Demonstrating Reliable High Level Waste Slurry Sampling Techniques to Support Hanford Waste Processing – 14194

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ASTRACT

The Hanford Tank Operations Contractor and the Hanford Waste Treatment and Immobilization Plant contractor are both engaged in demonstrating mixing, sampling, and transfer system capability using simulated Hanford High-Level Waste (HLW) formulations. This work represents one of the remaining technical issues with the high-level waste treatment mission at Hanford. The Tank Operations Contractor must demonstrate the ability to adequately mix and sample high-level waste feed to meet the Waste Treatment and Immobilization Plant Waste Acceptance Criteria and Data Quality Objectives. The sampling method employed must support both Tank Operations and Waste Treatment and Immobilization Plant requirements. To facilitate information transfer between the two facilities the mixing and sampling demonstrations are led by the One System Integrated Project Team. The One System team, Waste Feed Delivery Mixing and Sampling Program, has developed a full scale sampling loop to demonstrate sampler capability. This paper discusses the full scale sampling loops ability to meet precision and accuracy requirements, including lessons learned during testing.

Results of the testing showed that the Isolok®[1] sampler chosen for implementation provides precise, repeatable results. The Isolok® sampler accuracy, as tested, did not meet test success criteria. Expert review of test data and test platform following testing identified several issues regarding the diversion sampler used to provide reference material used to judge the Isolok®'s accuracy. Recommendations were made to obtain new data to evaluate the sampler's accuracy utilizing a different type of reference sampler.

INTRODUCTION

The U.S. Department of Energy, Office of River Protection manages the River Protection Project. The River Protection Project mission is to retrieve and treat Hanford's tank waste and close the tank farms to protect the Columbia River. As a result, the Office of River Protection is responsible for the retrieval, treatment and disposal of approximately 208 million liters of radioactive waste contained in the Hanford Site waste tanks.

The Waste Treatment and Immobilization Plant will process the waste feed it receives from the Tank Operations Contractor into its final disposal form. Waste staged as feed will be sampled to ensure it meets Waste Treatment and Immobilization Plant – Tank Operations Contractor interface agreements. The Tank Operations Contractor's Waste Feed Delivery Mixing and Sampling Program is tasked with developing and demonstrating waste feed capabilities.

Implementation of the sampling concept on a Hanford million gallon double-shell tank will utilize the tank's transfer pump for recirculating waste feed through a sampling loop where a small portion of the waste will be captured before the waste is returned to the tank. Sampling will occur while the tank is being mixed by two rotating jet mixer pumps. The sampling method must minimize contamination and be remotely operated to minimize operator exposure to radiation. The total amount of material to be

[1] Isolok® is a registered trademark of Sentry Equipment Corp. of Oconomowoc, WI

sampled for qualification of a feed tank will be between four and ten liters (most of the sampled material will be used for process evaluation, not analytical analysis). Sample container volume will be between 250 mL and 1000 mL; most likely 500 mL to best utilize current transportation systems.

The Waste Feed Delivery Mixing and Sampling Program must determine the range of waste physical properties that the Tank Operations Contractor could transfer to the Waste Treatment and Immobilization Plant and the ability characterize staged waste feed using the proposed system. Work reported here was performed to understanding our ability to characterize the feed. Characterization must be performed to precision and accuracy levels that allow disposition of interface requirements.

Two sampling methods are currently employed by the Tank Operations Contractor, core sampling and grab sampling. Neither of these methods was designed for meeting waste feed delivery sampling needs; each has issues which have led the Tank Operations Contractor to choose a new sampling method for feed characterization – a modified Isolok® MSE sampler, by Sentry, in a closed sampling loop. The Isolok® sampler is the same configuration modified for use in radioactive environments at the Waste Treatment and Immobilization Plant. Testing this sampling method full scale is required for its validation as well as for validation of interface requirements. Simulants used during testing represent the broad spectrum of rheological and particle properties potentially transferred to Waste Treatment and Immobilization Plant in high level waste feed batches and are outlined in TPP-PLAN-51625, Rev. 0, *Waste Fee Delivery Mixing and Sampling Program Simulant Definition for Tank Farm Performance Testing* [1]. Results are applicable to both the Tank Operations Contractor and Waste Treatment and Immobilization Plant sampling programs.

The specific objectives of the tests as outlined in the test plan, *RPP-PLAN-52623 Rev. 0, One System Waste Feed Delivery Mixing and Sampling Program System Performance Test Plan* [2], were:

- Verify the precision of sample volume – samples should be within 5% of the target volume (257 ml).
- Verify the precision of the sampled material – sample component variance attributed to the sampling method should have percent relative standard deviations (%RSD) of less than 10%.
- Verify the Accuracy of the sampler – sample solid component concentrations should be within 10% of its corresponding reference sample.

METHODS

For a complete outline of test information please see RPP-RPT-53930, Rev. 0, *One System Waste Feed Delivery Remote Sampler Performance Test Report* [3].

Test Matrix:
Waste feed delivery plans to use the same double-shell tank mixing and waste transfer operating parameters for all waste feed types and staging tanks. Therefore operational parameters, such as transfer rate of 530 +/-19 liters per minute and temperature at 21.1 +/- 1.1 ºC, were held constant and the design of experiments was based on simulant characteristics. The experiment was a full factorial array using three factors [2][3]:

- Solids composition: categorical, two levels (typical and high)
- Solids loading: continuous, two levels (9 and 13 weight percent un-dissolved solids)
- Supernate: categorical, three levels (low, typical, and high)

In addition to the twelve runs outlined by the test matrix using Newtonian supernates, two non-Newtonian tests were performed.

The use of simulants based on categories representing low, typical, and high properties enable testing to estimate system performance in both bounding and nominal operating regions with a minimal number of test runs.

Test Simulants:
Sampler performance was measured against different Hanford waste simulants, each simulant comprised of two basic components, undissolved solids and supernate, outlined in the test program's simulant definition document [1]. The simulant definition document outlines three solids categories, two of which were used for Isolok® performance testing – "typical" and "high" base solids formulas, see Tables 1 and 2. The third base solids simulant, categorized as "low", was based totally from small gibbsite and would not have provided a good measure for sampler performance, and was not included in the test matrix. The high base solids provided an upper bound to fast settling solids that could be seen in waste feed.

Table 1. Particle Component by Solids Simulant

Component	Particle Density (g/cm^3)	Typical Base		High Base	
		Volume Fraction	Mass Fraction	Volume Fraction	Mass Fraction
Small Gibbsite	2.42	0.30	0.27	--	--
Large Gibbsite	2.42	0.50	0.44	0.05	0.03
Small Sand	2.65	--	--	0.47	0.35
Medium Sand	2.65	0.13	0.13	--	--
Large Sand	2.65	--	--	0.28	0.21
Zirconium Oxide	5.7	0.05	0.10	0.05	0.08
Stainless Steel	8.0	0.02	0.06	0.15	0.33

Table 2. Particle Size Distributions by Particle Type

Cumulative Volume Fraction	Target Particle Size by Component (µm)						
	Small Gibbsite	Large Gibbsite	Small Sand	Medium Sand	Large Sand	Zirconium Oxide	Stainless Steel
d_{10}	0.542	2.94	34.0	88.3	228	0.745	62.9
d_{50}	1.29	10.1	57.1	148	383	6.02	112
d_{90}	3.09	20.7	96.4	250	646	22.7	187
d_{95}	3.95	24.7	112	290	749	28.6	221

Three levels of supernate compositions were also provided. Since the influence of supernate density and viscosity were assumed to be important factors regarding segregation of fast settling solids, the test matrix included all three regimes, Table 3.

Table 3. Supernate Composition for Remote Sampler Demonstration Testing

Component	Low	Typical	High
Description	Low density / low viscosity	Typical density / typical viscosity	High density / high viscosity
Density (g/ml)	1.1±0.06	1.29±0.06	1.37±0.07
Viscosity (cP)	1.0±0.5	3.6±0.5	15±3
Sodium Thiosulfate (wt%)	12	31.5	33.4
Glycerol (wt%)	0	0	19.5
Water (wt%)	88	68.5	47.1

The two non-Newtonian simulants where based off kaolin clay [1]. The simulants represented low and high shear stress slurries.

Table 4. Non-Newtonian Simulants (20°C)

Bingham Yield Stress (Pa)	Density (g/ml)	Kaolin Clay (wt%)	Solids
3	~1.15	22.5	Same amount of Zr and Stainless Steel as added for the run: high base solids, 13 wt % waste loading, typical supernate
10	~1.19	26.5	Same amount of Zr and Stainless Steel as added for the run: high base solids, 13 wt % waste loading, typical supernate

Analytical Methods:
Mass analysis of samples was performed at the test facility on all samples, ten per run. Volumes were determined by converting sample by using slurry densities as measured by the Coriolis meter [3].

The compositions of five samples from each run were determined by an off-site analytical laboratory. Analysis comprised of filtering/washing un-dissolved solids (for some supernate/solids compositions multi-stage filtering was employed) followed by digestion and analysis:

- Al(OH)3 (total gibbsite) – Microwave Digestion / ICP-AES
- SiO2 (total sand) – Borate Fusion / ICP-AES
- Stainless Steel (total) – Microwave Digestion / ICP-AES
- ZrO2 (total) – Microwave Digestion / ICP-AES

Due to the volume of the diversion samples, a subsampling method, coring, was developed. To understand error do to analytical work, diversion control samples were analyzed. Each sample was analyzed three times (replicates or sets of cores) and each analysis had eight subsamples. Results are shown in Table 5; analytical error is broken out by subsample – replicates are cores and subsamples are the ICP-AES subsamples. Results show that error due to the coring technique is minor for all except the simulants with the highest solids concentrations, high base solids and non-Newtonian fluids [3]. This could be due to channeling as the fast settling solids look for a path of least resistance on their way to the bottom of the beaker.

The lower volume of the Isolok® samples allowed filtering and washing of the complete sample prior to subsampling, digestion, and analysis.

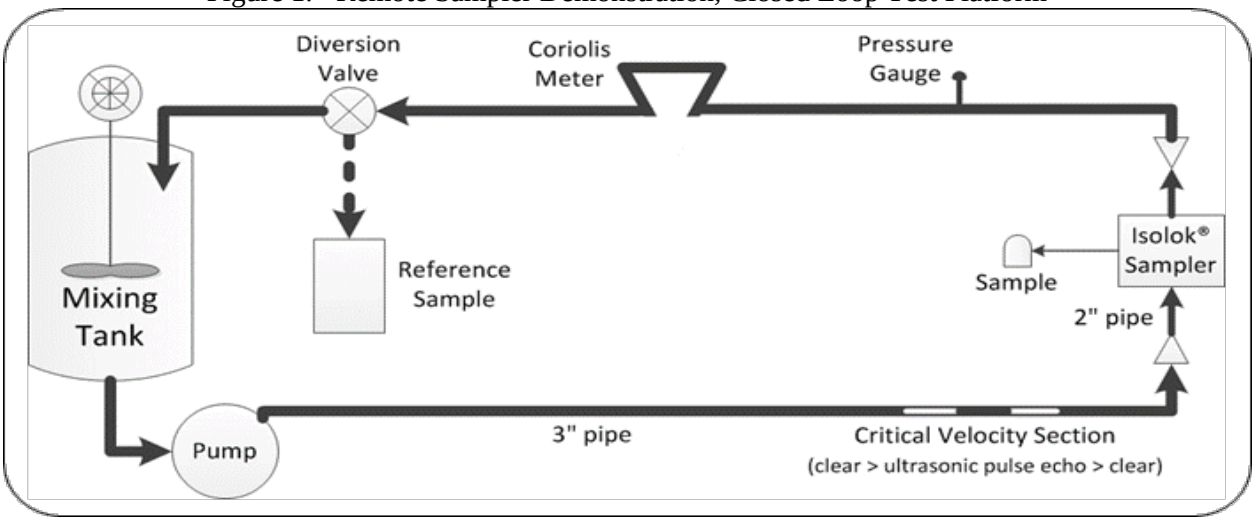
Table 5. Diversion Sample Uncertainty Analysis [3]

Base Solids Supernate		Description	Prepared Wt%	Recovered Mean Wt%	% Relative Lab Error	% RSD Total	% RSD Replicates (% of total variance)		% RSD Subsamples
Typical	Low	Gibbsite	71.1	66.9	-5.9	5.8	0.8	(2.1)	5.8
		Sand	12.9	12.3	-5.0	14.7	7.8	(28.0)	12.5
		ZrO_2	10.0	10.1	1.0	5.7	0.0	(0.0)	5.7
		SS	6.0	5.6	-7.0	30.9	0.0	(0.0)	30.9
		TOTAL	100.0	94.9					
	Typical	Gibbsite	71.0	67.9	-4.4	5.5	2.3	(17.8)	5.0
		Sand	13.0	12.5	-4.2	13.4	0.0	(0.0)	13.4
		ZrO_2	10.0	10.0	-0.5	4.6	1.0	(4.4)	4.5
		SS	6.0	5.7	-5.8	21.7	0.0	(0.0)	21.7
		TOTAL	100.0	95.9					
	High	Gibbsite	71.0	65.9	-7.1	5.8	1.2	(4.3)	5.7
		Sand	13.0	14.7	12.5	13.3	0.0	(0.0)	13.3
		ZrO_2	10.0	9.7	-3.9	5.7	0.0	(0.0)	5.7
		SS	6.0	6.3	5.9	20.3	0.0	(0.0)	20.3
		TOTAL	100.0	96.6					
High	Typical	Gibbsite	2.9	3.4	15.3	7.9	0.0	(0.0)	7.9
		Sand	56.1	56.3	0.4	6.1	0.9	(2.1)	6.1
		ZrO_2	8.0	5.4	-32.2	7.5	0.0	(0.0)	7.5
		SS	33.0	31.7	-3.9	9.4	5.0	(28.0)	8.0
		TOTAL	100.0	96.8					
	High	Gibbsite	2.9	3.1	5.2	9.4	2.2	(5.3)	9.1
		Sand	56.0	53.5	-4.5	6.7	4.8	(50.9)	4.7
		ZrO_2	8.1	8.8	9.4	7.8	3.2	(17.2)	7.1
		SS	33.0	31.2	-5.4	11.2	7.9	(49.8)	8.0
		TOTAL	100.0	96.6					
Non-Newtonian 10 Pa		ZrO_2	3.6	4.0	10.1	3.9	2.3	(35.0)	3.1
		SS	14.8	11.2	-24.1	31.7	16.1	(25.9)	27.3
		Kaolin Clay	81.6	86.4	5.9	2.6	2.0	(61.1)	1.6
		TOTAL	100.0	101.6					

Green = The mean of the measured values is not statistically different from the prepared value at the 95% confidence level.
Red = The mean of the measured values is statistically different from the prepared value at the 95% confidence level.

Test Platform:
The remote sampler demonstration test system utilized a small mixing tank (in place of a full scale Hanford double shell tank) coupled with a with full scale sampling system. Items that are full scale include: pipe size (diameter), flow rate, sampling system, and instrumentation. The system is shown in Figure 1.

Figure 1. Remote Sampler Demonstration, Closed Loop Test Platform



Mixing Tank / Pump: The mixing tank was designed to homogenize the waste as best possible and provide a consistent feed to the sample loop; not to represent mixing that occurs in Hanford's double-shell tanks. Modifications were made to the tank to minimize buildup of fast settling solids at the tank floor, see Figure 2. The tank volume was 680 L (0.914 m diameter). A diversion plate covered the discharge to minimize direct pass through of fast settling solids to the transfer pump. The transfer pump was a typical centrifugal pump, constructed from stainless steel.

Figure 2. Remote Sampler Demonstration Mixing Tank

Critical Velocity Section: The critical velocity section of the test loop was a located just downstream of the pump discharge [3][4]. The section was horizontal with 65 pipe diameters of straight pipe prior to the test section and 15 pipe diameters downstream of the test section. The test section was comprised of three sections – two 0.6 m transparent sections surrounding a roughly 0.6 m stainless steel section. The transparent sections were used to determine critical velocity visually, and the stainless steel section was used to allow critical velocity determination using an ultra-sonic pulse echo technique developed by

PNNL-22029, Rev 0, *Hanford Tank Farms Waste Feed Flow Loop Phase VI: PulseEcho System Performance Evaluation* [4]. The critical velocity work will not be reviewed here.

Isolok® Sampler: After the critical velocity section the flow turned upward and reduced from 7.62 cm (3") schedule 40 pipe to 5.08 cm (2") schedule 40 pipe. The Isolok® sampler was 8 degrees from true vertical due to design requirements supporting the Isolok®'s sample container adapter and mechanical handling testing apparatus (data not included in this report), which was also performed on the system during the same time frame.

The MSE version of the Isolok® is specifically designed for viscous and thixotropic fluids. The significant modifications to the factory equipment include:

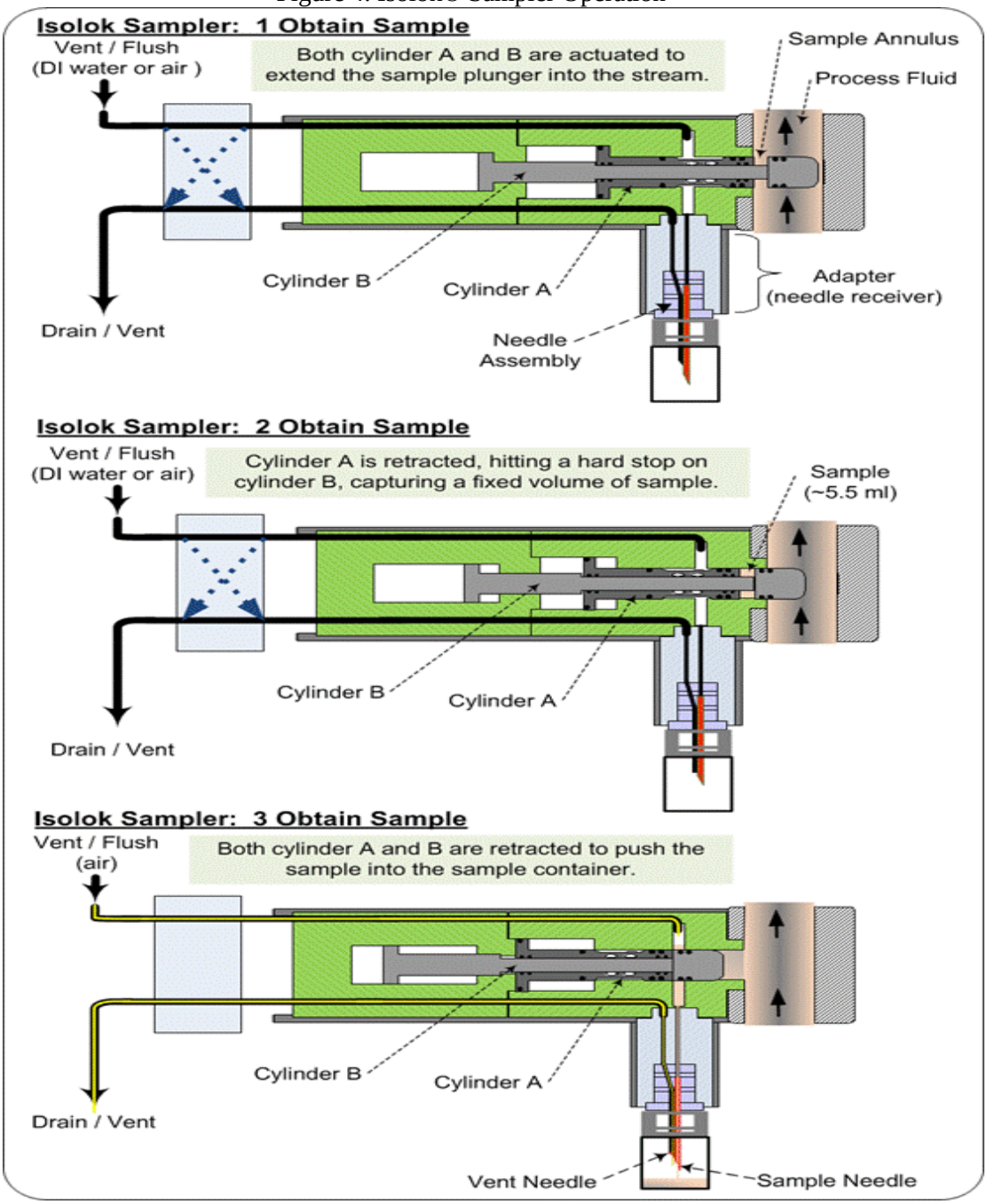
- Bolt-on (versus clamp-on) adapter mount
- Extension arm with needle (where the needle provides the path from the sampler to the sample container and is capable of being remotely replaced)
- Relocation of all air/water connections to the back of the sampler

and

- O-rings materials were changed to materials that meet site requirements for contact with tank waste.

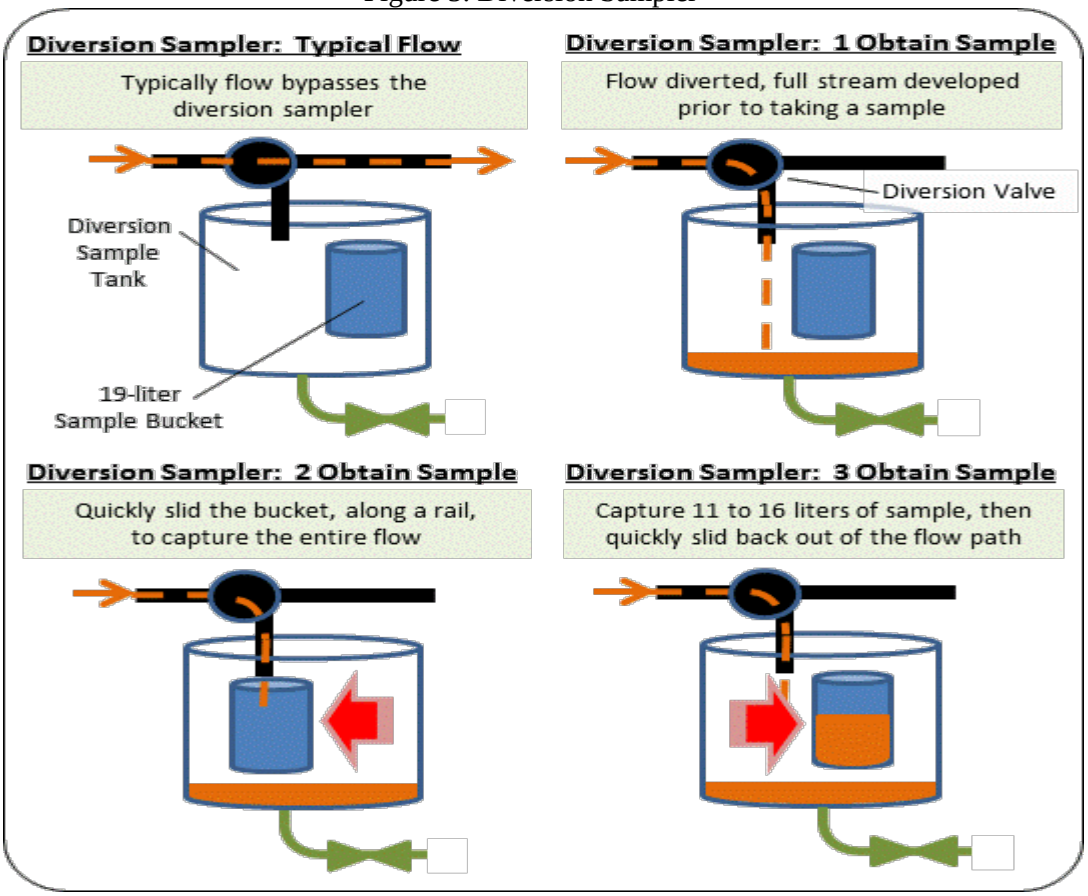
A simple outline of Isolok® operation is provided in Figure 4. One cycle or increment of the Isolok® sampler obtains about five milliliters of sample. The number of increments can be adjusted to any value enabling the use of a large selection of sampling containers. The Tank Operations Contractor will optimize the size of the sample container based on analytical and shipping criteria; likely resulting in a sample volume somewhere between 250 to 500 mL. For our testing a 250 mL container was used and 47 increments taken over a period of about ten minutes [3].

Figure 4. Isolok® Sampler Operation



Diversion Sampler: A full diversion sampler, used for obtaining reference samples, was located just before the simulant dropped back into the mixing tank. The sampler used a three way valve which diverted the flow from returning to the mixer tank through a the three inch line into a separate tank. Once the flow was diverted, an eleven to sixteen liter sample was taken by sliding a bucket into the flow. An outline of the sampling processes is show in Figure 5.

Figure 5. Diversion Sampler



DISCUSSION

Run order was randomized [2]. Order of events for each run was: measurement of critical velocity, obtain ten Isolok® samples, obtain diversion sample. All Isolok® samples were weighed and every other sample was sent to the laboratory along with the runs diversion sample for component analysis. Results were then compared to test goals [3].

Sample volumetric data is shown in Table 6; the Isolok® sampler consistently took the same volume of material for each sample.

Component data was obtained for five samples from each run; the laboratory analyzed each sample eight times [3]. Results for stainless steel and zirconium are shown in Tables 7 and 8; stainless steel represents the fast settling solids. See RPP-RPT-53930 [3] for gibbsite and sand results. Samples are compared against the corresponding diversion sample to estimate Isolok® sampler accuracy and the variance component due to sampling is broken out to quantify Isolok® precision.

Sample accuracy, % error, appears to be very poor for the Isolok® sampler with the concentration of stainless steel much higher and the zirconium concentration lower in the Isolok® sampler relative to the diversion sampler. A discussion of issues that likely resulted in the discrepancy is in the test errors

section below.

Table 6. Isolok® Volume Results

Run (solids-solids loading-supernate)	Isolok® Sample Volume (g/(g/ml)) Ave.	Min.	Max.
Typical-9-Low	257.4	254	259
Typical-9-Typical	259.3	255	274
Typical-9-High	245	241	249
Typical-13-Low	258.6	257	265
Typical-13-Typical	255.6	253	258
Typical-13-High	250.5	248	266
High-9-Low	266	261	271
High-9-Typical	263.7	254	268
High-9-High[a]	257.9	255	261
High-13-Low	266.2	264	271
High-13 -Typical	269.9	260	272
High-13-High	263.5	262	265
Non-Newtonian-3	264.5	262	266
Non-Newtonian-10	264.5	249	291

[a] Due to a sampler malfunction, the volume of the final sample collected was approximately one increment (5.47 ml) low.

The break out of variance components by level was performed by constructing a model where laboratory samples (analytical subsamples) are nested within Isolok® samples; performed using JMP® 10 by SAS®. Results show that in general, most of the variance is due to laboratory analysis [3]. No clear pattern is seen, except that for the high base solids, where 33% of the undissolved solids are stainless steel, nearly all of the variance for each run is attributed to the laboratory.

Table 7. Non-Newtonian Simulant Recovery Data for Stainless Steel and Zirconium

Base Simulant	Yield Stress (target % total solids)	Critical Velocity (m/s)	Sampler	Stainless Steel Mean	Stainless Steel % Error (a)	Stainless Steel Total	Stainless Steel Lab	Stainless Steel % RSD Smplr (% of total var. attributed by the smplr)	ZrO2 Mean	ZrO2 % Error (a)	ZrO2 Total	ZrO2 Lab	ZrO2 % RSD Smplr (% of total var. attributed by the smplr)
Non-Newtonian	3 Pa (26.5)	1.5	Div.	14.6	106.8	26.8	NA	NA	4.7	-7.8	7.6	NA	NA
			Iso.	30.2		21.7	20.7	6.5 (9.0)	4.3		9.3	9.1	1.9 (4.0)
	10 Pa (32.2)	1.6	Div.	17.8	57.1	39.0	NA	NA	4.6	-0.8	5.1	NA	NA
			Iso.	28.0		25.6	24.9	6.1 (5.6)	4.6		15.5	13.6	7.5 (23.5)

Div. = Diversion Sampler, Iso. = Isolok® Sampler

(a) % error is relative to the diversion (reference) sample {(100 x (Isolok® – Diversion)/Diversion)}.

Table 8. Newtonian Simulant Recovery Data for Stainless Steel and Zirconium

Base Solids	Base Solids Loading	Supernate	Critical Velocity (m/s)	Sampler	Stainless Steel (wt % solids)					ZrO2 (wt % solids)				
					Mean	% Error (c)	%RSD Total	%RSD Lab	%RSD Smplr (% of total var. from smplr)	Mean	% Error (c)	%RSD Total	%RSD Lab	%RSD Smplr (% of total var. from smplr)
Typical Base	9 wt%	Low	1.4	Div.	3.3	83.4	42.5	NA	NA	10.9	-10.1	6.9	NA	NA
				Iso.	6.0		29.1	25.9	13.4 (21.1)	9.8		7.3	6.9	2.4 (10.6)
		Typical	0.8	Div.	4.7	33.1	10.0	NA	NA	10.0	-3.0	6.7	NA	NA
				Iso.	6.3		27.1	25.7	8.4 (9.6)	9.8		6.7	6.5	1.4 (4.6)
		High	1.3	Div.	4.4	70.5	31.6	NA	NA	9.6	0.2	4.9	NA	NA
				Iso.	7.5		36.1	32.9	14.7 (16.7)	9.7		9.6	7.6	5.9 (37.6)
	13 wt%	Low	1.5	Div.	3.8	55.8	24.7	NA	NA	10.6	-8.5	3.7	NA	NA
				Iso.	6.0		23.9	23.9	0.0 (0.0)	9.7		7.8	7.8	0.0 (0.0)
		Typical	0.8	Div.	5.1	79.3	14.9	NA	NA	9.6	-5.7	7.9	NA	NA
				Iso.	9.2		23.6	21.9	8.9 (14.1)	9.1		6.8	6.4	2.3 (11.1)
		High	1.3	Div.	5.4	53.3	17.4	NA	NA	9.6	-2.1	6.6	NA	NA
				Iso.	8.2		22.7	21.3	7.9 (12.1)	9.4		7.0	7.0	0.0 (0.0)
High Base	9 wt%	Low(a)	2.1	Div.	9.1	62.0	27.6	NA	NA	10.9	-30.0	21.4	NA	NA
				Iso.	14.8		17.4	15.9	7.0 (16.2)	7.6		14.0	14.0	0.0 (0.0)
		Typical	1.6	Div.	26.3	42.7	22.1	NA	NA	8.6	-30.3	19.2	NA	NA
				Iso.	37.5		14.1	14.1	0.0 (0.0)	6.0		35.6	16.5	31.5 (78.5)
		High	1.2	Div.	33.9	15.3	12.5	NA	NA	8.0	-32.8	11.7	NA	NA
				Iso.	39.1		15.9	15.9	0.0 (0.0)	5.4		12.2	12.1	1.6 (1.7)
	13 wt%	Low	2.1	Div.	8.2	82.5	13.4	NA	NA	9.5	-23.6	5.3	NA	NA
				Iso.	15.0		24.9	24.9	0.0 (0.0)	7.3		19.4	17.9	7.4 (14.5)
		Typical	1.6	Div.	22.1	90.1	10.8	NA	NA	7.7	-39.6	6.5	NA	NA
				Iso.	42.1		16.6	16.6	0.0 (0.0)	4.7		20.7	18.1	10.1 (23.7)
		High	1.2	Div.	30.4	36.8	12.7	NA	NA	7.2	-26.4	7.6	NA	NA
				Iso.	41.6		11.7	11.7	0.0 (0.0)	5.3		16.5	16.5	0.0 (0.0)

Div. = Diversion Sampler, Iso. = Isolok® Sampler

(a) Two Isolok® samples were collected at 190 gpm instead of 140 gpm; these samples are omitted from the results.

(b) % error is relative to the diversion (reference) sample {(100 x (Isolok® – Diversion)/Diversion)}.

Test Errors:

This discussion is not intended to be a thorough review of all of the errors. The purpose of the discussion is to understand errors associated with this work; aiding the interpretation of the results. The system design concept was to provide good mixing and constant flow, such that little to no variation in concentration would be realized over time or during sampling.

A review of the system and test data was performed after testing was completed by a sampling expert RPP-RPT-56000, Rev. 0, *External Review of the Remote Sampler Demonstration Platform* [5]. The review targeted correct sampling practices as outlined in Pierre Gy's Sampling Theory and Sampling Practice [6]. The errors inherent in the Isolok® sampling system were known prior to testing, and although the Isolok® system inherently introduces error, given the highly radioactive material being sampled and sampling goals, the expert consultant agreed that the Isolok® system is the correct sampler for field implementation – provided its ability to meet data quality objectives is proven. The diversion

sampler however, which was developed to provide reference samples and allow quantification of Isolok® accuracy performance, did not follow Pierre Gy's recommendations to the extent needed for use as a reference sampler [6]; several of these which likely resulted in large sampling error were not identified until testing was completed. The key sampling errors inherent to the Isolok® and diversion samplers are:

- Isolok® Sampler
  - Not every part of the sample has an equal chance of being sampled
  - Does not employ cutting edges that equally respect particle size or shape – and, therefore, the center of gravity
  - Non-probabilistic; the amount of material captured does not change with the flow rate through the system
  - Plunger extension, the first step in acquiring a sample increment, restricts flow through the pipe causing a ~30 liter per minute drop in flow rate, Figure 6
  - Sampler performance may be better if oriented perfectly vertical
- Diversion Sampler
  - Captures all parts of the sample, not in an equiprobabilistic fashion (the leading edge was in the flow stream longer than the trailing edge); relative to the amount of sample obtained, error induced by oversampling one edged of the stream is expected to be small
  - Does not have a proportional cutting edge designed specifically for sampling
  - Obtains one large (several gallons or 1.7% of the lot) sample at one brief point in time; therefore does not account for any changes in the system that occur over time, and was not captured during Isolok® sampler operation
  - Diversion valve actuation increased the line resistance and dropped the flow rate ~120 liter per minute; at or below the critical velocities of most of the test simulants, Figure 7

Figure 6. Isolok® Actuation Effect on Flow Rate

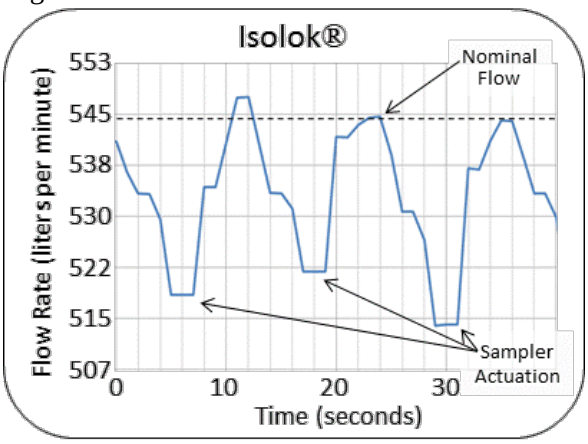
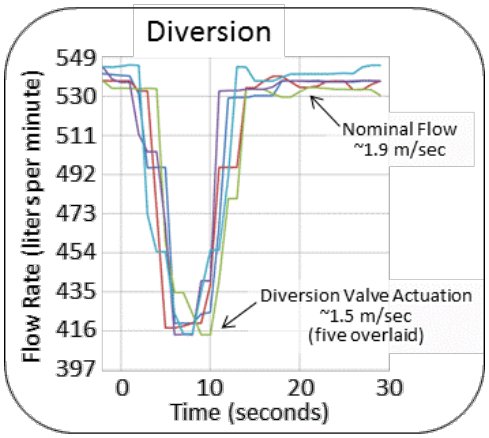


Figure 7. Diversion Actuation Effect on Flow Rate



Data for Isolok samples in Table 8 show that laboratory analytical error was high, especially for stainless steel. Most of the error associated with each test runs set of samples was associated to analytical error (or the laboratory variance component).

CONCLUSIONS

Twelve tests with Newtonian supernates were conducted representing a full factorial design that included two different solid compositions (typical and high); two mass loadings (9 and 13wt% solids); and three supernate density and viscosity combinations (low, typical, and high); two additional tests included non-Newtonian working fluids [2].

The remote sampler demonstration system performance tests evaluated the accuracy and precision of sampling simulated Hanford waste slurry with the Isolok® sampler [3]. Ten Isolok samples were taken during each test, and volume of the slurry collected was determined. Five of the samples were analyzed for chemical content. Table 9 summarizes the comparison of the Isolok® samples to the test criteria: green boxes indicate fully meeting the criteria, yellow indicates a few discrepancies, and red indicates failure.

Volume (within 5% of the target volume (257 ml)): 148 of the 150 Isolok® samples were within 5% of the targeted volume [3].

Precision (solids component concentrations having percent relative standard deviations (%RSD) less than 10%): The repeatability for the Isolok® sampler for gibbsite [$Al(OH)_3$], sand [SiO_2], and zirconium oxide [ZrO_2] met the success criteria in all but one test [3]. Results were consistent between the Newtonian and the non-Newtonian tests. Repeatability for stainless steel was mixed, being acceptable in high concentrations but not acceptable at low concentrations. Repeatability with the low concentrations of stainless steel was likely be due to analytical capabilities; although analytical capabilities are frequently stated as a percentage of the analyzed value, there is often a fixed, or inherent, error in the analysis which leads to more relative uncertainty in smaller values.

Accuracy (collecting representative samples with solids component concentration within 10% of a reference sample): The Isolok® samples did not meet the success criteria relative to the reference sample. However, based on posttest review by the team, including an expert sampling consultant [3][5], no conclusions should be drawn regarding the Isolok®'s ability to accurately sample Hanford waste simulants from these test results. The reference sampler employed had several inherent flaws as outlined by Pierre Gy [5][6]; major issues identified regarding the diversion sampler were:

- Sampling time frame and number of increments (one) were way too low to match the Isolok® sampler's performance
- Actuation of the sampler caused a major drop in system flow rate, likely dropping out fast settling solids and resulting in low concentrations of these components in the reference sample

Table 9. Test Results versus Success Criteria

Test Condition (base solids–mass loading –supernate)	Volume	Precision Goals Met				Accuracy Goals Met			
		Gibbsite	Sand	ZrO_2	SS	Gibbsite	Sand	ZrO_2	SS
Typ-9%-Low	Yes	Yes	Yes	Yes	No	No	No	Yes	No
Typ-9%-Typ	9/10	Yes	Yes	Yes	Yes	Yes	No	Yes	No
Typ-9%-High	Yes	Yes	Yes	Yes	No	Yes	No	Yes	No
Typ-13%-Low	Yes	Yes	Yes	Yes	Yes	No	No	Yes	No
Typ-13%-Typ	Yes	Yes	Yes	Yes	Yes	No	No	Yes	No
Typ-13%-High	Yes	Yes	No[a]	Yes	Yes[a]	Yes	No	Yes	No
High-9%-Low	Yes	Yes	Yes	Yes	Yes	No	Yes	No	No
High-9%-Typ	Yes	No[b]	No[b]	No[b]	Yes	No	No	No	No
High-9%-High	Yes	Yes	Yes	Yes	Yes	No	No	No	No
High-13%-Low	Yes	Yes	Yes	Yes	Yes	No	Yes	No	No
High-13%-Typ	Yes	No	Yes	No	Yes	No	No	No	No
High-13%-High	Yes	Yes	Yes	Yes	Yes	No	No	No	No
Non-Newtonian 3 Pa	Yes	NA[c]	NA[c]	Yes	Yes	NA[c]	NA[c]	Yes	No
Non-Newtonian 10 Pa	9/10	NA[c]	NA[c]	Yes	No	NA[c]	NA[c]	Yes	No

[a] Samples were collected on different days and have different sand and SS concentrations in the collected samples, which may have led to the higher variability in these samples.
[b] It is likely that the "No" determination is based on a sample labeling error rather than a performance issue.
[c] Non-Newtonian tests did not include Gibbsite or sand.

The Isolok® sampling system was demonstrated to collect repeatable samples; however, additional work is necessary to quantify the accuracy of the sampler [3]. In order to quantify the sampler accuracy, analytical methods must be improved and the test platform must be modified to employ a equiprobabilistic reference sampler. The reference sampler needs to follow good sampling practices, including multi-increment (as many increments as taken by the Isolok® sampler), equiprobabilistic collection of sample material, and be obtained during the same time period as the Isolok® samples [5]. The reference sampler must also not alter the flow conditions in the pipe (i.e., cause a temporary drop in flow rate). To ensure the Isolok® sampler is accurately assessed, modifications should include aligning the sampler in a truly vertical manner with at least ten pipe diameters immediately before and five pipe diameters immediately after the sampler.

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