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## **MONOSODIUM TITANATE MULTI-STRIKE TESTING**

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## LIST OF ACRONYMS

AA	Atomic Absorption
ADS	Analytical Development Section
Am	Americium
AMP	Ammonium Molybdophosphate
Cs	Cesium
DF	Decontamination Factor
DOE	Department of Energy
HLW	High-Level-Waste
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
MST	Monosodium Titanate
Np	Neptunium
PE	Polyethylene
PES	Polyethylsulfone
Pu	Plutonium
PuTTA	Plutonium Triphenyltrifluoroacetone Scintillation Analysis
PVDF	Polyvinylidenedifluoride
Sr	Strontium
SRTC	Savannah River Technology Center
SWPF	Salt Waste Processing Facility
TT&QAP	Task Technical and Quality Assurance Plan
U	Uranium
WAC	Waste Acceptance Criteria
WCS	Waste Characterization System
WPTS	Waste Processing Technology Section

## 1.0 EXECUTIVE SUMMARY

Research over the past decade has studied the adsorption of plutonium and uranium onto monosodium titanate (MST) in alkaline solutions. Tests showed that MST would remove the targeted radionuclides from simulated alkaline waste. Testing also indicated that Pu removal kinetics and Np capacity of the MST material impacts the size of equipment and waste blending plans for the SWPF. Additionally, calculations suggested the baseline MST process (MST concentration of 0.4 g/L) may not achieve the desired decontamination in wastes containing elevated concentrations of Pu and Np. In this task, the authors investigated the performance of non-baseline process parameters and their effectiveness for treating waste feed in the Salt Waste Processing Facility. The work addresses a DOE request in support of technical needs expressed, in part, by the Engineering, Procurement, and Construction Contractors for the Salt Waste Processing Facility. The work investigated the effect of increased MST addition (up to 1.2 g/L) and the benefit of extra filtration steps with multiple additions of MST to salt waste containing actinides and strontium. Both simulants and actual waste testing occurred. Actual waste tests utilized a Tank 39H composite waste solution. In addition, testing to determine desorption of actinides from residual MST occurred. The release of sorbed Sr and actinides from loaded MST during the washing stages in the Salt Waste Processing Facility is an unresolved process behavior. Desorption tests assessed this potential problem using loaded MST from the residue of the MST adsorption tests.

Programmatic conclusions drawn from this task follow.

- MST adsorption of Sr and actinides is minimally influenced by multi-strikes (alone) within the 24 h process cycle time.
- Use of intermediate filtration in conjunction with multiple MST strikes improves removal of Pu and Sr. The low starting concentration of Np does not permit determining the influence of intermediate filtration on Np removal.
- The use of intermediate filtration in conjunction with multiple MST strikes is ineffective for increasing U and Am removal.
- The solubility of Am appears to fall well below the Waste Characterization System estimates.
- Desorption of Sr and Pu during 24 h of solids washing does not pose a threat to process limits.

Testing did not identify problematic areas requiring further investigation. However, testing showed that the use of AMP may have influenced the removal of  $^{241}\text{Am}$ , possibly caused by a filtration effect. (Note: AMP is a solid that can be added to prepare samples for analysis by adsorbing Cs, after which it is filtered out of solution). Slow precipitation of Am also occurred obscuring the data. The authors recommend follow-up testing to investigate this observation if AMP is to be continued for use in testing requiring  $^{241}\text{Am}$  analysis. Analysis of non-radioactive Sr in the tests proved difficult due to the low concentration of non-radioactive Sr and its nearness to the method detection limit for ICP-MS. Efforts to utilize AMP to minimize dilution of actual waste for removal from the cell did not help for this analysis since instrument dilution still proved necessary due to the salt content.

## 2.0 INTRODUCTION

The Salt Waste Processing Facility (SWPF) at the Savannah River Site will use monosodium titanate (MST) for the removal of radioactive strontium (Sr), plutonium (Pu) and neptunium (Np). MST has been investigated for more than a decade for use at SRS. Initial research<sup>1</sup> studied the adsorption of Pu and uranium (U) onto MST in alkaline solutions. These tests showed that MST would remove the targeted radionuclides from simulated alkaline waste. Additional testing indicated that Pu removal kinetics impacts the size of equipment and waste blending plans for the SWPF. Additionally, calculations suggested the baseline MST process (MST concentration of 0.4 g/L) may not achieve the desired decontamination in wastes containing elevated concentrations of Pu.<sup>2</sup>

The actinide removal process tests described in this document evaluate additional process parameters and their effectiveness for treating waste feed in the SWPF. The work addresses a DOE request in support of technical needs expressed, in part, by the Engineering, Procurement, and Construction Contractors for the Salt Waste Processing Facility. The work scope also incorporates the use of an analytical development tool aimed at improving sensitivity and accuracy of analyses (primarily for non-radioactive Sr). The planned scope of work is documented in WSRC-RP-2003-00403, Rev. 1 (TT&QAP).<sup>3</sup> The work described in this document is only a portion of the entire work scope discussed in the referenced TT&QAP.

The requested task and work scope documented within are as follows.

Monosodium Titanate Multi-strike Demonstration – Determine the effect of increased MST addition (up to 1.2 g/L) and benefit of extra filtration steps with multiple additions of MST to salt waste containing actinides and strontium. Both simulant and actual waste testing are required. In addition, testing to determine desorption of actinides from residual MST is also requested.

## 3.0 DISCUSSION

Testing for this task consisted of two groups: radioactive simulant and actual high-level-waste. Each group included two types of testing – Sr and actinides adsorption onto MST and desorption from residual or “loaded” MST. Adsorption testing provides a basis for evaluating various parameters currently being considered for use in the SWPF. Desorption testing provides the first prototypical data evaluating the potential problem of release or desorption of sorbed Sr and actinides from residual MST during washing later in the process.

### 3.1 EXPERIMENTAL

#### 3.1.1 Simulant Adsorption Testing

Monosodium titanate has been used for some time to remove actinides and strontium from salt waste solutions.<sup>1</sup> The method of adsorption testing used in this task mimics the experimental methods used in prior tasks. The notable exception is that this task requires the testing of multiple MST concentrations, multiple MST strikes, and intermediate filtration to remove residual MST. Additionally, the filter size is changed to reflect intended plant use. Past studies used 0.45  $\mu$  filters. The current testing used 0.1  $\mu$  filters both for sampling and intermediate filtration.

Simulant adsorption testing represented the initial phase of testing for this task. Personnel prepared the standard simulant salt solution using a nitric acid prep to increase actinide solubility. The bulk salt composition of the simulant waste is described in Table 3-1. The simulant waste was spiked with target concentrations of  $^{238}\text{U}$  ( $\text{UO}_2^{2+}$ ) at 10,000  $\mu\text{g/L}$ ,  $^{239/240}\text{Pu}$  (IV) at 200  $\mu\text{g/L}$ ,  $^{237}\text{Np}$  (V) at 500  $\mu\text{g/L}$ , and  $^{241}\text{Am}$  (III) at 40  $\mu\text{g/L}$ . The target total strontium concentration was 600  $\mu\text{g/L}$  (from tramp contaminant in other salts), spiked with  $^{85}\text{Sr}$  tracer. The simulant solution equilibrated for 10 weeks prior to adsorption testing. Figure 3-1 provides a graph of the Sr and actinides during the equilibration period. Note the Am showed significant instability during the first 60 days of equilibration but stabilized prior to testing.

**Table 3-1 Simulant Salt Solution Composition**

Component	Concentration (M)
$\text{NaNO}_3$	2.60
$\text{NaOH}$	1.33
$\text{Na}_2\text{SO}_4$	0.521
$\text{NaAl}(\text{OH})_4$	0.429
$\text{NaNO}_2$	0.134
$\text{Na}_2\text{CO}_3$	0.0260
Total $\text{Na}^+$	5.6

Analysis was the same for the starting feed solution and subsequent adsorption tests. Sampling involved pulling approximately 4.0 mL of the test solution into a disposable 10-mL syringe and filtering the sample mixture through a 0.1- $\mu\text{m}$  syringe filter disk (PVDF membrane) and into a sample bottle. Three milliliter portions of each filtered sample were pipetted into a second sample bottle containing 3 mL of 5 M nitric acid. The diluted, acidified sample was manually shaken for approximately 15 seconds and then allowed to equilibrate for a minimum of 2 hours prior to submittal for analysis. Samples were analyzed for  $^{237}\text{Np}$ ,  $^{238}\text{U}$ ,  $^{239/240}\text{Pu}$ ,  $^{241}\text{Am}$ , and  $^{85}\text{Sr}$  concentrations by gamma spectroscopy, inductively coupled plasma-mass spectrometry (ICP-MS), Pu triphenyltrifluoroacetone scintillation analysis (PuTTA), and radiochemical methods. The soluble concentrations of  $^{85}\text{Sr}$  and the actinides prior to testing are shown in Table 3-2.

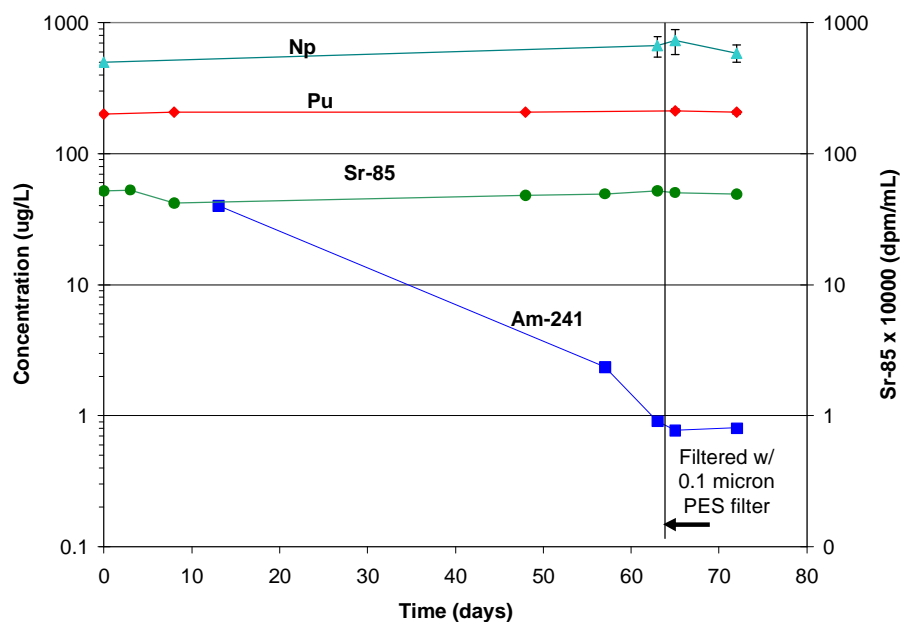


Figure 3-1 Equilibration of Stock Simulant Salt Solution

**Table 3-2 Soluble Strontium and Actinide Concentrations in the Simulant Salt Solution**

Component	Concentration (μg/L)
<sup>238</sup> U	11,650
<sup>239/40</sup> Pu	211
<sup>237</sup> Np	562
<sup>241</sup> Am	0.75
Total Sr	298

The initial experiments with simulated waste used test protocols A through F shown in Table 3-3. All tests occurred in 250-mL polyethylene bottles fitted with a cap. Researchers prepared each test by placing 120 mL of the equilibrated simulant solution in the bottle. The desired amount of MST was added to each test by pipette at the appropriate time (i.e., post-sampling and filtration for the multi-strike tests D and E and at 0 h for all other tests). The MST came from Optima Batch #00-QAB-417. Sample containers were continuously shaken using an orbital shaking (~175 rpm) bath at a constant temperature of  $25 \pm 3$  °C. For those tests involving intermediate filtration, the residual (post-sampling) bulk test solutions were filtered through 0.1 μ PES disposable cup filters. Sampling involved removing a test bottle from the shaker, manually shaking to produce a homogeneous mixture, pulling approximately 4.0 mL of the test mixture into a disposable 10-mL syringe, and filtering the sample mixture through a 0.1-μm syringe filter disk (PVDF) and into a sample bottle. Three milliliter portions of each filtered sample were pipetted into a second sample bottle containing 3 mL of 5 M nitric acid. The diluted, acidified sample was manually shaken for



approximately 15 seconds and then allowed to equilibrate for a minimum of 2 hours prior to submittal for analysis. Samples were analyzed using the same methods as previously noted for the simulant stock solution.

**Table 3-3 Simulant Adsorption Testing Protocols**

<b>Test ID</b>	<b>Description</b>
A	Addition of 0.4 g/L MST with sample analysis at 0, 6, 12, 24, 48, and 168 h
B	Addition of 0.8 g/L MST with sample analysis at 0, 6, 12, 24, 48, and 168 h
C	Addition of 1.2 g/L MST with sample analysis at 0, 6, 12, 24, 48, and 168 h. Experiment conducted in duplicate.
D	Add 0.4 g/L MST (incrementally) at 0, 6, and 12 h with sample analysis at 0, 6, 12, 24, 36, 48, and 168 h (prior to incremental additions at 6 and 12 h).
E	Add 0.4 g/L MST (incrementally) at 0, 6, and 12 h with filtration (0.1 $\mu$ m) prior to the second and third MST strikes and with sample analysis at 0, 6, 12, 24, 36, 48, and 168 h (prior to filtrations and incremental additions at 6 and 12 h). Experiment conducted in duplicate.
F	Control – no addition of MST with sample analysis at 0, 6, 12, 24, and 168 h.

### 3.1.2 Simulant Desorption Testing

A recently raised concern for the process is the potential for desorption or release of sorbed Sr and actinides from loaded (used) MST during the washing stages in the SWPF. Tests were performed to assess desorption from loaded MST using the residual solids from four of the previous MST adsorption tests: Tests A, B, C (actually Test C2 since duplicate C adsorption tests were conducted), and D. No replicate desorption tests occurred. The tests were conducted by concentrating the residual MST solids using a centrifuge (see Figure 3-2). The solids concentration target before testing was set at 2 wt % MST. However, the small volume of residual waste solution and solids made this concentration difficult to achieve. To concentrate the solids, we decanted as much supernate as possible from the centrifuged solids and then added the desired volume of supernate back to the solids to prepare a 2 wt % solids concentration. The mass of solids present was calculated assuming that the centrifuged volume of test solution contained the target concentration of MST added during the adsorption tests (i.e., if 0.4 g/L MST was added in the adsorption test, then the residual adsorption test volume contained 0.4 g/L MST solids). The actual mass of the centrifuged solids weigh significantly more than calculated since the residual solids were wet. Also recall that the residual test solutions with the solids were stored at room temperature for 152 days between the adsorption and desorption tests. The decanted residual supernate from each test was sampled and analyzed to determine both the amount of Sr and Pu loaded on the MST during its 152 days of storage as well as the residual soluble concentration added back to the centrifuged MST solids.

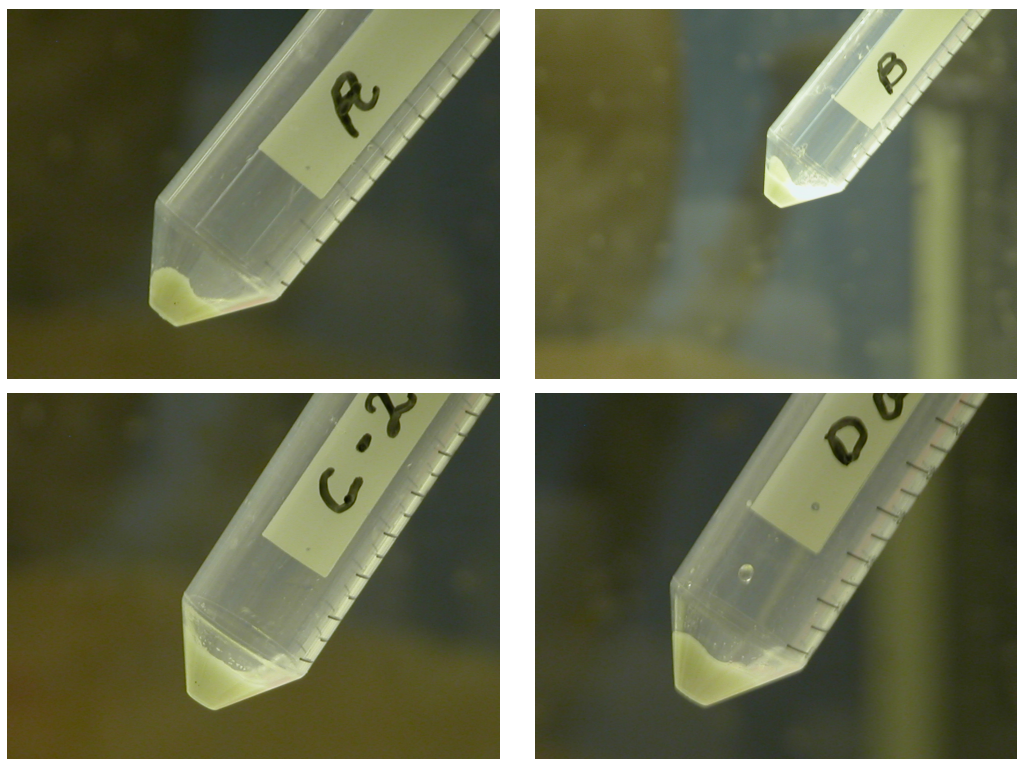


Figure 3-2 Residual MST (Post-Centrifuging) Solids prior to Desorption Testing

The residual solids and supernate were kept in the centrifuge tube and diluted with distilled, deionized water (rather than inhibited or process water as expected for the facility) to simulate washing of the solids. The exact level of dilution was based upon the amount of water required to reduce the residual supernate's measured sodium concentration (5.6 M) to a theoretical final sodium concentration of 0.5 M (i.e., approximately an 11 fold dilution). After dilution, the residual material was continuously shaken using an orbital shaking ( $\sim 175$  rpm) bath at a constant temperature of  $25 \pm 3$  °C. The tests were sampled at 4 h, 8 h, 12 h, and 24 h. The sample procedure was similar to that outlined in the simulant adsorption tests except volumes and dilutions were adapted for the smaller test volumes. Sample volumes were either 1 or 2 mL and dilution factors were either 5 or 2.5, respectively. Dilution was achieved using 2 M  $\text{HNO}_3$ . Samples were submitted for analysis by PuTTA and gamma analysis for  $^{85}\text{Sr}$ .

### 3.1.3 Actual Waste Adsorption Testing

Testing with actual high-level waste represented the second part of this task. Much of the test methods and parameters duplicated those from the simulant adsorption testing. Testing of multiple MST concentrations, multiple MST strikes, and intermediate filtration to remove residual MST was repeated. Additionally, the same filter types and size ( $0.1 \mu$  PVDF and PES filters) were used both for sampling and intermediate filtration. Actual waste samples from Tank 39H were provided to SRTC for two tasks – this task as well as supernate characterization for the SWPF. The samples (HTF-E 82-86) arrived July 10-11, 2003 at SRTC. The as-received waste samples were combined, sampled, and analyzed for both

sodium and anions by inductively coupled plasma-emission spectroscopy (ICP-ES) and ion chromatography (IC). At the time this document is being written, a comprehensive characterization of the waste is being performed by M. E. Stallings<sup>3</sup> and as such was not repeated in this scope of work.

The sodium concentration of the as-received waste was  $6.7 \pm 0.3$  M. The waste solution was diluted (in two steps) with 1.66 M NaOH to produce a 5.6 M sodium salt solution. The 5.6 M Na waste equilibrated for 12 weeks prior to its use in adsorption testing. The waste solution was sampled periodically during the twelve weeks to monitor the stability of Sr and actinides. Sampling involved pulling approximately 4.0 mL of the test solution into a disposable 10-mL syringe and filtering the sample mixture through a 0.1- $\mu$ m syringe filter disk (PVDF) and into a sample bottle. One milliliter portions of each filtered sample were pipetted into a second sample bottle containing 49 mL of 2 M HNO<sub>3</sub>. The diluted, acidified sample was manually shaken for approximately 15 seconds and then allowed to equilibrate for a minimum of 2 hours prior to submittal for analysis. Samples were analyzed for <sup>237</sup>Np, U, <sup>238</sup>Pu, <sup>239/240</sup>Pu, <sup>241</sup>Am, and <sup>90</sup>Sr concentrations by ICP-MS, PuTTA, and radiochemical methods. The soluble concentrations of <sup>90</sup>Sr and the actinides during the 12 weeks of equilibration are shown in Figure 3-3. Concentrations measured during equilibration and at the start of testing are shown in Table 3-4.

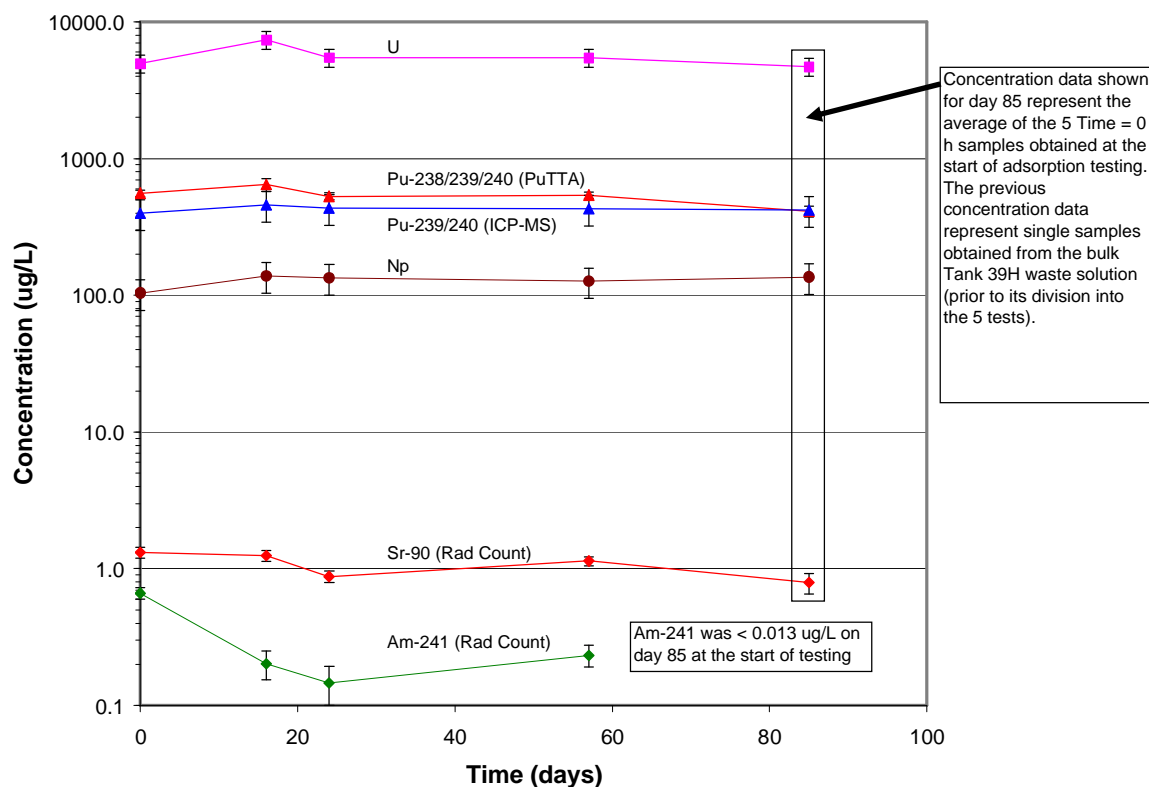


Figure 3-3 Equilibration of Tank 39H Waste Feed Solution

**Table 3-4 Equilibration of Soluble Strontium and Actinides in Tank 39H Waste Solution**

TIME (days)	Concentration (µg/L)					
	Sr-90 (Rad Count)	Total Pu (PuTTA)	Pu-239/240 (ICP-MS)	Np-237 (ICP-MS)	Total U (ICP-MS)	Am-241 (Rad Count)
0	1.31 ± 0.12	560 ± 29	397 ± 60	104 ± 16	4960 ± 744	0.663 ± 0.063
16	1.25 ± 0.12	647 ± 70	459 ± 69	139 ± 21	7400 ± 1110	0.202 ± 0.048
24	0.878 ± 0.081	530 ± 31	434 ± 65	134 ± 20	5480 ± 822	0.147 ± 0.046
57	1.14 ± 0.08	542 ± 29	431 ± 65	127 ± 19	5460 ± 819	0.233 ± 0.043
85*	0.791 ± 0.134	411 ± 39	421 ± 63	136 ± 3	4690 ± 50	< 0.013

\*Adsorption testing started on day 85. Data shown represents the average of 5 Time = 0 h samples and the reported error is their standard deviation.

Examination of Figure 3-3 shows that the soluble concentration of <sup>241</sup>Am was not stable prior to testing. The cause of the significant drop after day 58 is unknown. The decrease may result from the use of AMP in the last feed samples and that the AMP improved filtration and removed fine particulate Am. Alternatively, the decrease may result from slow precipitation of Am lasting nearly 60 days similar to that observed for simulated waste [see Figure 3-1]. Neptunium, uranium, and plutonium all show fairly stable soluble concentrations prior to testing. The <sup>90</sup>Sr concentration shows a small degree of instability.

Actual waste MST adsorption experiments used the test protocols A, B, E, G, and H shown in Table 3-5. Protocols A, B, and E duplicated experiments from the simulant adsorption testing. However, Test E was not conducted in duplicate (as was done with simulant). Testing occurred in the SRTC Shielded Cells Facility. Testing used 250-mL polyethylene bottles fitted with a cap. Researchers prepared each test by placing 120 mL of the equilibrated simulant solution in the bottle. The desired amount of MST was added to each test using pre-dosed aliquots at the appropriate time (i.e., post-sampling and filtration for the multi-strike tests E and H and at 0 h for the remaining experiments). The MST came from Optima Batch #00-QAB-417 (as in the simulant testing). Sample bottles were continuously stirred (magnetically) in a water bath at a constant temperature of 25 ± 4 °C. Figure 3-4 is a photograph of the waterbath apparatus prior to installation in the SRTC Shielded Cells. For those tests (E and H) involving intermediate filtration, the residual (post-sampling) bulk test solutions were filtered through 0.1 µ PES disposable cup filters. (For Test H, the test solution was centrifuged prior to filtration to collect the first strike MST solids for desorption testing – see Section 3.1.4). Sampling involved removing a test bottle from the waterbath, manually shaking to produce a homogeneous mixture, pulling approximately 7.0 mL of the test mixture into a disposable 10-mL syringe, and filtering the sample mixture through a 0.1-µm syringe filter disk (PVDF) and into a sample bottle. Five milliliter portions of each filtered sample were pipetted into a second sample bottle containing 20 mL of 2 M nitric acid. The diluted, acidified samples were manually shaken for approximately 15 seconds and then allowed to equilibrate for a minimum of 2 hours.

Prior to this testing, work was performed with simulants using ammonium molybdophosphate (AMP) to assess its affect on various salt solutions.<sup>3</sup> Previously, the bright yellow powder had been used analytically to remove cesium from waste solutions.

The results of the testing showed AMP, under the conditions employed, to be effective for removing cesium while not affecting the strontium or actinide concentrations.<sup>4</sup> Since the AMP demonstration was successful with simulant, DOE requested that the researchers use AMP as part of this task's sample analysis protocols. The methodology involved transferring the diluted, acidified (~1 M residual acid) sample to a second sample bottle which contained AMP (0.002 g/mL). The mixture was manually shaken for ~ 30 seconds and then immediately filtered using a 0.45  $\mu$  disposable cup filter (cellulose nitrate membrane). The filtered samples were then analyzed for  $^{237}\text{Np}$ , U,  $^{238}\text{Pu}$ ,  $^{239/240}\text{Pu}$ ,  $^{241}\text{Am}$ , and  $^{90}\text{Sr}$  concentrations by ICP-MS, PuTTA, and radiochemical methods.

**Table 3-5 Actual Waste Adsorption Test Protocols**

Test ID	Description
A	Addition of 0.4 g/L MST with sample analysis at 0, 6, 12, 24, 48, and 168 h.
B	Addition of 0.8 g/L MST with sample analysis at 0, 6, 12, 24, 48, and 168 h.
E	Add 0.4 g/L MST (incrementally) at 0, 6, and 12 h with filtration (0.1 $\mu$ m) prior to the second and third MST strikes and with sample analysis at 0, 6, 12, 24, 36, 48, and 168 h (prior to filtrations and incremental additions at 6 and 12 h).
G	Control – no addition of MST with sample analysis at 0, 6, 12, 24, and 168 h.
H	Add 0.2 g/L MST (incrementally) at 0, 6, and 12 h with filtration (0.1 $\mu$ m) prior to the second and third MST strikes and with sample analysis at 0, 6, 12, 24, and 168 h (prior to filtrations and incremental additions at 6 and 12 h).

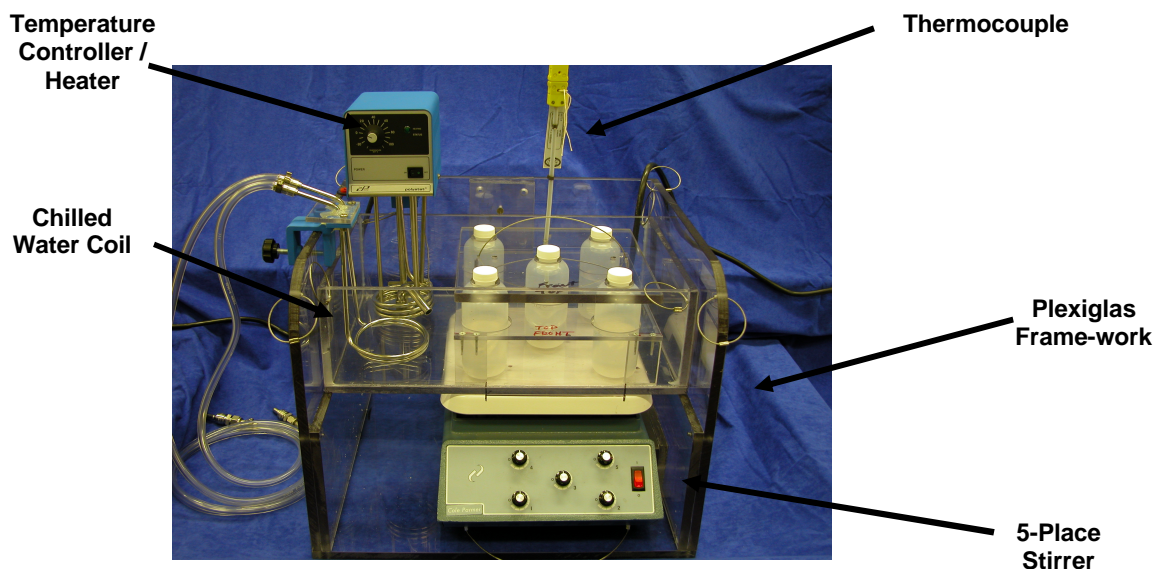


Figure 3-4 Actual Waste Adsorption Test Equipment



### 3.1.4 Actual Waste Desorption Testing

Actual waste desorption tests used the residual MST from two of the previous actual waste MST adsorption tests: Test A and the MST solids from the first strike in Test H. In the case of Test H, the solids were held in a 3 mL sample of its waste solution until after all adsorption tests completed. Similar to the simulant desorption tests, the tests were conducted by concentrating the residual MST solids using a centrifuge (see Figure 3-5). Again, the target solids concentration was 2 wt % MST. However, the small volume of residual waste solution and solids made this difficult to achieve. As with the simulant testing, we decanted as much supernate as possible off the centrifuged solids and then added the desired volume of supernate back to the solids to prepare a 2 wt % solids concentration. The mass of solids present was calculated assuming the centrifuged volume of test solution contained the target concentration of MST added during the adsorption tests (i.e., if 0.4 g/L MST was added in the adsorption test, then the residual adsorption test volume contained 0.4 g/L MST solids). Unlike the simulant desorption tests, the residual test solutions and centrifuged solids were only held for a period of 1 to 2 weeks between the adsorption and desorption tests. The decanted residual supernate from each test was sampled and analyzed to determine both the amount of Sr and Pu loaded on the MST during its adsorption testing as well as the residual soluble concentration added back to the centrifuged MST solids.

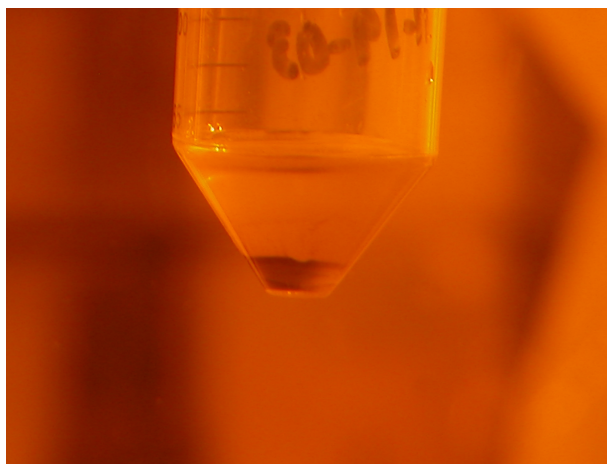


Figure 3-5 Residual MST (Post-Centrifuging) Solids from Test H prior to Desorption Testing

The residual solids and supernate were transferred to a custom-built glass vessel and diluted with distilled, deionized water (rather than inhibited or process water as expected in the facility) to simulate washing of the solids (see Figure 3-6). The exact level of dilution was based upon the amount of water required to reduce the residual supernate's measured sodium concentration (5.6 M) to a theoretical final sodium concentration of 0.5 M (i.e., approximately an 11 fold dilution). After dilution, the residual material was continuously stirred (magnetically) at a constant temperature of  $25 \pm 3$  °C in the same apparatus as used in the adsorption tests. The tests were sampled at 4 h, 8 h, 12 h, and 24 h. The sample procedure was similar to that outlined in the simulant desorption tests. Sampling involved removing a test bottle from the waterbath, manually shaking to produce a homogeneous

mixture, pulling approximately 2.0 mL of the test mixture into a disposable 10-mL syringe, and filtering the sample mixture through a 0.1- $\mu$ m syringe filter disk (PVDF) and into a sample bottle. One milliliter portions of each filtered sample were pipetted into a second sample bottle containing 4 mL of 2 M nitric acid. The diluted, acidified sample was manually shaken for approximately 15 seconds and then allowed to equilibrate for a minimum of 2 hours. Unlike the actual waste adsorption tests, AMP was not employed since the residual cesium concentration was low (due to decanting off the supernate and dilution with water). The filtered samples were analyzed for  $^{238}\text{Pu}$ ,  $^{239/240}\text{Pu}$ , and  $^{90}\text{Sr}$  by PuTTA and radiochemical methods.

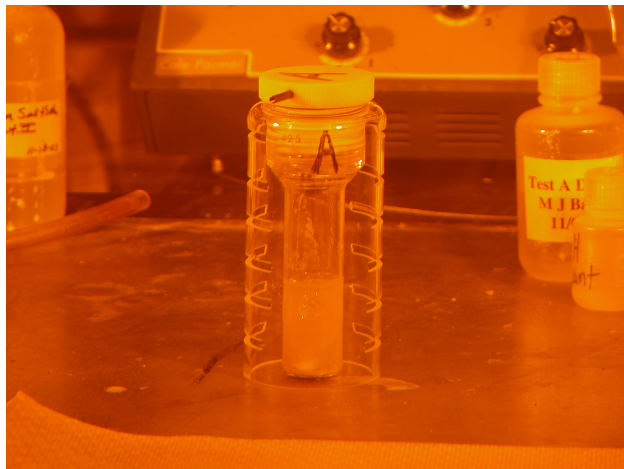


Figure 3-6 Desorption Test Vessel with Residual MST Solids and Diluted Supernate

## 3.2 RESULTS

### 3.2.1 Simulant Adsorption Testing

The objective of both the simulated and actual waste adsorption tests was to investigate the influence of multiple MST additions and the use of intermediate filtration on the efficiency of MST to treat high-level-waste. Simulant testing offered the first attempt at understanding the effect of these parameters. Radioactivity and mass concentration data from the tests are contained in the Appendix (see Table 7-1 and Table 7-2, respectively). Note that uranium data are not provided in the first table since its radioactivity concentration is so low. Figure 3-7 through Figure 3-14 provides graphical representation of the data for the actinides as well as  $^{85}\text{Sr}$ . A close examination of the Pu data in Figure 3-8 indicates that use of multi-strikes alone does not influence Pu adsorption or significantly improve the process cycle time (i.e., two strikes of 0.4 g/L MST yield a similar solution concentration as a single strike of 0.8 g/L MST, likewise three strikes of 0.4 g/L MST yield a similar solution concentration as a single strike of 1.2 g/L MST). Comparison of the Pu data also indicates that increasing the MST concentration alone provides minimal cycle time improvement. However, Figure 3-7 effectively demonstrates that the use of intermediate filtration between MST strikes does significantly increase the total amount of Pu sorbed by the cumulative MST. The cause of this increase is presumably due to the removal of sorbed Pu from the solution, thus shifting the adsorption curve to a lower starting Pu concentration (i.e., providing a conventional “stage efficiency” for sequential separations).

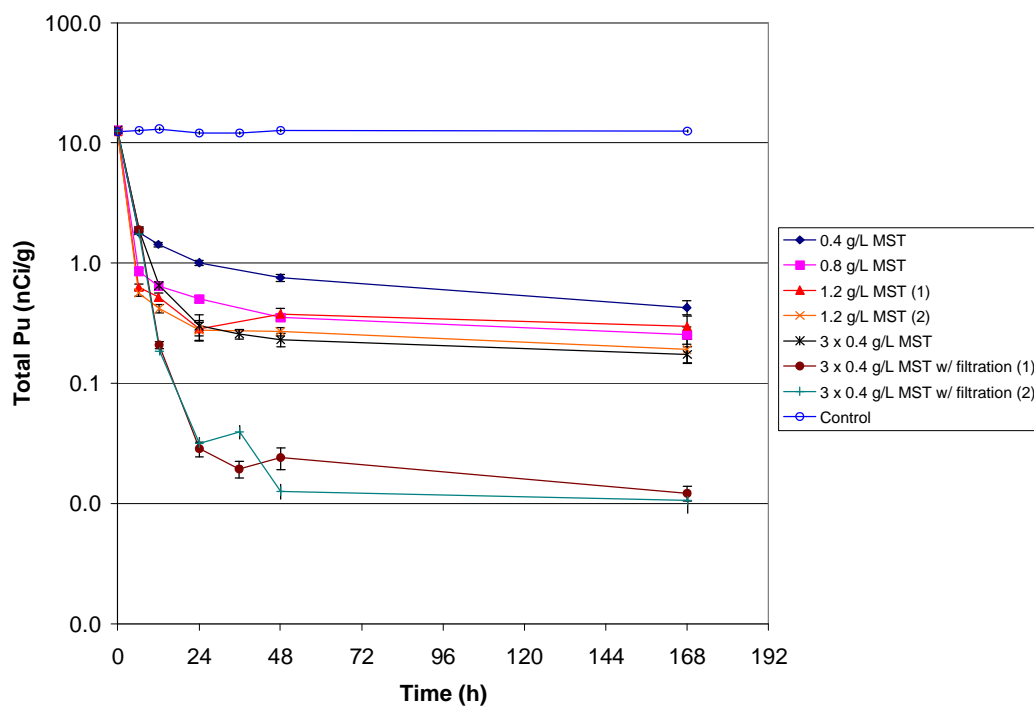


Figure 3-7 Simulant Adsorption Test Plutonium Results

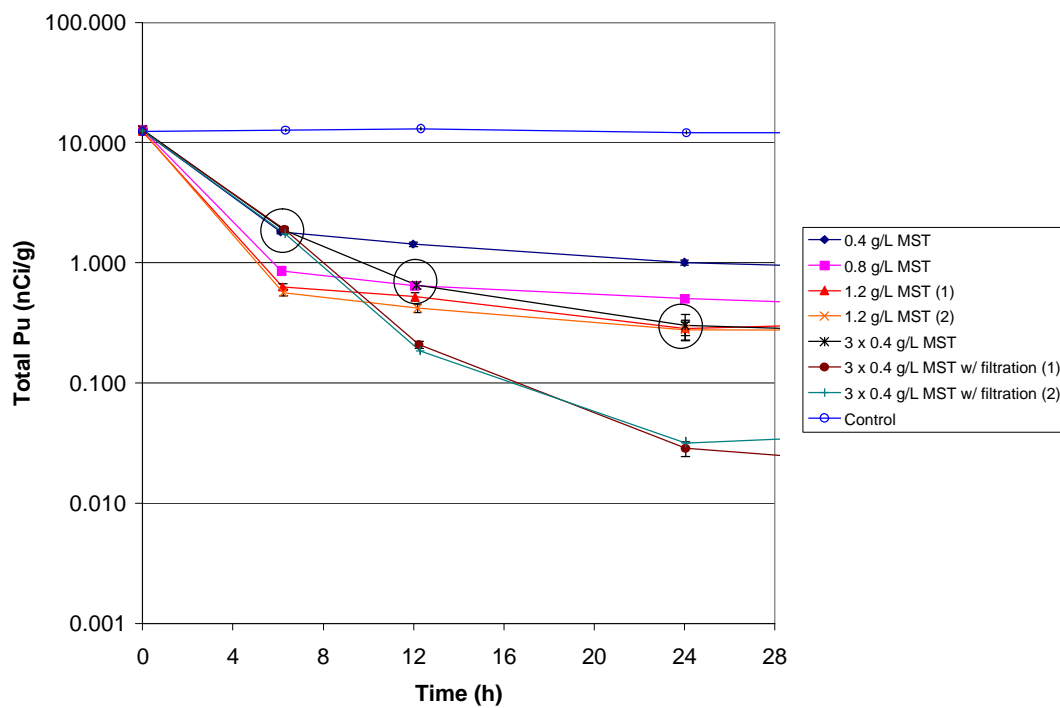


Figure 3-8 Simulant Adsorption Test Plutonium Results – Expanded View



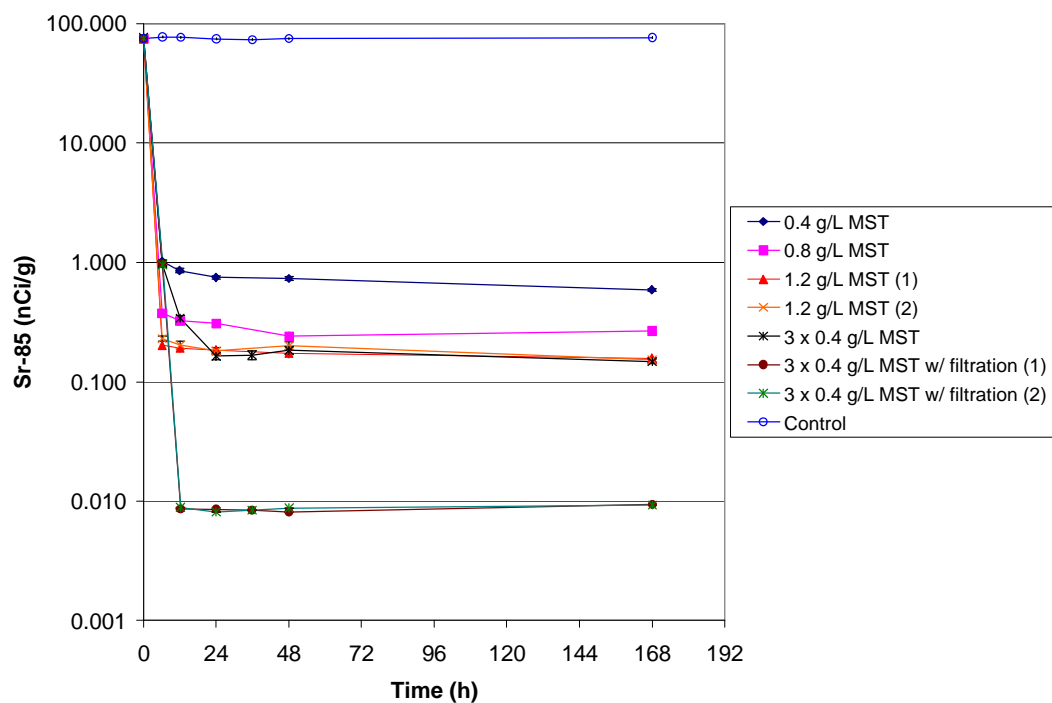


Figure 3-9 Simulant Adsorption Test  $^{85}\text{Sr}$  Results

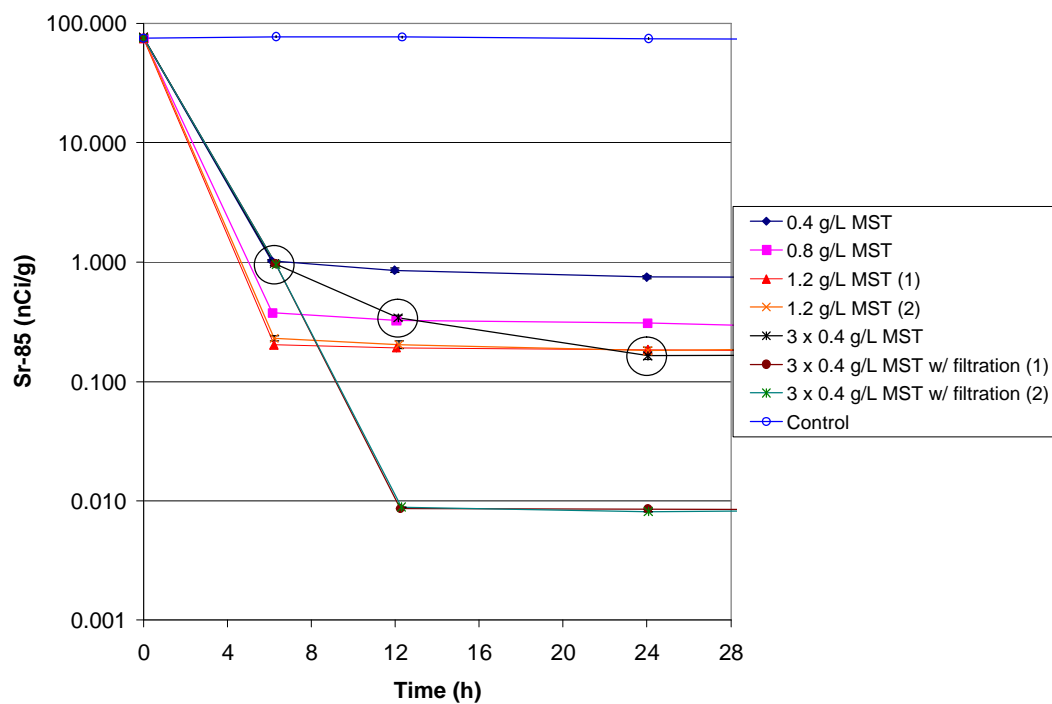


Figure 3-10 Simulant Adsorption Test  $^{85}\text{Sr}$  Results – Expanded View

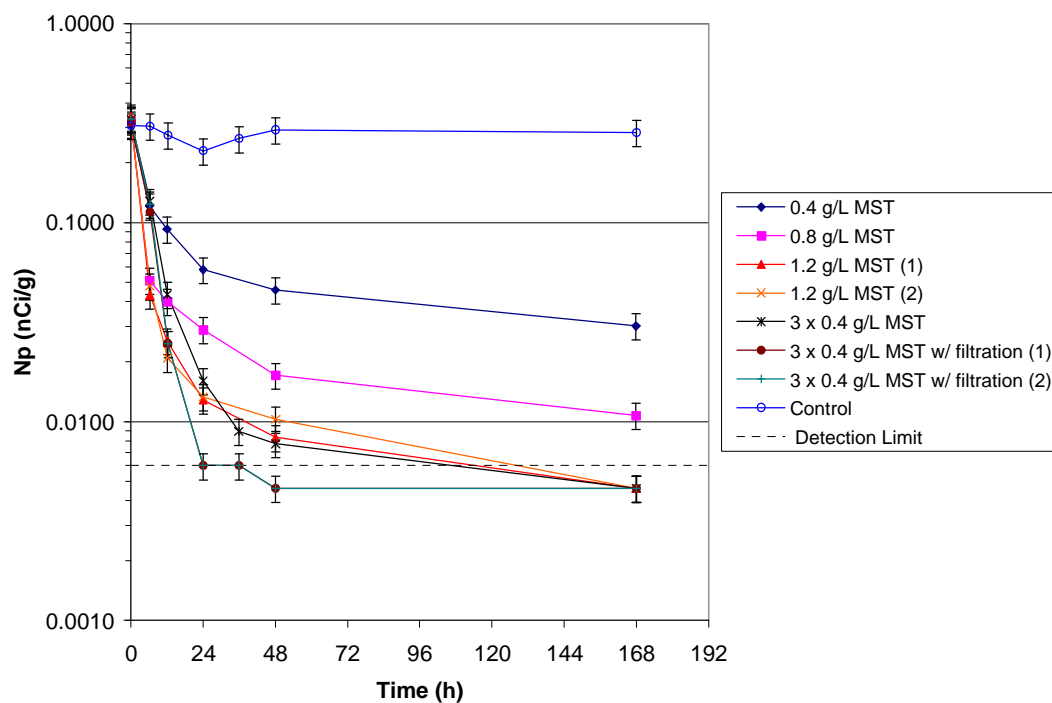


Figure 3-11 Simulant Adsorption Test  $^{237}\text{Np}$  Results

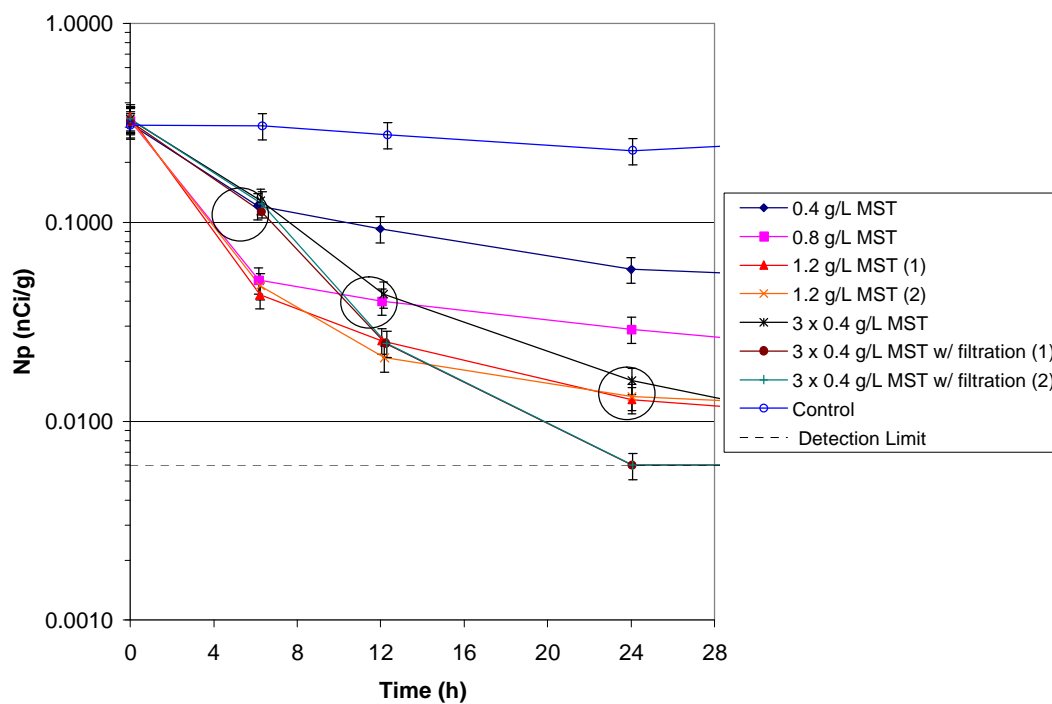


Figure 3-12 Simulant Adsorption Test  $^{237}\text{Np}$  Results – Expanded View

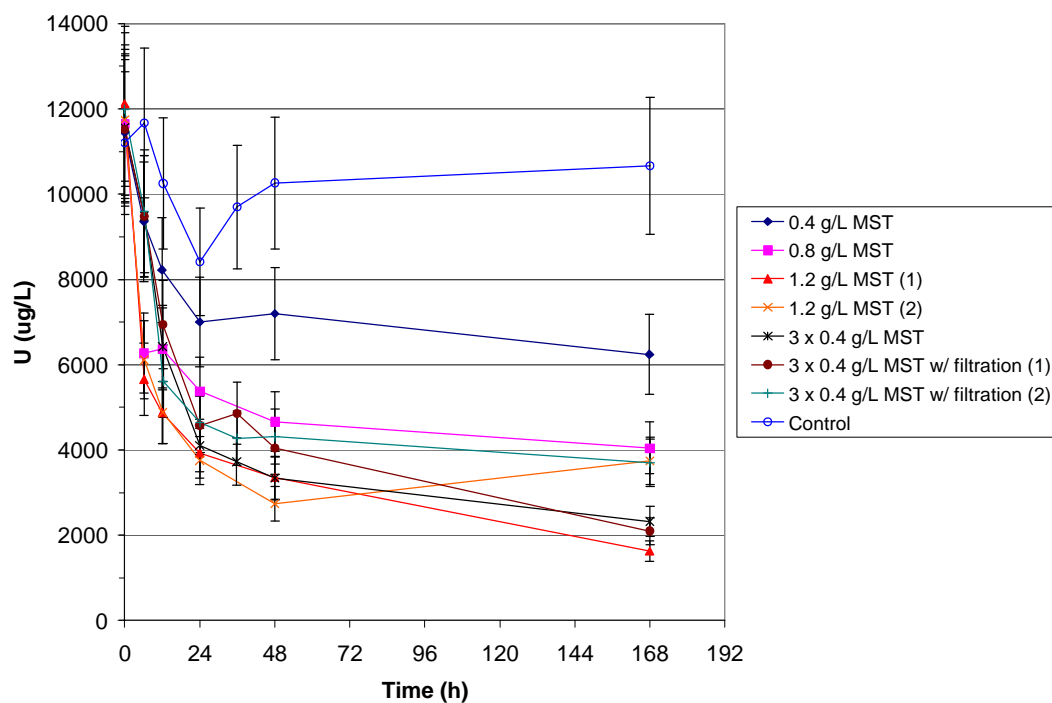


Figure 3-13 Simulant Adsorption Test U Results

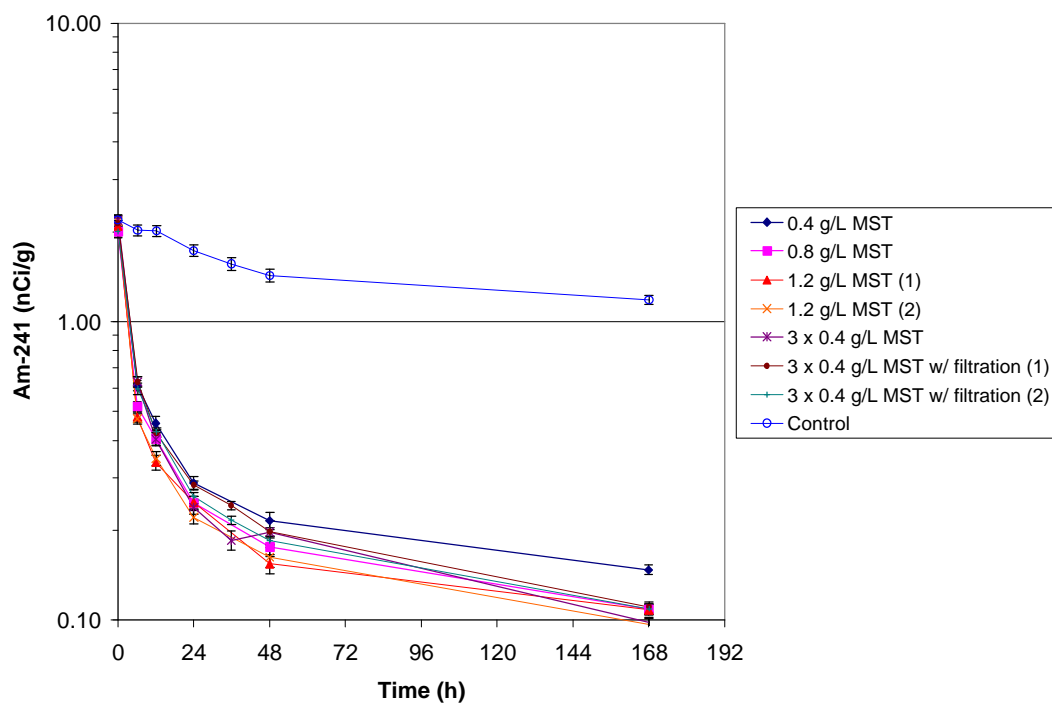


Figure 3-14 Simulant Adsorption Test <sup>241</sup>Am Results

Figure 3-9 and Figure 3-10 demonstrate the same observations for  $^{85}\text{Sr}$  adsorption onto MST. However, the detection limit of  $^{85}\text{Sr}$  obscures the full extent of the influence of intermediate filtration on the species. Both data sets showed exceptional agreement between replicates. Figure 3-11 and Figure 3-12 show similar trends for Np as observed with the Pu and  $^{85}\text{Sr}$  data. However, since the 1.2 g/L MST and intermediate filtration test data fall below the detection limit early in the test, the increased influence of intermediate filtration cannot be certain. In the case of uranium, the data shows more variability than the other data sets (this is also due in part to the graph not using a log scale for the U concentration) as evidenced in Figure 3-13. Similar to the Pu,  $^{85}\text{Sr}$ , and  $^{237}\text{Np}$  data, the U data demonstrate that multi-strike is ineffective in increasing removal efficiency. Unlike the other three species, the influence of intermediate filtration was not observed with the U data. Figure 3-14 shows that the behavior of  $^{241}\text{Am}$  is unique among the test species. Little if any increase in sorption from added MST is observed and intermediate filtration was ineffective. The Control test shows a small decrease in soluble  $^{241}\text{Am}$  during the testing leading to speculation that the mechanism of  $^{241}\text{Am}$  removal is likely due to precipitation rather than adsorption.

Decontamination factors (DFs) were calculated for each of the tests and each Analyte as reported in Table 3-6. Comparison of the DFs between comparable tests is reliable since the tests all used the same simulant and temperature. The DFs reflect the same observations as the graphs. Specifically, that Pu,  $^{85}\text{Sr}$ , and Np all show increased adsorption with the combination of increased addition of MST and the use of intermediate filtration. Uranium DFs also demonstrate the increase associated with increased MST addition. However, intermediate filtration did not produce a measurable increase in DF for U. Lastly,  $^{241}\text{Am}$  did not demonstrate increase in DF due to either an increase of MST or the use of intermediate filtration.

**Table 3-6 Simulant Adsorption Test Decontamination Factors**

	<u>Pu</u>		<u>Sr-85</u>		<u>Np</u>		<u>U</u>		<u>Am-241</u>	
	24 h	168 h	24 h	168 h	24 h	168 h	24 h	168 h	24 h	168 h
0.4 g/L MST	13	30	100	130	5.4	10	1.6	1.8	7.3	14
0.8 g/L MST	27	52	240	280	11	30	2.2	2.9	8.1	18
1.2 g/L MST (avg)	44	55	410	490	26	72	3.1	5.3	9.1	21
3 x 0.4 g/L MST	44	74	470	520	21	72	2.8	5.0	9.2	22
3 x 0.4 g/L MST filtered (avg)	450	1190	9010	8050	55	71	2.6	4.4	7.6	19
Control	1.0	1.0	1.0	1.0	1.3	1.1	1.3	1.0	1.3	1.9

### 3.2.2 Simulant Desorption Testing

The release of sorbed Sr and actinides from loaded MST during the washing stages in the SWPF is of concern. Desorption tests were performed to assess this potential problem using loaded MST from four of the previous MST adsorption tests: Tests A, B, C (actually Test C2 since duplicate C adsorption tests were conducted), and D. The use of a centrifuge to concentrate the residual MST solids proved adequate. The target solids concentration and diluted sodium concentrations were obtained with little difficulty. Table 3-7 provides the test characteristics of interest. Specifically, the theoretical maximum Pu and  $^{85}\text{Sr}$  concentrations are well above the instruments' detection limit. The theoretical max concentrations represent

the maximum concentrations that would result if all of the species desorbed into the washwater. Examination of the data contained in Table 3-8 shows that all but one of the data points are below the instrumental detection limit. While the exact value of each species released is unknown, the less than values serve to bound the release rate at a very small value, if any. The magnitude of the detection limit demonstrates that release of sorbed species from loaded MST are well below the Waste Acceptance Criteria (WAC) limits and should not be a problem during washing in the SWPF.

**Table 3-7 Simulant Desorption Test Characteristics**

Test ID	Theoretical Solids Concentration (wt%)*	Pu Loading (umole/g MST)	Theoretical Max [Pu] (nCi/g)#	Sr-85 Loading (umole/g MST)	Theoretical Max [Sr-85] (nCi/g)#
A	2.1	2.2	88	1.1E-04	530
B	2.0	1.1	45	5.7E-05	260
C	2.1	0.72	29	3.9E-05	180
D	2.0	0.74	30	3.9E-05	180

\*The theoretical solids concentration is calculated from the mass of MST added to the original adsorption test, the residual volume of test solution from the adsorption test, and the volumes of supernate and wash water added to the desorption test.

#The theoretical maximum Pu and <sup>85</sup>Sr concentrations represent the maximum concentrations that would result if all of the species desorbed into the washwater.

**Table 3-8 Simulant Desorption Test Data – Concentration Units of nCi/g**

Test ID	Reaction Time (h)	Concentration (nCi/g)			
		<sup>85</sup> Sr	±	Pu	±
Test A	0.0	0.007	0.002	< 0.02	mda
Test A	4.0	< 0.076	mda	< 0.92	mda
Test A	8.0	< 0.068	mda	< 0.08	mda
Test A	12.0	< 0.067	mda	< 1.34	mda
Test A	24.1	< 0.066	mda	< 10.0	mda
Test B	0.0	0.003	0.001	< 0.03	mda
Test B	4.1	< 0.034	mda	< 0.19	mda
Test B	8.0	< 0.035	mda	< 0.18	mda
Test B	12.0	< 0.016	mda	< 0.15	mda
Test B	24.1	< 0.032	mda	< 0.42	mda
Test C	0.0	< 0.002	mda	< 0.02	mda
Test C	4.0	< 0.035	mda	< 11.4	mda
Test C	8.0	< 0.039	mda	< 0.16	mda
Test C	12.0	< 0.018	mda	< 1.07	mda
Test C	24.1	< 0.031	mda	< 0.22	mda
Test D	0.0	< 0.002	mda	< 0.01	mda
Test D	4.0	< 0.032	mda	< 0.20	mda
Test D	8.1	< 0.032	mda	< 0.61	mda
Test D	12.1	< 0.018	mda	< 0.08	mda
Test D	24.1	< 0.030	mda	< 0.12	mda

mda = minimum detectable analysis

### 3.2.3 Actual Waste Adsorption Testing

Actual waste adsorption testing was performed with tank 39H waste diluted to 5.6 M sodium. Actinide analysis by ICP-MS of the Tank 39H feed after 12 weeks of testing showed the waste to be unique in composition (see Table 3-9). Analysis shows the waste to contain a

high percentage of  $^{235}\text{U}$  as well as measurable quantities of  $^{241}\text{Pu}$  and  $^{242}\text{Pu}$ . Additionally, the  $^{241}\text{Am}$  was determined to be significantly lower than observed in previous feed sample analyses (i.e., sample analyses performed during feed equilibration prior to testing). Speculation is that the difference between earlier feed samples and the equilibrated samples at the start of testing is caused by an AMP filtration effect, although this possible explanation of the difference remains unproven.

**Table 3-9 Soluble Actinides Composition of 5.6 M Na Tank 39H Waste**

Mass #	Average Starting Concentration (ug/L)	Uncertainty (ug/L) (n = 5)
$^{234}\text{U}$	118	5.8
$^{235}\text{U}$	1491	7.7
$^{236}\text{U}$	476	4.0
$^{237}\text{Np}$	136	2.8
$^{238}\text{U}$	2608	50.3
$^{239}\text{Pu}$	374	12.5
$^{240}\text{Pu}$	47.1	3.6
$^{241}\text{Pu}$	8.92	1.63
$^{242}\text{Pu}$	38.4	2.6
$^{241}\text{Am}$ (Rad)	< 0.0134	upper limit

Actual waste adsorption testing repeated four of the five test protocols from simulant testing. The fifth test involved three additions of 0.2 g/L MST with intermediate filtration. The objectives of the tests were the same as that of the simulant testing. Radioactivity and mass concentration data from the tests are contained in the Appendix (see Table 7-3 and Table 7-4, respectively). Note that U data again are not provided in the first table since its radioactivity concentration is so low. Figure 3-15 through Figure 3-17 provides graphical representation of the data for Pu,  $^{90}\text{Sr}$ , and U. Both  $^{237}\text{Np}$  and  $^{241}\text{Am}$  data were not presented graphically since the bulk of their data fell below their detection limits.

Figure 3-15 demonstrates the same findings as observed in simulant adsorption testing. Specifically, increased MST provides for increased adsorption with minimal cycle time improvement. The use of intermediate filtration increased Pu removal. In fact, only the two multi-strike tests with intermediate filtration reached the Pu process limit (18 nCi/g) for this waste within 24 h. Figure 3-16 demonstrates many of the same findings for  $^{90}\text{Sr}$ , with a few notable exceptions. All tests met the process limit for  $^{90}\text{Sr}$ . The behavior of the two multi-strike tests with intermediate filtration were contrary to common logic (i.e., the 3 x 0.2 g/L MST outperformed the 3 x 0.4 g/L MST). The cause of this later discrepancy is unknown.

Figure 3-17 shows that U behavior is similar to that observed in the simulant tests. Specifically, adsorption increased with increasing MST concentration. In the case of  $^{237}\text{Np}$ , the low starting concentration prevented full observation of MST test influences (i.e., the detection limits cover the test results). However, results from six hours of testing followed the predicted behavior.

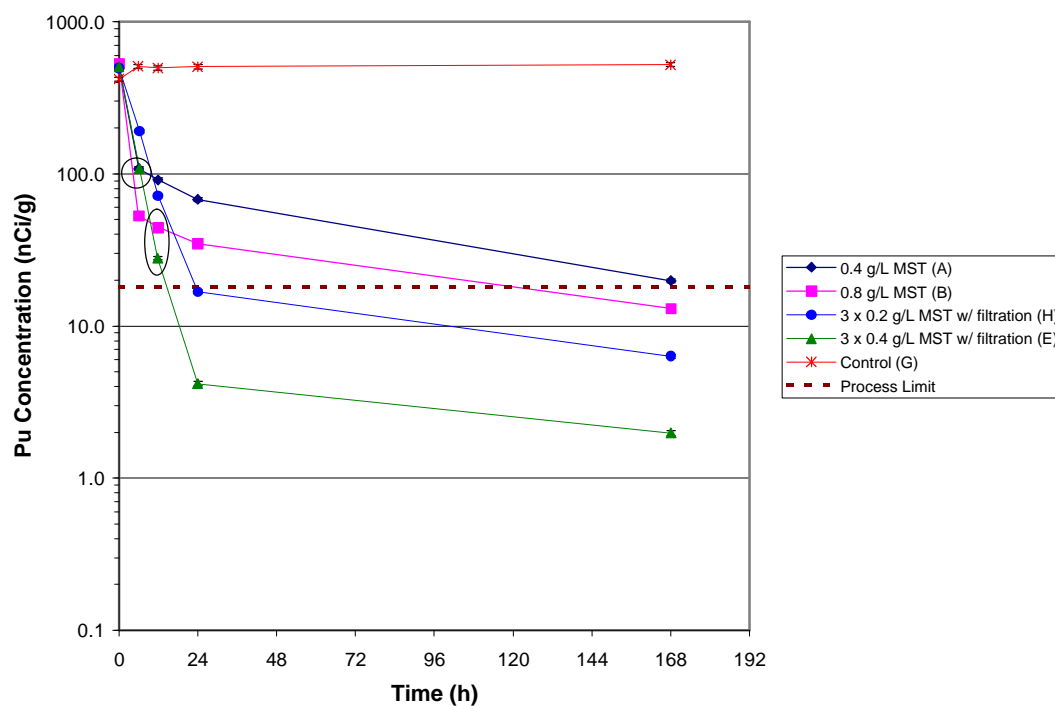


Figure 3-15 Actual Waste Adsorption Test  $^{238/39/40}\text{Pu}$  Results

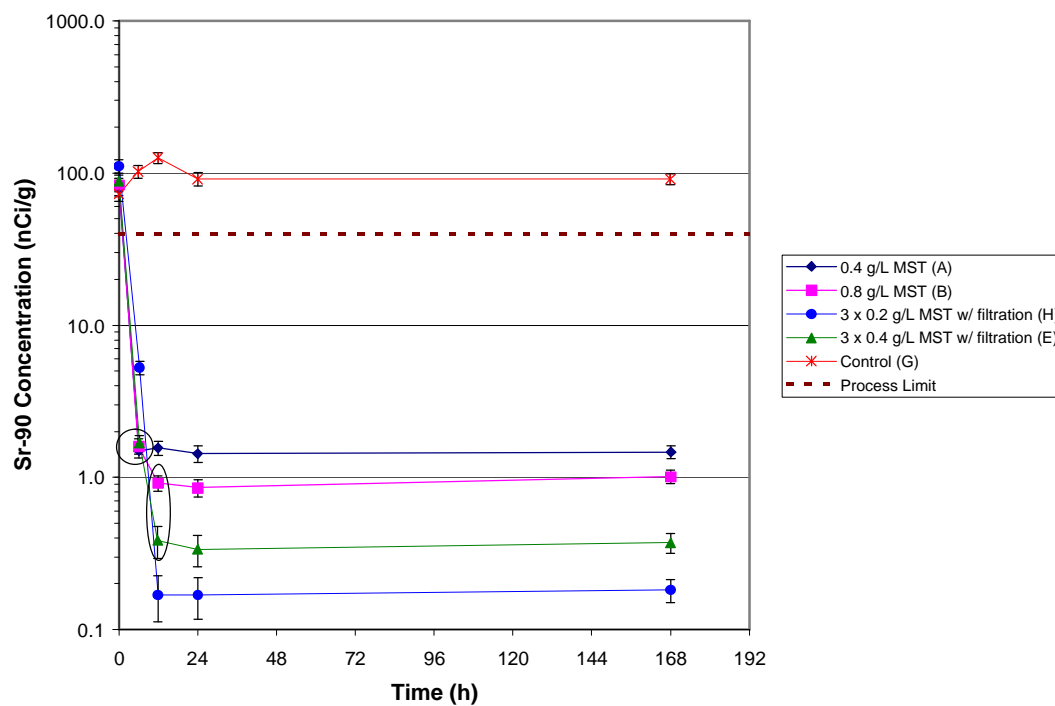


Figure 3-16 Actual Waste Adsorption Test  $^{90}\text{Sr}$  Results

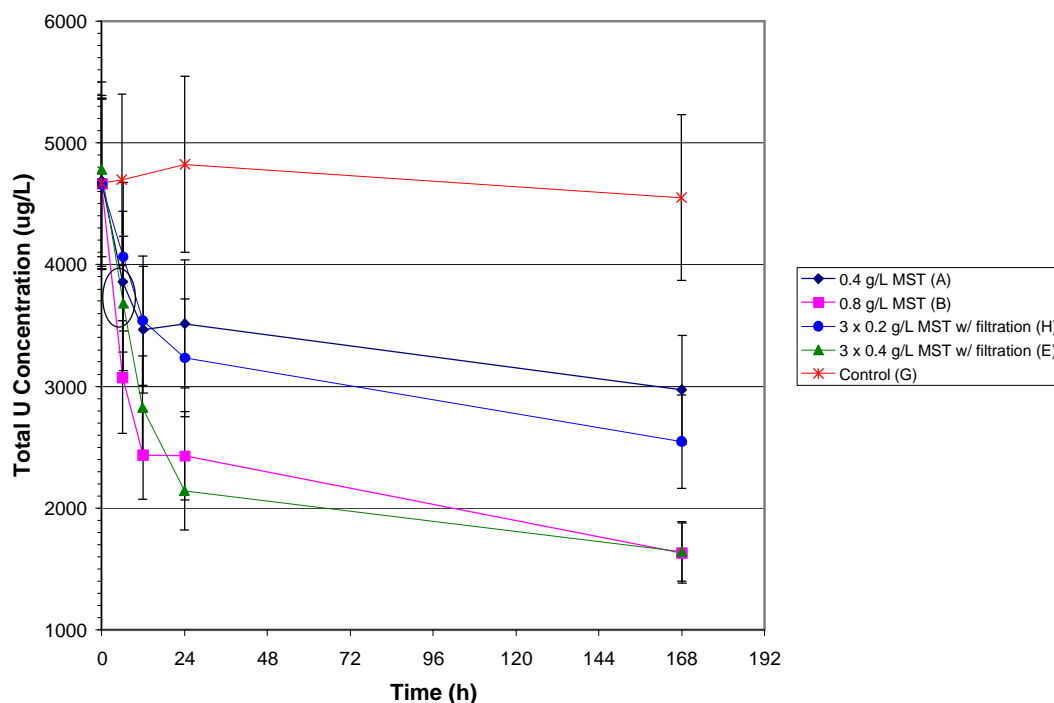


Figure 3-17 Actual Waste Adsorption Test U Results

Decontamination factors were calculated for each analyte as reported in Table 3-10. Again, comparison of the DFs between comparable tests is possible since the tests all used the same starting material and temperature. The DFs reflect the same observations as the graphs. Specifically, that Pu and  $^{90}\text{Sr}$  all show increased adsorption with increased addition of MST and the use of intermediate filtration. Uranium DFs also demonstrate the increase associated with increased MST addition.

Table 3-10 Actual Waste Adsorption Test Decontamination Factors

Test	Pu		Sr-90	
	24 h	168 h	24 h	168 h
0.4 g/L MST (A)	7.8	25	55	54
0.8 g/L MST (B)	15	40	98	83
3 x 0.2 g/L MST (H)	33	68	660	NA
3 x 0.4 g/L MST (E)	120	320	260	240
Control (G)	0.86	0.81	0.80	0.80

Test	Np		U	
	6 h#	168 h	24 h	168 h
0.4 g/L MST (A)	2.1	> 2.1	1.3	1.6
0.8 g/L MST (B)	3.8	> 2.2	1.9	3.0
3 x 0.2 g/L MST (H)	1.6	> 2.2	1.5	1.8
3 x 0.4 g/L MST (E)	2.1	> 2.2	2.3	3.1
Control (G)	1.0	1.2	0.99	1.0

#DFs for Np were reported for 6 h instead of 24 h since the data was all below the detection limit after 6 h.



### 3.2.4 Actual Waste Desorption Testing

Actual waste desorption tests were conducted to investigate the release of sorbed Sr and Pu from loaded MST during washing. Tests used loaded MST from two of the previous Actual Waste MST adsorption tests: Tests A and H (first strike solids only). As with the simulant desorption testing, the use of a centrifuge to concentrate the residual MST solids proved adequate. The target solids concentration and diluted sodium concentrations were obtained with little difficulty. Table 3-11 provides the test characteristics of interest. Specifically, the theoretical maximum Pu and  $^{90}\text{Sr}$  concentrations are well above the analytical detection limit. The concentrations represent the maximum concentrations that would result if all of the species desorbed into the washwater. Examination of the data contained in Table 3-12 shows that  $^{90}\text{Sr}$  desorption was minimal (if any) during the 24 h test period. Desorption of  $^{239/40}\text{Pu}$  was not detected in the 4, 8, 12, or 24 h samples from either test. Desorption of  $^{238}\text{Pu}$  was detected in the 12 and 24 h samples in both tests, although at concentrations well below the WAC process limits. Less than 0.01 % of the loaded  $^{238}\text{Pu}$  desorbed in 24 h. However, the data shows an increasing trend and is insufficient to predict the level of desorption at the end of the cycle time.

**Table 3-11 Actual Waste Desorption Test Characteristics**

Test ID	Theoretical Solids Concentration (wt%)*	Pu Loading (umole/g MST)	Theoretical Max [Pu] (nCi/g)#	Sr-90 Loading (umole/g MST)	Theoretical Max [Sr-90] (nCi/g)#
A	1.9	4.3	3600	0.020	560
H	2.4	8.6	7200	0.054	1600

\*The theoretical solids concentration is calculated from the mass of MST added to the original adsorption test, the residual volume of test solution from the adsorption test, and the volumes of supernate and wash water added to the desorption test.

#The theoretical maximum Pu and  $^{90}\text{Sr}$  concentrations represent the maximum concentrations that would result if all of the species desorbed into the washwater.

**Table 3-12 Actual Waste Desorption Test Data – Concentration Units of nCi/g**

Test ID	Reaction Time (h)	Concentration (nCi/g)					
		$^{90}\text{Sr}$	$\pm$	$^{238}\text{Pu}$	$\pm$	$^{239/40}\text{Pu}$	$\pm$
AW-Test A	0.0	0.23	0.02	< 0.101	mda	0.032	0.008
AW-Test A	4.0	0.18	0.05	< 0.036	mda	< 0.118	mda
AW-Test A	8.0	< 0.14	mda	< 0.574	mda	< 0.037	mda
AW-Test A	12.0	< 0.12	mda	0.084	0.017	< 0.153	mda
AW-Test A	24.0	0.34	0.06	0.230	0.046	< 0.089	mda
AW-Test H	0.0	0.41	0.04	< 2.363	mda	< 0.112	mda
AW-Test H	4.1	< 0.11	mda	< 0.287	mda	< 0.068	mda
AW-Test H	8.2	0.30	0.06	< 0.104	mda	< 0.036	mda
AW-Test H	12.0	0.17	0.05	0.247	0.049	< 0.054	mda
AW-Test H	24.1	0.27	0.05	0.549	0.626	< 0.064	mda
Blank	4.0	0.21	0.06	< 0.035	mda	< 0.145	mda
Blank	12.0	0.14	0.05	0.065	0.013	< 0.064	mda

mda = minimum detectable analysis

### 3.2.5 Mathematical Modeling

#### 3.2.5.1 Prediction of Sorption Performance

An earlier report determined the Dubinin-Astashov (DA) model best fitted actinide sorption on MST.<sup>5</sup> That report determined optimal parameters for the Dubinin-Astashov model by regression of a large data set for MST sorption. Personnel used this model to predict actinide loading on MST under the conditions used in this study. To perform the calculations personnel used Jump<sup>®</sup> software (version 5.03) from the SAS institute.

The authors determined the solute final concentration and the amount of solute loaded on the MST sorbent to assess the sorption prediction. Researchers determined the final solute concentration from the intercept of the operating line and the predicted isotherm (see Figure 3-18). The operating line (OL) represents the mass balance equation between the final and initial solute concentration and the solute loaded on the sorbent. The slope of the operating line is defined as the ratio of solution volume to mass of sorbent. The slope and intercept of the operating line with the predicted isotherm varied with experimental conditions. Correspondingly, the predicted final solute concentration (i.e., the intercept with the isotherm curve) varied. In the case of several batch additions to a solution, the slope of the operating line decreased with the sequential additions since the ratio of solution volume to solid decreased. The final concentration as determined from the intercept of the OL with DA model decreased. If filtration is conducted between batch additions, the slope of the OL remains the same but the line is offset (shifted) to lower concentrations with each filtration and addition of MST. Consequently, the final actinide concentration decreases even more than for sequential strikes without filtration. The effect of multiple MST strikes and filtration is shown in (The numbers on the operating lines (OL) indicate the order of the MST strike to the solution.) Figure 3-19. With the information above, researchers predicted the final actinide

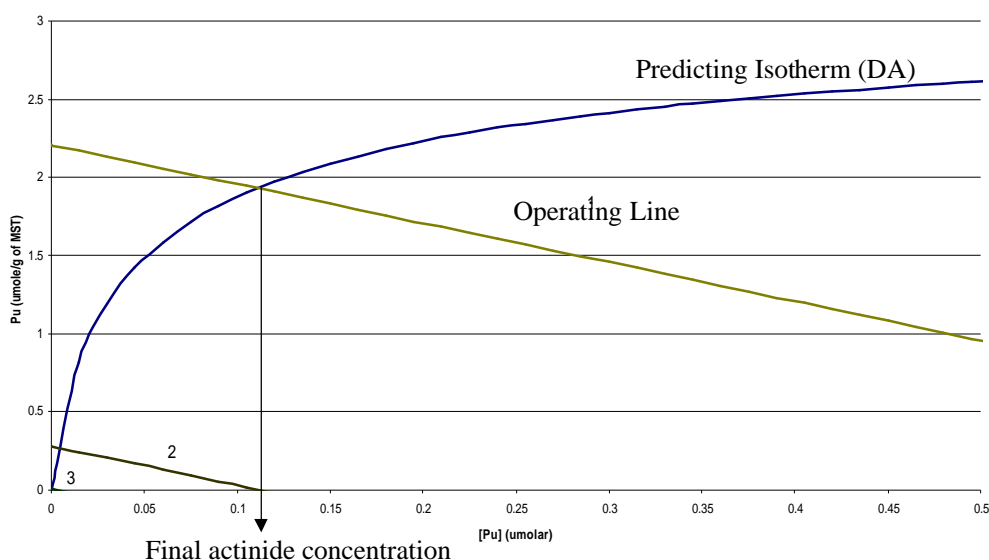
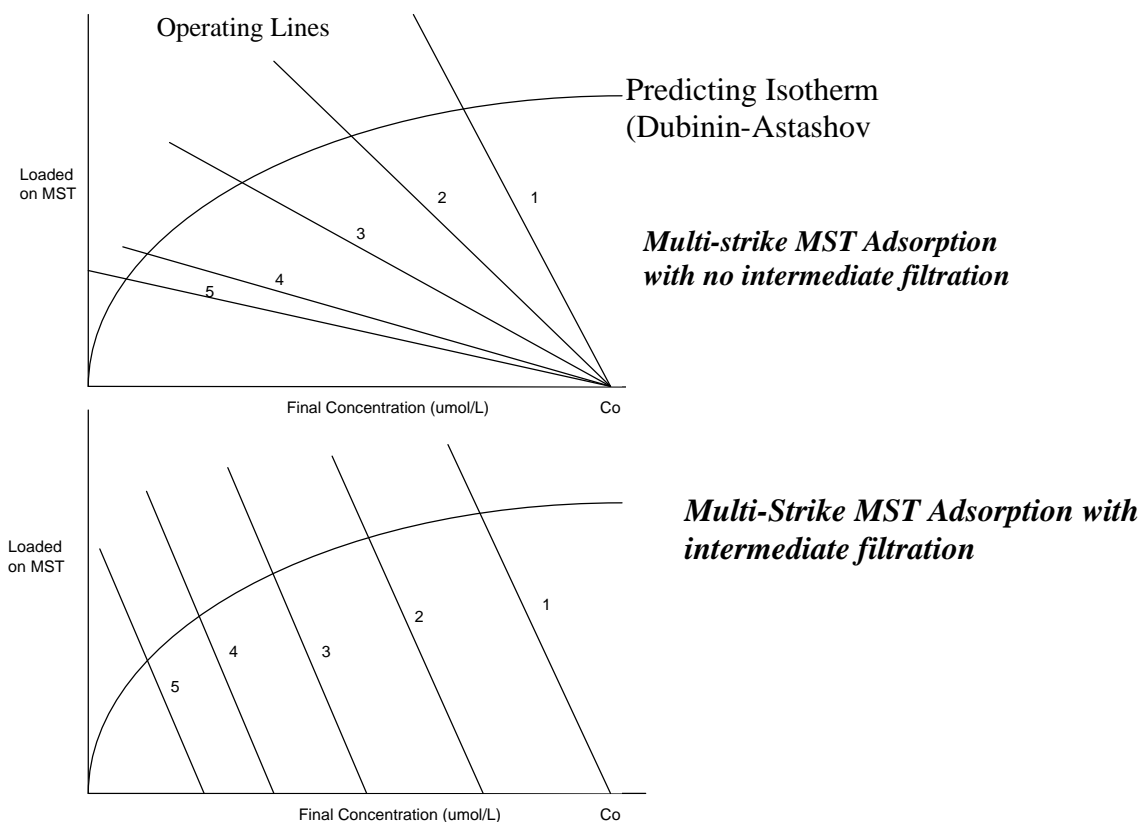


Figure 3-18 The X-value of the intercept of the operating line with the predicting isotherm gives the final concentration for a given batch contact.



(The numbers on the operating lines (OL) indicate the order of the MST strike to the solution.)

Figure 3-19 The effect of multiple MST strikes and filtration on the final actinide concentration

and Sr concentrations for the various monosodium titanate addition strategies. The predictions contained errors determined from the individual prediction confidence limits of the isotherm.

Table 3-13 compares the measured versus predicted concentrations of Sr and actinides for simulant adsorption testing. Table 3-14 compares the measured versus predicted concentrations of Sr and actinides for actual waste adsorption testing. Examination of the measured versus predicted data shows that, in general, the model over predicts or is close to the measured Pu concentration and under predicts or is close to the measured Sr, Np, and U concentrations. A factor which affects the comparison is that in the case of multiple strikes (with or without filtration), is that the measured values are not at equilibrium (the model is based upon equilibrium data and better predicts data collected after longer times like 168 h, note that equilibrium is assumed to have occurred by 168 h). In general, the model does a credible job of predicting the trends in solution concentrations. The model generally appears to offer the best level of predictability for the isotopes with the following order: Pu > U > Sr > Np.

**Table 3-13 Measured Versus Predicted Solution Concentrations for Simulant Adsorption Tests**

Test ID	MST (g/L)	Analysis Time (h)*	Plutonium (μM)		Strontium (μM)		Neptunium (μM)		Uranium (μM)	
			Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured	Predicted
Test A	0.4	168	0.03	0.11	0.02	0.02	0.221	0.27	40	32.4
Test B	0.8	168	0.02	0.02	0.012	0.008	0.08	4.00E-03	27	23
Test C	1.2	168	0.017	0.013	0.007	0.005	0.034	5.00E-05	17	16.3
Test D - 1st Strike	1 x 0.4	6 h	0.129	0.110	0.026	0.020	0.94	0.27	40	32.4
Test D - 2nd Strike	2 x 0.4	12 h	0.045	0.025	0.012	0.008	0.32	4.00E-03	27	23
Test D - 3rd Strike	3 x 0.4	24 h	0.021	0.013	0.007	0.005	0.03	5.00E-05	17	16.3
Test D - 3rd Strike	3 x 0.4	168 h	0.012	0.013	0.007	0.005	0.0034	5.00E-05	16	16.3
Test E - 1st Strike	1 x 0.4 w/ filtration	6 h	0.13	0.11	0.045	0.020	0.83	0.27	40	32.4
Test E - 2nd Strike	2 x 0.4 w/ filtration	12 h	0.014	0.005	0.0004	7.2 E-5	0.18	2.00E-13	26	20.3
Test E - 3rd Strike	3 x 0.4 w/ filtration	24 h	0.002	0.0005	0.0004	4.1 E-7	<0.4	< 4.E-19	19	11.1
Test E - 3rd Strike	3 x 0.4 w/ filtration	168 h	0.001	0.013	0.006	0.005	<0.4	< 2.E-21	12	8

\*The analysis time refers to the time at which the measured data was obtained. Correspondingly, all predicted data assume equilibrium was obtained.

**Table 3-14 Measured Versus Predicted Solution Concentrations for Actual Waste Adsorption Tests**

Test ID	MST (g/L)	Analysis Time (h)*	Plutonium (μM)		Strontium (μM)		Neptunium (μM)		Uranium (μM)	
			Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured	Predicted
AW - Test A	0.4	168	0.07	0.66	0.03	0.01	0.035	6.50E-07	14.22	10.73
AW - Test B	0.8	168	0.05	0.16	0.024	3 E-04	0.024	4.00E-13	6.9	5.33
AW-Test E - 1st Strike	1 x 0.4 w/ filtration	6 h	0.37	0.66	0.04	0.01	0.58	0.7	14.5	11.03
AW-Test E - 2nd Strike	2 x 0.4 w/ filtration	12 h	0.053	0.093	0.009	3.97 E-05	0.27	0.18	9.11	4.28
AW-Test E - 3rd Strike	3 x 0.4 w/ filtration	24 h	0.002	0.015	0.008	2.45 E-07	<0.24	0.03	4.29	0.31
AW-Test E - 3rd Strike	3 x 0.4 w/ filtration	168 h	NA	NA	0.0085	2.45 E-07	NA	NA	1.07	0.31
AW-Test H - 1st Strike	1 x 0.2 w/ filtration	6 h	0.64	1.22	0.1	0.02	0.56	0.7	16.31	14.54
AW-Test H - 2nd Strike	2 x 0.2 w/ filtration	12 h	0.24	0.68	0.003	1.84E-04	0.35	0.11	13.22	10.11
AW-Test H - 3rd Strike	3 x 0.2 w/ filtration	24 h	0.055	0.220	0.003	1.80E-06	<0.24	0.012	<9.04	6.33
AW-Test H - 3rd Strike	3 x 0.2 w/ filtration	168 h	0.03	0.22	NA	NA	NA	NA	6.97	6.33

\*The analysis time refers to the time at which the measured data was obtained. Correspondingly, all predicted data assume equilibrium was obtained.

## 4.0 CONCLUSIONS

Research over the past decade has studied the adsorption of plutonium and uranium onto MST in alkaline solutions. Tests showed that MST would remove the targeted radionuclides from simulated alkaline waste. Testing indicated that Pu removal kinetics and Np capacity of the MST material impacts the size of equipment and waste blending plans for the SWPF. Additionally, calculations suggested the baseline MST process (MST concentration of 0.4 g/L) may not achieve the desired decontamination in wastes containing elevated concentrations of Pu and Np. In this task, the authors investigated the performance of non-baseline process parameters and their effectiveness for treating waste feed in the Salt Waste Processing Facility. The work investigated the effect of increased MST addition (up to 1.2 g/L) and the benefit of extra filtration steps with multiple additions of MST to salt waste containing actinides and strontium. Both simulant and actual waste testing were performed. Actual waste tests utilized a Tank 39H composite waste solution. In addition, testing to determine desorption of actinides from residual MST was conducted. The release of sorbed

Sr and actinides from loaded MST during the washing stages in the Salt Waste Processing Facility is an unresolved process behavior. Desorption tests were performed to assess this potential problem using loaded MST from the residue of the MST adsorption tests.

Programmatic conclusions drawn from this task follow.

- MST adsorption of Sr and actinides is minimally influenced by multi-strikes (alone) within the 24 h process cycle time.
- Use of intermediate filtration in conjunction with multiple MST strikes improves removal of Pu and Sr. The low starting concentration of Np does not permit determining the influence of intermediate filtration on Np removal.
- The use of intermediate filtration in conjunction with multiple MST strikes is ineffective for increasing U and Am removal.
- The solubility of Am falls well below Waste Characterization System estimates.
- Desorption of Sr and Pu during 24 h of solids washing does not pose a threat to process limits.

## **5.0 FUTURE WORK**

Testing did not identify problematic areas requiring further investigation. However, the authors do recommend further desorption tests to explore desorption from MST well after the 24 h process time. Furthermore, testing showed that the use of AMP may have influenced the removal of <sup>241</sup>Am (possibly caused by a filtration effect). The authors recommend follow-up testing to investigate this observation if AMP is to be continued for use in testing requiring <sup>241</sup>Am analysis. Lastly, attempts to analyze non-radioactive Sr in the tests have proven the current method to be susceptible to higher than acceptable detection limits.

## **6.0 ACKNOWLEDGEMENTS**

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## 7.0 APPENDIX

Table 7-1 Simulant Adsorption Test Data – Concentration Units of nCi/g

SAMPLE	TIME (h)	Concentration (nCi/g)							
		<sup>85</sup> Sr	±	Pu	±	<sup>237</sup> Np	±	<sup>241</sup> Am	±
Test A	0	74.9	1.4	12.6	0.3	0.312	0.047	2.09	0.09
Test A	6.1	1.02	0.029	1.80	0.06	0.121	0.018	0.612	0.024
Test A	12.0	0.856	0.033	1.43	0.06	0.0926	0.0139	0.457	0.025
Test A	24.0	0.748	0.024	1.00	0.05	0.0579	0.0087	0.288	0.015
Test A	48.0	0.735	0.029	0.756	0.052	0.0458	0.0069	0.215	0.015
Test A	168	0.590	0.015	0.428	0.057	0.0302	0.0045	0.147	0.005
Test B	0.0	75.2	1.4	12.8	0.3	0.324	0.049	2.00	0.08
Test B	6.1	0.376	0.016	0.859	0.050	0.0513	0.0077	0.520	0.021
Test B	12.0	0.324	0.019	0.646	0.046	0.0402	0.0060	0.406	0.020
Test B	24.0	0.307	0.014	0.503	0.036	0.0290	0.0043	0.247	0.012
Test B	48.0	0.241	0.017	0.353	0.025	0.0171	0.0026	0.176	0.013
Test B	168	0.267	0.007	0.253	0.041	0.0107	0.0016	0.109	0.005
Test C1	0.0	74.2	1.4	12.4	0.3	0.339	0.051	2.09	0.09
Test C1	6.2	0.202	0.012	0.630	0.039	0.0431	0.0065	0.480	0.020
Test C1	12.1	0.191	0.014	0.523	0.038	0.0254	0.0038	0.338	0.020
Test C1	24.0	0.185	0.012	0.285	0.038	0.0128	0.0019	0.248	0.012
Test C1	48.0	0.174	0.014	0.379	0.043	0.0083	0.0012	0.155	0.012
Test C1	168	0.157	0.006	0.299	0.064	< 0.0046	mda	0.108	0.004
Test C2	0.0	76.4	1.5	12.5	0.3	0.329	0.049	2.17	0.09
Test C2	6.2	0.230	0.012	0.565	0.034	0.0479	0.0072	0.473	0.019
Test C2	12.2	0.203	0.015	0.418	0.033	0.0208	0.0031	0.348	0.019
Test C2	24.0	0.181	0.012	0.279	0.055	0.0133	0.0020	0.221	0.012
Test C2	48.1	0.200	0.015	0.270	0.022	0.0103	0.0015	0.163	0.012
Test C2	168	0.152	0.006	0.191	0.042	< 0.0046	mda	0.097	0.004
Test D	0.0	76.3	1.5	12.9	0.3	0.330	0.050	2.19	0.08
Test D	6.2	0.974	0.029	1.87	0.08	0.128	0.019	0.630	0.024
Test D	12.1	0.341	0.019	0.651	0.045	0.0435	0.0065	0.404	0.020
Test D	24.0	0.164	0.010	0.300	0.072	0.0160	0.0024	0.239	0.012
Test D	35.9	0.167	0.015	0.257	0.024	0.0089	0.0013	0.185	0.014
Test D	48.1	0.183	0.013	0.231	0.030	0.0077	0.0012	0.198	0.018
Test D	168	0.148	0.007	0.174	0.027	< 0.0046	mda	0.098	0.004
Test E1	0.0	75.0	1.4	12.5	0.3	0.322	0.048	2.14	0.09
Test E1	6.3	0.967	0.028	1.90	0.06	0.113	0.017	0.629	0.024
Test E1	12.3	< 0.009	mda	0.208	0.014	0.0247	0.0037	0.422	0.012
Test E1	24.0	< 0.009	mda	0.028	0.004	< 0.0060	mda	0.284	0.009
Test E1	35.9	< 0.008	mda	0.019	0.003	< 0.0060	mda	0.243	0.008
Test E1	48.1	< 0.008	mda	0.024	0.005	< 0.0046	mda	0.198	0.007
Test E1	168	< 0.009	mda	0.012	0.002	< 0.0046	mda	0.111	0.004
Test E2	0.0	75.3	1.4	12.5	0.3	0.332	0.050	2.00	0.10
Test E2	6.3	0.956	0.028	1.75	0.06	0.124	0.019	0.596	0.025
Test E2	12.3	< 0.009	mda	0.185	0.011	0.0246	0.0037	0.428	0.013
Test E2	24.1	< 0.008	mda	0.032	0.004	< 0.0060	mda	0.260	0.008
Test E2	35.9	< 0.008	mda	0.040	0.005	< 0.0060	mda	0.216	0.007
Test E2	48.1	< 0.009	mda	0.013	0.002	< 0.0046	mda	0.185	0.006
Test E2	168	< 0.009	mda	0.011	0.002	< 0.0046	mda	0.109	0.004
Test F	0.0	75.3	1.4	12.4	0.3	0.307	0.046	2.19	0.09
Test F	6.3	77.2	1.5	12.7	0.4	0.304	0.046	2.02	0.08
Test F	12.3	76.6	1.5	13.1	0.3	0.275	0.041	2.02	0.08
Test F	24.1	74.5	1.4	12.1	0.3	0.229	0.034	1.73	0.08
Test F	36.0	73.4	1.4	12.1	0.3	0.264	0.040	1.56	0.08
Test F	48.1	74.9	1.4	12.7	0.4	0.292	0.044	1.43	0.07
Test F	168	76.5	1.4	12.5	0.3	0.284	0.043	1.18	0.04

**Table 7-2 Simulant Adsorption Test Data – Concentration Units of µg/L**

SAMPLE	TIME (h)	Concentration (µg/L)									
		<sup>85</sup> Sr ±		Pu ±		<sup>237</sup> Np ±		U ±		<sup>241</sup> Am ±	
Test A	0	3.89E-03	7.4E-05	212	5	540	81	11400	1720	0.745	0.034
Test A	6.1	5.31E-05	1.5E-06	30.0	0.9	210	32	9360	1400	0.218	0.009
Test A	12.0	4.44E-05	1.7E-06	23.6	0.9	160	24	8220	1230	0.163	0.009
Test A	24.0	3.88E-05	1.3E-06	16.8	0.7	100	15	7000	1050	0.103	0.005
Test A	48.0	3.81E-05	1.5E-06	12.6	0.8	79.4	11.9	7200	1080	0.0767	0.0052
Test A	168	3.06E-05	7.6E-07	7.1	0.9	52.4	7.9	6240	936	0.0525	0.0019
Test B	0.0	3.90E-03	7.4E-05	214	6	562	84	11600	1750	0.714	0.029
Test B	6.1	1.95E-05	8.3E-07	14.2	0.7	89.0	13.3	6270	940	0.185	0.007
Test B	12.0	1.68E-05	1.0E-06	10.3	0.7	69.6	10.4	6370	955	0.145	0.007
Test B	24.0	1.59E-05	7.5E-07	7.9	0.5	50.2	7.5	5370	806	0.0881	0.0044
Test B	48.0	1.25E-05	9.0E-07	6.0	0.3	29.6	4.4	4670	700	0.0628	0.0045
Test B	168	1.38E-05	3.9E-07	4.1	0.6	18.6	2.8	4050	607	0.0388	0.0017
Test C1	0.0	3.85E-03	7.3E-05	207	5	588	88	12100	1820	0.745	0.034
Test C1	6.2	1.05E-05	6.3E-07	10.2	0.5	74.7	11.2	5660	848	0.171	0.007
Test C1	12.1	9.89E-06	7.3E-07	8.7	0.6	44.1	6.6	4880	732	0.120	0.007
Test C1	24.0	9.58E-06	6.4E-07	4.8	0.6	22.3	3.3	3930	589	0.0884	0.0044
Test C1	48.0	9.01E-06	7.3E-07	6.3	0.7	14.4	2.2	3350	502	0.0551	0.0042
Test C1	168	8.15E-06	3.1E-07	4.8	0.9	8.0	1.2	1630	244	0.0386	0.0015
Test C2	0.0	3.96E-03	7.6E-05	209	5	570	86	11700	1760	0.772	0.033
Test C2	6.2	1.19E-05	6.4E-07	9.2	0.5	83.0	12.5	6110	916	0.168	0.007
Test C2	12.2	1.05E-05	7.6E-07	7.0	0.5	36.1	5.4	4880	732	0.124	0.007
Test C2	24.0	9.37E-06	6.1E-07	4.6	0.9	23.0	3.5	3750	563	0.0788	0.0041
Test C2	48.1	1.04E-05	7.8E-07	4.4	0.3	17.8	2.7	2740	411	0.0579	0.0044
Test C2	168	7.88E-06	3.1E-07	3.1	0.7	8.0	1.2	3740	562	0.0346	0.0016
Test D	0.0	3.96E-03	7.6E-05	216	6	572	86	11600	1730	0.781	0.030
Test D	6.2	5.05E-05	1.5E-06	30.9	1.2	222	33	9490	1420	0.224	0.009
Test D	12.1	1.77E-05	9.9E-07	10.9	0.7	75.5	11.3	6420	964	0.144	0.007
Test D	24.0	8.48E-06	5.3E-07	4.9	1.2	27.8	4.2	4100	616	0.0851	0.0044
Test D	35.9	8.67E-06	7.8E-07	4.4	0.4	15.5	2.3	3720	559	0.0660	0.0049
Test D	48.1	9.48E-06	6.8E-07	3.9	0.5	13.4	2.0	3340	500	0.0704	0.0064
Test D	168	7.65E-06	3.4E-07	2.9	0.4	8.0	1.2	2320	348	0.0349	0.0014
Test E1	0.0	3.89E-03	7.4E-05	209	5	558	84	11500	1730	0.763	0.031
Test E1	6.3	5.02E-05	1.5E-06	31.0	1.0	196	29	9480	1420	0.224	0.009
Test E1	12.3	< 4.5E-07	mda	3.5	0.2	42.8	6.4	6940	1040	0.151	0.004
Test E1	24.0	< 4.4E-07	mda	0.45	0.06	10.4	1.6	4570	686	0.101	0.003
Test E1	35.9	< 4.4E-07	mda	0.31	0.05	10.4	1.6	4860	729	0.0864	0.0028
Test E1	48.1	< 4.2E-07	mda	0.40	0.08	8.0	1.2	4040	607	0.0704	0.0025
Test E1	168	< 4.9E-07	mda	0.19	0.03	8.0	1.2	2100	315	0.0395	0.0015
Test E2	0.0	3.91E-03	7.5E-05	209	6	576	86	12000	1800	0.714	0.035
Test E2	6.3	4.96E-05	1.5E-06	29.1	1.0	215	32	9600	1440	0.213	0.009
Test E2	12.3	< 4.6E-07	mda	3.1	0.2	42.6	6.4	5610	842	0.152	0.005
Test E2	24.1	< 4.2E-07	mda	0.48	0.05	10.4	1.6	4640	696	0.0925	0.0030
Test E2	35.9	< 4.4E-07	mda	0.37	0.04	10.4	1.6	4270	641	0.0770	0.0023
Test E2	48.1	< 4.5E-07	mda	0.20	0.02	8.0	1.2	4310	647	0.0659	0.0022
Test E2	168	< 4.8E-07	mda	0.17	0.04	8.0	1.2	3710	556	0.0389	0.0015
Test F	0.0	3.90E-03	7.5E-05	208	6	532	80	11200	1680	0.782	0.033
Test F	6.3	4.00E-03	7.6E-05	213	6	528	79	11700	1750	0.721	0.030
Test F	12.3	3.97E-03	7.6E-05	220	6	476	71	10200	1540	0.719	0.030
Test F	24.1	3.86E-03	7.3E-05	204	6	397	60	8410	1260	0.617	0.027
Test F	36.0	3.80E-03	7.3E-05	203	5	458	69	9700	1460	0.557	0.028
Test F	48.1	3.88E-03	7.5E-05	213	6	507	76	10300	1540	0.509	0.025
Test F	168	3.97E-03	7.5E-05	211	5	492	74	10700	1600	0.422	0.015

Table 7-3 Actual Waste Adsorption Test Data – Concentration Units of nCi/g

SAMPLE	TIME (h)	Concentration (nCi/g)											
		<sup>90</sup> Sr	±	<sup>238</sup> Pu	±	<sup>239/240</sup> Pu	±	Total Pu	±	<sup>237</sup> Np	±	<sup>241</sup> Am	±
AW-Test A	0.0	79.2	7.9	479	20	24.7	1.3	504	21	0.0770	0.0192	< 0.034	mda
AW-Test A	6.1	1.49	0.16	103	3	5.3	0.2	108	3	0.0368	0.0092	< 0.030	mda
AW-Test A	12.0	1.56	0.17	87.0	2.4	4.5	0.2	91.5	2.6	< 0.032	mda	< 0.039	mda
AW-Test A	24.0	1.43	0.17	64.9	1.8	3.1	0.1	68.0	1.9	< 0.027	mda	< 0.029	mda
AW-Test A	168	1.47	0.14	18.8	0.5	1.0	0.0	19.8	0.5	< 0.036	mda	< 0.036	mda
AW-Test B	0.0	83.5	8.4	503	17	26.7	1.0	530	18	0.0753	0.0188	< 0.030	mda
AW-Test B	6.0	1.60	0.18	50.5	1.3	2.5	0.1	53.0	1.4	0.0198	0.0049	< 0.036	mda
AW-Test B	11.9	0.92	0.11	42.3	1.1	2.2	0.1	44.5	1.2	< 0.032	mda	< 0.035	mda
AW-Test B	24.0	0.86	0.11	33.0	0.9	1.8	0.1	34.8	1.0	< 0.026	mda	< 0.028	mda
AW-Test B	168	1.01	0.10	12.4	0.3	0.7	0.0	13.0	0.4	< 0.034	mda	< 0.035	mda
AW-Test E	0.0	88.0	8.8	480	15	24.8	0.9	505	16	0.0772	0.0193	< 0.035	mda
AW-Test E	6.2	1.68	0.21	103	3	5.3	0.2	108	3	0.0361	0.0090	< 0.032	mda
AW-Test E	11.9	0.39	0.09	26.6	0.7	1.3	0.1	27.9	0.8	< 0.032	mda	< 0.032	mda
AW-Test E	24.0	0.34	0.08	4.0	0.1	0.2	0.0	4.2	0.1	< 0.027	mda	< 0.028	mda
AW-Test E	168	0.37	0.05	1.9	0.1	0.1	0.0	2.0	0.1	< 0.036	mda	< 0.050	mda
AW-Test G	0.0	72.5	7.3	399	12	21	1	419	13	0.0789	0.0197	< 0.032	mda
AW-Test G	5.9	102	10	485	13	25	1	509	14	0.0767	0.0192	< 0.032	mda
AW-Test G	11.9	126	10	472	15	24	1	496	16	< 0.033	mda	< 0.042	mda
AW-Test G	24.0	91.0	9.1	481	15	24	1	505	16	0.0963	0.0241	< 0.032	mda
AW-Test G	168	91.2	7.4	494	14	25	1	520	15	0.0677	0.0169	< 0.044	mda
AW-Test H	0.0	111	11	470	20	25.7	1.3	496	21	0.0750	0.0188	< 0.054	mda
AW-Test H	6.3	5.27	0.54	181	5	9.2	0.3	190	5	0.0469	0.0117	< 0.029	mda
AW-Test H	11.9	0.17	0.06	68.1	1.8	3.5	0.1	71.6	1.9	< 0.032	mda	< 0.030	mda
AW-Test H	24.1	0.17	0.05	16.0	0.4	0.8	0.1	16.8	0.5	< 0.029	mda	< 0.035	mda
AW-Test H	168	0.18	0.03	6.0	0.2	0.4	0.0	6.3	0.2	< 0.035	mda	< 0.030	mda
Blank	0.0	0.15	0.05	0.28	0.02	< 0.17	mda	< 0.46	mda	< 0.003	mda	< 0.028	mda
Blank	5.9	0.77	0.11	0.30	0.03	< 0.34	mda	< 0.64	mda	< 0.003	mda	< 0.027	mda
Blank	12.0	0.20	0.05	< 0.02	mda	< 0.05	mda	< 0.07	mda	< 0.032	mda	< 0.033	mda
Blank	24.1	0.31	0.07	0.56	0.04	< 0.29	mda	< 0.84	mda	< 0.026	mda	< 0.044	mda
Blank	168	0.24	0.05	< 0.02	mda	0.02	0.01	< 0.03	mda	< 0.034	mda	< 0.028	mda

mda = minimum detectable analysis

Table 7-4 Actual Waste Adsorption Test Data – Concentration Units of µg/L

SAMPLE	TIME (h)	Concentration (µg/L)													
		<sup>90</sup> Sr	±	<sup>238</sup> Pu	±	<sup>239/240</sup> Pu	±	Total Pu	±	<sup>237</sup> Np	±	U	±	<sup>241</sup> Am	±
AW-Test A	0.0	0.721	0.072	35.0	1.4	380	20	415	21	136	34	4690	703	< 0.012	mda
AW-Test A	6.1	0.0136	0.0014	7.49	0.19	81.5	2.5	89.0	2.7	65.1	16.3	3860	579	< 0.011	mda
AW-Test A	12.0	0.0142	0.0015	6.35	0.17	68.8	3.7	75.2	3.9	< 56	mda	3470	520	< 0.014	mda
AW-Test A	24.0	0.0130	0.0016	4.73	0.13	48.4	2.1	53.1	2.2	< 47	mda	3510	527	< 0.011	mda
AW-Test A	168	0.0134	0.0013	1.37	0.04	15.4	0.7	16.8	0.8	< 64	mda	2970	446	< 0.013	mda
AW-Test B	0.0	0.761	0.076	36.7	1.3	411	16	447	17	133	33	4660	699	< 0.011	mda
AW-Test B	6.0	0.0146	0.0017	3.68	0.09	39.2	1.5	42.8	1.6	35.0	8.8	3080	461	< 0.013	mda
AW-Test B	11.9	0.0084	0.0010	3.09	0.08	33.2	1.6	36.3	1.7	< 57	mda	2440	366	< 0.013	mda
AW-Test B	24.0	0.0078	0.0010	2.41	0.06	28.1	1.3	30.5	1.3	< 46	mda	2430	365	< 0.010	mda
AW-Test B	168	0.0092	0.0009	0.90	0.02	10.2	0.5	11.1	0.5	< 60	mda	1630	245	< 0.013	mda
AW-Test E	0.0	0.802	0.080	35.0	1.1	381	14	417	15	137	34	4780	717	< 0.013	mda
AW-Test E	6.2	0.0153	0.0019	7.50	0.20	82.1	2.4	89.6	2.6	64.0	16.0	3680	552	< 0.012	mda
AW-Test E	11.9	0.0035	0.0008	1.94	0.05	20.4	1.2	22.3	1.2	< 56	mda	2830	424	< 0.012	mda
AW-Test E	24.0	0.0031	0.0007	0.29	0.01	3.23	0.39	3.52	0.40	< 48	mda	2140	321	< 0.010	mda
AW-Test E	168	0.0034	0.0005	0.14	0.01	1.17	0.23	1.30	0.24	< 64	mda	1640	247	< 0.018	mda
AW-Test G	0.0	0.661	0.066	29.1	0.9	316	11	345	12	140	35	4670	700	< 0.012	mda
AW-Test G	5.9	0.934	0.090	35.4	1.0	379	11	414	12	136	34	4700	704	< 0.012	mda
AW-Test G	11.9	1.15	0.09	34.4	1.1	372	13	407	14	< 58	mda	1260	189	< 0.015	mda
AW-Test G	24.0	0.829	0.083	35.1	1.1	366	14	401	15	171	43	4820	723	< 0.012	mda
AW-Test G	168	0.831	0.067	36.1	1.0	392	12	428	13	120	30	4550	683	< 0.016	mda
AW-Test H	0.0	1.01	0.10	34.3	1.4	395	20	430	21	133	33	4660	699	< 0.020	mda
AW-Test H	6.3	0.0480	0.0049	13.2	0.3	141	4	154	4	83.1	20.8	4060	610	< 0.010	mda
AW-Test H	11.9	0.0015	0.0005	4.97	0.13	53.4	2.0	58.4	2.1	< 57	mda	3540	531	< 0.011	mda
AW-Test H	24.1	0.0015	0.0005	1.17	0.03	12.0	0.8	13.2	0.9	< 51	mda	3230	485	< 0.013	mda
AW-Test H	168	0.0017	0.0003	0.43	0.01	5.88	0.41	6.3	0.4	< 61	mda	2550	382	< 0.011	mda
Blank	0.0	0.0013	0.0004	0.0207	0.0017	< 2.69	mda	< 2.71	DL	< 4.7	mda	< 19	mda	< 0.010	mda
Blank	5.9	0.0070	0.0010	0.0218	0.0020	< 5.20	mda	< 5.22	DL	< 4.6	mda	< 22	mda	< 0.010	mda
Blank	12.0	0.0018	0.0005	< 0.001	mda	< 0.78	mda	< 0.78	DL	< 56	mda	< 224	mda	< 0.012	mda
Blank	24.1	0.0028	0.0006	0.0406	0.0026	< 4.44	mda	< 4.48	DL	< 46	mda	< 186	mda	< 0.016	mda
Blank	168	0.0022	0.0004	< 0.001	mda	0.24	0.08	< 0.24	DL	< 60	mda	< 240	mda	< 0.010	mda

mda = minimum detectable analysis



## 8.0 REFERENCES

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- <sup>3</sup> Barnes, M. J., Hobbs, D. T., Peters, T. B., Stallings, M. E., and Fink, S. D. *Task Technical and Quality Assurance Plan for Waste Characterization Support*, WSRC-RP-2003-00403, Rev. 1, Savannah River Site, Aiken, SC 29808 (November 18, 2003).
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- <sup>5</sup> F. F. Fondeur, D. T. Hobbs, M. J. Barnes and S. D. Fink, *Sorption Modeling of Sr, Pu, U and Np adsorption on Monosodium Titanate*, WSRC-TR-2003-00180, Savannah River Site, Aiken, SC 29808 (May 2003).