Statistical Evaluation of Small Scale Mixing Demonstration Sampling and Batch Transfer Performance – 12093

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ABSTRACT

The ability to effectively mix, sample, certify, and deliver consistent batches of High Level Waste (HLW) feed from the Hanford Double Shell Tanks (DST) to the Waste Treatment and Immobilization Plant (WTP) presents a significant mission risk with potential to impact mission length and the quantity of HLW glass produced. DOE's Tank Operations Contractor, Washington River Protection Solutions (WRPS) has previously presented the results of mixing performance in two different sizes of small scale DSTs to support scale up estimates of full scale DST mixing performance [1]. Currently, sufficient sampling of DSTs is one of the largest programmatic risks that could prevent timely delivery of high level waste to the WTP. WRPS has performed small scale mixing and sampling demonstrations to study the ability to sufficiently sample the tanks. The statistical evaluation of the demonstration results which lead to the conclusion that the two scales of small DST are behaving similarly and that full scale performance is predictable will be presented. This work is essential to reduce the risk of requiring a new dedicated feed sampling facility and will guide future optimization work to ensure the waste feed delivery mission will be accomplished successfully.

This paper will focus on the analytical data collected from mixing, sampling, and batch transfer testing from the small scale mixing demonstration tanks and how those data are being interpreted to begin to understand the relationship between samples taken prior to transfer and samples from the subsequent batches transferred. An overview of the types of data collected and examples of typical raw data will be provided. The paper will then discuss the processing and manipulation of the data which is necessary to begin evaluating sampling and batch transfer performance. This discussion will also include the evaluation of the analytical measurement capability with regard to the simulant material used in the demonstration tests. The paper will conclude with a discussion of the analysis results illustrating the relationship between the pre-transfer samples and the batch transfers, which support the recommendation regarding the need for a dedicated feed sampling facility.

INTRODUCTION

Hanford HLW will be staged in 3785-cubic meter (1-million gallon), underground DSTs prior to delivery to the WTP for treatment. HLW is a combination of liquid and undissolved solids that settle and form sludge in the bottom of the DSTs. The DSTs are approximately 23 meters (75 feet) in diameter and 12 meters (40 feet) high, with equipment access provided through risers located in the dome of the tank. The baseline design for transferring the HLW to the WTP for treatment includes using two 760,000 BTU/hr (300-horsepower) centrifugal mixer pumps with two opposed nozzles each to mobilize the sludge particles, and one submerged inlet centrifugal transfer pump to deliver the HLW slurry through pipelines to the WTP. The HLW feed certification and delivery strategy includes mixing and sampling the waste in a 3785- cubic meter (1- million gallon) staged DST, certifying it as compliant with WTP requirements, and then transferring multiple 475-cubic meter (125,000-gallon) batches to the WTP [2]. The level of

accuracy for certifying waste feed is still being finalized, but the feed must be shown to meet the regulatory, safety basis, and operational requirements within the defined tolerance band [3].

Traditional methods used in Hanford's tank farms to sample DSTs consist of individual grab sample (liquids) or core sample (settled solids) events while the tanks are quiescent (that is, not being mixed). However, these methods cannot provide a representative slurry sample of the waste that would be transferred to the WTP. Hanford's Waste Feed Delivery Mixing and Sampling Demonstration program is focused on identifying representative sampling techniques and demonstrating that consistent 475-cubic meter (125,000-gallon) transfers can be made from existing DSTs.

DEMONSTRATION PROGRAM

The Mixing and Sampling Demonstration Program is structured to define appropriate DST sampling techniques that result in representative samples, and to define batch transfer techniques that will deliver consistent batches of HLW to the WTP. The program builds on information gained from progressively larger and more complex small-scale mixing platforms that are scaled to match the full-size DST configuration. The mixing demonstration strategy is built around the following progressive concepts:

- Demonstrate DST mixing and batch transfer phenomena with small-scale "scouting studies"
- Develop small-scale mixing and batch transfer performance data using two different small-scale mixing platforms
- Demonstrate representative sampling capability on two different small-scale mixing platforms

The information gained from the small-scale demonstrations will be compared against emerging WTP data tolerance requirements. Once a functional sampling and batch transfer system is defined, full-scale performance will be confirmed with a full-scale demonstration of system capabilities in the DST designated for WTP commissioning. This document focuses on the evaluation of data collected from the small-scale mixing demonstration batch transfer testing performed from April through August, 2011.

SMALL SCALE DEMONSTRATIONS

Mixing Platform

The mixing and sampling demonstration program initially focuses on the first HLW planned for transfer to WTP, (from tank AY-102). Consequently, small-scale demonstration tanks were geometrically scaled by linear dimensions to match AY-102. AY-102, is approximately 23 meters (75 feet) in diameter, with an operating liquid height of 9.2 meters (364 inches) and a sludge (settled solids) height of 1.4 meters (55 inches). The particle size range of the solids in AY-102 ranges from 2.5 to 16.8 (99th percentile) microns, and density varies from 2.4 to 11.4 g/cm3 [3]. The baseline tank configuration will include two mixer pumps, with opposing 15 cm (6 inches) diameter nozzles that will circulate tank waste at approximately 315 liters per second (5000 gallons per minute) per nozzle. The mixer pumps can be rotated such that the nozzles cover a full 360° of rotation. AY-102 also contains 22 air lift circulators (ALCs) that are currently not functional. The ALCs are cylindrical obstructions in the tank, each 0.8 meters (30 inches) in diameter extending down to within 0.8 meters (30 inches) of the tank floor.

The Small Scale Mixing Demonstration (SSMD) platform contains two functionally equivalent tanks of approximately 1/20th and 1/8th scales. The smaller tank size of 1.1 meters (43 inches) diameter was selected to match the size of the scouting study tank at Savannah River National Laboratory (SRNL) that has been operating since March 2009 [4]. This size allows direct comparison of performance data between the two tanks. The larger tank size of 3.0 meters (120 inches) was selected to provide a noticeable scale difference (nearly three times) from the small tank while still remaining small enough to allow clear visual observation of mixing performance. The two scaled tanks and associated operating equipment are shown in Fig. 1. The small scale of the demonstration tanks made it impractical to mechanically duplicate the rotating, centrifugal mixer pumps found in the full scale DSTs. The scaled flow characteristics of the mixer pumps were duplicated in a similar manner to those used in the SRNL tank. A pump external to the tank provides the suction through a central column and mixer flow through annulus fed jet nozzles that were scaled appropriately to match the full-scale mixer pump configuration.



Fig. 1. Small Scale Mixing Demonstration (SSMD) Platform

Simple to complex particulate simulants were selected to represent a broad spectrum of potential mixing conditions and build correlations with the data collected at SRNL and the expected waste conditions in AY-102. The simulant used was a combination of Gibbsite $(Al(OH)_3)$, zirconium oxide (ZrO_2) , silicon carbide (SiC), bismuth oxide (Bi_2O_3) , and stainless steel (SS) with particle size distributions specifically selected to match potential tank waste characteristics [5]. The first phase of demonstrations was focused on mixing performance in the two tanks under varying conditions. The objective of the mixing phase was to understand mixing performance in the two scales to the extent necessary to provide confidence that both tanks can be operated under similar conditions. Once equivalent operating envelopes are defined for each tank, the second phase of demonstrations could begin. The second phase was focused on demonstrating batch transfer performance and the ability to collect representative samples. The primary objective of the batch transfer testing was to determine whether the samples taken from the recirculation loop prior to the actual transfers could be used to determine the composition of the material in the batch transfers. The sampling demonstration portion of the second phase

would be focused on how to obtain representative samples from the recirculation loop. The batch transfer demonstration performance will be discussed here.

Data Collection

The batch transfer performance testing consisted of a series of tests performed in the two scaled tanks (identified as TK201 and TK301) at specified mixer pump flow rates. See Table I for a list of the tests performed. Note that the nozzle velocity is calculated based on the flow rate and the pump configuration for each tank. The term velocity as used in the remainder of this document represents the fluid velocity as it emerges from the mixer pump nozzles.

Test ID	Tank	Flow rate (gpm)	Nozzle velocity (ft/sec)
23	TK201	6.5	16.9
24	TK201	8.5	22.1
25	TK301	70	22.3
26	TK301	90	28.7
27	TK201	10.6	27.6
28	TK201	10.6	27.6
29	TK201	9.5	24.7
30	TK301	111	35.4
31	TK201	10.6	27.6
34	TK301	100	31.9
37	TK201	10.6	27.6

Table I. Batch Transfer Test Runs

For each test, the simulant material was mixed in the tank for a prescribed time interval, and a small pre-transfer sample was taken from a recirculation loop in the mixing platform. This pretransfer sample was obtained by diverting the entire flow from the recirculation loop into a container for a short period of time. After obtaining the pre-transfer sample, five scale-sized batches were sequentially transferred from the mixing tank into temporary receipt containers. Analytical samples were obtained from the pre-transfer sample and each of the batch transfers. These analytical samples were then sent to a laboratory for analysis of the chemical composition. The results of the lab analysis were the dry weight fraction of each of the simulant components. Table II provides an example of the analytical lab data obtained. Note that multiple subsamples were obtained for lab analysis from each pre-transfer sample (identified as "FD" in the column Batch), as well as each batch transfer.

Test ID	Tank	Flow rate (gpm)	Nozzle velocity (ft/sec)	Batch	C_SiC	C_Bi2O3	C_ZrO2	C_AI(OH)3	C_SS
23	TK201	6.5	16.9	FD	0.014	0.009	0.588	0.365	0.023
23	TK201	6.5	16.9	FD	0.009	0.013	0.593	0.380	0.004
23	TK201	6.5	16.9	1	0.007	0.003	0.750	0.232	0.008
23	TK201	6.5	16.9	1	0.021	0.013	0.695	0.264	0.008
23	TK201	6.5	16.9	1	0.020	0.028	0.647	0.288	0.019
23	TK201	6.5	16.9	1	0.005	0.005	0.748	0.229	0.012

Table II. Example Analytical Lab Data

Test	Tank	Flow	Nozzle	Batch	C_SiC	C_Bi2O3	C_ZrO2	C_AI(OH)3	C_SS
ID		rate	velocity						
		(gpm)	(ft/sec)						
23	TK201	6.5	16.9	2	0.008	0.006	0.650	0.328	0.007
23	TK201	6.5	16.9	2	0.017	0.009	0.671	0.297	0.006
23	TK201	6.5	16.9	2	0.008	0.012	0.551	0.421	0.006
23	TK201	6.5	16.9	2	0.009	0.006	0.725	0.248	0.012
23	TK201	6.5	16.9	3	0.005	0.010	0.600	0.377	0.007
23	TK201	6.5	16.9	3	0.012	0.025	0.670	0.287	0.007
23	TK201	6.5	16.9	3	0.014	0.009	0.670	0.293	0.013
23	TK201	6.5	16.9	3	0.007	0.008	0.665	0.315	0.004
23	TK201	6.5	16.9	4	0.034	0.014	0.623	0.315	0.014
23	TK201	6.5	16.9	4	0.007	0.007	0.654	0.325	0.007
23	TK201	6.5	16.9	4	0.014	0.009	0.721	0.251	0.005
23	TK201	6.5	16.9	4	0.008	0.006	0.709	0.272	0.004
23	TK201	6.5	16.9	5	0.024	0.01	0.57	0.39	0.006
23	TK201	6.5	16.9	5	0.007	0.01	0.671	0.301	0.011
23	TK201	6.5	16.9	5	0.002	0.009	0.617	0.357	0.015
23	TK201	6.5	16.9	5	0.008	0.008	0.73	0.249	0.004

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DATA ANALYSIS

Although the primary focus of the data analysis was on understanding the relationship between the pre-transfer samples and the batch transfer samples, a number of different analyses were performed to better understand the quality of the data and different kinds of information that could be obtained from the data. The analyses shown were most helpful in supporting the overall decision-making process. All analyses shown here were produced using JMP statistical software [6].

Control charts for the variability of the constituent concentrations were used to better understand the inherent variability in the lab measurements, as well as to identify possible questionable observations. Fig. 2 shows the standard deviation (or S) control charts for the constituent concentrations.



Note: 19 samples were excluded

Fig. 2. Standard Deviation Control Charts for Concentrations

These control charts are based on the standard deviation of the concentration (or dry weight fraction) of the specified constituent obtained from each pre-transfer and batch transfer sample. The standard deviation is calculated from the multiple lab analysis samples which were obtained from each pre-transfer and batch transfer sample. In the case of the pre-transfer samples, there were two lab analysis samples obtained for each test run. For the batch transfers, some batches had two lab analysis samples while others had four lab analysis samples. This standard deviation represents the lab analytical variability as well as the subsampling variability.

Therefore, samples which have a larger standard deviation could be an indication of an error in the lab analysis, an error in the subsampling procedure, or inconsistency in the pre-transfer or batch transfer sample material. In the figure, there are numerous observations which are identified by circled points and an asterisk. These are values which have been identified as unusual, based on the control limits. Note that the majority of the unusual observations occurred earlier in the time sequence, suggesting that the general operations may be improving with experience. The control limits are calculated to represent the expected variability in the data. For the purposes of this analysis, the unusual observations have been identified for investigation and possible re-analysis by the lab. The intent is to determine whether there were identifiable problems associated with these values. If one assumes that all these unusual data have reasonable explanations, based on investigation, then the control chart limits shown provide an "ideal case" estimate of the expected variability in the calculations of the control limits. The estimates of expected variability are shown in Table III.

Table III. "Ideal Case" Constituent Standard Deviations

	SiC	Bi2O3	ZrO2	AI(OH)3	Stainless Steel
Standard deviation (weight %)	0.8%	0.3%	3.8%	3.8%	0.5%

One approach to understanding the relationship between the pre-transfer samples and the batch transfer samples is to examine the constituent concentrations in the samples, as measured by the dry weight fraction of the constituents. Fig. 3 provides a suite of graphs which plot the difference between the measured concentration of each constituent from the batch transfer sample and the pre-transfer sample on the vertical axis (labeled as D_C_X, where X represents the constituent name) against the batch transfer sequence number on the horizontal axis. The graphs shown in the left column represent the data from the small scale mixing tank (identified as tank "TK201"); those in the right column represent the data from the large scale mixing tank (identified as tank "TK301"). Different curves on the graphs represent the different flow rates used in the testing, as identified in the key on the right side.



Fig. 3. Difference in Concentration (Batch minus Pre-Transfer) by Batch, Tank, Flow Rate

There are several items of note in the graphs shown in Fig. 3. In general, the behavior displayed is similar in the two different scaled tanks. When looking at SiC, Bi_2O_3 , and SS, the differences shown tend to be negative, which indicates that the concentration in the pre-transfer sample tends to be higher than the concentration in the subsequent batch transfer samples. For the two constituents which comprise the bulk of the material, $AI(OH)_3$ and ZrO_2 , the concentrations in the pre-transfer sample on average tend to be very similar to the concentrations in the batch transfer samples.

Another approach to the analysis is looking at the relative difference in the mass of the constituents, similar to the concentrations shown previously. This is shown in Fig. 4.



Fig. 4. Difference in Mass (Batch minus Pre-Transfer) by Batch, Tank, Flow Rate

The data shown in Fig. 4 are the same format as that shown in Fig. 3; the difference is that the relative difference in the mass, rather than concentration, is shown in Fig. 4. Since the volume of the pre-transfer samples was different than the volume of the batch transfers, the mass in the pre-transfer sample needs to be "scaled-up" to the same volume as the batch transfers, based on the estimated volumes of both. The difference between the batch transfer mass and the scaled-up pre-transfer mass is then divided by the scaled-up pre-transfer mass to provide the relative difference shown on the vertical axes (labeled as D_M_X r%, where X represents the constituent name) in Fig. 4.

Inspecting the graphs in Fig. 4, it again appears that the behavior for most of the constituents is similar between the two different tanks; $AI(OH)_3$ appears to be somewhat different. Also, with the exception of $AI(OH)_3$, the relative differences in the constituent masses are generally negative, and trending generally downward with subsequent batches, suggesting that the

batches tend to transfer less of the constituent, on a relative basis, as the batch transfers progress.

Additional analyses were performed to better understand the relationship between the batch transfer performance and the mixer pump flow rate (or velocity) used in each tank. Since ultimately it will be necessary to estimate the mixer pump velocity which produces acceptable batch transfer performance at the full scale tank size, it is useful to understand how the batch transfer performance is related to the scale of the tanks being used. One model for describing the scaling relationship is given by:

— = — (Eq. 1)

where u_i represents the velocity in tank at scale i, T_i represents the diameter of tank at scale i, and *a* represents a scale factor exponent. While the exponent *a* may have a theoretical basis, it is also possible to estimate *a* empirically by identifying the corresponding velocities in two different scaled tanks that produce "equivalent" behavior.

In the case of batch transfer testing, one approach to defining equivalent behavior is to use the concentrations of the individual constituents for the pre-transfer samples and the batch transfer samples. Conceptually, when the concentrations between the two scaled tanks are "equivalent", the corresponding velocities for each scaled tank are then the appropriate velocities to be used to empirically determine the scaling exponent *a*. Once the scaling exponent is determined, the scaling model can then be used to identify the corresponding velocity at any scale which would produce "equivalent" behavior.

One difficulty in determining the corresponding velocities is having enough different velocities at the multiple scales to be able to identify behavior that is equivalent. Another difficulty in this particular approach of using the constituent concentrations to define equivalent behavior is the inherent constraint on the concentrations. Although there are five constituents, six when you also include the water, the sum of the concentrations must always equal 1 (or 100%). So there really aren't five (or six) independent values, and the analysis methodology needs to appropriately deal with this constraint.

To address the first difficulty, rather than make pair-wise comparisons between the various velocities used for the two scaled tanks, which is limited to the actual velocities used, a statistical model can be fit to the velocity data for each tank. This will then allow identification of the velocities for each tank that produce equivalent behavior, based on the model predictions. In addition, the form of the model used allows direct estimation of the scale exponent *a* which best fits the data.

In order to deal with the second difficulty, a log-ratio transformation of the concentration data is used [7]. This log-ratio transformation allows for the use of standard statistical analyses to be performed, as well as an inverse transformation back to the original concentrations. The actual equation used to perform the transformation is given by

 $_{-} = \log - - - (Eq. 2)$

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Where X represents the wet weight fraction of constituent X (SiC, Bi_2O_3 , ZrO_2 , Al(OH)₃, or SS) and *Water* represents the wet weight fraction of water. These five log-ratio variables are then used as the response variables for the model fitting, with the model given by:

$$= + \times \times \frac{120}{43.2} + \times \times \frac{120}{43.2} + \times \times \frac{120}{43.2} + \times \times \frac{120}{43.2} + \times h \qquad (Eq. 3)$$

$$301: = + \times + \times + \times + \times h$$

Where *a* and b_i represent the model coefficients to be estimated from the data, *u* represents the velocity, and *Batch* represents a numeric batch number to account for the decrease in concentration on successive batches observed previously. The values 120 and 43.2 represent the diameters of the large and small tanks, respectively. The general model form, which requires a nonlinear fitting routine, is essentially a cubic polynomial in velocity (chosen for simplicity), with the velocity in the small tank scaled by the exponent *a*, based on the scaling model.

Although the complete analysis results will not be shown here, Fig. 5 illustrates the results for the analysis of SiC. Similar results were obtained for the other constituents.



Fig. 5. Actual, Predicted Concentration of SiC vs Velocity, Scaled Velocity

The graph on the left side of Fig. 5 shows the measured concentration of SiC (open circles) and the predicted concentration of SiC (solid dots) versus the actual nozzle velocity tested. The graph on the right side of Fig. 5 shows the same values versus the scaled velocity, as determined from the fitted model. The scaled velocity for tank TK301 is the actual velocity. The scaled velocity for tank TK201 is calculated from Eq. 1, based on the actual velocity used in tank TK201 and the exponent *a* determined from the fitted model. Although there is still a fair amount of variability in the measured values, it also seems clear from the right hand graph that the concentration values are more "equivalent" than on the left hand graph. Based on this, it appears that a reasonable scale model can be identified for SiC, which would allow predictions of concentration at different velocities at different scales.

CONCLUSION

Based on the analyses of the data, it appears that the pre-transfer sample can be used to provide an estimate of the constituents in the subsequent batch transfer samples. For most constituents, the concentration of the pre-transfer sample is higher than that obtained from the subsequent batch transfer samples. For the other constituents, the concentration of the pre-transfer sample appears to be similar to that obtained from the batch transfer samples. Also, for most constituents, the mass of the constituent obtained from the batch transfer is less than what

would be projected from the pre-transfer sample. These results suggest that the pre-transfer sample provides a conservative estimate, i.e., an over-estimate, of the material which is transferred in the subsequent batches. Also, it appears possible to identify reasonable models for predicting transfer performance, as measured by constituent concentration, at different velocities and different scales. These results all support the recommendation that a dedicated facility for feed sampling is not currently needed to support the first WTP feed delivery.

Additional analysis is needed to further refine the transfer performance modeling. This includes developing intervals to bound the predictions, as well as determining appropriate methodology to use for verifying the models on the scaled equipment.

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