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Determination of Actinide Loadings onto Monosodium Titanate (MST) under Conditions Relevant to the Actinide Removal Process Facility

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Abstract: Researchers at the Savannah River Site (SRS) have studied adsorption of uranium, plutonium, and neptunium onto monosodium titanate (MST) at conditions relevant to operation of the Actinide Removal Process (ARP) facility. This study measured actinide loadings at a large phase ratio of simulated wastes solution to mass of MST. The large phase ratio was designed to mimic the maximum phase ratio that would occur during a single process cycle of the ARP facility. Uranium and plutonium loadings measured in this study proved much higher than previous measurements at lower phase ratios.

Keywords: Actinide strontium removal, MST

INTRODUCTION

The Actinide Removal Process (ARP) removes radioactive strontium and alpha activity from high-level waste solutions at the Savannah River Site (SRS). High-level waste is transferred into a batch reactor and contacted with MST. After 24 hours of contact, the suspension is filtered to separate the solids and the decontaminated solution. The decontaminated waste solution is transferred on to either the Modular Caustic-Side Solvent Extraction Unit (CSSX) or the Saltstone Disposal Facility. The MST solids remain in the batch reactor. This operation is repeated until sufficient solids are

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accumulated for transfer into the Defense Waste Processing Facility (DWPF). Current operational planning indicates that as many as 17 batch contacts will occur in a single process cycle.

Testing in support of the down selection of technology for SRS waste pretreatment technology measured uranium loadings onto MST well above the maximum value determined under conditions relevant to the now-abandoned In-Tank Precipitation (ITP) process (1). The operational strategy of up to 17 batch contacts provides an opportunity for much higher actinide loadings onto the MST in the ARP facility compared to the ITP facility. Since waste solutions at SRS contains both enriched uranium and weapons-grade plutonium, determination of loadings of uranium, plutonium, and neptunium under conditions relevant to the ARP facility is needed to evaluate nuclear criticality safety (2, 3).

EXPERIMENTAL

Reagents

Plutonium (principally ^{239}Pu and ^{240}Pu) and neptunium were provided by onsite stocks produced by reprocessing of nuclear materials at SRS. Uranium was purchased as depleted uranyl nitrate hexahydrate from Noah Chemical. ^{85}Sr was supplied by Perkin Elmer Life Science Products (Catalog #NEZ-082). All other reagents used to prepare the simulated waste solutions were purchased as reagent-grade chemicals and used without further purification.

Analytical Measurements

Determination of plutonium content occurred by first separating the plutonium from uranium and neptunium, followed by alpha spectrometry analyses of the separated plutonium. Plutonium separation occurred by extracting the acidified sample with thenoyl-triflouroacetone (TTA). Each extraction was traced with a ^{236}Pu tracer to quantify plutonium recoveries. The Pu-TTA extractant was flame mounted on stainless steel planchets. The alpha planchets were analyzed on one of two systems:

i. a multiplexed array of Canberra Industries quad-alpha spectrometers with 16 passivated implanted planar silicon (PIPS) detectors, interfaced with a Canberra Industries Genie-2K PC based multichannel analyzer, or
ii. a Canberra Alpha Analyst alpha spectrometry system with 24 PIPS detectors.

The ^{85}Sr was analyzed by gamma spectrometry. The gamma spectrometry system was based on an Advanced Measurement Technology, Inc.'s 50% relative efficiency N-Type high purity germanium GMX detector. The detector was enclosed in a Changer Lab's lead shielded, automated robotic sample changer and was interfaced to a PC-based Canberra Industries Genie 2K multichannel analyzer system.

Uranium and neptunium were measured by inductively coupled plasma mass spectrometry (ICP-MS) using a VG Elemental Plasma Quad 2 Inductively Coupled Plasma Mass Spectrometer.

Radioactive Solution Preparation and Equilibrium

The tests used a simulated waste solution developed at SRNL for testing MST performance in support of salt processing at the Savannah River Site (4–6). Table 1 provides the target chemical and radiochemical composition of the simulant. The quantities of the actinide components were selected to maximize the loading of actinides onto the MST. The selected target actinide concentrations are considered reasonable approximations of the maximum soluble concentrations anticipated for ARP operations based on feed stream predictions.

A high concentration of strontium could potentially reduce the loading of the actinide elements onto the MST. Thus, we minimized the concentration of stable strontium in the simulant to reduce the potential for loading strontium onto the MST. We prepared the simulant using reagent grade chemicals

Table 1. Target composition of simulated waste solution

Component	Target concentration	Measured concentration
$NaNO_3$	2.60 *M*	2.59 *M*
NaOH	1.33 *M*	1.34 *M*
Na_2SO_4	0.521 *M*	0.508 *M*
$NaAl(OH)_4$	0.429 *M*	0.381 *M*
$NaNO_2$	0.134 *M*	0.133 *M*
Na_2CO_3	0.0260 *M*	0.0198 *M*
Total Na^+	5.60 *M*	5.13 *M*
Cold strontium	~100 μg/L[a]	NM
^{85}Sr	50,000 dpm/mL	30,400 dpm/mL
^{237}Np	500 μg/L	477 ug/L
^{238}U	25,000 μg/L	26,500 ug/L
$^{239/40}$Pu	1200 μg/L	885 ug/L

NM = not measured

[a] We did not deliberately add stable strontium. In such cases $\leq$100 μg/L of stable strontium typically enters the simulant as impurities from the chemical reagents, based on measurements of prior simulants prepared in this manner.

and deionized distilled water (DDI) in accordance with the established procedure (7).

To satisfy all the goals of this study, the researchers followed a series of steps detailed below. Simulant solution was prepared and verified to contain the proper quantities of ^{85}Sr, ^{238}U, and $^{239/240}$Pu. During a two-week equilibration period, the solution was sampled for supernatant radioisotope concentrations. At the end of two weeks, it appeared that equilibration was complete and the simulant was deemed ready for use. The simulant solution was split into three experimental bottles (Bottles #1, #2, #3) and one control bottle. Each bottle contained 8.5 L of the simulant solution.

0.2 g of MST solids (Optima Chemicals, Inc., Batch #00-QAB-417) was added to each bottle (0.0235 g MST/L) to each bottle and allowed contact for seven days. The solutions in the bottles were agitated using a magnetic stirrer. During the seven-day period, technicians sampled the supernatant in the experiment and control bottles at 4, 6, 8, 24, 96, 168, and 336 or 384 hours (depending on which bottle). At the completion of testing, the MST was filtered from the solutions using a removable 0.45-μm nylon filter and the MST was retained for analyses.

The three bottles used in the experiments were replicates of each other. Control samples (simulant solution without MST solids) were pulled at the same time of the experimental samples, except for the 336- or 384-hour samples. All experiments were performed at ambient laboratory temperature and pressure. The temperature was monitored at least once per day and ranged from 18.8 to 21.8°C over the testing period.

Sampling Methodology

For each solution sample, we removed a sub-surface aliquot from the test bottle. We filtered through a 0.1-μm polyvinylidene difluoride (PVDF) syringe filter to remove any solids and acidified a measured volume of the filtrate with an equal volume of 5.0 M HNO_3. We inspected the acidified samples after allowing them to stand for a minimum of 2 hours for evidence of solids. The presence of solids could introduce an error into the determination of fissile concentrations. All acidified samples were found to be clear with no evidence of any solids.

At the conclusion of the experiment we recovered the MST solids by filtration. We attempted to dissolve the recovered solids in a 1:1 mixture of concentrated sulfuric acid and water. Previous testing found that MST solids loaded with plutonium and uranium readily dissolved in this acidic solution (8). We observed that the solids from Bottle #3 readily dissolved in the sulfuric acid solution. However, the solids recovered from Bottles #1 and #2 did not completely dissolve.

Additions of HNO_3, H_2O_2, and NaF also failed to completely dissolve these solids. Thus, we filtered the dissolution suspensions and recovered the undissolved solids and the filtrate from this step. The filtrate was recovered, diluted to a known volume, and submitted to determine titanium and actinide content. We recovered the undissolved solids and performed a sodium peroxide fusion to convert the solids to a form that would dissolve in acid. The peroxide fusion proved successful and we determined the titanium and actinide content of these solids as well.

RESULTS

The chemical and radiochemical composition of the simulant met the target concentrations for all components except plutonium. The plutonium concentration measured 885 μg/L compared to the target of 1200 μg/L. This result is not unexpected, as a value of 885 μg/L falls within the confidence interval for plutonium solubility for a solution having the chemical composition as listed in Table 1.

The plutonium concentration in this simulant is approximately a factor of 4 higher than that used in previous simulant testing (4–6) and a factor of 2 higher than that in actual waste testing (9). At the conclusion of the experiment, the solution contained approximately 120 μg/L of plutonium. This indicates that the system contained sufficient total mass of plutonium for achieving high mass loadings onto the MST solids. Note that the solution concentration of plutonium was continuing to decrease with the final sampling time (ca. 2 weeks). This indicates that the system may not have reached equilibrium at the time we concluded the experiment.

At the end of the experiment, the loaded MST solids were recovered by filtration. MST solids recovery ranged from 33.4% to 78.1%, as measured by titanium content upon dissolution of the recovered solids. Solids losses likely reflect retention of the small quantities of solids (44–130 mg MST) within the large 10-liter carboys used for the tests.

Recovered solids from two of the tests contained a small amount of solids that did not dissolve in sulfuric acid even upon addition of additional oxidizing and complexing agents, hydrogen peroxide and fluoride, respectively. We believe that these solids were aluminosilicates formed from the aluminum in the simulant and silicates leached from the filtering glassware. We dissolved these solids using a peroxide fusion technique developed by the Analytical Development Section of SRNL. Analysis of the solutions produced by this dissolution technique revealed that these residual solids showed negligible amounts of actinides or strontium.

Plutonium Results

Table 2 provides the solution concentrations of plutonium at each sampling time for the test and control bottles by two methods: Plutonium Thenoyl Trifluoroacetone (PuTTA) and ICP-MS. Figure 1 shows the averaged

Table 2. Plutonium concentrations

	$^{239/240}$Pu Values by PuTTA μg/L				$^{239/240}$Pu Values by ICP-MS μg/L			
Time (hours)	Bottle 1	Bottle 2	Bottle 3	Control	Bottle 1	Bottle 2	Bottle 3	Control
0[a]	882 ± 95				885 ± 177			
4	740 ± 41	727 ± 36	855 ± 53	731 ± 37	706 ± 141	717 ± 143	686 ± 137	798 ± 160
6	684 ± 40	692 ± 40	665 ± 35	789 ± 42	701 ± 140	685 ± 137	675 ± 135	788 ± 158
8	679 ± 34	678 ± 33	716 ± 35	732 ± 35	687 ± 137	671 ± 134	680 ± 136	788 ± 158
24	597 ± 28	637 ± 29	628 ± 31	852 ± 42	646 ± 129	637 ± 127	637 ± 127	802 ± 160
96	505 ± 26	470 ± 22	473 ± 25	858 ± 41	473 ± 95	499 ± 100	484 ± 97	786 ± 157
168	315 ± 16	351 ± 19	32 ± 19	856 ± 50	330 ± 66	349 ± 70	313 ± 63	792 ± 158
336	121 ± 6	NA	NA	NA	129[b] ± 26	NA	NA	NA
384	NA	113 ± 6	80.1 ± 3.7	NA	NA	137[b] ± 27	141[b] ± 28	NA

NA = sample not pulled.

[a] Time 0 is before MST addition and is the average of four samples taken at various times before the test.

[b] The time = 338 and 384 samples report and use only ^{239}Pu values.

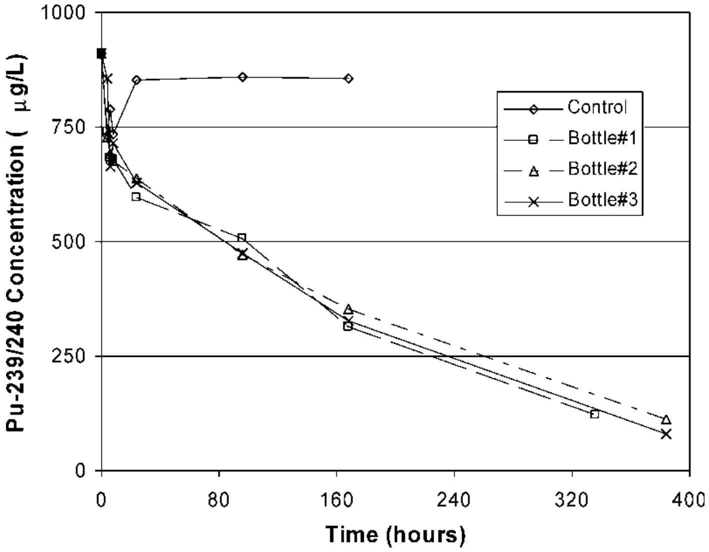

Figure 1. Plutonium concentration versus time.

(PuTTa and ICP-MS) data. The results show a high degree of precision among the three replicates and the analytical methods.

Due to the large liquid:MST ratio, the rate of removal and final DF values is less than under lower phase ratio and lower initial sorbate concentrations. After approximately two weeks of contact, the average final DF measured 7.65. Caution should be exercised when using the short-term (4-hour) data. The short contact time data is difficult to distinguish from the control when evaluated with the analytical uncertainty.

Uranium Results

Table 3 provides the solution concentrations of uranium at each sampling time for the test and control bottles as determined by the ICP-MS method. Figure 2 is the graphical representation of the data. As with plutonium, the uranium results show a high degree of precision among all three replicates. Prior to 96 hours of contact, we cannot conclusively determine the degree of uranium removal. At or after 96 hours contact, the solution data indicate increasing uranium removal with increasing contact time.

Neptunium Results

Table 4 provides the solution concentrations of neptunium at each sampling time for the test and control bottles as determined by the ICP-MS method. Figure 3 is

Table 3. Uranium concentrations

Time (hours)	$^{235/238}$Uranium Values by ICP-MS μg/L			
	Bottle 1	Bottle 2	Bottle 3	Control
0[a]	$(2.65 \pm 0.53)10^4$			
4	$(2.49 \pm 0.50)10^4$	$(2.49 \pm 0.50)10^4$	$(2.45 \pm 0.49)10^4$	$(2.49 \pm 0.50)10^4$
6	$(2.51 \pm 0.50)10^4$	$(2.43 \pm 0.49)10^4$	$(2.43 \pm 0.49)10^4$	$(2.47 \pm 0.49)10^4$
8	$(2.49 \pm 0.40)10^4$	$(2.43 \pm 0.49)10^4$	$(2.45 \pm 0.49)10^4$	$(2.47 \pm 0.49)10^4$
24	$(2.57 \pm 0.51)10^4$	$(2.53 \pm 0.51)10^4$	$(2.51 \pm 0.50)10^4$	$(2.51 \pm 0.50)10^4$
96	$(2.47 \pm 0.49)10^4$	$(2.47 \pm 0.49)10^4$	$(2.43 \pm 0.49)10^4$	$(2.49 \pm 0.50)10^4$
168	$(2.37 \pm 0.47)10^4$	$(2.39 \pm 0.48)10^4$	$(2.37 \pm 0.47)10^4$	$(2.47 \pm 0.49)10^4$
336	$(2.21 \pm 0.44)10^4$	NA	NA	NA
384	NA	$(2.19 \pm 0.44)10^4$	$(2.15 \pm 0.43)10^4$	NA

NA = sample not pulled.

Numbers in parenthesis are single standard deviation of analytical method.

[a] Time 0 is pre-MST and is the average of four pre-MST values.

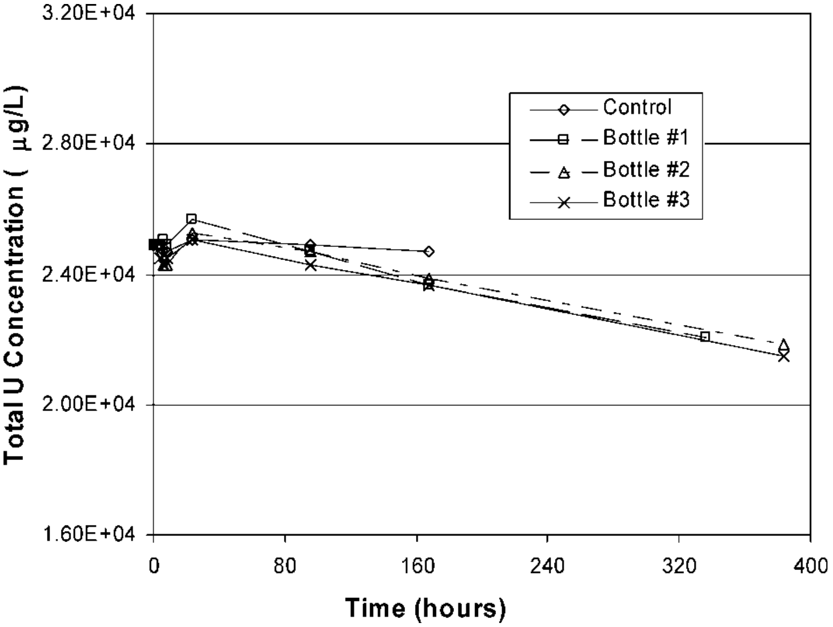

Figure 2. Uranium concentration versus time as measured by ICP-MS.

the graphical representation of the data. As with plutonium and uranium, the neptunium results show a high degree of precision among all three replicates. Prior to 96 hours of contact, we cannot conclusively determine the degree of neptunium removal. At or after 96 hours contact, the solution data indicate increasing neptunium removal with increasing contact time.

Table 4. ^{237}Neptunium concentration over time

	^{237}Neptunium Values by ICP-MS µg/L			
Time (hours)	Bottle 1	Bottle 2	Bottle 3	Control
0[a]	477 ± 95			
4	400 ± 80	412 ± 82	396 ± 79	426 ± 85
6	406 ± 81	392 ± 78	400 ± 80	418 ± 83
8	404 ± 81	390 ± 78	390 ± 78	420 ± 84
24	422 ± 84	416 ± 83	418 ± 84	434 ± 87
96	376 ± 75	378 ± 75	378 ± 76	432 ± 86
168	328 ± 66	340 ± 68	328 ± 66	426 ± 85
336	222 ± 44	NA	NA	NA
384	NA	228 ± 46	216 ± 43	NA

NA = sample not pulled.

[a] Time 0 is pre-MST and is the average of 4 pre-MST values.

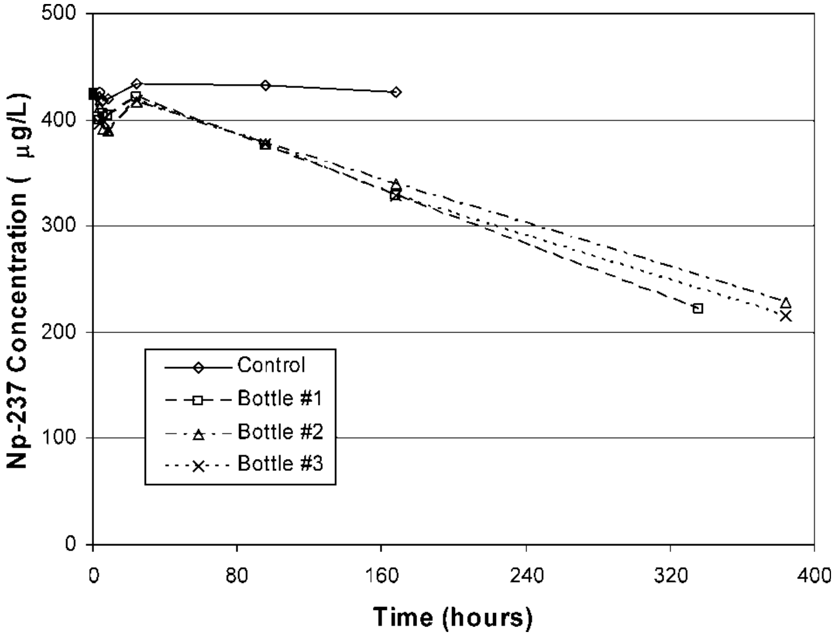

Figure 3. Neptunium concentration versus time as measured by ICP-MS.

Actinide Loading onto MST

Table 5 provides the measured loadings of plutonium, uranium, and neptunium onto MST for each test. Starred values at 336 or 384 hours of contact time are those measured from the recovered solids. All other values are those calculated based on the quantity of actinide removed from solution and the quantity of MST added to each test bottle. Loading values are provided on a µg Pu/g MST basis.

Mass Balance

One check of data consistency is whether the sum of the analytical results from the filtrate and solids matches the known amounts of actinide in solution before the addition of the MST. The actinide in the final filtrate sample and the actinide on the MST solids were compared to the actinide in solution before the addition of MST (Table 6). For the actinide in the filtrate before MST addition, the value is the average of the four samples. For the actinide on MST, the amount of plutonium captured on all the MST was corrected for the recovered quantity of MST. The percent mass balance term was derived by dividing the sum of the actinide in the filtrate and actinide on the MST by the actinide in the filtrate before MST addition. A second mass balance check can be calculated

Table 5. Actinide loading on MST

Sample time	Pu loading (μg Pu/g MST)		U Loading (μg Pu/g MST)	Np Loading (μg Pu/g MST)
	PuTTa	ICP-MS		
4	$(4.57 \pm 2.99)10^3$	$(7.93 \pm 0.67)10^3$	$(7.60 \pm 0.99)10^4$	$(3.14 \pm 0.35)10^3$
6	$(8.57 \pm 0.59)10^3$	$(8.62 \pm 0.56)10^3$	$(8.45 \pm 1.97)10^4$	$(3.28 \pm 0.30)10^3$
8	$(8.12 \pm 0.92)10^3$	$(8.94 \pm 0.35)10^3$	$(8.46 \pm 1.30)10^4$	$(3.48 \pm 0.34)10^3$
24	$(1.11 \pm 0.09)10^4$	$(1.06 \pm 0.02)10^4$	$(5.06 \pm 1.31)10^4$	$(2.46 \pm 0.13)10^3$
96	$(1.70 \pm 0.08)10^4$	$(1.72 \pm 0.06)10^4$	$(8.46 \pm 0.98)10^4$	$(4.21 \pm 0.05)10^3$
168	$(2.34 \pm 0.08)10^4$	$(2.38 \pm 0.08)10^4$	$(1.19 \pm 0.05)10^5$	$(6.14 \pm 0.29)10^3$
336/384	$(3.30 \pm 0.09)10^4$	$(3.21 \pm 0.03)10^4$	$(2.01 \pm 0.13)10^5$	$(1.08 \pm 0.03)10^4$
336/384[a]	$(3.82 \pm 0.52)10^4$	$(3.14 \pm 0.11)10^4$	$(1.32 \pm 0.25)10^5$	$(9.55 \pm 0.47)10^3$

numbers in parenthesis are single standard deviation of analytical method.

[a] Starred data is derived from the loaded MST solids analyses whereas the rest of the data is derived from the filtrate data.

Table 6. Actinide mass balance

Method	Plutonium mass balance	Uranium mass balance	Neptunium % mass balance
PuTTA	114 ± 12.7%	NA	NA
ICP-MS	98.2 ± 3.34%	93.8 ± 1.10%	93.8 ± 1.07%

by comparing the ~360 hour filtrate against the solids data. A good mass balance will have both values close to each other.

DISCUSSION OF RESULTS

The operational strategy for the ARP facility is considerably different than that planned for the Salt Waste Processing Facility (SWPF). Due to the small batch reactor size, multiple small batches must be conducted to accumulate sufficient MST solids for washing and transferring to the DWPF for disposal. Current plans include as many as seventeen batch contacts during a single process cycle. This has the effect of exposing the MST solids to multiple contacts with fresh waste solutions. Consequently, the loading of strontium and fissile elements could be higher than that in the SWPF, which will contact the MST in most cases with a single batch of waste solution and no more than two batches if additional sorbate removal is required. Given the potential for higher loadings of fissile isotopes of uranium and plutonium, we undertook a study to measure actinide loadings at conditions that would represent the highest phase ratio that any of the MST solids would experience during the 17-batch process cycle in ARP.

The operating plan for the ARP facility designates the addition of 0.4 g/L of fresh MST to each batch contact. However, since the previous batch of MST is retained in the reactor, the effective phase ratio decreases with each succeeding batch assuming fresh MST is added. Thus for the 17th contact, the effective phase ratio decreases to a value of 6.67 g/L assuming no losses of the MST solids.

To provide a conservative upper estimate of actinide loadings onto the MST over the 17-batch contact cycle we measured the loading at a phase

Table 7. Solution vs solids data

Method	Plutonium μg/g loading	Uranium μg/g loading	Neptunium μg/g loading
Solution	$(3.26 \pm 0.08)10^4$	$(2.01 \pm 0.13)10^5$	$(1.08 \pm 0.03)10^4$
Solids	$(3.48 \pm 0.50)10^4$	$(1.32 \pm 0.25)10^5$	$(9.55 \pm 0.47)10^3$

Numbers in parenthesis are single standard deviation of analytical method.

ratio of 0.0235 g/L MST. This represents the case in which a single strike of MST at 0.4 g/L MST is carried through the entire 17-contact process cycle without any further MST additions. In normal operations only 1/17th of the total quantity of MST solids present at the conclusion of the process cycle would have contacted waste solution at this equivalent phase ratio.

To provide additional conservatism, we measured the loadings in contact with a simulated waste solution that contained very high concentrations of uranium, plutonium, and neptunium. The selected concentrations represent the highest expected values for waste solutions that will be processed through the ARP facility. Thus, the measured fissile loadings reported in this document should provide conservative upper values for actinide loadings for planned operations in the ARP facility.

At the conditions testing we measured (average of filtrate and solids data) fissile loadings of $(3.37 \pm 0.29)10^4$ μg/g for plutonium, $(1.67 \pm 0.19)10^5$ μg/g for uranium, and $(1.02 \pm 0.036)10^4$ for neptunium. Calculations indicate good mass balance agreement for all three actinides. The measured loadings for uranium and plutonium in this study are considerably higher than those previously reported in support of the In-Tank Precipitation Facility. The closest previous data set for the plutonium is from previous work done in 1993 (10). The previous data was collected at 19°C after a 168-hour contact time over a range of MST concentration ranging from 0.05–0.5 g/L MST. From a loading curve derived from that work, the maximum plutonium loading at 0.0235 g MST per liter of simulant (the conditions of this work) was approximately 15 times lower than reported in this work. For the uranium, the closest previous data set is from previous work done in 2002 (1). The previous data was collected at 19°C for one week, at 0.2 g MST per liter of simulant. From that work, the maximum estimated U loading was approximately half of the value reported in this work. No body of previous data makes a comparison for neptunium. The higher loadings are consistent with that expected given the much higher phase ratio and higher fissile concentrations in the simulated waste solution.

The theoretical maximum cation capacity of MST is 5.0 milliequivalents per gram based on an empirical formula of $NaTi_2O_5H$ and assuming all of the sodium ions are exchangeable (11). In the strongly alkaline and high ionic strength salt conditions, we believe the predominant cationic species of plutonium, uranium, and neptunium that exchange for sodium in the MST are Pu^{4+}, UO_2^{2+}, and NpO_2^{+}, respectively. Converting the measured actinide loadings into the equivalent cation values we calculate that the total actinide loadings represent 35% of the theoretical cation capacity.

High-resolution transmission electron microscopy revealed that the MST particles consist of a fibrous outer region and an amorphous glasslike inner core (12). Analysis of the strontium-sorbed MST located the strontium only in the outer fibrous region of the particle suggesting that the inner glasslike region is not readily accessible for sorption/ion exchange. If we assume that only the fringe region of the MST particle is involved with actinide loading, then the effective capacity of the MST is estimated at 2.32 meq/g. At this lower estimate of capacity, the measured total actinide loading represents 75% of the estimated effective cation capacity of the MST. Inspection of the graphs showing the solution phase actinide concentrations as a function of contact time suggests that the tests may not have reached equilibrium when we terminated the tests. If this is the case, additional loading of the MST could occur with longer contact times. Thus, the measured loadings for actinides in this study indicate a high degree of loading, but not necessarily the maximum possible loading of actinides onto the MST.

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