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In-Tank Elutriation Test Report and Independent Assessment

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EXECUTIVE SUMMARY

The Department of Energy (DOE) Office of Environmental Management (EM) funded Technology Development and Deployment (TDD) to solve technical problems associated with waste tank closure for sites such as Hanford Site and Savannah River Site (SRS). One of the tasks supported by this funding at Savannah River National Laboratory (SRNL) and Pacific Northwest Laboratory (PNNL) was In-Tank Elutriation. Elutriation is the process whereby physical separation occurs based on particle size and density [Kirk-Othmer, 2006].

This report satisfies the first phase of Task WP-1.3.1.1 In-Tank Elutriation, which is to assess the feasibility of this method of separation in waste tanks at Hanford Site and SRS. This report includes an analysis of scoping tests performed in the Engineering Development Laboratory of SRNL, analysis of Hanford's inadvertent elutriation, the viability of separation methods such as elutriation and hydrocyclones and recommendations for a path forward.

This report will demonstrate that the retrieval of Hanford salt waste tank S-112 very successfully decreased the tank's inventories of radionuclides. Analyses of samples collected from the tank showed that concentrations of the major radionuclides, Cs-137 and Sr-90, were decreased by factors of 250 and 6 and their total curie tank inventories decreased by factors of 60,000 and 2000. The total tank curie loading decreased from 300,000 Ci to 55 Ci. The remaining heel was nearly all innocuous gibbsite, $\text{Al}(\text{OH})_3$. However, in the process of tank retrieval approximately 85% of the tank gibbsite was also removed. Significant amounts of money and processing time could be saved if more gibbsite could be left in tanks while still removing nearly all of the radionuclides.

There were factors which helped to make the elutriation of Tank S-112 successful which would not necessarily be present in all salt tanks.

1. The gibbsite particles in the tank were surprisingly large, as much as 200 μm . The gibbsite crystals had probably grown in size over a period of decades.
2. The radionuclides were apparently either in the form of soluble compounds, like cesium, or micrometer sized particles of actinide oxides or hydroxides.
3. After the initial tank retrieval the tank contained cobble which is not conducive to elutriation. Only after the tank contents were treated with thousands of gallons of 50 wt% caustic, were the solids converted to sand which is compatible with elutriation.

Discussions between SRNL and PNNL resulted in plans to test elutriation in two phases; in Phase 1 particles would be separated by differences in settling velocity in an existing scaled tank with its associated hardware and in Phase 2 additional hardware, such as a hydrocyclone, would be added downstream to separate slow settling particles from liquid. Phase 1 of in-tank elutriation was tested for Proof of Principle in the Engineering Development Laboratory of SRNL in a 41" diameter, 87 gallon tank. The tank had been previously used as a 1/22 scale model of Hanford Waste Tank AY-102. The objective of the testing was to determine which tank operating parameters achieved the best separation between fast- and slow-settling particles.

For Phase 1 testing a simulated waste tank supernatant, slow-settling particles and fast-settling particles were loaded to the scaled tank. Because this was a Proof of Principle test, readily available solids particles were used that represented fast-settling and slow-settling particles. The tank contents were agitated using rotating mixer jet pumps (MJP) which suspended solids while liquid and solids were drawn out of the tank with a suction tube. The

goal was to determine the optimum hydraulic operating conditions to achieve clean separation in which the residual solids in the tank were nearly all fast-settling particles and the solids transferred out of the tank were nearly all slow-settling particles. Tests were conducted at different pump jet velocities, suction tube diameters and suction tube elevations. Testing revealed that the most important variable was jet velocity which translates to a downstream fluid velocity in the vicinity of the suction tube which can suspend particles and potentially allow their removal from the tank. The optimum jet velocity in the vicinity of the suction tube was between 1.5 and 2 ft/s (4 – 5 gpm). During testing at lower velocities a significant amount of slow-settling particles remained in the tank. At higher velocities a significant amount of fast-settling particles were elutriated from the tank. It should be noted that this range of velocities is appropriate for this particular geometry and particles. However, the principle of In-Tank Elutriation was proved.

In-tank elutriation has the potential to save much money in tank closure. However, more work, both analytical and experimental, must be done before an improved version of the process could be applied to actual waste tanks. It is recommended that testing with more prototypic simulants be conducted. Also, scale-up criteria for elutriation and the resulting size of pilot scale test equipment require investigation during future research. In addition, it is recommended that the use of hydrocyclones be pursued in Phase 2 testing. Hydrocyclones are a precise and efficient separation tool that are frequently used in industry.

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LIST OF ABBREVIATIONS

DST	Double Shell Tank
DWPF	Defense Waste Processing Facility
EDL	Engineering Development Laboratory
HLW	High Level Waste
LAW	Low Activity Waste
MJP	Mixer Jet Pump
PNNL	Pacific Northwest National Laboratory
PSD	particle size distribution
PVC	polyvinyl chloride
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SST	Single Shell Tank
TPA	Tri Party Agreement
WTP	Waste Treatment Plant

1.0 Introduction

1.1 Background

Hanford Site and Savannah River Site both have dozens of waste tanks that contain approximately one million gallons of radioactive waste each. Both sites are in the process of emptying tanks and then grouting them for long term disposition. In 1989 the U. S. Department of Energy (DOE), which operates the Hanford Site, the U. S. Environmental Protection Agency, and the State of Washington signed a comprehensive cleanup and compliance agreement called the Tri Party Agreement (TPA). The TPA goal for emptying tanks is to remove waste until less than 360 cubic feet of waste is left [Barton 2009]. Starting in 2003 Hanford Site inadvertently discovered a method involving elutriation that has the potential to greatly decrease the amount of radioactive contaminants left in a salt waste tank. Therefore, it may be possible to leave more non-hazardous material in the tank and reduce the cost of tank closure.

1.2 Benefits of Elutriation

Elutriation is a process of separating particles from one another based on size and density relationships [Kirk-Othmer]. Usually performed up-flow, it might be thought of as the inverse of differential settling. As the result of an inadvertent elutriation, retrieval of Tank S-112 waste led to a heel composed almost entirely of large (~100 μm) particles of pure gibbsite, $\text{Al}(\text{OH})_3$. Concentrations of radionuclides in the heel, probably in the form of soluble compounds or micron sized particles, were greatly reduced relative to the pre-retrieved waste, e.g., the plutonium-239 concentration was a factor of 6 lower in the heel, curium 16 times lower, strontium 8 times lower and cesium was 246 times lower. These reductions occurred through a combination of dissolution, caustic digestion, and elutriation. Presumably, if the physical separation during retrieval was carried out intentionally, as with in-tank elutriation hardware, even better radionuclide removal from the gibbsite heel could have been obtained. If state and federal regulators could be convinced that this purified gibbsite heel does not represent a significant environmental hazard -- even if it exceeded 360 ft^3 in volume -- then the heel could be grouted in place during tank closure. Most of the aluminum compounds left in the tank would not be delivered to the Waste Treatment Plant (WTP), thus reducing the sodium hydroxide demand for aluminum dissolution in the WTP and decreasing the treatment time.

The primary benefit associated with successful implementation of In-Tank Elutriation would be a reduction in the volume of immobilized low-activity waste (LAW) glass to be disposed at Hanford, due to the reduced amount of aluminum in the feed to WTP and the reduced amount of sodium added at WTP for processing that aluminum. This would lead to lower costs and shorter treatment time.

2.0 Analysis of Tank S-112 Elutriation –Independent Assessment

2.1 Description of Tank S-112 Retrieval and Inadvertent Elutriation

This section is a summary of previous reports and presentations.

Cantrell, et al. [2008] summarized the retrieval of Hanford Tank S-112, shown in Figure 1. The tank went into service in 1952 and was declared inactive in 1976. The tank diameter and height are 75' and 25', respectively. Over its service life it was used to store waste from the redox process, evaporator bottoms, and recycling streams from 242-S Evaporator-Crystallizer. Waste

retrieval from Tank S-112 occurred in two phases. Phase I, from September 2003 to May 2005 used salt cake dissolution and modified sluicing (using nozzles to spray the salt with water or salt solution) and removed all but 28,000 gal or 95% retrieval. In the second phase the Remote Water Lance (RWL) was installed to retrieve the hard heel. This was followed by two 25% caustic additions, enhanced sluicing and 50 wt% caustic addition. After final retrieval the residual waste volume was 2390 gal.

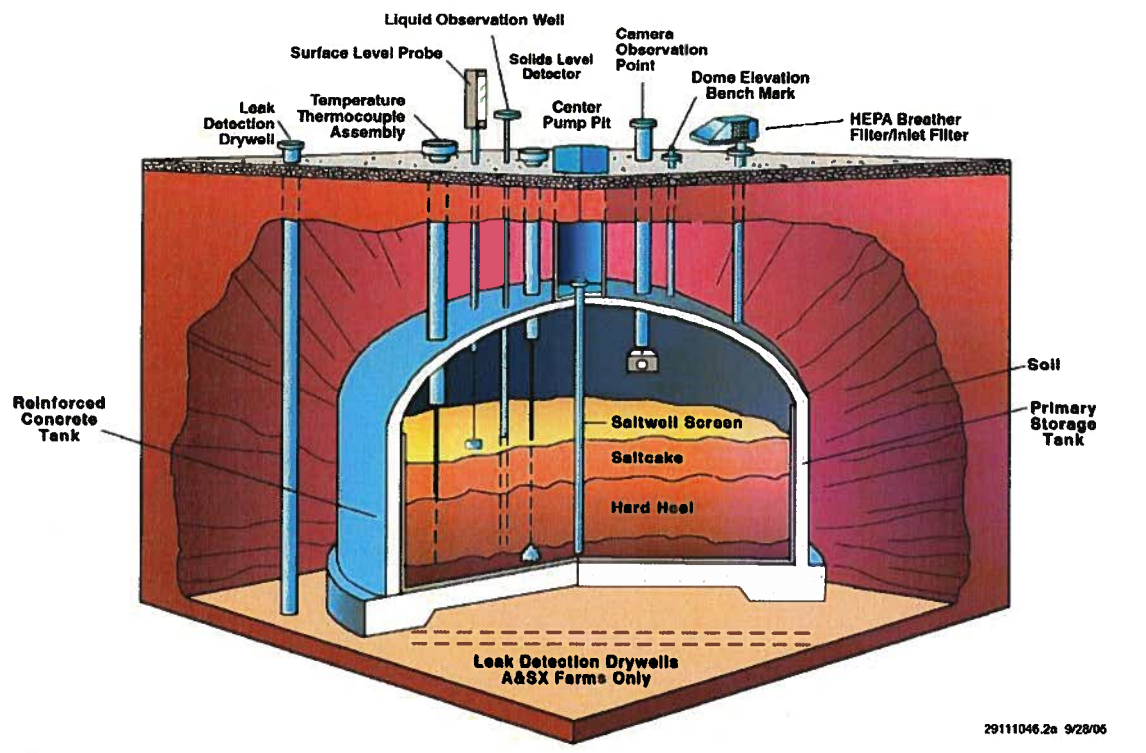


Figure 1 Generalized Drawing of 100 Series Hanford Tank

Eacker, et al [2003] described the plans for retrieving Tank S-112 and four other waste tanks at Hanford prior to retrieval. The retrieval technologies did not use previous sluicing practice which required large quantities of water. S-112 retrieval was performed with fresh water sluicing and high flow pumping systems and was expected to require two or three weeks. They planned to maintain liquid level below known tank wall leaks. S-112 retrieval was scheduled to be completed by Fall, 2003. Retrieval was to begin at the center of the tank and progress outward and use up to three directional sluicing nozzles and an automatic dual nozzle tank cleaning device. Planned pressure and flow were 550 kPa and 375 L/min (75 psig and 100 gpm). The transfer pump was to be a 100 gpm progressive cavity pump or vertical turbine pump. The report included SEM images of particles as large as 300 μm .

Dodd [2008] reported that an estimated 53 million gallons of high-level trans-uranic and low level radioactive waste is stored underground in 149 Single Shell Tanks (SST) and 28 Double Shell Tanks (DST). The SST range in size from 55,000 gallon to 1,000,000 gallon. TPA requires removal of as much waste as technically possible, with waste residues not to exceed 360 ft^3 in 530,000 gallon or larger tanks. Exceptions may be requested. Formerly they used Past Practice Hydraulic Sluicing with large volumes of DST supernatant and water. Concern over possible leaks led to the need for retrieval methods that used smaller volumes of water in a more controlled manner. As of 2008 waste retrieval had been completed at seven Hanford Site, SST

including S-112. S-112 retrieval was completed 2/28/2007 meeting the TPA Limits of less than 360 ft³ using salt cake dissolution, modified sluicing, in-tank remote controlled vehicle with high pressure water spray, and caustic dissolution. Some tanks were used in cascades such that solids separated and settled and less radioactive liquids flowed on to another tank. About 75% of the radioactivity is from Sr-90, 24% is Cs-137 and the remainder is actinides.

Barton [2009] presented a discussion of the retrieval of Tank S-112. Prior to retrieval, the tank contained 614,000 gal of mostly soluble salt. Barton expressed the opinion that the actinides in the tank, which constituted a small fraction of the total mass, were in the form of insoluble micrometer sized oxide or hydroxide particles. Starting in 2003 Hanford Site began to de-inventory Salt Tank S-112 at Hanford Site as part of final disposition for the tank, which is 75' diameter and 24' tall. Hanford Site is somewhat limited in their options for performing waste removal from tanks like this because there is no steam or ventilation available and limited access to water and electricity. In preparation for tank retrieval, workers used a water lance to dig a hole in the center of the tank and lowered a 90 gpm pump into the hole. Then they simultaneously used sluicing nozzles at the periphery of the tank. The nozzle pressure and flow were 100 psig and 70 - 90 gpm. This was done for 300 - 500 hours. When 25,000 gallons of salt were left, the surface was hard and relatively impermeable, like a salt lick, so they used a Salt Mantis with high pressure water to break it up. The resulting solids were light tan in color and consisted of cobble (chunks of solids the size of cobblestones) and sand. During the period from June to August 2006 they attempted to leach the residuals using 8 molar caustic solution. It was not clear that the caustic had any beneficial effect, but they were able to sluice out about one thousand additional gallons of waste from the tank.

In February 2007, Hanford workers pumped 19 M NaOH solution into the tank in anticipation that the higher viscosity and density of that liquid would facilitate solids removal. It was not necessary to purchase additional caustic because caustic was needed for corrosion control in a downstream tank. When they returned after a weekend, three days later, they saw foaming which was evidence of reaction. The cobble had disintegrated into sand. Pumpout without sluicing retrieved 1500 gal. When they were finished, the tank contained 2400 gallons, mostly gibbsite in the size range from 70 μm to 200 μm. Gibbsite that large was surprising since it is difficult to purchase gibbsite that large. Gibbsite crystals may have grown over 30 years in the tank. The removal pump does not effectively lift particles larger than 100 μm. During the period June – September 2007 tank solids were sampled and analyzed. Some of the solids samples were treated with different concentrations and temperatures of caustic. The dissolution tests showed no reaction at 8 M and ambient temperature and complete reaction with 19 M in less than two weeks. About three gallons of caustic was required per gallon of solids. Heating would greatly speed dissolution but probably is not possible for SST.

2.2 Chemical Analysis of Tank S-112 Contents Before and After Retrieval

The contents of Tank S-112 were sampled at different locations at least four times over a period of years before the tank contents were retrieved and again on 6/7/2007 after retrieval. The remaining solids, mostly gibbsite Al(OH)₃, were sampled after salt removal and were analyzed for a suite of elements and anions. Aluminum and silicon were tested both by acid digestion and fusion with potassium hydroxide. Acid digestion can be incomplete when both aluminum and silicon are present. Consequently, fusion results were used to calculate average tank concentration when results from both methods were available. Also, composite core samples rather than sub-segment samples were used for the “before” samples because they are more representative of tank contents. The results for chemical species and radionuclides are summarized in Table 8 and Table 9. Significant reduction in the radionuclide inventory was realized during the retrieval of Tank S-112 with the most important results summarized as follows.

1. There were large decreases in the concentrations of nitrate and nitrite by factors of 17,000 and 1000 because those species are very soluble in water.
2. Because other species were preferentially depleted, the concentration of aluminum increased by a factor of 35. However, most of the mass of aluminum was removed from the tank and the final mass of aluminum was one-seventh of the original mass.
3. The two biggest sources of radioactive loading (curies) were Cs-137 and Sr-90. Their concentrations were decreased by factors of 250 and 6 and their tank inventories decreased by 60,000 and 2000.
4. The final curie inventory was estimated to be 55. According to Appendix F in a report by the National Research Council [2006] the curie inventory in Hanford Tank C-203, for which retrieval is considered completed, was 36. Therefore, the total curie inventory for Tank S-122 is comparable to a completed tank.

In summary, the sampling process used by Hanford in assessing the inadvertent elutriation was considered fairly representative with consideration of the uncertainties addressed below.

1. There is uncertainty in characterizing a tank using a limited number of small samples, rarely more than six. At worst the standard deviation of the samples was one-third of the average value.
2. For the purpose of computing total percent or total mass, aluminum was assumed to be in the form of gibbsite, molecular weight 78, silicon was assumed to be silicate, molecular weight 76 and sulfur was assumed to be sulfate, molecular weight 96. Using those molecular weights, as shown in Table 1 the sums of the mass percentages of the major species for "before" and "after" were 87% and 97%. Therefore, the sampling process was fairly representative.

2.3 Evaluation of Hanford's Inadvertent Elutriation

Elutriation of Hanford Tank S-112 would not have been as successful without the addition of large quantities of 50 wt% caustic. The consequences of adding large quantities of caustic to other tanks should be analyzed with the following considerations:

- Addition of caustic impacts available Double Shell Tank (DST) space and will require the evaporation of an additional 4 – 6 L water per liter waste and the estimated cost to evaporate is \$10/gal. The minimum batch size is probably 10,000 gal of 50% caustic. Three truckloads of caustic or 12,000 gallons will react with 4200 gal of gibbsite solids.
- If high caustic is used to break up cobble, the solids should first be washed with water to remove phosphate and oxalate which would otherwise precipitate with the caustic treatment. The concept may be implemented without changes to modified sluicing retrieval systems.

In summary, based upon the Hanford experience of the inadvertent elutriation, in-tank elutriation was shown (i) to be useful in salt waste tanks and (ii) successful implementation of elutriation requires preliminary steps which have costs associated with them. The preliminary steps required for elutriation are removal of soluble salts, breaking up hard salt pan, and digesting cobble to sand using caustic solution. Since the salt waste tanks probably can not be heated, this probably means treatment with stronger than 25% caustic, although maybe not 50%. Therefore, a cost and benefit analysis should be performed first. The ability to leave more innocuous gibbsite in a waste tank has benefits, but the introduction of large quantities of caustic into the waste stream has costs. The cost and benefit analysis should also consider how many tanks could use elutriation.

3.0 Elutriation Scoping Tests at SRNL

3.1 Purpose of Scoping Tests

The purpose of the scoping tests was to demonstrate that waste tank particles can be elutriated or separated in the tank by using an existing SRNL mixing and transfer demonstration system. Specifically, parameters that influence the elutriation process were to be varied over a range. The scoping tests used the AY-102 simulant on hand with slow-settling particles (gibbsite, 30 μm , sp. gr. 2.42) and fast-settling particles (stainless steel, 75 μm – 100 μm , sp. gr. 8.03).

3.2 Description of equipment

A photograph and schematic of the elutriation tests system are shown in Figure 2 and Figure 3. This test system was used earlier for batch transfer demonstrations (Adamson 2009). The mixing tank wall and bottom are transparent to allow visual observations. The mixing tank (40.5" diameter, 30" height) is a 1/22nd scale replica of Hanford Tank AY-102. Table 1 provides pertinent design and operating parameters for the full size AY-102 waste tank and the 1/22nd scale test tank.

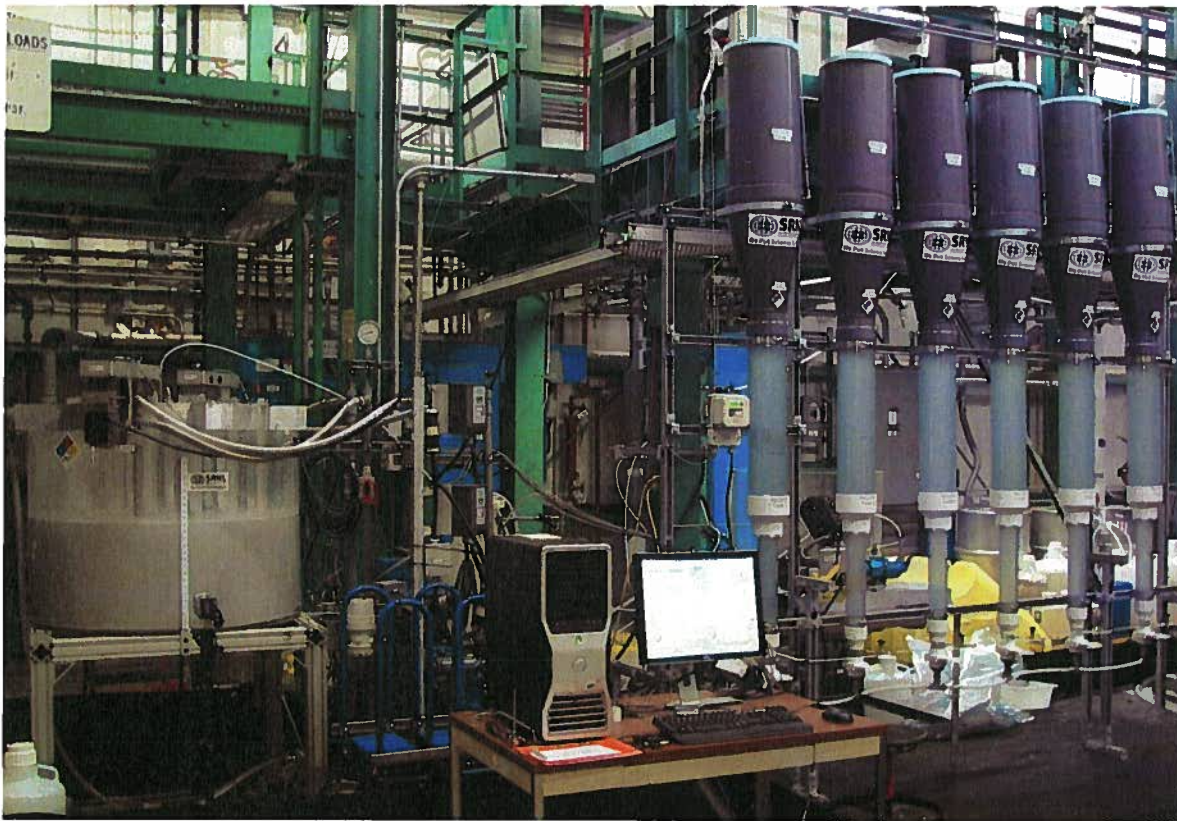


Figure 2 Photograph of the Mixing/Transfer Demonstration System used for the In-Tank Elutriation Scoping Tests

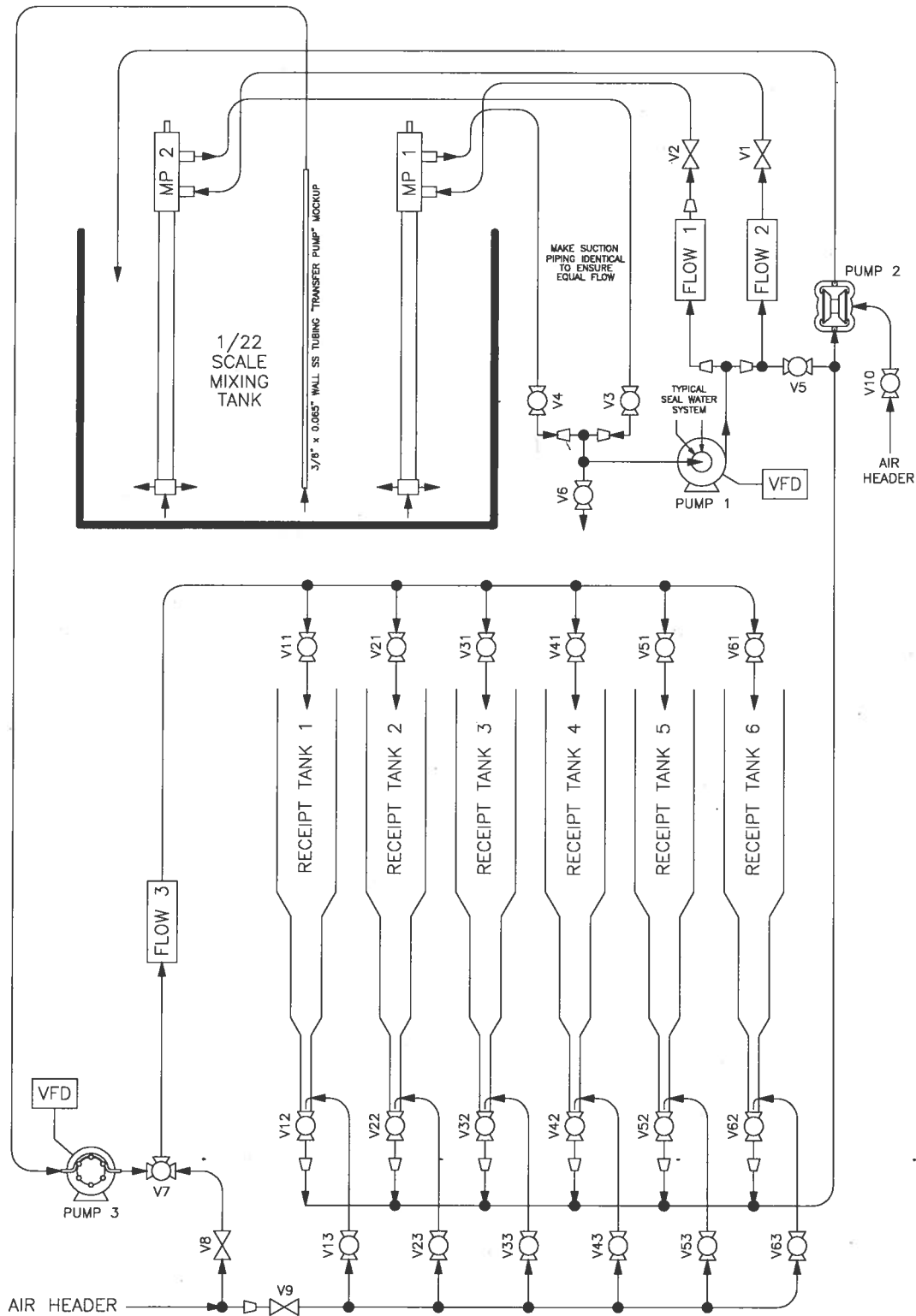


Figure 3: Schematic of the Elutriation Test System

Table 1: Parameters Associated with Hanford Tank and SRNL Scaled Tank

Parameter Description	Full Scale Hanford	SRNL@ $1/22=0.045$ scale factor
Tank diameter	75 ft (900 inches)	40.5 inches (actual 40.9")
Tank operating height	364 inches	16.5 inches
Total waste height	347 inches	15.6 inches
Sludge height	55 inches	2.49 inches (actual 1.75")
Supernatant height	292 inches	13.2 inches
Total waste volume	955,085 gal	87 gallons
Batch volume to WTP	160,000 gal	14.3 gal
Residence mixing time AY-102	45.2 minutes	
Flow for scaled model to have full scale residence time	-	0.47 gal/min/nozzle
Nozzle velocity at full-scale residence	-	2.6 ft/sec
Pump location from tank center	22 ft	11.9 inches
Pump above tank bottom	6 inches	0.27 inches
Nozzle diameter	6 inches	0.27 inches
Nozzle location from bottom of pump	9 inches	0.41 inches
Pump rotational speed	0.2 rpm	1.6 rpm
Pump flowrate	5280 gal/min/nozzle	10.8 gal/min/nozzle
Nozzle Exit Velocity	60 ft/sec	60 ft/sec
U_oD	30 ft ² /s	1.35 ft ² /s
U_oD at a flowrate of 8 gpm MJP		0.504 ft ² /s
U_oD at a flowrate of 5 gpm MJP		0.315 ft ² /s
1/22 nd Pump flowrate (8 gpm)	-	4 gal/min/nozzle
Nozzle Exit Velocity		22.4 ft/sec/nozzle
1/22 nd Pump flowrate (5 gpm)	-	2.5 gal/min/nozzle
Nozzle Exit Velocity	-	14 ft/sec/nozzle
Pump flowrate (power cal.)	5280 gal/min/nozzle	5.0 gal/min/nozzle
Nozzle velocity (power calculation)	60 ft/sec	28 ft/sec
U_oD (power calculation)	-	0.63 ft ² /s
Liquid density	1,150 kg/m ³	1,289 kg/m ³
Solids density	2,500 kg/m ³	Gibbsite: 2,420 kg/m ³ Stainless steel: 8,000 kg/m ³
Viscosity of liquid	2.8 cP	2.55 cP
Air Lift Circ. (ALC) dia. (22 in tank)	30 inches	1.35" (used 1.25")
ALC elevation above tank bottom	30 inches	1.35 inches
Heating coil dia. (1 in tank)	40.375 inches	1.8" (used 1.75")
Transfer pump outer diameter	12 inches	0.54" (used 3/8" sst tube)
Transfer pump inlet diameter	2.25 inches	0.1" (used 0.125")
Transfer pump above tank bottom	5 inches	0.23 inches
Transfer pump, pump rate	90 – 140 gpm	0.58 gpm
Transfer pump, velocity	3.9 ft/s, 6.1 ft/s	3.95 ft/s in 3/8", 0.025 ft/s in 3"
Batch transfer volume	160,000 gal	14.3 gal
Particle size distribution	2.5 ~ 16.8 μ m	stainless steel - 75 to 106 μ m gibbsite 30 μ m

The test tank was provided with internal obstructions to achieve prototypic geometry. The tank has prototypical obstructions, a transfer pump feed line and two Mixer Jet Pumps (MJPs). The slurry pump that fed the mixer jets was located outside the test tank and its speed was controlled by a variable speed drive. Fluid was pumped from the test tank and then circulated back to the 1/22nd scale MJPs. Each MJP has two nozzles oriented 180° apart.

The six Receipt Tanks shown in Figures 2 and 3 are transparent PVC over most of their length to allow for measurement of the volume of solids that were transferred in each test. The Receipt Tanks hold approximately 22 gallons each.

Testing was conducted per the R&D Direction summarized in Section 3.4. Test results were also recorded in Laboratory Notebook, SRNL-NB-2010-00124.

3.3 Simulant

The supernatant used in all of the scoping in-tank elutriation testing was prepared for a previous task at SRNL. The supernatant was intended to represent waste in an average Hanford waste tank. The supernatant itself is transparent. The recipe for this simulant is given in the “Revised Preparation of Simulated Feed Solution for Pilot Plant”, CH2M-0701541.1 report (July, 2007). The recipe was modified from the report recipe by the removal of the sodium dichromate to prevent the simulant from being classified as a hazardous solution. The composition of the supernatant is shown in Table 2.

Table 2 Supernatant Simulant Used in Elutriation Scoping Tests

Chemical	g/liter
NaAlO ₂ *H ₂ O	29.18
NaOH	24.49
Na ₂ CO ₄	65.71
Na ₂ C ₂ O ₄	0.81
KNO ₃	1.80
NaNO ₃	279.63
NaNO ₂	35.88
NaSO ₄	18.41
Na ₃ PO ₄ *12H ₂ O*1/4NaOH	18.09
NaCl	4.30
NaF	0.42

The supernatant was spiked with particles that represented both fast-settling and slow-settling particles in tank waste. The critical parameter in elutriation is the settling velocity which considers both the particle size and the density of the particle. Since large-size gibbsite particles representative of those in the Hanford S-112 tank (i.e., greater than 200 μm) are not commercially available, 100 μm stainless steel was utilized in its place since the settling velocities were comparable. Small size gibbsite particles at 30 μm represented the slow-settling particles that would represent radionuclides in the tanks. The solid particles used for these tests are characterized in Table 3. The fast-settling particles were dark and the slow-settling particles were light to allow visual evaluation on the performance of the separation. Figure 4 is a photograph of the simulant in the mixing tank prior to mixing. The light color slow-settling particles are seen

resting above the thin dark color fast-settling particles. This color intensity distinction was used in the analysis of the test results.

Table 3 Particles Used in Elutriation Scoping Tests

Particle	Composition	Size (µm)	Density (kg/m ³)	Color	Mass in simulant, lb.
Fast-settling	Stainless steel	~100	8,000	dark	23.2
Slow-settling	gibbsite	~30	2,420	light	82.6



Figure 4 Photograph of Scaled Mixing Tank with Lighter Colored, Slow-Settling Particles on Dark, Fast-Settling Particles

3.4 Test Matrix

A total of seven elutriation tests were performed as shown in Table 4. The following three parameters were identified that may have an impact on the Phase 1 elutriation tests:

- mixing pump flowrate
- suction tube diameter
- suction tube elevation above tank bottom

As shown in Table 4, the flow rate for each mixing pump was varied over a range of 4 – 8 gpm, because 4 and 8 gpm were considered to be bounding cases. For the slurry transfer out of the mixing tank two suction tube sizes were tested. The first five tests used a 3/8" OD, 0.065" wall stainless steel tube providing a velocity of about 4 ft/s at the suction point. The last two tests were performed with a 3" SCH 40 pipe section at the suction point resulting in a suction velocity of

0.025 ft/s. An additional variable was the suction tube elevation above the tank bottom. Four tests were performed at a tube elevation of 0.25" and three at 1.25". Both mixing pumps rotated at a constant speed of 1.6 rpm for all seven tests. The orientations of the nozzles of the two mixing pumps were not synchronized to yield a randomized mixing operation.

Table 4 In-Tank Elutriation Test Matrix

Test No.	Test Date	Flow Rate per Mixing Pump*, gpm	Suction Tube OD, inch	Suction Tube ID, inch	Suction Tube Elevation above Tank Bottom, inch
1	9/1/2010	5	0.375	0.245	0.25
2	9/13/2010	4	0.375	0.245	0.25
3	9/16/2010	6	0.375	0.245	0.25
4	9/22/2010	5	0.375	0.245	1.25
5	9/27/2010	4	0.375	0.245	1.25
6	10/1/2010	5	3.5	3.068	0.25
7	10/4/2010	8	3.5	3.068	1.25

* Each mixing pump has two nozzles

3.5 Summary of test procedure

As discussed above, a total of seven elutriation tests were performed by following detailed set of step by step instructions, which are summarized below.

1. Load the Mixing Tank with Fractional Crystallization simulant, which was also a AY-102 simulant, as described in section 3.2 (about 87 gallons, 82.6 lbs of gibbsite (< 30 micron) and 23.2 lbs of stainless steel particles (nominally, 100 micron). Note: the simulant was mixed in 2008 so some of the sodium hydroxide reacted to form additional sodium carbonate.
2. Set the suction tube elevation as shown in the test matrix, Table 2 above.
3. Rotate the MJPs at 1.6 rpm.
5. Provide both MJPs with 10 gpm flow each to mix the tank contents for 10 minutes.
6. Pull samples from the mixed tank for particle size distribution (PSD)-analysis.
7. Reduce the MJP flow rate according to the test matrix.
8. Observe and record photographically any formation of no mixing zones ("dead zones").
9. Start transfer of first batch (about one-sixth of total inventory) out of the mixing Tank at 0.58 gpm using Pump 3.
10. At the completion of the first batch, continue transferring the subsequent five batches. Note that Pump 3 is not turned off in between different batches.
11. At the conclusion of all six transfers, stop the flow and rotation of the MJPs.
12. Collect a representative sample of the mixing tank heel contents for PSD analysis.

13. Record the levels in the Receipt Tanks for total amount of slurry and solids levels after solids have settled.
14. Mix the contents of Receipt Tanks using air sparging and collect representative samples for PSD analyses from Receipt Tanks 1, 3 and 5.
15. Transfer contents of all Receipt Tanks back to the mixing tank to prepare for the next test per the test matrix. Use air sparging as needed to empty out the Receipt Tanks.

3.6 Analysis of Elutriation Scoping Results

The elutriation results from varying the tank parameters: (i) mixing jet pump flowrate, (ii) suction tube diameter, and (iii) suction tube elevation are discussed in this section. The elutriation results were quantified using three methods:

- 1) **Visual Observation** - The first method of quantification was visual observation, which included both observations of solids remaining in the mixing tank and in the receipt tanks. The simulant was spiked with both fast-settling particles (dark color) and slow-settling particles (light color) to allow a quick visual determination of separation effectiveness. The impact of the test parameters can be immediately assessed visually and corresponded to the physical measurements used in methods 2) and 3) discussed below.
- 2) **Solids Measurement (Volume)** – The second method included measurement of the solids heights in the six Receipt Tanks after each of the tests and computation of the approximate volumes of the fast-settling and slow-settling solids. The tanks had been previously calibrated to give volume as a function of solids height [Adamson, et al. 2010].
- 3) **Particle Size Distribution Measurement** – The third method involved analytical measurement of the samples removed from the mixing tank.

3.6.1 Visual Observation

The most significant parameter tested that impacted separations was the mixing jet pump (MJP) flowrate. Figures 5 through 7 illustrate the effectiveness of the MJP flowrate in achieving separation between the particles remaining in the tank and those that are removed to the six (6) receipt tanks. In Figure 5, the separation effectiveness of using a MJP velocity of 5 gpm (Test 1) is shown in that most of the darker fast-settling solids remain in the mixing tank with very little light-colored slow-settling solids. In Figure 6, the opposite effect is shown in that most of the light-colored slow-settling solids were removed to the Receipt Tanks with very little dark-colored solids. In Figure 7, both dark and light colored solids are shown in the Receipt Tank which would be expected when the MJP flowrate was increased to 8 gpm (Test 7) to the point that even the fast-settling solids are entrained with the slow-settling solids and removed to the Receipt Tanks. Several samples were taken from both the Receipt Tank and the mixing tank to obtain particle size distribution of the solids.

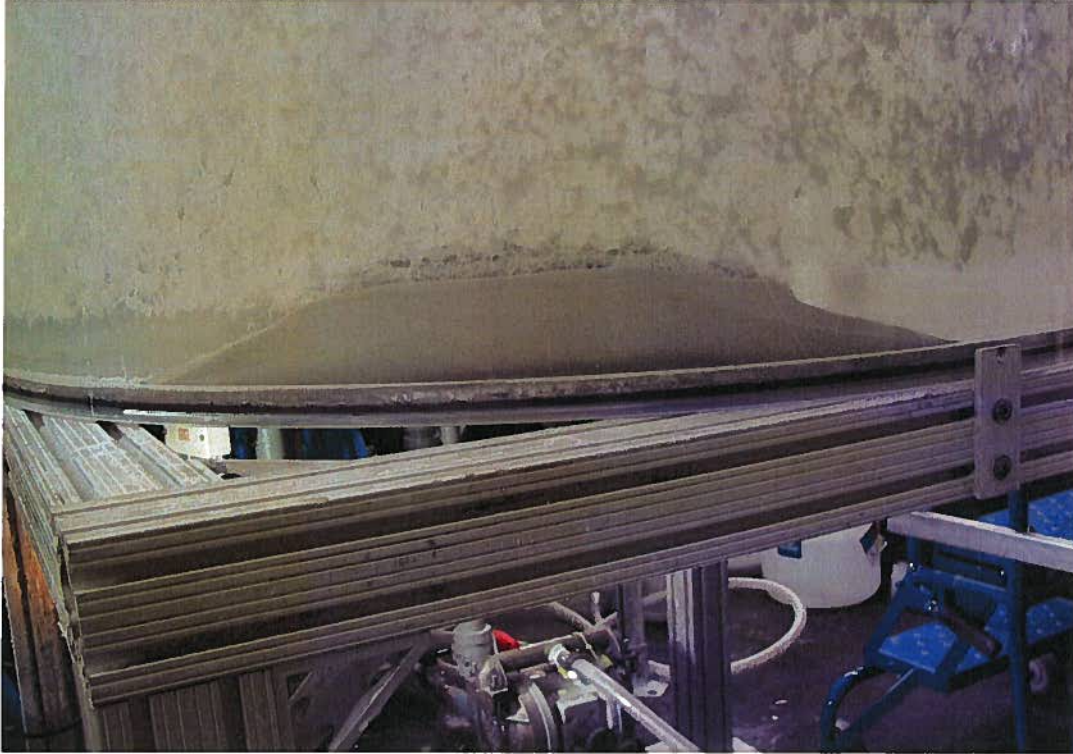


Figure 5 Mound of Dark Fast-Settling Solids Left in Mixing Tank of Test 1 at Optimum Pump Flow Rate (5 GPM)

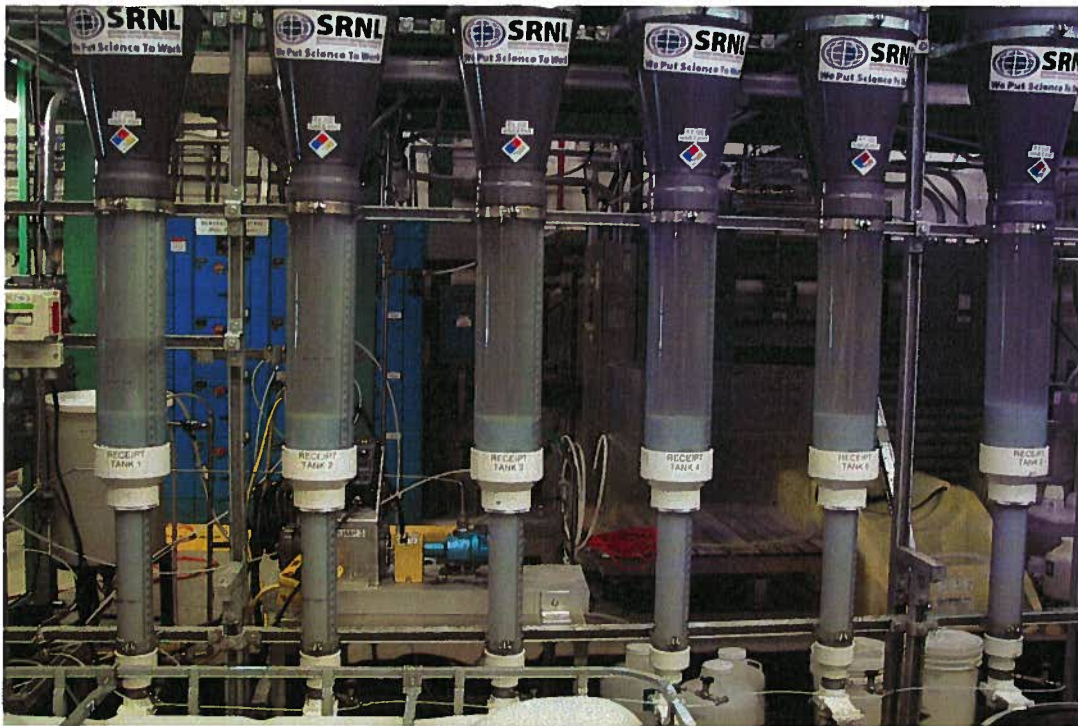


Figure 6 Light Colored Slow-Settling Solids in the Receipt Tanks of Test 1 at Optimum Pump Flow Rate (5 GPM)

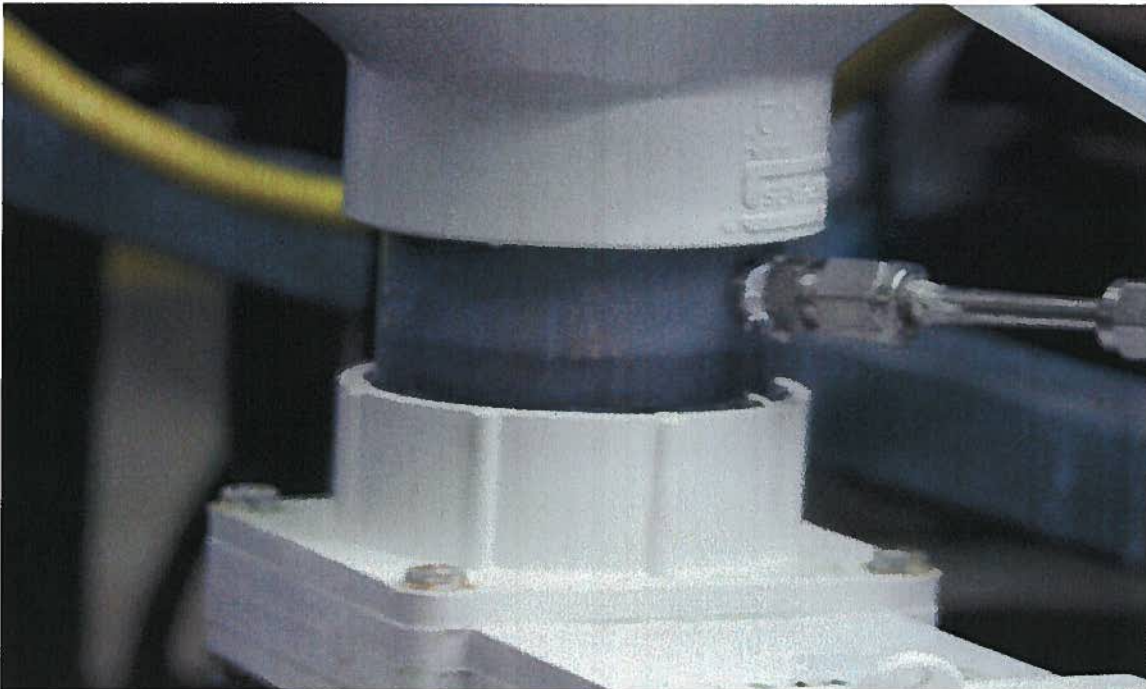


Figure 7 Dark, Fast-Settling Solids entrained and in Receipt Tank of Test 7 at High Pump Flow Rates (8 GPM)

3.6.2 Solids Measurement

For optimum separation efficiency, the goal is to operate the MJP flow rate and other tank parameters such that minimal fast-settling solids are entrained into the Receipt Tanks (RTs). Table 5 gives volumes of fast and slow-settling solids for the six Receipt Tanks (RT1-6) resulting from the seven test runs shown in Table 3. The following are observations and comments about the data shown in Table 5.

1. As expected, testing showed that less than optimum separation efficiency resulted at higher pump flow rates due to the entrainment of fast-settling solids with the slow-settling solids.
 - a. For example, tests run at the higher pump flow rates of 6 to 8 gpm (Tests 3 and 7) resulted in the most fast-settling solids in the RTs as shown in Table 5.
 - b. For tests run at 4 and 5 gpm (Tests 1, 2 and 5), Table 5 shows there was little or no fast-settling solids in the RTs.
2. The test results showed there is an optimum MJP flow rate that results in optimum separation. In this testing, tests run at MJP flow rates of 5 gpm resulted in optimum separation where the heavier fast-settling solids remained in the MDT and the lighter slow-settling solids were removed to the RTs. Test runs at higher MJP flow rates resulted in heavier solids that were entrained at the higher flow rates interfering in the removal of the lighter solids that normally would have been separated and removed to the RTs.
 - c. For test runs at the higher flow rates of 6 and 8 gpm (Tests 3 and 7) and at the higher suction tube elevation (Test 4), the RTs contained less than the average volume of slow-settling solids indicating that some slow-settling remained behind in the MDT.

Table 5 Volumes of Fast and Slow-Settling Solids

Test #	RT-1	RT-2	RT-3	RT-4	RT-5	RT-6	Total (gal)
Fast-Settling Solids							
1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3	0.25	0.24	0.23	0.23	0.23	NR*	1.17
4	0.04	0.04	0.04	0.04	0.04	0.04	0.22
5	0.00	0.00	0.00	0.00	0.00	0.00	0.00
6	0.04	0.04	0.04	0.04	0.04	0.04	0.22
7	0.14	0.10	0.11	0.10	0.11	0.00	0.56
Total							2.17
Slow-Settling Solids							
1	1.76	1.67	1.63	1.65	1.69	1.74	10.13
2	2.26	2.14	2.10	1.98	2.06	NR	10.54
3	1.70	1.72	1.67	1.61	1.70	NR	8.41
4	1.83	1.76	1.75	1.74	1.75	0.92	9.76
5	2.08	2.04	1.99	1.96	1.91	0.96	10.95
6	2.51	2.67	2.73	2.78	2.63	2.27	15.59
7	1.57	1.56	1.54	1.55	1.57	0.51	8.30
Total							

* Not measured

- b. For test runs at 4 and 5 gpm (Tests 1, 2 and 5), nearly all of the slow-settling was transferred to the Receipt Tanks. Also for Tests 1, 2 and 5 little or no fast-settling particles transferred to the Receipt Tanks.
 - c. For test runs at higher MJP flow rates and at higher suction tube elevations (Test 3, 4, 6 and 7, significant amount of fast-settling particles were transferred. For Test 3, all or nearly all of the fast-settling particles transferred.
3. The testing demonstrated excellent tank mixing and consistency in the batch transfers to each of the RTs. This consistency was demonstrated by the test results that showed an even distribution of the fast-settling solids between the six RTs. Also, for a given test, the volumes of the light- colored slow-settling solids in the six Receipt Tanks were approximately equal.
 4. There were sources of uncertainty in the data:
 - a. In the previous section of this report it was noted that 82.6 pounds of gibbsite (slow-settling solids) was initially added to the mixing tank. The “loose pack” bulk density of gibbsite was measured and was approximately 0.86 g/mL. Note, that this density is much less than the published density of non-porous gibbsite, 2.42 g/mL. The mass of gibbsite translates to a volume of 11.5 gal. The total volumes of gibbsite in Table 5 ranged from 8.3 gal to 15.59 gal. It is not known why for Test 6 there appeared to be more total gibbsite volume in the Receipt Tanks than was originally in the mixing tank.

- b. In the previous section of this report it was noted that 23.2 pounds of stainless steel powder were initially added to the mixing tank. The “loose pack” bulk density of stainless steel powder was measured and was approximately 2.55 g/mL. Note, that this density is much less than the published density of stainless steel, 8.03 g/mL. The mass of stainless steel translates to a volume of 1.09 gal.
- c. Figure 6 shows that some of the plastic fittings in the Receipt Tanks are opaque. Therefore, there is large uncertainty associated with some of the stainless steel volumes. For example, the readings of zero volume may actually be 0.01 gallon. Also, the uncertainty for stainless steel volumes for Test 7 is $\pm 35\%$.
- d. Gibbsite heights for RT-6 are uncertain because the readings were obscured by an opaque 6” reducer.

3.6.3 Particle Size Distribution Method

The final method for quantifying separation performance was by use of Particle Size Distributions (PSDs). Figure 8 shows the PSD of a sample from the mixing tank that contains both fast-settling and slow-settling particles prior to testing and compares it to the PSDs of vendor samples for both fast- and slow-settling particles. The fast-settling, stainless steel particles had been sieved by the manufacturer to be 106 μm or less, however the PSD volume peaks were at 134 μm as shown in Figure 8. The vendor’s method of cold-working the stainless steel altered the crystal structure to make the particles magnetic and allowed some of the particles to form clumps as large as 300 μm . However, in the RTs, the sizes were at a maximum of 100 microns because the clumps were sheared apart inside the transfer and mixing pumps. Figure 8 shows the bi-modal size distribution in the simulant which is anticipated with the addition of both the larger fast-settling stainless steel particles and the smaller slow-settling particles (approximate peak at 23 μm).

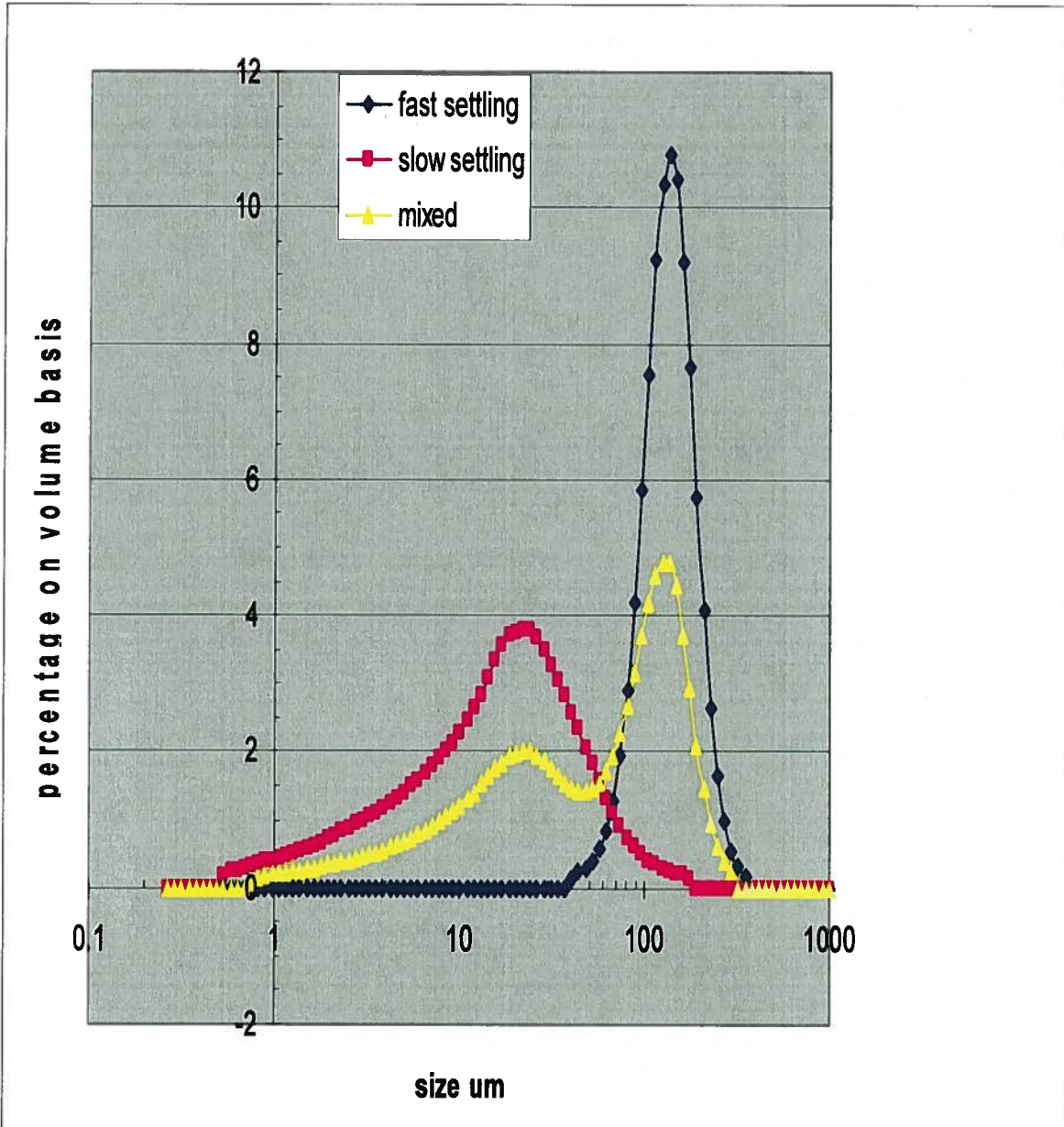


Figure 8 Bimodal Particle Size Distribution in Elutriation Simulant

Figure 9 shows the PSDs for the residual solids in the tank after the completion of elutriation as well as the PSD for the as-received (vendor) fast settling particles. For the as-received fast-settling particles there were no particles with sizes less than or equal to 37 μm. However, in some of the tests, particularly tests conducted at slower MJP velocities of 4 gpm (Tests 2 and Test 5), the PSDs indicated a significant number of particles with sizes less than or equal to 37 μm. Note those tests had the lowest flow rate of 4 gpm per MJP. These results indicate that tests at the lower pump velocity of 4 gpm did not result in optimum elutriation efficiency. The velocity was too low to remove all of the slow-settling particles to the receipt tank, and hence, the reason for smaller size particles remaining in the Mixing Tank.

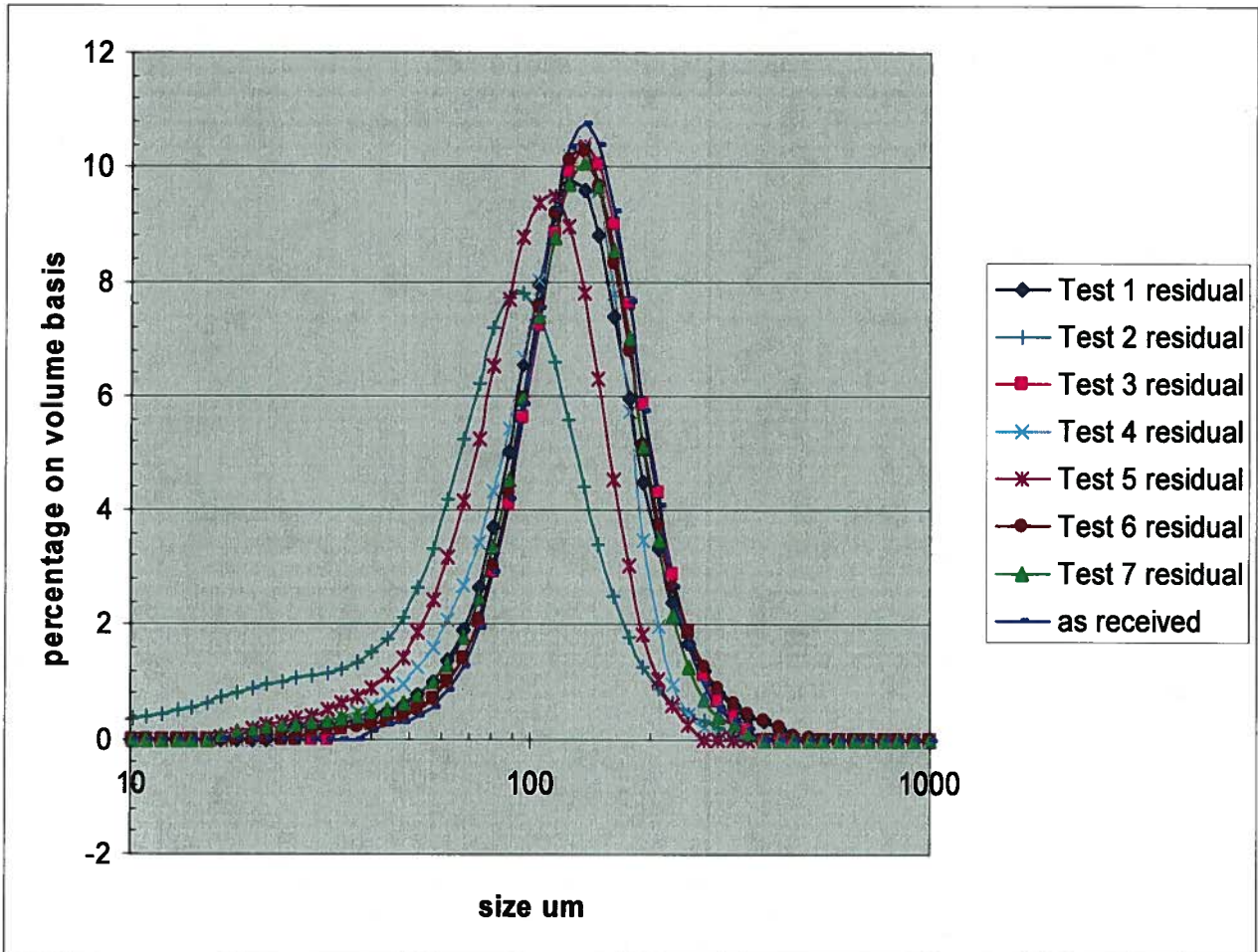


Figure 9 Particle Size Distributions for Tank Residue

Figure 10 shows the PSDs for the contents of the Receipt Tanks in Test 7. This figure shows that the PSD for Test 7 RT3 had two maxima, one at 20 μm for slow-settling particles and one at 74 μm for fast-settling particles. The bimodal distributions indicate that both fast- and slow-settling particles were present. These results would be anticipated since the mixing pump flow rate was the highest tested at 8 gpm which would have removed a large portion of both slow- and fast-particles from the MDT to the Receipt Tanks.

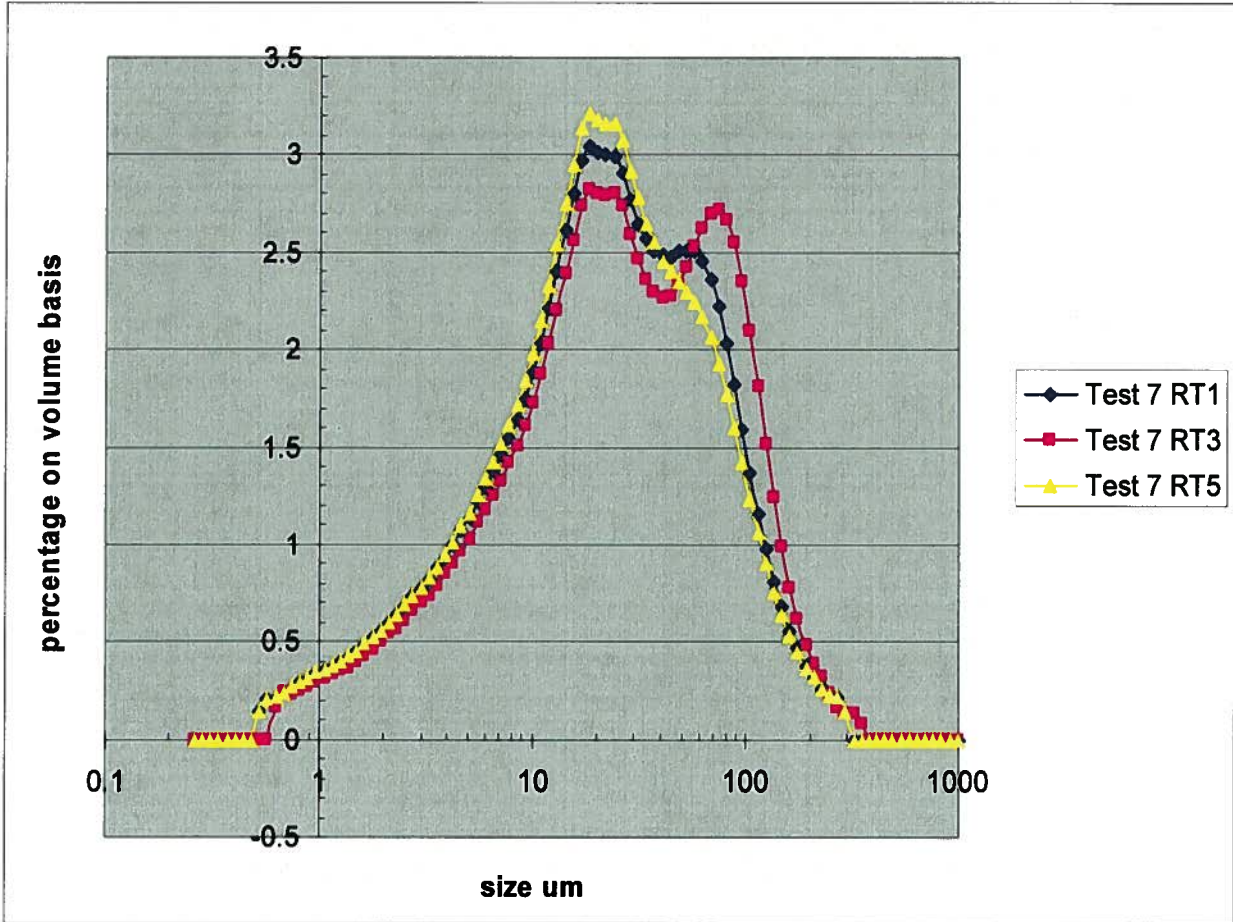


Figure 10 Particle Size Distributions for Receipt Tanks, Test 7

3.7 Summary of Results

The elutriation scoping test have shown that the most significant factor that impacts separation efficiency of particles is the MJP flow rate, which is directly related to jet velocity in the vicinity of the removal tube. The jet velocity that suspends solids and allows them to be removed from the tank is the velocity of the jet in the vicinity of the removal tube. Blevins [1984] gives the maximum velocity downstream of an axisymmetric jet.

$$U_m = 12 r_0 U_0 / x$$

where r_0 is the radius of the nozzle, U_0 is the velocity at the nozzle and x is the distance downstream of the nozzle. This equation was used to compute the jet velocities entered into Table 6.

Table 6 SRNL Elutriation Jet Velocities

Test #	Date	MJP Flow (gpm)	Jet Velocities (ft/sec)
1	9/1/2010	5	1.89
2	9/13/2010	4	1.51
3	9/16/2010	6	2.27
4	9/22/2010	5	1.89
5	9/27/2010	4	1.51
6	10/1/2010	5	1.89
7	10/4/2010	8	3.03

The goal of the elutriation scoping testing was to separate the particles leaving the fast-settling particles in the mixing tank while removing the slow-settling particles into the Receipt Tanks. The results are summarized in Figure 11 which plots the volume fraction of fast-settling particles in the tank residue and the Receipt Tanks using two measurement methods. Three significant trends were observed in Figure 11:

1. Increasing the MJP flow rate, and hence, jet velocity resulted in an increasing amount of fast-settling solids removal from the MDT into the Receipt Tanks.
2. Low pump speeds (4.0 gpm, jet velocity of 1.5 ft/sec) resulted in significant amounts of slow-settling solids remaining in the MDT. Figure 10 illustrates this by the percentage of fast-settling solids remaining in the MDT decreasing from the near 100% shown in runs at higher pump flow rates to an approximate 80%.
3. An optimum MJP flow rate of 5.0 gpm (jet velocity of 1.89 ft/sec) resulted in the minimum amount of heavy fast-settling particles from being entrained into the Receipt Tanks and the maximum amount of lighter slow-settling particles being separated into the RTs.

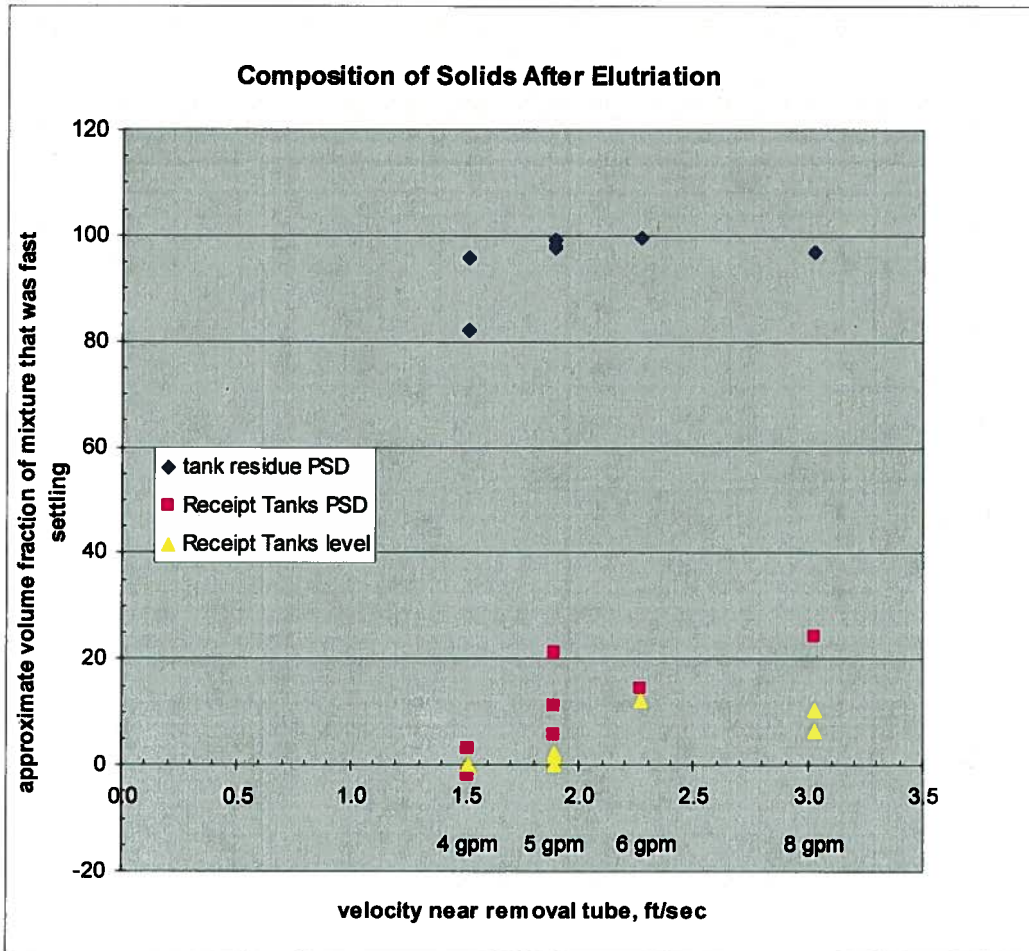


Figure 11 Trends – Increasing Fast-Settling Solids in Receipt Tanks with Increasing Jet Velocity

3.8 Calculation of Data in Figure 11

The fraction of fast-settling particles in the Receipt Tanks was calculated two ways. The first way was to divide the volume of fast-settling particles listed in Table 5 by the total particle volume. The results are plotted in Figure 11. Increasing velocity generally increases the fraction of the contents of the Receipt Tanks that is fast-settling particles.

The second way that fraction fast-settling particles was calculated was more complicated and based on PSDs. Figure 10 shows that the PSD for Test 7 RT3 had two maxima, one at 20 μm for slow-settling particles and one at 74 μm for fast-settling particles. Ideally the Receipt Tanks would contain 100% slow-settling particles with no fast-settling particles. The following steps were used to estimate the fraction of fast-settling particles in the Receipt Tanks. The slow-settling PSD in Figure 9 was integrated from 74 μm to 1000 μm . The same integration was performed for the PSDs for the contents of the Receipt Tanks for the seven tests. In each case the fast-settling PSD integral was subtracted from the Receipt Tank integral to give an estimate of what fraction of fast-settling particles larger than 74 μm was in the Receipt Tank. The PSD for fast-settling particles is approximately symmetrical so the volume of fast-settling particles less than 74 μm is equal to the volume greater than 74 μm . Therefore, the amounts of fast-settling particles greater than 74 μm were doubled to get the total amount of fast-settling particles. The

results are plotted in Figure 11 as a function of velocity in the vicinity of the removal tube. The contents of the Receipt Tanks contained almost no fast-settling particles for a velocity of 1.5 ft/sec and increasing amounts of fast-settling particles for velocities of 1.9 ft/sec and greater. The two methods for computing the fraction of fast-settling particles in the Receipt Tanks give different absolute values, but the same trends.

PSDs were also used to estimate the fraction of slow-settling particles in the mixing tank residue. Figure 8 shows that none of the as-received fast-settling particles had a size of 37 μm or less. Figure 9 shows that some of the PSDs for the residues in the tank had significant numbers of particles smaller than 37 μm as the result of admixture of slow-settling particles. Ideally the residue would be 100% fast-settling particles with no slow-settling particles. The following steps were used to estimate the fraction of slow-settling particles in the residues. The PSD for slow-settling particles in Figure 8 was integrated from zero to 37 μm and it was determined that 83% of the total volume of slow-settling particles had a size of 37 μm or less. Then each of the PSDs for residues plotted in Figure 9 was integrated from zero to 37 μm to give the fraction of total volume that was less than or equal to 37 μm . That fraction was divided by 0.83 to get an estimate of the fraction of slow-settling particles in the sample. The results are plotted in Figure 11 as a function of jet velocity in the vicinity of the removal tube. The residue contained a significant amount of slow-settling particles for a jet velocity of 1.5 ft/sec and little slow-settling particles for velocities of 1.9 ft/sec or greater.

Table 7 Elutriation Tests Data Summary

Test #	Date	MJP Flow (gpm)	Residue $\leq 37 \mu\text{m}$	% Gibbsite in Residue	Receipt Tank 1 $\geq 74 \mu\text{m}$	Receipt Tank 3 $\geq 74 \mu\text{m}$	Receipt Tank 5 $\geq 74 \mu\text{m}$
1	9/1/2010	5	1.48	98.21	22.00	14.90	8.70
2	9/13/2010	4	14.60	82.33	7.80	6.10	4.60
3	9/16/2010	6	0.41	99.50	9.30	12.80	13.60
4	9/22/2010	5	1.82	97.80	6.30	7.50	8.50
5	9/27/2010	4	3.49	95.78	4.60	5.20	1.20
6	10/1/2010	5	0.74	99.10	10.60	10.30	9.30
7	10/4/2010	8	2.57	96.89	15.20	21.30	13.80

Newitt, et al. [1955] measured velocities in pipe that were necessary to just suspend sand and gravel off the bottom of the pipe with a water flow. The velocity to just suspend 100 μm sand was 2 ft/sec. This is approximately consistent with the velocity that suspends significant quantities of stainless steel which results in stainless steel being transported to the Receipt Tanks.

4.0 Conclusions and Recommendations for Path Forward

The successful In-tank Elutriation in Hanford Tank S-112 and the successful Phase 1 elutriation tests at SRNL show that the concept has potential to significantly reduce the cost of tank closure. The testing and previous results were related to Salt Waste tanks; however, benefits of elutriation could be shown to be applicable to Sludge Waste tanks as well. There were certain limitations to the simulant used in the elutriation testing performed at SRNL, specifically with the magnetic effects of the stainless steel materials, however, the proof of concept was evident in the testing. The development of the concept will require additional work, listed below.

1. Elutriation of Tank S-112 required large quantities of 50 wt% caustic to break up the hard cobble in that salt waste tank. The consequences of adding large quantities of caustic to other tanks should be analyzed. Addition of caustic impacts tank space and will require additional evaporation. Another consideration in elutriation is that washing of solids will be required to remove phosphate and oxalate, which would otherwise precipitate with the caustic treatment.
2. In-Tank Elutriation was shown at Hanford to be useful in salt waste tanks, and successful implementation of elutriation required preliminary steps, which have costs associated with them. The preliminary steps are removal of soluble salts, breaking up hard salt pan, and digesting cobble to sand using caustic solution. Since the salt waste tanks probably can not be heated, this probably means treatment with stronger than 25% caustic, although perhaps not 50%. Therefore, a cost and benefit analysis should be performed. The ability to leave more innocuous gibbsite in a waste tank has benefits, but the introduction of large quantities of caustic into the waste stream has costs. The cost and benefit analysis should also consider how many tanks could use elutriation.
3. Laboratory analysis of actual tank waste solids is critical in evaluating the efficacy of elutriation for a waste tank (including sludge waste tanks). Tank waste samples are archived in the Hanford 222-S Laboratory and are available for analyses. There are two types of analyses that are needed to develop a representative simulant:
 - a. Solids Density and Size Measurement - Based on chemical and physical analyses of the waste solids, determine whether elutriation is likely to be possible by calculation of the settling velocities of the solids by first measuring the solid density and size.
 - b. Volume Fraction of Solids -For use in waste tanks, develop a representative simulant by closely matching the volume fractions of fast-settling and slow-settling particles if data is available from tank characterization.

If after evaluating the settling velocities and elutriation is considered possible, then a representative sample can be generated and elutriation testing performed.

4. Perform testing with a hydrocyclone in a scaled test with the representative simulant. Hydrocyclones have the potential to further separate tank solids that have been elutriated from the tank with less required liquid.
5. Research whether leaving larger quantities of sandy material in a waste tank will compromise the integrity of tank grouting.

5.0 References

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Table 8 Species Before and After Elutriation

species	units	"before" average concentration	"before" st. dev. concentration	"before" average concentration	"after" average concentration	"after" st. dev. concentration	"before"/"after" concentration	"before" mass	"after" mass	"before"/"after" masses
aluminum	ug/g	6313	1977	224623	2818	0.028	23514	3250.3	7	
hydroxide	ug/g	10575	1261	35833	1202	0.3	39392	518.5	76	
nitrate	ug/g	389850	139317	23	8	16781.0	1452198	0.3	4319965	
nitrite	ug/g	9304	5724	9	5	1024.4	34656	0.1	263715	
oxalate	ug/g	5180	3010	131	0	39.4	19294	1.9	10148	
silicon	ug/g	1365	180	919	16	1.5	5085	13.3	383	
sodium	ug/g	259500	7578	45786	1536	5.7	966642	662.5	1459	
sulfur	ug/g	12498	5785	1812	49	6.9	46553	26.2	1776	
uranium	ug/g	4958	96	1812	32	2.7	18467	26.2	704	
water	%	13	5	23	1	0.6	477826	3320.8	144	
total percent	%	87	23	97	2					
total mass	kg						3231247	14040		

Table 9 Radionuclides Before and After Elutriation

isotope	units	"before" average concentration	"before" st. dev. concentration	"after" average concentration	"after" st. dev. concentration	"before"/"after" concentration	"before" kCi	"after" kCi	"before"/"after" as curies
Am-241	uCi/g	0.02891	0.01135	0.00077	0.00002	37.3	0.107686	1E-05	9615
Cs-137	uCi/g	44.61	13.67	0.18	0.04	245.8	166.1544	0.0026	63274
Curium-243/244	uCi/g	0.00409	0.00051	0.00026	0.00001	15.9	0.015217	4E-06	4081
Pu-238	uCi/g	0.00222	0.00028	0.00081	0.00002	2.7	0.008279	1E-05	703
Pu-239	uCi/g	0.00973	0.00563	0.00160	0.00014	6.1	0.036235	2E-05	1562
Sr-90	uCi/g	24.50	8.61	3.02	0.15	8.1	91.2629	0.0437	2091
Tc-99	uCi/g	3.09	1.12	0.49	0.04	6.3	11.50824	0.0071	1615
Th-230	uCi/g	0.892	0.114	0.003	0.000	281.2	3.320945	5E-05	72384
Th-232	uCi/g	3.54	1.56	0.13	0.04	27.1	13.20332	0.0019	6989
total	kCi						286	0.055	5157

Distribution:

A. B. Barnes, 999-W
D. A. Crowley, 773-43A
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
J. H. Scogin, 773-A
W. R. Wilmarth, 773-A
H. H. Burns, 773-41A