$ or one-third the original area. Therefore, the normalized velocity was three times larger for the same

volumetric slurry flow. The overall hydraulic resistance of the modified sampling valve was only slightly greater than for the original valve because most of the pressure drop was in the vial.

As was the case for the testing of the original sampling valve, the primary method for tracking performance was the use of slurry density. Slurry density ratios for the modified sampling valve are also plotted in Figure 7. Sampling accuracy improved greatly and was nearly independent of velocity ratio. The maximum divergence in density ratio from unity decreased by a factor of more than four. The average density ratio for all of the data points for the modified sampler was 0.99998, implying essentially perfect sampling on average. The modified sampling valve drew an accurate sample for all vial flows, transfer flows, both types of slurry and different slurry dilutions.

## Summary and Conclusion

Two test facilities were constructed and tested for the purpose of improving slurry sampling at the SRS Vitrification Facility. In both cases the original hardware gave unacceptably inaccurate sampling. For the Melter Feed Facility the slurry stream sent to the melter contained too little glass frit. For the Sampling Valve Facility the sampled stream usually contained too much glass frit. Simple and easy to implement hardware changes were made in both test facilities that took into account results from similar experiments reported in the open literature. Tests were conducted over the full range of slurry rheologies and velocities expected. The hardware changes greatly improved the accuracy of sampling in both test facilities. Shortly after the conclusion of testing the same two hardware changes were installed at the Vitrification Facility.

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## References

deVera, E. R., Simmons, B. P., Stephens, R. D. and Storm, D. L., "Samplers and Sampling Procedures for Hazardous Waste Streams", EPA-600/2-80-018, 1980.

Nasr-El-Din, H., Afacan, A., Masliyah, J. H., "Solids Segregation in a Slurry Flow Through a T-Junction with a Horizontal Approach", **Int. J. Multiphase Flow**, Vol. 15, pp. 659-671, 1989.

Nasr-El-Din, H, Shook, C. A., and Esmail, M. N., "Wall Sampling in Slurry Systems", **Can. J. Chem. Eng.**, Vol. 63, pp. 746-753, 1985.

Nasr-El-Din, H, Shook, C. A., and Esmail, M. N., "Isokinetic Probe Sampling from Slurry Pipelines", **Can. J. Chem. Eng.**, Vol. 62, pp. 179-185, 1984.

Steimke, J. L., "Results of Tests of TFL Melter Feed Loop", WSRC-TR-94-0570, November 1994.

Steimke, J. L., "Results of Tests of TFL Hydragard Loop", WSRC-TR-94-0598, February 1995.