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Investigating Suspension of MST, CST, and Simulated Sludge Slurries in a Pilot-Scale Waste Tank

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LIST OF ACRONYMS

ABS	Acrylonitrile butadiene styrene
ARP	Actinide Removal Process
CST	Crystalline Silicotitanate
DWPF	Defense Waste Processing Facility
EDL	Engineering Development Laboratory
ICP-MS	Inductively Coupled Plasma–Mass Spectroscopy
MST	Monosodium Titanate
P&ID	Process and Instrument Diagram
RMF	Rotary Microfilter
rpm	revolutions per minute
RMJ	Rotating Mixer Jet
SCIX	Small Column Ion Exchange
SMP	Submersible Mixer Pumps
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
VFD	Variable Frequency Drive
VSL	Vitreous State Laboratory

1.0 SUMMARY

The Small Column Ion Exchange (SCIX) process is being developed to remove cesium, strontium, and actinides from Savannah River Site (SRS) Liquid Waste using an existing waste tank (i.e., Tank 41H) to house the process. Savannah River National Laboratory (SRNL) is conducting pilot-scale mixing tests to determine the pump requirements for suspending and resuspending monosodium titanate (MST), crystalline silicotitanate (CST), and simulated sludge. The purpose of this pilot scale testing is for the pumps to resuspend the MST, CST, and simulated sludge particles so that they can be removed from the tank, and to suspend the MST so it can contact strontium and actinides.

The pilot-scale tank is a 1/10.85 linear scaled model of Tank 41H. The tank diameter, tank liquid level, pump nozzle diameter, pump elevation, and cooling coil diameter are all 1/10.85 of their dimensions in Tank 41H. The pump locations correspond to the proposed locations in Tank 41H by the SCIX program (Risers B5, B3, and B1). Previous testing showed that three Submersible Mixer Pumps (SMPs) will provide sufficient power to initially suspend MST in an SRS waste tank, and to resuspend MST that has settled in a waste tank at nominal 45 °C for four weeks.

The conclusions from this analysis follow.

- Three SMPs will be able to resuspend more than 99.9 % of the MST and CST that has settled for four weeks at nominal 45 °C. The testing shows the required pump discharge velocity is 84 % of the maximum discharge velocity of the pump.
- Three SMPs will be able to resuspend more than 99.9 % of the MST, CST, and simulated sludge that has settled for four weeks at nominal 45 °C. The testing shows the required pump discharge velocity is 82 % of the maximum discharge velocity of the pump.
- A contact time of 6 – 12 hours is needed for strontium sorption by MST in a jet mixed tank with cooling coils, which is consistent with bench-scale testing and actinide removal process (ARP) operation.

2.0 INTRODUCTION

Savannah River Remediation (SRR) is developing the SCIX process to remove cesium, strontium, and select actinides from SRS Liquid Waste using an existing waste tank (i.e., Tank 41H) to house the process. The process adds MST as a slurry to the waste tank (i.e., Tank 41H) to chemically sorb the strontium and select actinides, removes the MST and entrained sludge with an in-riser rotary microfilter (RMF), and removes cesium from the RMF filtrate with ion-exchange columns containing CST. The RMF returns the concentrated solids (i.e., MST and entrained sludge) to the waste tank. After being loaded with cesium, the CST is ground to reduce its size and transferred as a slurry to Tank 40H (baseline case) or Tank 41H (the feasibility of adding ground CST to Tank 41H is under evaluation). The MST, sludge, and CST (if transferred to Tank 41H) in the waste tank will be periodically transported to a sludge batch preparation tank (Tank 42H or Tank 51H). Both the MST and CST streams will ultimately be transferred to the Defense Waste Processing Facility (DWPF) for vitrification.

To assist SRR in designing the SCIX process, SRNL is conducting pilot-scale testing to determine the number, type, and size of pumps needed to suspend/resuspend the solid particles (i.e., MST, CST, and sludge) in Tank 41H.[1]

The purpose of this mixing application is for the MST to adequately contact the strontium and actinide containing liquid and to remobilize the solid particles so they can be removed from the tank. There are no requirements for the MST to be homogeneous in the tank during either mixing activity.

The objectives of the pilot-scale testing follow:

- To determine the strontium sorption by MST as a function of time
- To determine the pump requirements (i.e., number, size, and speed) to suspend solids for adequate contact with salt solution and resuspend settled solids after sitting in the tank for an extended period of time.

This document describes the pump requirements to resuspend MST plus CST slurries and MST plus CST plus simulated sludge slurries that have settled for four weeks at nominal 45 °C, and the required contact time for strontium sorption by MST in a jet mixed tank with cooling coils.

A previous document[2] describes the tests conducted to determine the pump requirements to suspend MST in Tank 41H with and without settling for four weeks at nominal 45 °C.[a]

3.0 TEST EQUIPMENT AND TESTING DESCRIPTION

3.1 PILOT SCALE TEST FACILITY DESCRIPTION

To meet the above objectives, SRNL personnel designed, fabricated, and assembled a 1/10.85 scale type IIIA waste tank in the Engineering Development Laboratory (EDL). The main tank is a nominal 8 foot diameter, 41 inch high transparent acrylic tank of wall thickness 1 inch. The pilot-scale tank contains cooling coils, which were fabricated from ¼ inch stainless steel tubing (0.25 inch OD) and linearly scaled to the diameter of the cooling coils in Tank 41H. The coils were arranged to replicate the cooling coils in Tank 41H. The acrylic tank sits on a 38 inch high open steel stand. This design facilitated direct visual observation of the cleaning radius from underneath since the tank bottom is transparent. At the center of the tank, a stainless steel center column is provided. A false bottom is provided to keep the tank contents heated to 45 °C for prolonged periods while the tank contents are settling. For more details on fabrication of the pilot-scale tank, see reference [2]. A photograph of the clear tank with scaled Tank 41H cooling coils is shown in Figure 1.

[a] Temperature = 45 ± 5 °C

Figure 1. Pilot-Scale Waste Tank

Following the MST resuspension test[2], significant leakage was observed from the false bottom into the main tank. The leakage was due to degradation of the custom molded insert. Because of this leakage, the tank was redesigned. The cooling coil assembly was removed from the tank, and the cooling coils were detached from the half circle acrylic plates. A new acrylic section was placed below the tank and sealed with an O-ring, creating a chamber approximately 1.25 inches thick. Water was recirculated through this chamber to heat the tank contents. The cooling coils were connected together with tie rods and installed in the tank. They were not physically connected to the tank bottom. This configuration was used for the resuspension tests with MST plus CST and MST plus CST plus simulated sludge. The strontium sorption tests were conducted with the tank configuration described in reference [2].

Figure 2 shows a top view of the pilot-scale tank with the riser locations corresponding to Tank 41H. The black and green line segments show the cooling coils. The pumps were placed in Risers B5, B3, and B1.

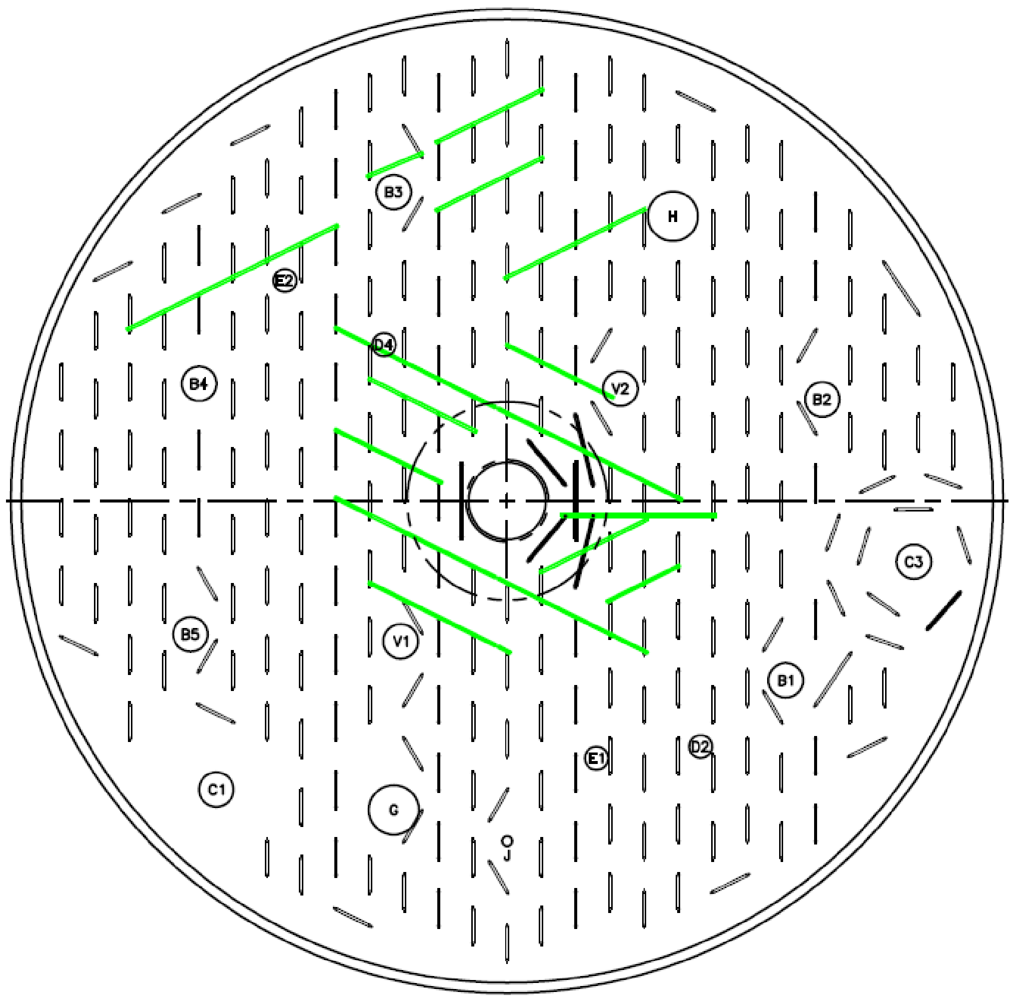

Figure 2. Pilot-Scale Tank Top View

Personnel placed three radial lines extending from each of the pumps on the tank bottom (see Figure 3). One of the lines from each pump extended from the pump to the center column. The other two lines extended from the pump to the tank wall, reaching the wall at the point corresponding to the midpoint between the pump and the closest pump on that side of the tank. From Figure 3, one can see the maximum cleaning radius needed to completely suspend the solid particles with three pumps is 47 inches (42.6 feet in Tank 41H) in the pilot-scale tank.

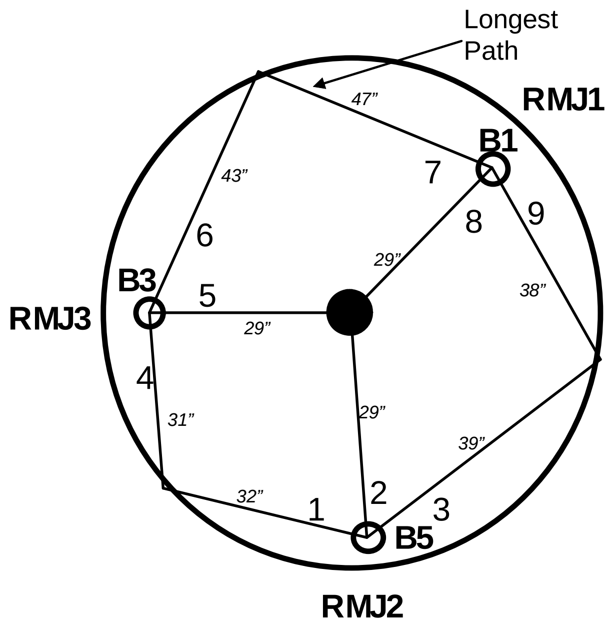

Figure 3. Cleaning Radius Measurement Locations for Three Pump Configuration

Figure 4 shows the process and instrumentation diagram (P&ID) for the Pilot-Scale Test Facility. It schematically shows the mixing tank, three mixer pumps, the external recirculation pump for mixers, and the instrumentation to measure flow rates and process temperatures. The tank heating system is also shown. The heating fluid in the false bottom is circulated independently by a separate pump through a 6700 watt heater. The fluid temperature at the heater outlet is measured to control the heater, which turns on or off as needed. For the settling and resuspension tests, the temperature was controlled to 45 ± 5 °C. The main circulation pump is controlled by a variable frequency drive (VFD) controller. This controller varies the main pump motor speed to achieve different flow rates through the mixer pumps. Additional flow balancing in the mixer pump is achieved by throttling a valve in each flow path.

Two additional vane type pumps are also provided for chemical transfers in and out of the tank. These vane pumps are also used to prime the centrifugal pump and vent the air out of the flow loop.

The liquid volume during testing was 800 gallons, which is geometrically-scaled to the expected volume in Tank 41H [1,000,000 gallons/$(10.85)^3$) = 780 gallons ~ 800 gallons]. The nozzle diameters are linearly scaled to the actual pumps. All tests were conducted with scaled SMPs. Details on the pump assembly and pump heads are provided in a previous technical report.[2]

The initial pump heads were fabricated from acrylonitrile butadiene styrene (ABS) by a rapid prototype method. The heads were inspected before and during testing. Following several months of testing, the ABS heads showed significant degradation. The pump heads were removed and replaced with polycarbonate heads for a short term (< one month) and stainless steel heads were used thereafter.[b] Prior to installation of the stainless steel heads, the authors conducted flow visualization tests with dye to verify that the discharge jet produced by the stainless steel pump heads behaved the same as the discharge jet produced by the polymeric heads. The failure of the ABS heads is likely from the rapid prototype method which does not provide significant crosslinking of the polymer.

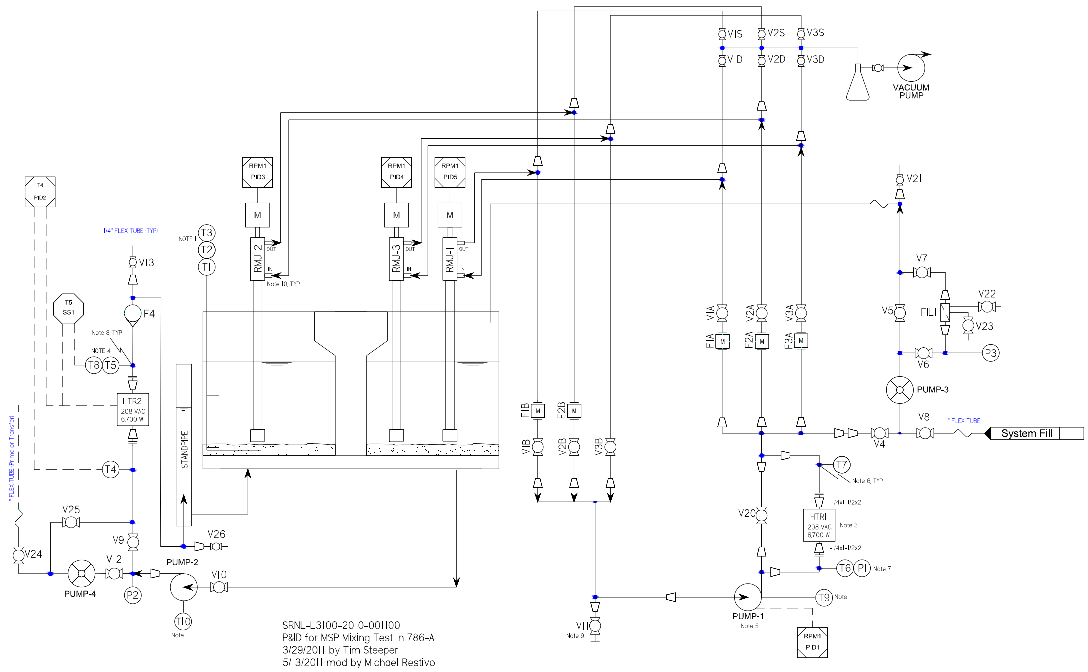

Figure 4. Process and Instrumentation Diagram of Pilot Scale Test Facility

The fluid coming out the nozzles is parallel to the tank bottom as in Tank 41H. The flow rate to each mixer pump is proportional to the jet velocity at the discharge point. The discharge velocity is the most important parameter for resuspension of solids in the tank. It can be calculated using Equation (1) below

$$U_0 = 4\,Q/\pi D^2 \qquad [1]$$

where U_0 is the average pump nozzle discharge velocity, Q is the flow rate through the pump nozzle, and D is the nozzle diameter. Additionally, the mixer pumps are designed to rotate the

[b] The ABS pump heads were used for the MST suspension tests and the MST resuspension tests with two pumps described in reference [2]. The ABS pump heads were also used for the strontium sorption tests described in this report. The polycarbonate pump heads were used for the MST resuspension test with three pumps described in reference [2]. The stainless steel pump heads were used for the MST-CST and MST-CST-sludge resuspension tests described in this report.

pump heads. For the present tests, all mixing pump heads were rotated at 3.6 revolutions per minute (rpm) (for basis, see reference 3). The nozzle orientation for different heads was not synchronized. Instead, it was kept at a random orientation to mimic prototypic conditions.

3.2 EXPERIMENTAL STEPS – RESUSPENSION TESTS

Pilot-scale resuspension tests were performed in the following manner. The feed tank was filled with 800 gallons of simulated supernate solution (see Table 1) by adding liquid to the premeasured mark in the tank. To this solution, appropriate amounts of MST, CST, and simulated sludge were added. Table 2 shows the amounts of MST, CST, and sludge added in each test. The MST was from the same batch being used at the ARP and provided by SRR. It was a 15 wt % slurry with a median particle size of 16 micron. The CST (IE-911) was manufactured by UOP and ground by the Vitreous State Laboratory (VSL) using a Hockmeyer immersion mill. It was a 20 wt % slurry with a median particle size of 2.5 – 8.2 micron. The sludge was simulated Sludge Batch 6. It was a 10.5 wt % insoluble solids slurry produced by Optima[4], and had a median particle size of 23 micron.

Table 1. Simulated Supernate Solution Recipe

Ionic Species	Tank 37F, Molar
Na^+	6.44
NO_3^-	2.26
NO_2^-	0.74
OH^-	2.57
AlO_2^-	0.35
CO_3^{-2}	0.11
SO_4^{-2}	0.15
SiO_3^{-2}	0.004[c]

Table 2. Amounts of MST, CST, and Simulated Sludge added to the Pilot-Scale Tank

Test	MST+CST	MST+CST+Sludge
Amount MST (15 wt % slurry)	2244 g	2244 g
Amount CST (20 wt % slurry)	28,600 g	28,600 g
Amount Sludge (10.35 wt % slurry)	none	4878 g

The solids (MST, CST, and simulated sludge) were allowed to settle for four weeks at 45 ± 5 °C. Following the settling, the mixer pumps were operated at increasing flow rates and the cleaning radius was measured at each flow rate. Researchers measured the cleaning radius along each of three radial lines extending from each pump (see Figure 3). The recorded cleaning radius was the minimum of the measured values that were less than the distance to the wall or center column. The flow rate was increased until all solid particles were resuspended, the maximum pump flow rate was reached, or increased flow would not support removal of additional material.

[c] Based on SRS average salt solution

3.3 STRONTIUM SORPTION TESTS

Researchers performed the strontium sorption tests in the following manner. They filled the tank with approximately 800 gallons of simulated salt solution. To this solution, they added sufficient strontium nitrate to produce a concentration of 5 mg/L strontium, mixed the tank for an hour, and allowed the solution to equilibrate for at least 24 hours. After 24 hours, they collected a liquid sample, filtered it through a 0.1 micron filter, and submitted it for analysis of cold strontium. They added 1210 grams of MST to produce an MST concentration of 0.4 g/L. They collected samples periodically (for one week), filtered them through a 0.1 micron filter, and submitted them for analysis of cold strontium.

After a week, additional strontium nitrate was added to the tank to increase the bulk concentration by 5 mg/L, the tank was mixed for an hour, and the solution was allowed to equilibrate for at least 24 hours. A sample was collected, filtered, and analyzed for cold strontium. Personnel added additional MST to increase the bulk tank concentration by 0.4 g/L. They collected samples periodically (for 48 hours), filtered them through a 0.1 micron filter, and submitted them for analysis of cold strontium. Three additional MST strikes were conducted for a total of five MST strikes.

SRNL performed the cold strontium analysis in the following manner. They took a 15 mL sample of filtered supernate and added a ^{85}Sr tracer to it. They added 10 mL of 16 M nitric acid to the sample. This sample was processed through an Eichrom Sr resin to capture strontium. After the sample was processed, the Eichrom Sr resin was rinsed with 25 mL of 8 M nitric acid. After rinsing, the resin was eluted with 0.0001 M nitric acid. The eluate was analyzed for ^{85}Sr by gamma to evaluate tracer recover and by Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS) for cold strontium.

4.0 RESULTS

4.1 SCALING OF TEST RESULTS

The pilot-scale results must be applied to the full-scale waste tanks. The scaling of test results from pilot-scale to full-scale is described in another SRNL document.[3] In that document, a conservative estimate of the scaling function for particle suspension was developed and is described by equation [2]

$$U_{0\text{-full-scale}} = 1.3\ U_{0\text{-pilot-scale}} \qquad [2]$$

where U_0 is the nozzle discharge velocity. The nozzle discharge velocity in the full-scale tank is 30% larger than the nozzle discharge velocity in the pilot-scale tank for equivalent solid particle suspension. This scaling is conservatively based on matching the shear stress at the solid-liquid interface in both tanks. The cooling coils will cause less drag and less reduction in jet velocity in the full-scale tank compared with the pilot-scale tank.[3] Therefore, the recommended pump nozzle velocity in the full-scale tank contains some conservatism.

The pump rotation rate in the pilot-scale tests should be 8.35 - 10.85 times the pump rotation rate in the full-scale tank.[3] Since previous SRNL testing showed a slower rotation rate may provide improved solids suspension[5], SRNL increased the pump rotation rate 10.85 times rather than 8.35 times to be conservative. Given typical pump rotation rates in the SRS Tank Farms are 1/5 – 1/3 rpm, the pump rotation rate in these tests was 3.6 rpm (0.33 rpm * 10.85 = 3.6 rpm).

Since the strontium sorption process is controlled by film diffusion[6], the pilot-scale mixer pump jet velocity was selected to produce equal power per unit volume in the pilot-scale and full-scale tanks.[3] Equation [3] describes this scaling.

$$U_{0\text{-full-scale}} = 2.2\ U_{0\text{-pilot-scale}} \qquad [3]$$

In addition, all MST particles were verified to be in suspension during the test.

The strontium sorption tests have been scaled to produce equal solid-liquid mass transfer coefficients between the pilot-scale and full-scale tanks. The rate of mass transfer of strontium (or an actinide) between the bulk liquid and the MST particles is described by equation [4]

$$N_A = k\ a_s\ (C_S - C_L) \qquad [4]$$

where N_A is the rate of mass transfer, k is the mass transfer coefficient, a_s is the MST surface area, C_S is the strontium concentration on the MST, and C_L is the strontium concentration in the bulk liquid. The tests were performed to produce the same mass transfer coefficient in both tanks. The initial bulk liquid and MST strontium concentrations and the MST surface area per MST volume will be the same in both tanks. Since the full-scale tank contains 10.85^3 = 1277 times the MST as the pilot-scale tank, the surface area will be 1277 times larger in the full-scale tank. Therefore, the rate of strontium transfer will be 1277 times faster in the full-scale tank. Since the full-scale tank will contain 1277 times the strontium as the pilot-scale tank, the time required to reach the same reduction in strontium concentration will be comparable in both tanks assuming that the scaled concentration gradient of the strontium matches at both scales.[3]

4.2 MST PLUS CST RESUSPENSION TEST WITH THREE PUMPS

The MST plus CST slurry was readily resuspended by the equivalent of three SMPs. Table 3 shows the data. The second column shows the pump parameters needed to resuspend 99.8 % of the solid particles, and the third column shows the pump parameters needed to resuspend 99.98 % of the solid particles. At a U_0D of 1.5 ft^2/s, all of the solid particles were resuspended except for two spots on the tank bottom. The spots measured approximately 2 inches x 3 inches and 2 inches x 4 inches. Comparing the area of these spots to the area of the tank bottom shows they occupy 0.2 % of the tank bottom. At a U_0D of 1.74 ft^2/s, all of the solid particles were resuspended except for two spots, described above, on the tank bottom. The spots measured approximately 3/4 inches x 3/4 inches (see Figure 5) and 3/4 inches x 1-1/4 inches. The spots were not found in a region expected to have poor mixing. Comparing the area of these spots to the area of the tank bottom shows they occupy 0.02 % of the tank bottom. The table shows three SMPs will be able to resuspend more than 99.9 % of the MST and CST that has settled for four weeks at nominal 45 °C, with 16 % conservatism. Figure 6 shows sample cleaning radii as a function of U_0D. The cleaning radii along the three radial lines are linear with U_0D and approximately equal.

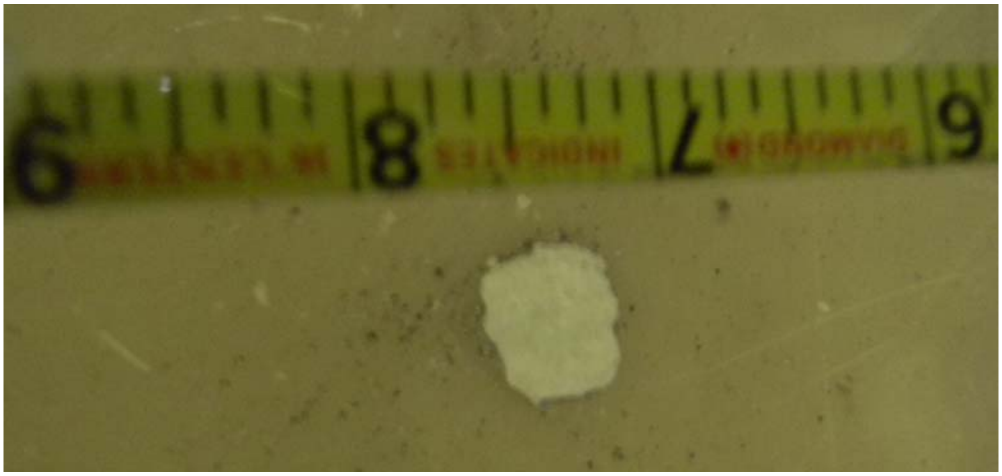

Figure 5. "Spot" on Tank Bottom

Table 3. Parameters Needed to Resuspend MST Plus CST that Settled for Four Weeks at 45 °C

Pump	3 SMP	3 SMP
$U_0D_{pilot\text{-}scale}$	1.50 ft^2/s	1.74 ft^2/s
Amount resuspended (%)	99.8 %	99.98 %
$U_0D_{full\text{-}scale}$ (equation [2])	21.2 ft^2/s	24.5 ft^2/s
Max $U_0D_{full\text{-}scale}$	29.0 ft^2/s	29.0 ft^2/s
Percent of Max $U_0D_{full\text{-}scale}$	73 %	84 %

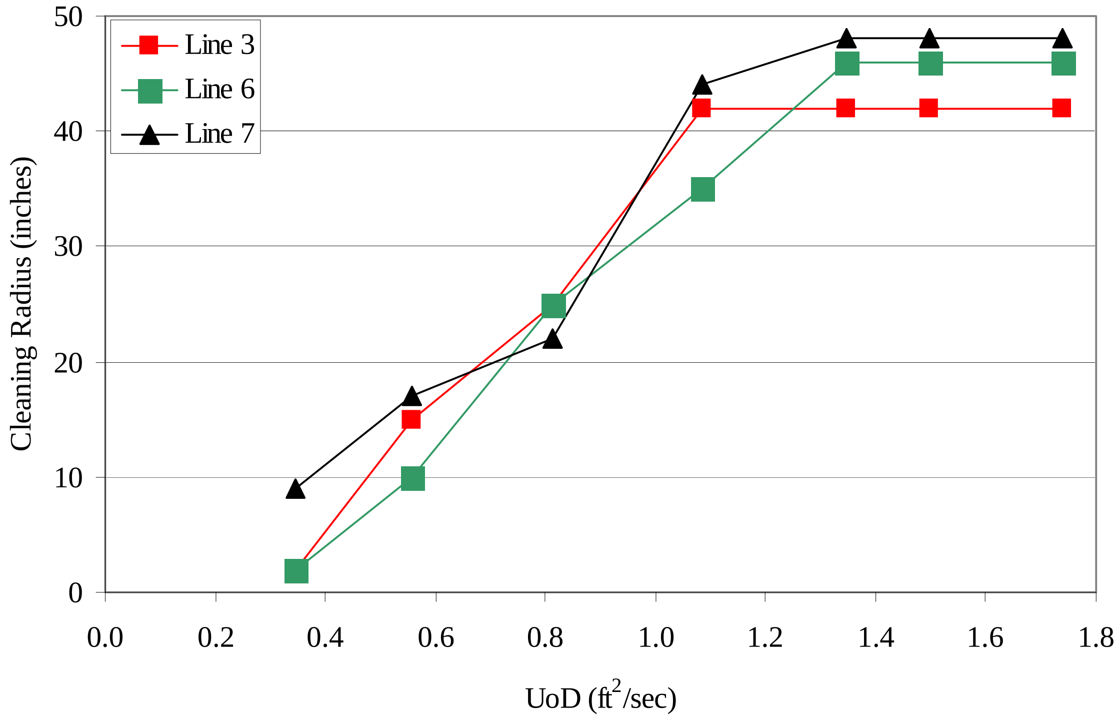

Figure 6. Sample Cleaning Radii during Resuspension Test with MST and CST

4.3 MST PLUS CST PLUS SLUDGE RESUSPENSION TEST WITH THREE PUMPS

The MST plus CST plus simulated sludge slurry was readily resuspended by the equivalent of three SMPs. Figure 7 shows sample cleaning radii as a function of U_0D. This figure shows relatively good agreement between the measured cleaning radii along the three radial lines. Table 4 shows the data. The second column shows the pump parameters needed to resuspend 99.8 % of the solid particles, and the third column shows the pump parameters needed to resuspend 99.92 % of the solid particles. At a U_0D of 1.36 ft^2/s, all of the solid particles were resuspended except for two spots on the tank bottom. The spots measured approximately 1.5 inches x 5.5 inches and 1 inch x 6.5 inches. These spots were in the same location as the spots described in section 4.2. Comparing the area of these spots to the area of the tank bottom shows they occupy 0.2 % of the tank bottom. At a U_0D of 1.69 ft^2/s, all of the solid particles were resuspended except for two spots (same spots described above) on the tank bottom. The spots measured approximately 2 inches x 1.5 inches and 1 inch x 2.5 inches. Comparing the area of these spots to the area of the tank bottom shows they occupy 0.08 % of the tank bottom. The table shows three SMPs will be able to resuspend more than 99.9 % of the MST, CST, and simulated sludge that has settled for four weeks at nominal 45 °C, with 18 % conservatism.

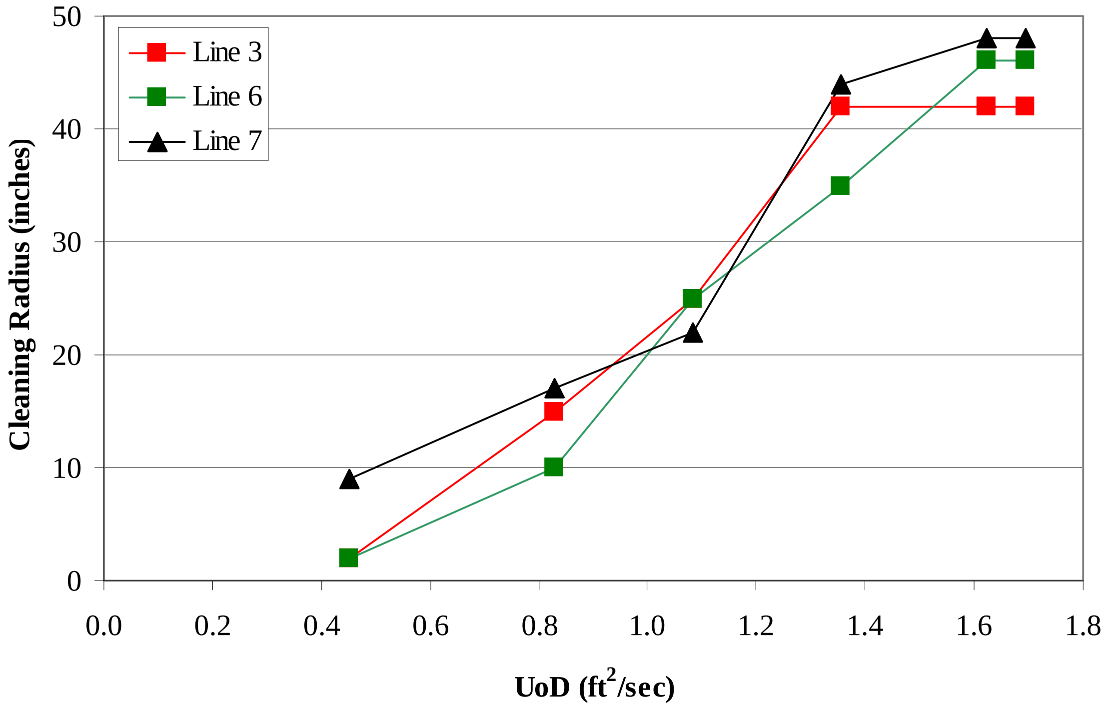

Figure 7. Sample Cleaning Radii during Resuspension Test with MST, CST, and Simulated Sludge

Figure 8 compares the cleaning radii along Line 7 for the tests with MST only, MST plus CST, and MST plus CST plus sludge. While the measured cleaning radii differ significantly at low and intermediate U_0D, above 1.3 ft^2/s, the cleaning radii agree well. Therefore, the pump requirements for resuspension do not differ significantly between the three feed slurries.

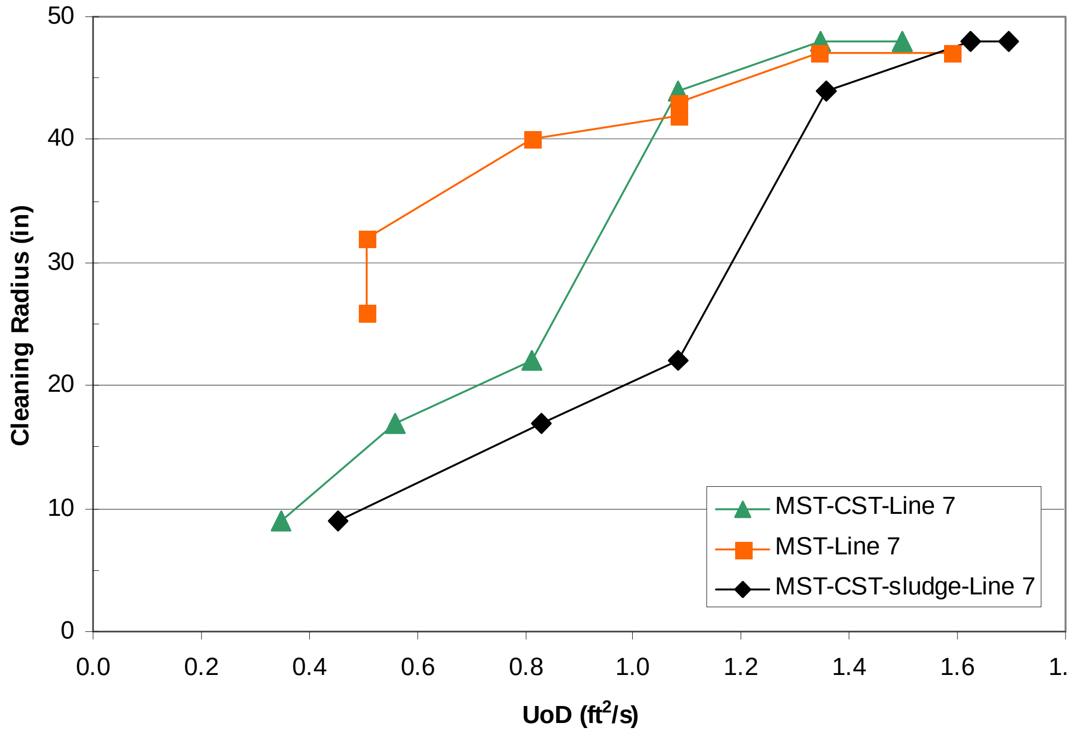

Figure 8. Comparison of Measured Cleaning Radii with Different Feed Slurries

Table 4. Parameters Needed to Resuspend MST Plus CST Plus Sludge that Settled for Four Weeks at 45 °C

Pump	3 SMP	3 SMP
$U_0D_{pilot\text{-}scale}$	1.36 ft^2/s	1.69 ft^2/s
Amount resuspended (%)	99.8 %	99.92 %
$U_0D_{full\text{-}scale}$ (equation [2])	19.2 ft^2/s	23.8 ft^2/s
Max $U_0D_{full\text{-}scale}$	29.0 ft^2/s	29.0 ft^2/s
Percent of Max $U_0D_{full\text{-}scale}$	66 %	82 %

4.4 STRONTIUM SORPTION TESTS

Figure 9 shows the measured strontium concentration as a function of time after the addition of MST in the first strike. Figure 10 shows the measured strontium concentration for all five strikes. Table 5 shows the measured strontium concentration prior to each MST strike and after 9 hours. The measured soluble strontium concentration prior to each MST strike was less than the calculated concentration in the tank based on the amount of strontium nitrate added and the liquid volume (5 mg/L). This difference is likely from the precipitation of strontium due to its low solubility, precipitation with carbonate, or sorption on the MST heel from previous strikes. The bench-scale testing saw a similar trend with the measured soluble strontium concentration being 0.86 mg/L versus a target concentration of 6 mg/L.[7] Following the MST addition, strontium quickly decreases in concentration, and it reaches steady-state within 6 – 12 hours. Subsequent strikes show reduced starting strontium concentrations, due to the MST heel in the tank and carbon dioxide sorption that leads to additional carbonate in the tank. This data matches the sorption kinetics for strontium in small scale testing, and the time to reach steady-state is comparable to the time to reach steady-state in bench-scale (3 L) testing (6 hours)[7] and the current strike time (12 hours) in the ARP.[7] SRNL examined the MST solids following the five strikes from this test and found the particles contained 1.7 ± 0.4 wt % strontium. Since the feed contained 5 mg/L strontium and 0.4 g/L MST, the maximum average strontium concentration on the particles would be 1.25 wt %. Because of differences in the supernate solution, the strontium solubility in this matrix, and the method by which the strontium was added, a direct comparison of this data with other MST sorption test data was not performed.

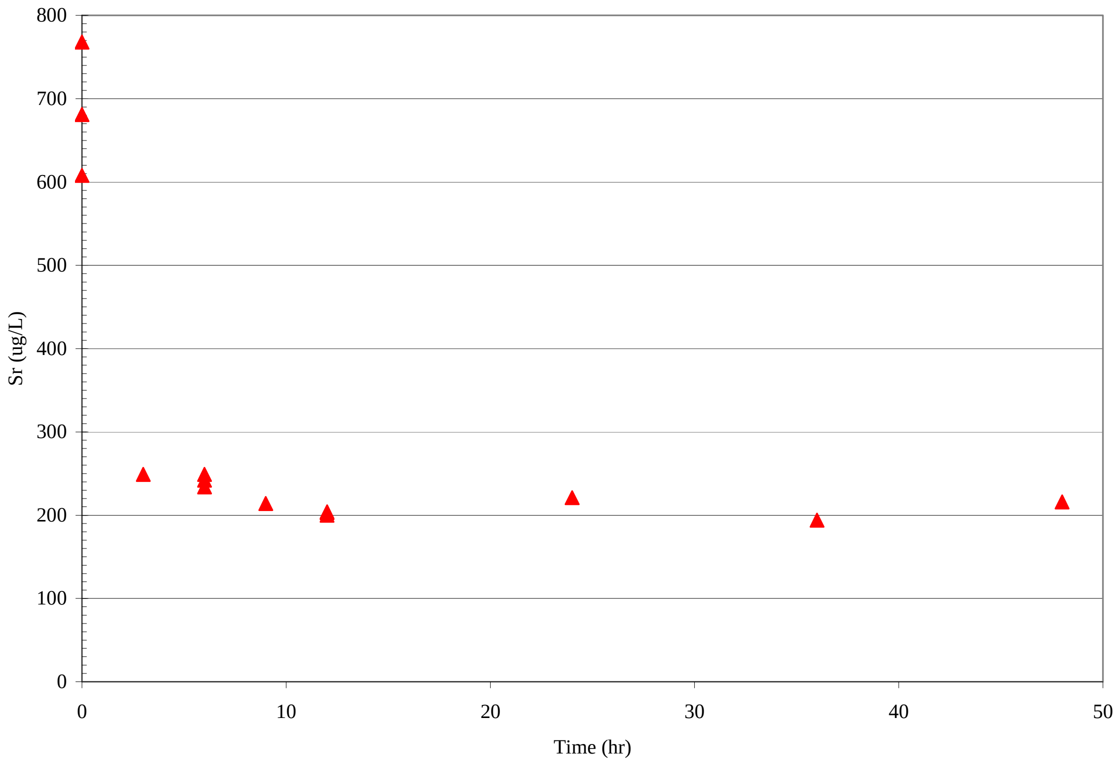

Figure 9. Strontium Concentration as a Function of Time during the First MST Strike

The test results show that the residual MST does not adversely impact the ability of the "fresh" MST to sorb strontium. In fact, the strontium concentration in solution is reduced with subsequent strikes. In addition, the presence of the MST heel allows strontium sorption to occur prior to the addition of new MST, as evidenced by the decrease in strontium concentration at time zero with successive strikes.

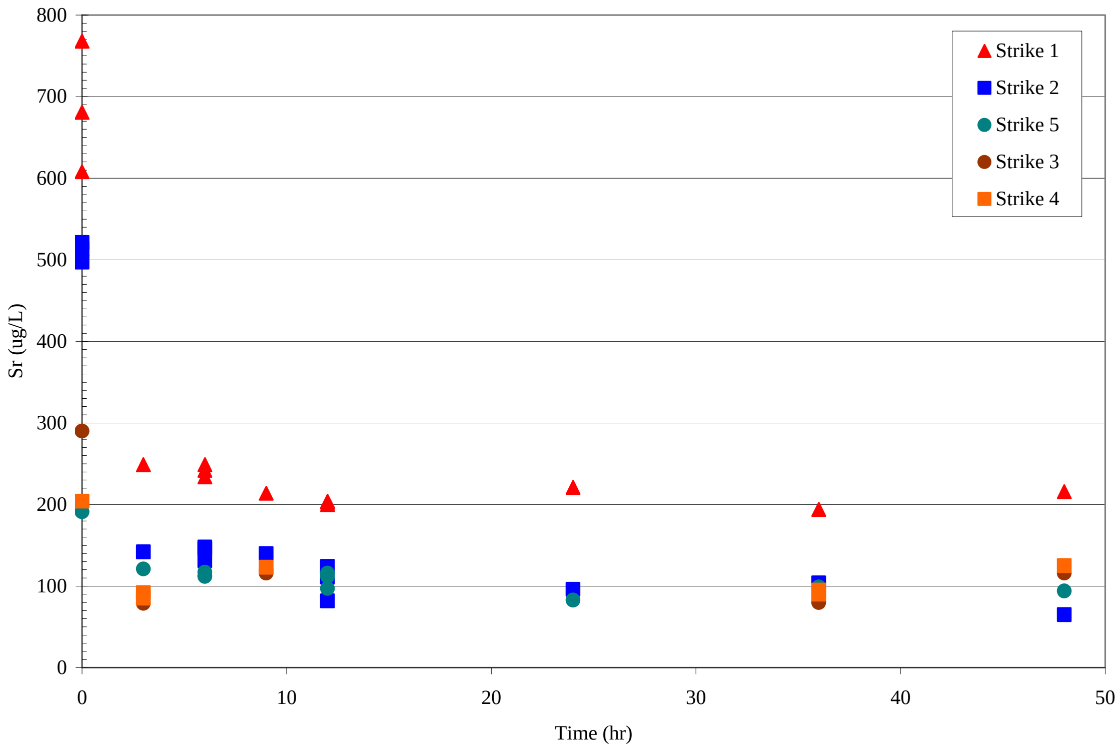

Figure 10. Strontium Concentration during Multiple MST Strike

Table 5. Average Strontium Concentration at Selected Times

Time (hr)	Strike 1 Sr (ug/L)	Strike 2 Sr (ug/L)	Strike 3 Sr (ug/L)	Strike 4 Sr (ug/L)	Strike 5 Sr (ug/L)
0	686	510	290	204	183
9	214	140	116	123	119

5.0 CONCLUSIONS

The conclusions from this analysis follow.

- Three SMPs will be able to resuspend more than 99.9 % of the MST and CST that has settled for four weeks at nominal 45 °C. The testing shows the required pump discharge velocity is 84 % of the maximum discharge velocity of the pump.
- Three SMPs will be able to resuspend more than 99.9 % of the MST, CST, and simulated sludge that has settled for four weeks at nominal 45 °C. The testing shows the required pump discharge velocity is 82 % of the maximum discharge velocity of the pump.
- A contact time of 6 – 12 hours is needed for strontium sorption by MST in a jet mixed tank with cooling coils, which is consistent with bench-scale testing and ARP operation.

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