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Review of a non-probabilistic sampler versus a Vezin sampler on low weight percent solids slurries

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The Hanford Tank Operations Contractor (TOC) and the Hanford Waste Treatment and Immobilization Plant (WTP) contractor are both engaged in demonstrating mixing, sampling, and transfer system capability using simulated Hanford High-Level Radioactive Waste (HLW) formulations. This work represents one of the remaining technical issues with the high-level waste treatment mission at Hanford – the TOC's ability to adequately sample high-level waste feed to meet the Waste Treatment and Immobilization Plant (WTP) Waste Acceptance Criteria Data Quality Objectives. A full-scale sampling loop was used at a cold test facility to evaluate sampler capability. The sampler under investigation for deployment is non-probabilistic but radioactive environment friendly. A Vezin sampler (probabilistic) was used to obtain reference samples and accurately characterize the simulant as it flowed through the test loop. The two samplers are located in series, allowing for multiple samples to be taken from both samplers over the same time period (sample pairs) and direct sample comparison. The Vezin sampler was modified to minimize material build up allowing for steady-state operation. This report discusses modifications made to the Vezin sampler and the results of sampler comparison.

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 litres 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 sample 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 sampled for qualification of a feed tank will be between four and ten litres (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.

A modified Isolok® MSE sampler, by Sentry, is the sampler of choice to meet safety, handling, and volume flexibility requirements. Because mixing cannot be assumed to produce a consistent homogenous feed the test loop, a custom two stage Vezin sampler, manufactured by FLSmidth USA Inc., was used to obtain reference samples during the same time period as the Isolok® samples were taken. A sketch of the test loop is below in Figure 1.

The test loop is primarily 3" schedule 40 pipe which is prototypic and allows for visual measurement of critical velocity through two clear sections; the method was developed during prototype testing for an ultrasonic pulse echo method for determine critical velocity.[1] A Coriolis meter was used to monitor flow rate. Temperature of slurry was control to approximately 21 °C using a chiller. The Isolok® is located ten pipe diameters above a 90° elbow and transition from 80 mm schedule 40 pipe to 50 mm schedule 40 pipe. The Isolok® captures a fixed sample volume using a plunger and cylinder which are each independently controlled pneumatically. A cut away figure of the Isolok® sampler for testing is show in Figure 2, and an animation of the Liquid Isolok® MSE sampler can be found at http://sentry-equip.com/Resources/Sentry-product-videos.htm. Each Isolok® sample was comprised of 115 increments; the final volume was ~630 mL. The two-stage Vezin sampler used is shown in Figure 3. The primary stage took approximately 77 cuts, and the secondary Vezin took approximately 170 cuts of the primary's sample; the final volume was ~1900 mL.

Two simulants (slurries) were used for testing.[2,3] Both slurries utilized the same carrier fluid, 31 % thiosulfate in water having a density of 1.29 g/mL and a viscosity of 3.3 cP. Six undissolved solids were used in the proportions outlined in Table 1. The typical

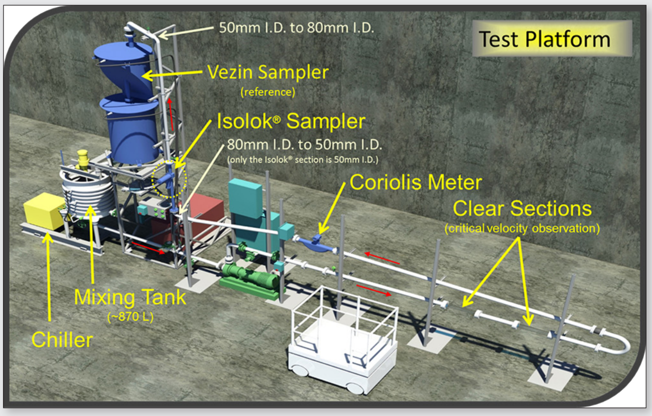


Figure 1. Test Loop

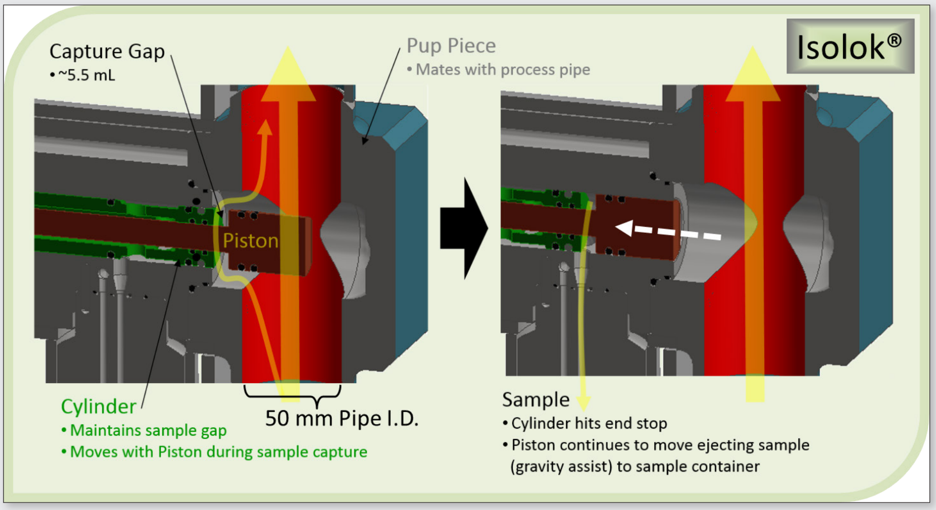


Figure 2. Isolok® Sampler

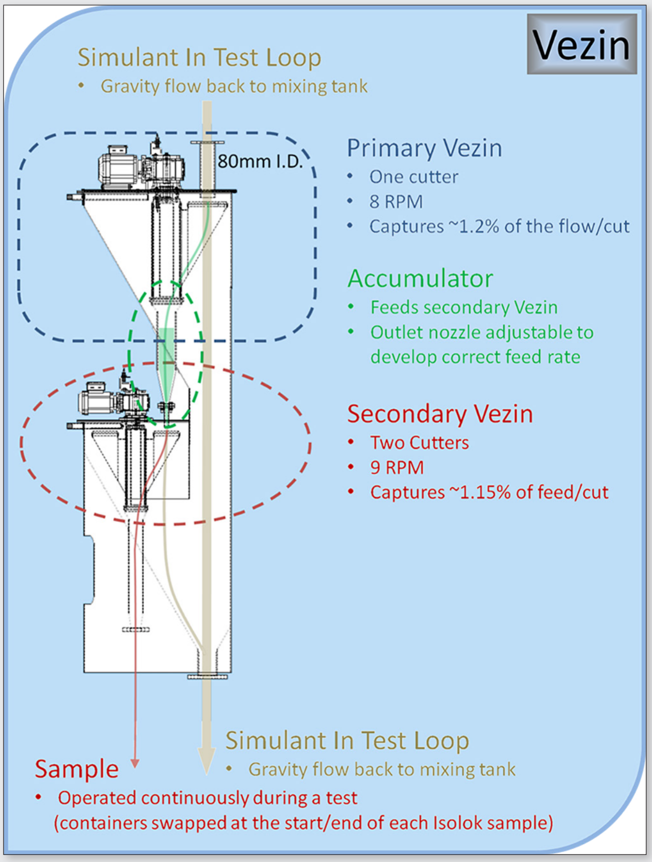


Figure 3. Two Stage Vezin Sampler

simulant recipe is primarily fine particulate, less than 75 µm, with a minor amount of fast settling solids – solids >75 µm. The high simulant recipe is a conservative (at the upper limit of fast settling solids relative to planned WTP feed) mix with a higher percentage of fast settling particles. Only the fast settling solids were targeted for analysis by sieving; large sand <710 µm (25 mesh) and >180 µm (80 mesh) and stainless steel <180 µm and >75 µm (170 mesh). All analytical sieves were American Society for Testing and Materials (ASTM) E161-12

Break-in and final test operations

Testing was performed in two phases[3] – a break-in test using the high simulant followed by final testing where both simulants were sampled and formal analytical data obtained. The break-in test was designed to allow the operators to practice sampling (capturing Isolok® and Vezin samples simultaneously) and work out any issues with the system. Two issues were resolved.

Accurate capture of Vezin samples for this type of test typically requires flushing of the cutters to make sure all material cut by the Vezin is included with the sample. Flushing the Vezin sampler is both time-consuming and has many steps, which increases the likelihood of operator error. The first goal of the break-in test was to determine if steady-state sampling could be performed. Material build up in the Vezin over time was estimated and modifications to the sampler were made to reduce material build up. Inspections showed that little, to no, material was left in the primary Vezin. Since only about 2.3% of the primary cutter material would be expected to be caught by the secondary Vezin, no changes were made to the primary Vezin.

Material did build up in the secondary Vezin. Rinsing the cutter and the flow path between the cutters and sample container separately resulted in an estimate that 40% of the material that was held up in the Vezin was held up by the cutters and 60% in the flow path. The cutters were modified to remove a lip at the bottom of the cutter where build up was most visible. The flow path was modified by reducing the size of the last section of pipe between the rotating cutters and sample container. See Figures 4 and 5 for photos and sketches of modifications made to the sampler.

Based on data review, the sampler was allowed to run 40 minutes before test samples were taken, and the modifications to the sampler reduced material build up during one sampling period (~9.5 minutes) from about 1 gram to less than 0.34 grams. Ideally, for sampling of slurries where concentration of material is the goal, the flow paths through the Vezin should be sized appropriately to the flow that they will carry. In the case presented here the secondary Vezin was sized identically to the primary Vezin, resulting in excess surface area along the flow path.

The second issue found during break-in testing was foaming caused by the free fall of slurry through the Vezin sampler. The

Table 1. Simulant Solids Components.

Component	Particle Density	Particle Size (d_{50})	Mass Fraction of Undissolved Solids By Simulant	
	g/cm^3	µm	Typical	High
Small Gibbsite[a]	2.42	2.2	0.27	0
Large Gibbsite[b]	2.42	9.9	0.44	0.053
Small Sand[b]	2.65	20.8	0.09	0.616
Large Sand[c]	2.65	414.3	0.04	0.074
Zirconium Oxide[a]	5.7	17.6	0.10	0.141
Stainless Steel[d]	8.0	122.3	0.06	0.116
Bulk Solids Density (g/cm^3)			2.7	3.1
Solids Loading in Slurry (wt %)			9.0	5.3

[a] Verified to be less than 63 µm.

[b] Pre-sieved through a 63 µm mesh.

[c] Pre-sieved, passed 710 µm and captured on 210 µm mesh.

[d] Pre-sieved, passed 150 µm and captured on 90 µm mesh.

Note: All pre-sieving was performed with sieves having 70% of the tolerances specified in ASTM E11-13.

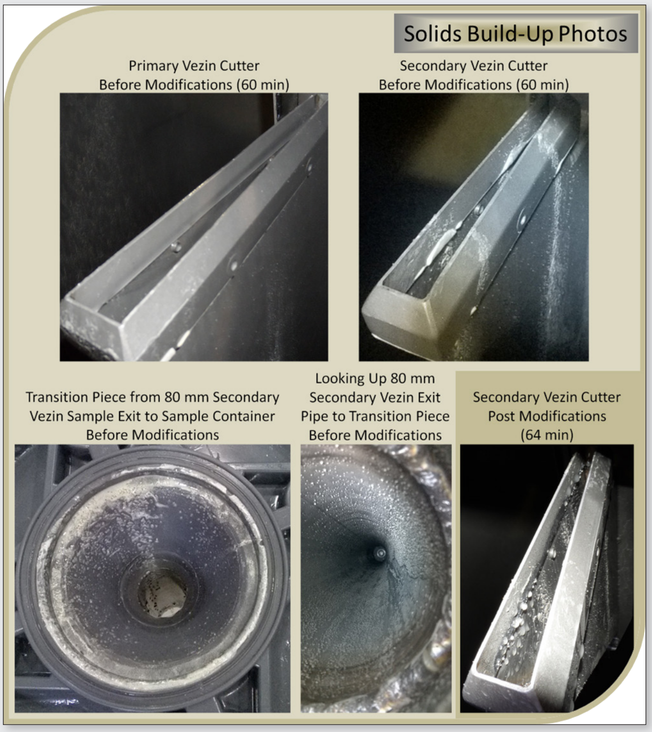


Figure 4. Vezin Inspection Photographs

addition of ~30 mL of a silicon based defoamer eliminated foaming in the high simulant and 3x that amount in the typical simulant.

During two formal test runs, 34 sample pairs were taken for each test — 30 (pairs 3 through 32) were analyzed for stainless steel and large sand. Parameters controlled during testing were flow rate, 530 ± 20 Lpm, and temperature 21 ± 1.7 °C [3]. During first test, using the typical solids slurry, sample number 14 was mishandled and could not be submitted to the laboratory for analysis and the second sample pair was analyzed as a replacement. The only other issue occurring during the formal tests was increased Isolok® volume, from ~650 mL to over 800 mL, during the high simulant test. The root cause was found to be worn Isolok® surfaces (due to previous testing using harsh simulants) and worn O-rings. Components estimated to have been removed from the test were added back to the test loop, sampler O-rings were replaced, and the test was repeated. Only the results of the repeat high simulant test are reviewed here.

Results and data review

Data obtained for each sample pair was:

- Critical velocity of slurry.
- Density, sample mass/sample volume.
- Concentration of solids <710 µm and >180 µm, captured on a #80 mesh – primarily large sand.
- Concentration of solids <180 µm and >75 µm, captured on a #170 mesh – primarily stainless steel.
- Limited results on slow settling solids, <75 µm.

Analytical method

Five control sample pairs were sent to the laboratory mixed with test samples for each test. By pre-sieving solids with one full mesh size on each side of the analytical sieve, analytical error was very low. See Table 2. The low analytical error is also evident by the tight spread of data, percent relative standard deviation (%RSD), over the course of the 30 sample pairs analyzed for each test.

Typical and high slurry

Critical velocity was determined for each test simulant before and after testing to verify the simulants were within test parameters. This was determined by incrementally dropping the test loop flow rate and observing the solids flow along the bottom of the clear sections using a high resolution video camera. The flow rate at which a stationary bed was formed was designated the slurry's critical velocity. The typical simulant had initial and final critical velocities of 0.82 m/s, and the high simulant had a starting critical velocity of 1.25 m/s and a final critical velocity of 1.22 m/s.

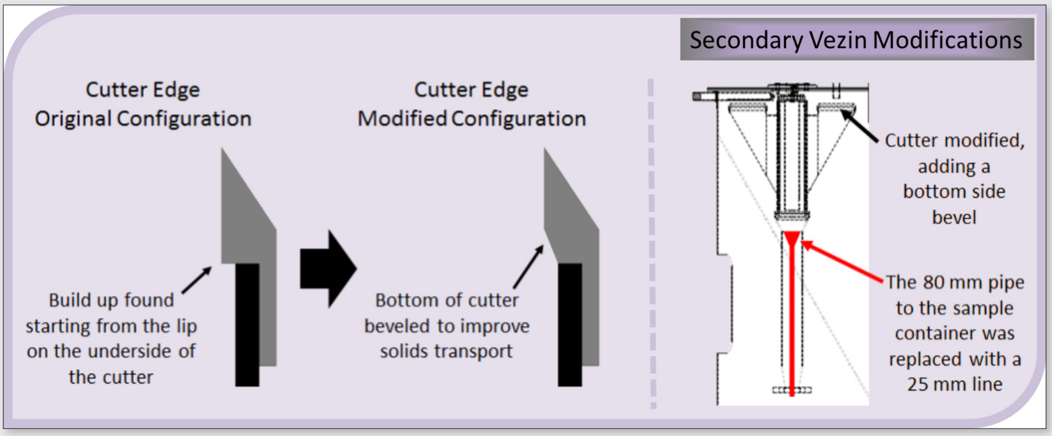


Figure 5. Modifications to Secondary Vezin Sampler

Table 2. Control Sample Data (for samples where slow settling solids were analysed)

Sample	Analysis By Sieve (g)			Prepared Mass (g)			% Recovered	
	<75 µm	75 µm	180 µm	<75 µm	M_{SS}	M_{LS}	<75 µm	% Total
Typical-Isolok® RSD-0804	108.1	7.3	4.8	109.7	7.3	4.9	98.5	98.5
Typical-Vezin RSD-0805	217.5	14.8	9.6	219.3	14.6	9.7	99.2	99.3
Typical-Isolok® RSD-0828	109.2	7.3	4.9	109.7	7.3	4.9	99.1	99.2
Typical-Vezin RSD-0829	217.9	14.6	9.7	219.3	14.6	9.7	99.4	99.4
High-Isolok® RSD-1023	53.9	13.6	8.4	58.0	13.3	8.4	93.0	95.3
High-Vezin RSD-1024	112.3	27.1	17.1	116.1	26.6	19.9	96.7	98.1

Note: SS = stainless steel and LS = large sand.

Sample pair densities and analytical data in terms of solids concentrations of material on each sieve are shown in Figure 6 for the typical slurry and Figure 7 for the high slurry. Both samplers were very consistent, sample to sample, without taking into account dynamic relationships (i.e., we assumed that the simulant did not change as material was removed and simply grouped all samples); see percent relative standard deviation data, Table 3.

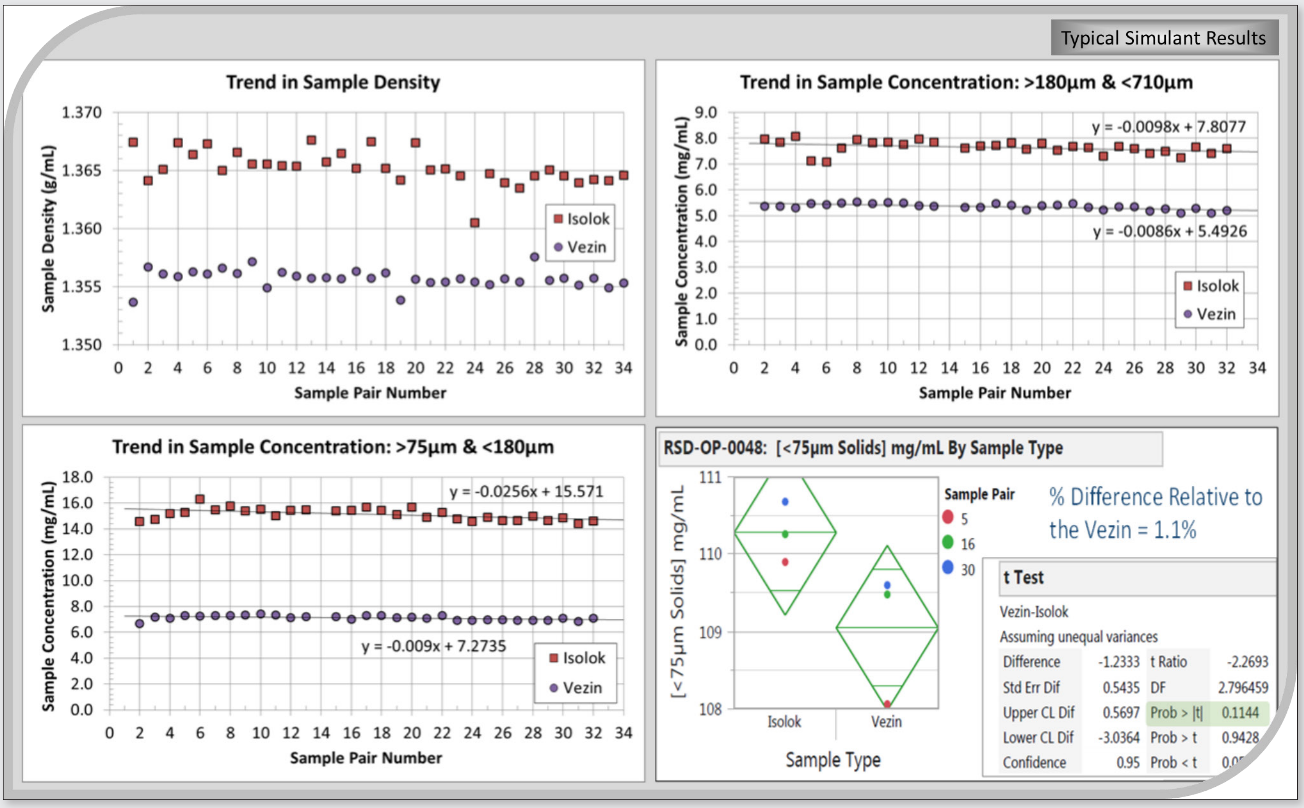
Table 3. Sampler Consistency Review – % Relative Standard Deviation

Sampler	Sample Property	N	Typical Slurry % RSD	High Slurry % RSD
Isolok®	Volume	34	0.58%	0.61%
	Density	34	0.11%	0.12%
	[180 µm]	30	3.20%	2.23%
	[75 µm]	30	2.97%	3.21%
Vezin	Volume	34	0.26%	0.30%
	Density	34	0.06%	0.04%
	[180 µm]	30	2.21%	2.84%
	[75 µm]	30	2.54%	2.84%

As is quickly evident from review of Figure 6 and Figure 7, the accuracy for fast settling solids was highly biased for the Isolok® sampler, as shown in the figures. Although only three samples from each test were analyzed for particles less than <75 µm (particle density about the same as for large sand), the very good analytical performance allows the conclusion that the typical slurry may have a slight bias and the high slurry bias may be slightly higher, around 3.7%. These biases are much less than the bias found for the large sand in these two simulants.

Review of the data was also performed by using variogram technique,[4,5] further confirming Isolok® sampler performance – as it relates to over sampling particles based on size and density. The variograms are shown in Figure 8 and Figure 9. The patterns in the plots for all three parameters, density, 180 µm sieve, and 75 µm sieve, are different between the two samplers. Changes in the Vezin plots are more smooth and orderly. This means that the Isolok® see's patterns that are not there, but given the relative standard deviation of the samples this error is most likely acceptable.

From information in Figure 6 and Figure 7 we know that the Isolok® was removing material at rates different from the bulk flow concentrations. The Vezin sampler variogram review is key to understanding the resulting slurry changes and therefore providing



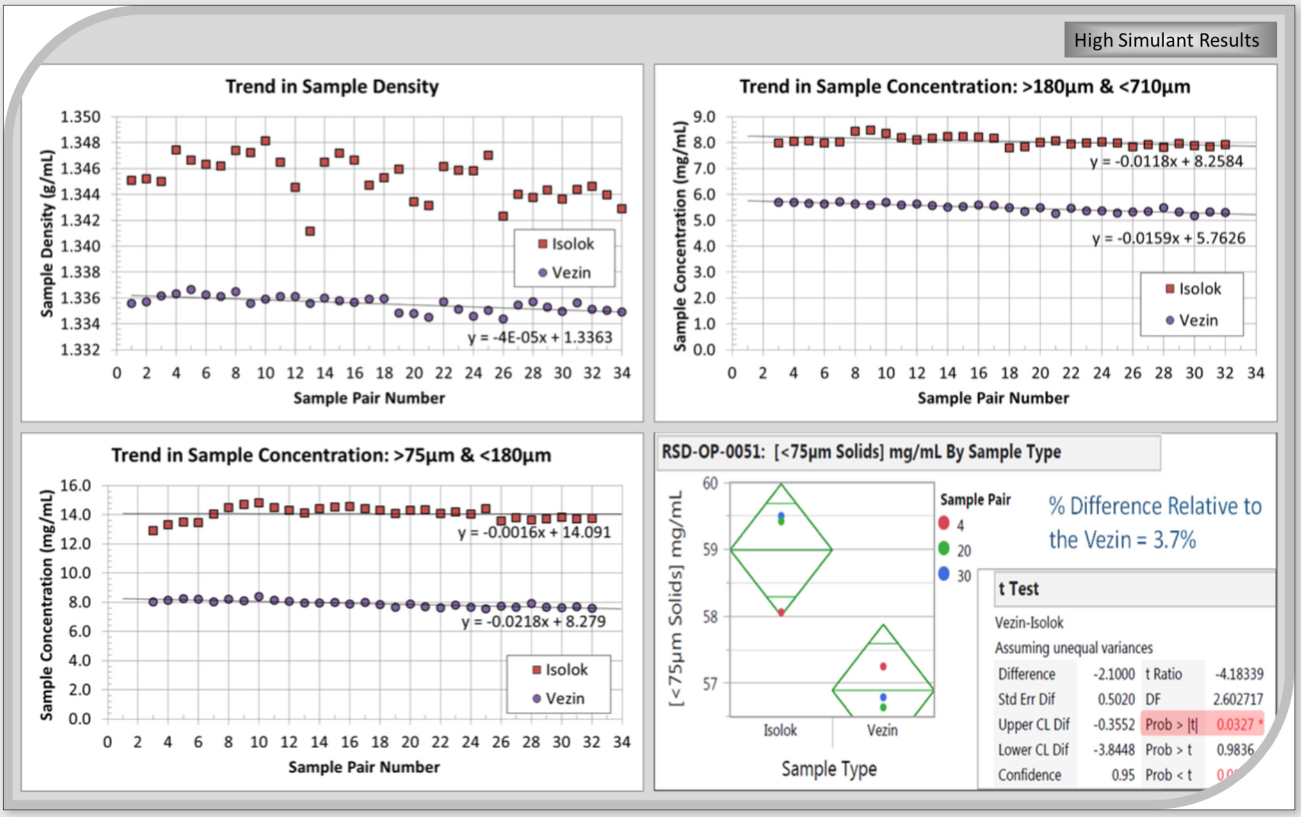
t Test

Vezin-Isolok

Assuming unequal variances

Difference	-1.2333	t Ratio	-2.2693
Std Err Dif	0.5435	DF	2.796459
Upper CL Dif	0.5697	Prob > |t|	0.1144
Lower CL Dif	-3.0364	Prob > t	0.9428
Confidence	0.95	Prob < t	0.0

Figure 6. Typical Simulant – data Run Charts and Slow Settling Solids Analysis



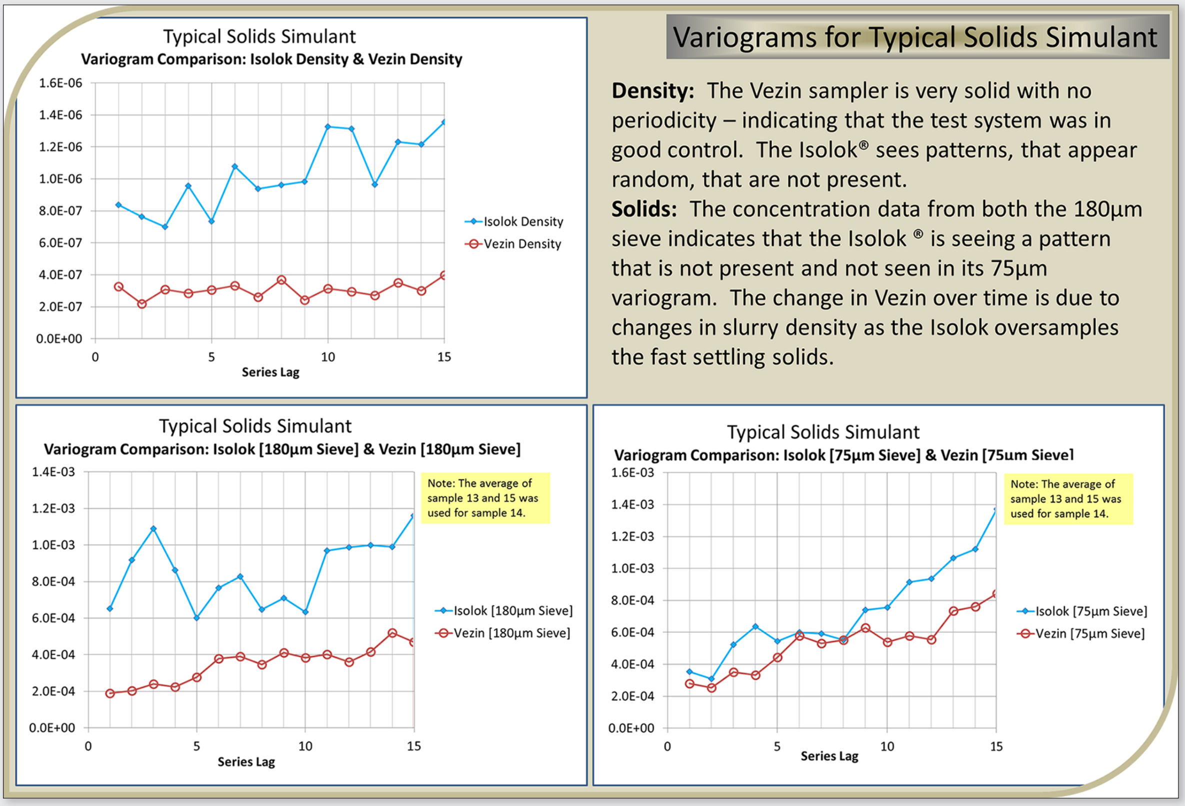
t Test

Vezin-Isolok

Assuming unequal variances

Difference	-2.1000	t Ratio	-4.18339
Std Err Dif	0.5020	DF	2.602717
Upper CL Dif	-0.3552	Prob > |t|	0.0327 *
Lower CL Dif	-3.8448	Prob > t	0.9836
Confidence	0.95	Prob < t	0.0

Figure 7. High Simulant – Data Run Charts and Slow Steeling Solids Analysis

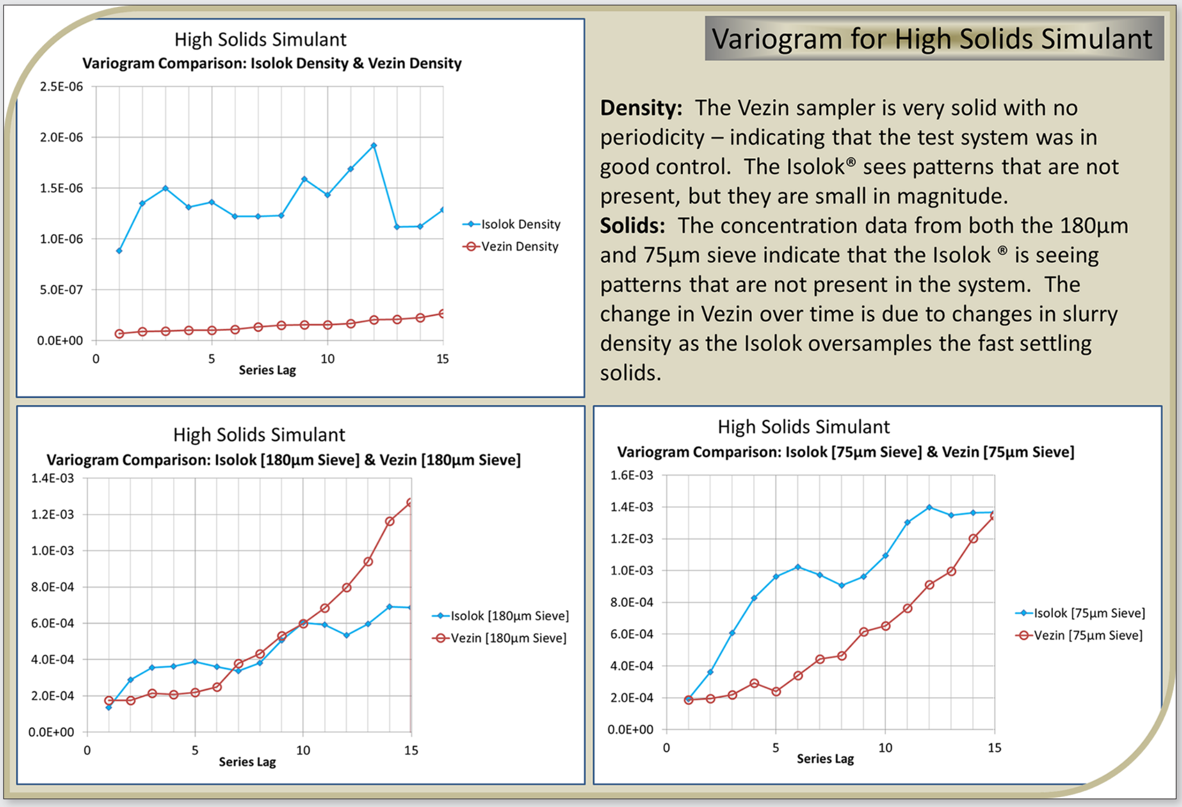


Variograms for Typical Solids Simulant

Density: The Vezin sampler is very solid with no periodicity – indicating that the test system was in good control. The Isolok® sees patterns, that appear random, that are not present.

Solids: The concentration data from both the 180µm sieve indicates that the Isolok ® is seeing a pattern that is not present and not seen in its 75µm variogram. The change in Vezin over time is due to changes in slurry density as the Isolok oversamples the fast settling solids.

Figure 8. Typical Simulant – Variogram Review



Variogram for High Solids Simulant

Density: The Vezin sampler is very solid with no periodicity – indicating that the test system was in good control. The Isolok® sees patterns that are not present, but they are small in magnitude.

Solids: The concentration data from both the 180µm and 75µm sieve indicate that the Isolok ® is seeing patterns that are not present in the system. The change in Vezin over time is due to changes in slurry density as the Isolok oversamples the fast settling solids.

Figure 9. High Simulant – Variogram Review

Table 4. Isolok® Bias by Slurry Type

Sample Property	Typical Slurry (%Bias)	High Slurry (%Bias)
Density	0.7 ± 0.1%	0.7 ± 0.1%
[180 µm] (g/mL)	43.0 ± 4.4%	46.9 ± 3.6%
[75 µm] (g/mL)	112.6 ± 4.4%	78.2 ± 7.1%
[<75 µm] (g/mL)	Not statistically significant at the 95% confidence interval.	3.7%

more insight to Isolok® sampler performance. The Vezin typical slurry variograms have trends, but they are not strong or without the presence of noise. Slow settling solids make up 90 wt% of the solids in the typical slurry, and are 78% gibbsite (the smallest and lightest particles in the simulant). The lack of strong trends relative to sampling noise, and no measurable change in critical velocity from start to end of test shows there was little change in slurry properties over the course of this test.

The variograms for the high slurry Vezin samples show a different picture; trends are clear and with little noise. All three figures, slurry density as well as the 180 µm sieve, and 75 µm sieve concentrations, have very precise and predictable trends. The weight percent of fast settling solids between the two slurries was very similar, about 0.9 wt % for the typical and about 0.95 wt % for the high. The slow settling solids in the high slurry solids was primarily (76%) sand, larger and denser than the gibbsite used in the typical slurry. A slight drop in critical velocity was measured from start to end of the high slurry test. (If sampling was ideal, no change in simulant composition would occur and the critical velocity would be constant.)

Therefore, review of both the standard data analysis techniques and variograms show that the Isolok® oversamples particles at different rates based on particle size, particle density, and simulant component make up. This supports the <75 µm sample analysis results. The typical simulant showed no (or possibly a small) bias is present for the slow-settling solids. For the high simulant there was a marketable drop in the estimated sand bias from ~45% for the large sand to ~5% for the small sand.

Conclusions

Vezin samplers are well documented as being equiprobabilistic, proportional, and following good sampling protocol [4]. But more than just selection of the Vezin type should be considered during sampler design and installation. Results presented here show modification of features that can instigate particle accumulation should be performed during design and construction. Surface areas which are in excess of those needed to ensure a smooth flow path from the sampler's cutter to sample container should be minimized. Implementing modifications to mitigate these items allowed testing to be performed much more efficiently and most likely with less error. Review of Vezin data by standard run charts and variogram analysis showed that:

- The test loop was consistent throughout each test run.
- No spikes in slurry densities were observed from start to end of testing.
- The Vezin itself was telling the truth, i.e., it reflected what was in the test loop during any given sample pair.

The Sentry Liquid Isolok® MSE sampler, which does not follow good sampling protocol, was tested to determine its performance versus a two stage Vezin sampler for two relatively low weight percent solids slurries. Review of Isolok® data versus the Vezin data show that the Isolok® sampler oversamples undissolved solids based on particle size and density. See Table 4. As either particle size or density increase, so does the rate of over sampling. The rate of over sampling is also influenced by other particles and their concentrations in the slurry. The Isolok® also saw patterns in the test loop that were not there, however the error due to these signals were not significant compared to the sampling bias.

When reviewing the Isolok®, we should remember that it was designed for sampling of homogeneous liquids. See Table 5. However, due to its features (compact, enclosed, and easily automated), its use can easily be desired for applications outside its application; provided its limitations are understood and accounted for. The use of an Isolok® MSE sampler for obtaining Hanford's radioactive waste material will be based on its sampling performance (including data presented here) and its physical attributes as they relate to operational goals and data quality objectives to be applied to the sampled material. The data quality objectives have not been defined yet.

Table 5. Isolok® Bias by Slurry Type

Isolok®	Vezin
■ Cons ▪ Not Equiprobabilistic ☐ Delimitation Error ☐ Extraction Error ☐ Segregation Error ☐ Periodic Heterogeneity Fluctuation Error ■ Pros ▪ Handling ☐ Preparation Error ☐ Good Contamination Control ▪ Size / Increment ☐ Fundamental Error ☐ Long-Range Heterogeneity Fluctuation Error ☐ Periodic Heterogeneity Fluctuation Error	■ Cons ▪ Handling ☐ High Possibility of External Contamination ■ Pros ▪ Handling ☐ Preparation Error ▪ Equiprobabilistic ☐ Delimitation Error ☐ Extraction Error ☐ Segregation Error ☐ Periodic Heterogeneity Fluctuation Error ▪ Size / Increment ☐ Fundamental Error ☐ Long-Range Heterogeneity Fluctuation Error ☐ Periodic Heterogeneity Fluctuation Error

Acknowledgements

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