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Stable Sr vs ^{85}Sr Sorption from Simulated Waste Solutions by MST and mMST

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EXECUTIVE SUMMARY

A series of tests were performed to examine the sorption of stable Sr versus the sorption of ^{85}Sr by monosodium titanate (MST) and modified monosodium titanate (mMST) from simulated waste solutions. Earlier testing indicated a discrepancy between the decontamination factors (DFs) obtained by measuring the stable Sr concentrations by inductively coupled plasma – mass spectroscopy (ICP-MS) and the ^{85}Sr activities by gamma spectroscopy. One hypothesis to explain this discrepancy was that the stable Sr and ^{85}Sr were in different chemical forms in the simulated solutions. Several simulants were prepared using different methods for adding the Sr and performance tests were carried out using MST and mMST to determine the Sr and ^{85}Sr DFs with the various simulants. Testing indicated no discrepancy between the Sr and ^{85}Sr DFs in tests with these simulants.

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LIST OF ABBREVIATIONS

DF	decontamination factor
ICP-MS	inductively coupled plasma – mass spectroscopy
MST	monosodium titanate
mMST	modified monosodium titanate
PuTTA	plutonium thenoyltrifluoroacetone scintillation
PVDF	polyvinylidene fluoride
SCIX	small column ion exchange

1.0 Introduction

Earlier Sr and actinide sorption testing with monosodium titanate (MST) and modified monosodium titanate (mMST) in support of the Small Column Ion Exchange (SCIX) Program showed a discrepancy between the decontamination factors (DFs) calculated for stable Sr and for ^{85}Sr.[1] Stable Sr concentration is measured using inductively coupled plasma – mass spectroscopy (ICP-MS), while the ^{85}Sr activity is measured using gamma spectroscopy. In the earlier testing many of the ^{85}Sr DFs were found to be about an order of magnitude higher than the stable Sr DFs calculated using the ICP-MS concentrations. In other cases the stable Sr concentration fell below the method detection limit, resulting in greater than values for the DFs. These tests were conducted for a total of 6 weeks; however, after 72 hours all of the stable Sr concentrations were below the detection limit, so only the data for the first 72 hours is presented in Table 1-1. It was hypothesized that the discrepancy may be due to the stable Sr and ^{85}Sr radiotracer being in different chemical forms in the simulant. The different forms of Sr originate from $Sr(NO_3)_2$ being the source of Sr^{2+} for the stable Sr, and the ^{85}Sr source containing both radioactive and stable Sr in an HCl solution. In addition to the bulk chemical matrices, other impurities particularly in the ^{85}Sr source could be complexing with some fraction of the Sr resulting in a different chemical form. Two sets of simulants were prepared to test this hypothesis.

Table 1-1. Sr DFs obtained in the earlier SCIX testing. The numbers in parentheses represent 1 sigma uncertainty.

Test ID	**SCIX-1**	**SCIX-2**	**MST-Shaker**	**mMST-Shaker**
Sorbent	**MST**	**mMST**	**MST**	**mMST**
^{85}Sr – 6 h	67.2 (20.2)	> 118	61.7 (18.4)	> 120
Stable Sr – 6 h	6.11 (1.73)	> 11.8	> 11.8	> 11.8
^{85}Sr – 12 h	58.9 (13.5)	84.2 (20.2)	> 85.3	> 114
Stable Sr – 12 h	3.35 (0.947)	> 12.2	> 12.2	> 12.2
^{85}Sr – 24 h	85.9 (23.5)	> 65.7	> 66.0	140 (21.5)
Stable Sr – 24 h	7.36 (2.53)	> 11.2	> 11.2	9.78 (2.77)
^{85}Sr – 48 h	87.9 (17.9)	> 132	132 (33.9)	132 (40.5)
Stable Sr – 48 h	> 5.38	> 5.38	> 5.38	1.95 (0.558)
^{85}Sr – 72 h	77.4 (12.5)	101 (20.5)	88.6 (17.8)	> 117
Stable Sr – 72 h	4.53 (1.28)	5.73 (1.62)	> 7.20	> 7.20

The general method for preparing the simulants involves dissolving all sorbates and the $Al(NO_3)_3$ in a nitric acid solution, as well as adding the radiotracers. Sodium hydroxide is then added to this solution to bring the free hydroxide concentration to the target value (generally about 2.2 M). The remainder of the sodium salts are then added, and the solution is diluted to the final concentration. Due to the relatively short half-life of ^{85}Sr, this radiotracer is often respiked into older simulants so that they can be used for additional testing. In this situation, the ^{85}Sr is being added to an alkaline solution, rather than an acidic solution.

In the first set of tests two simulants were prepared. The first simulant was prepared following the general procedure described in the previous paragraph, adding the stable Sr in the form of $Sr(NO_3)_2$ and the ^{85}Sr tracer to the simulant separately, but while the solution was still acidic. In the second simulant, the $Sr(NO_3)_2$ was first dissolved in a small volume of water. The ^{85}Sr tracer was then added to this solution, and the solution was evaporated to dryness removing the bulk of the mineral acids (HNO_3 and HCl). The residue containing both forms of the Sr was then redissolved in distilled water and added to the simulant solution, again while still acidic.

In the second set of tests two additional simulants were prepared. The first simulant was prepared following the general procedure described above. For preparation of the second simulant, the ^{85}Sr was not added until the simulant had been fully prepared and diluted to the final concentration. This simulant was meant to simulate the common practice of respiking older simulants with additional ^{85}Sr, where the radiotracer is being added to an alkaline solution.

2.0 Experimental Procedure

2.1 Simulant Preparation

In the first set of tests two simulants were prepared, SWS-2-2011 A and B. The first simulant (A – normal prep) was prepared following the general procedure described in the previous paragraph, adding the stable Sr in the form of $Sr(NO_3)_2$ and the ^{85}Sr tracer to the simulant separately, but while the solution was still acidic. In the second simulant (B – combined addition), the $Sr(NO_3)_2$ was first dissolved in a small volume of water. The ^{85}Sr tracer was then added to this solution, and the solution was evaporated to dryness. The residue containing both forms of the Sr was then redissolved in distilled water and added to the simulant solution, again while still acidic. The composition of these simulants is shown in Table 2-1.

In the second set of tests two additional simulants were prepared, SWS-5-2011 A and B. The first simulant (A – normal prep) was prepared following the general procedure described above. For preparation of the second simulant (B – post-prep spiked), the ^{85}Sr was not added until the simulant had been fully prepared and diluted to the final concentration. This simulant was meant to simulate the common practice of respiking older simulants with additional ^{85}Sr, where the radiotracer is being added to an alkaline solution. The composition of these simulants is shown in Table 2-2.

Table 2-1. Composition of SWS-2-2011 A and B.

Component	Target Concentration	Measured Concentration (A – normal prep)	Measured Concentration (B – combined addition)
$NaNO_3$	2.03 M	2.11 ± 0.21 M	2.11 ± 0.21 M
NaOH	2.21 M	2.08 ± 0.21 M	1.89 ± 0.19 M
Na_2SO_4	0.14 M	0.149 ± 0.015 M	0.148 ± 0.015 M
$NaAl(OH)_4$	0.28 M	0.280 ± 0.028 M	0.281 ± 0.028 M
$NaNO_2$	0.50 M	0.563 ± 0.056 M	0.554 ± 0.055 M
$NaCO_3$	0.15 M	0.171 ± 0.017	0.177 ± 0.018
Total Na	5.6 M	5.66 ± 0.57 M	5.61 ± 0.56 M
Total Sr	6.0 mg/L	0.763 ± 0.153 mg/L	0.925 ± 0.185 mg/L
^{85}Sr	≥10,000 dpm/mL	18,300 ± 915 dpm/mL	16,300 ± 815 dpm/mL
Total Pu	0.2 mg/L	0.213 ± 0.011 mg/L	0.215 ± 0.011 mg/L
^{237}Np	0.5 mg/L	0.523 ± 0.105 mg/L	0.516 ± 0.103 mg/L
Total U	10 mg/L	10.5 ± 2.1 mg/L	10.6 ± 2.1 mg/L
^{137}Cs	≥30,000 dpm/mL	308,000 ± 15400 dpm/mL	307,000 ± 15400 dpm/mL

Table 2-2. Composition of SWS-5-2011 A and B.

Component	Target Concentration	Measured Concentration (A – normal prep)	Measured Concentration (B – post-prep spiked)
$NaNO_3$	2.03 M	2.00 ± 0.20 M	2.24 ± 0.21 M
NaOH	2.21 M	1.95 ± 0.20 M	2.00 ± 0.20 M
Na_2SO_4	0.14 M	0.146 ± 0.015 M	0.150 ± 0.015 M
$NaAl(OH)_4$	0.28 M	0.281 ± 0.028 M	0.285 ± 0.029 M
$NaNO_2$	0.50 M	0.524 ± 0.052 M	0.576 ± 0.058 M
$NaCO_3$	0.15 M	0.38 ± 0.04	0.16 ± 0.02
Total Na	5.6 M	5.79 ± 0.58 M	5.79 ± 0.58 M
Total Sr	6.0 mg/L	0.961 ± 0.192 mg/L	1.22 ± 0.24 mg/L
^{85}Sr	≥10,000 dpm/mL	36,000 ± 1800 dpm/mL	36,000 ± 1800 dpm/mL
Total Pu	0.2 mg/L	0.508 ± 0.025 mg/L	0.622 ± 0.031 mg/L
^{237}Np	0.5 mg/L	0.413 ± 0.083 mg/L	0.453 ± 0.091 mg/L
Total U	10 mg/L	9.10 ± 1.82 mg/L	10.1 ± 2.0 mg/L
^{137}Cs	≥30,000 dpm/mL	272,000± 13,600 dpm/mL	320,000 ± 16000 dpm/mL

2.2 Uptake Testing

All tests were performed by contacting MST or mMST with appropriate simulant solution in a temperature controlled orbital shaker oven at 25 °C and 175 rpm shaker speed. The first set of tests was performed as follows. Fifty mL of simulant SWS-2-2011 A or B was placed in each of 10 60-mL polyethylene bottles (5 each). MST and mMST were then added to the appropriate bottles at concentrations of 0.4 g/L and 0.2 g/L, respectively. Each sorbent was tested in duplicate for each simulant, and each simulant had one control containing no sorbent. The MST used in these experiments was supplied by Optima, Lot # 00-QAB-417. The mMST used in these experiments, batch LS-10, was prepared in the lab by treating a sample of the Optima 00-QAB-417 material with hydrogen peroxide following a previously published procedure.[2] The bottles were then placed in the shaker oven and agitated for 24 hours. After 24 hours, samples were removed and filtered through a 0.1 μm polyvinylidene fluoride (PVDF) syringe filter to remove the solids. An aliquot of the filtrate was acidified with an equal volume of 5 M nitric acid and submitted for inductively coupled plasma – mass spectroscopy (ICP-MS), gamma scan, and plutonium thenoyltrifluoroacetone scintillation (PuTTA) analyses.

Results from the initial tests resulted in many of the sorbate concentrations being below the analytical method detection limit, making it difficult to draw any conclusions regarding the difference in stable Sr and ^{85}Sr uptake. Therefore, a second set of tests was performed using the same conditions as test set 1, only the MST and mMST concentrations were reduced to 0.1 g/L and 0.05 g/L, respectively. Samples from these tests were submitted for gamma scan and ICP-MS analysis only.

The third set of tests involved the use of the second set of simulants, SWS-5-2011 A and B. The same procedure used for test set 1 was followed, adding MST and mMST at the reduced concentrations of 0.1 g/L and 0.05 g/L, respectively. Samples from these tests were submitted for ICP-MS, gamma scan, and PuTTA analyses.

3.0 Results and Discussion

The 24 hour decontamination factors (DFs) obtained for each set of tests are shown in Table 3-1. In addition the 24 hour DFs from test sets 2 and 3 are plotted in Figures 3-1 and 3-2. The concentrations are provided in the Appendix. Both the stable Sr and ^{85}Sr concentrations fell below the method detection limit in the first set of tests, resulting in greater than values obtained for the DFs. Therefore, no conclusions could be drawn regarding a difference in stable Sr versus ^{85}Sr uptake. In test set 2, all of the stable Sr concentrations for Simulant B (combined addition) fell below the method detection limit. However, as can be seen from the results with Simulant A (normal prep), there does not appear to be a discrepancy between the stable Sr and ^{85}Sr DFs. These results indicate that the normal method for preparing the simulant, i.e. adding the stable Sr and ^{85}Sr separately, but before adding the sodium hydroxide, does not result in different uptake of the two forms of Sr.

The final set of tests, test set 3, was designed to determine if the addition of ^{85}Sr to an already prepared, alkaline simulant caused a difference in the uptake of stable Sr and ^{85}Sr. Based on the results in Table 3-1 and Figure 3-2, there is excellent agreement between the Sr DFs obtained by the two different methods, indicating no difference in uptake of the two different forms of Sr.

Table 3-1. Decontamination Factors (DF) for Test Sets 1-3. The value in parenthesis represents one sigma uncertainty.

Test Set 1 – SWS-2-2011 A&B, 0.4 g/L MST, 0.2 g/L mMST

Sorbate (method)	Simulant A – Normal Prep				Simulant B – Combined Addition			
	MST		mMST		MST		mMST	
	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
^{85}Sr (gamma)	> 31.8	> 33.8	> 32.0	> 31.5	> 31.5	> 40.7	> 31.5	> 33.5
Stable Sr (ICP-MS)	> 3.36	> 3.36	> 3.36	> 3.36	> 3.09	> 3.09	> 3.09	> 3.09
^{237}Np (gamma)	0.847 (0.156)	1.31 (0.413)	0.845 (0.152)	> 1.01	1.11 (0.341)	0.757 (0.136)	> 0.784	0.927 (0.309)
Pu (PuTTA)	3.26 (0.217)	3.49 (0.227)	29.0 (1.98)	24.2 (1.70)	3.59 (0.270)	3.72 (0.293)	35.6 (2.89)	29.2 (2.36)
U (ICP-MS)	1.21 (0.341)	1.22 (0.346)	1.00 (0.283)	1.03 (0.292)	1.24 (0.351)	1.21 (0.343)	0.990 (0.280)	0.987 (0.279)

Test Set 2 – SWS-2-2011 A&B, 0.1 g/L MST, 0.05 g/L mMST

Sorbate (method)	Simulant A – Normal Prep				Simulant B – Combined Addition			
	MST		mMST		MST		mMST	
	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
^{85}Sr (gamma)	3.26 (0.238)	7.86 (0.856)	3.03 (0.214)	5.88 (0.618)	9.06 (1.42)	13.5 (2.92)	12.5 (2.51)	> 24.2
Stable Sr (ICP-MS)	2.69 (0.760)	> 5.70	3.01 (0.851)	> 5.70	> 7.01	> 7.01	> 7.01	> 7.01
^{237}Np (ICP-MS)	1.29 (0.366)	1.19 (0.337)	1.32 (0.372)	1.70 (0.480)	0.996 (0.282)	0.975 (0.276)	1.16 (0.327)	1.21 (0.342)
Pu (ICP-MS)	1.52 (0.431)	1.37 (0.689)	2.19 (0.621)	2.56 (0.723)	1.65 (0.533)	2.24 (0.634)	2.79 (0.790)	> 2.85
U (ICP-MS)	1.02 (0.287)	1.04 (0.296)	1.00 (0.296)	0.998 (0.282)	1.04 (0.295)	1.07 (0.301)	0.994 (0.281)	1.05 (0.297)

Test Set 3 – SWS-5-2011 A&B, 0.1 g/L MST, 0.05 g/L mMST

Sorbate (method)	Simulant A – Normal Prep				Simulant B – Post-prep Spiked			
	MST		mMST		MST		mMST	
	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
^{85}Sr (gamma)	4.69 (0.332)	2.96 (0.209)	8.46 (0.598)	3.95 (0.279)	8.96 (0.634)	3.58 (0.253)	6.48 (0.458)	5.13 (0.363)
Stable Sr (ICP-MS)	3.72 (1.05)	3.19 (0.902)	8.02 (2.27)	4.03 (1.14)	9.64 (2.73)	3.70 (1.05)	6.52 (1.85)	5.37 (1.52)
^{237}Np (ICP-MS)	1.04 (0.295)	1.09 (0.308)	1.27 (0.359)	1.10 (0.312)	1.19 (0.337)	1.18 (0.334)	1.30 (0.368)	1.24 (0.352)
Pu (PuTTA)	1.72 (0.112)	1.59 (0.102)	3.81 (0.245)	2.80 (0.176)	2.14 (0.137)	1.51 (0.105)	3.31 (0.223)	2.88 (0.195)
U (ICP-MS)	1.03 (0.293)	1.03 (0.290)	1.00 (0.283)	1.04 (0.294)	1.03 (0.293)	1.02 (0.288)	0.989 (0.280)	0.956 (0.270)

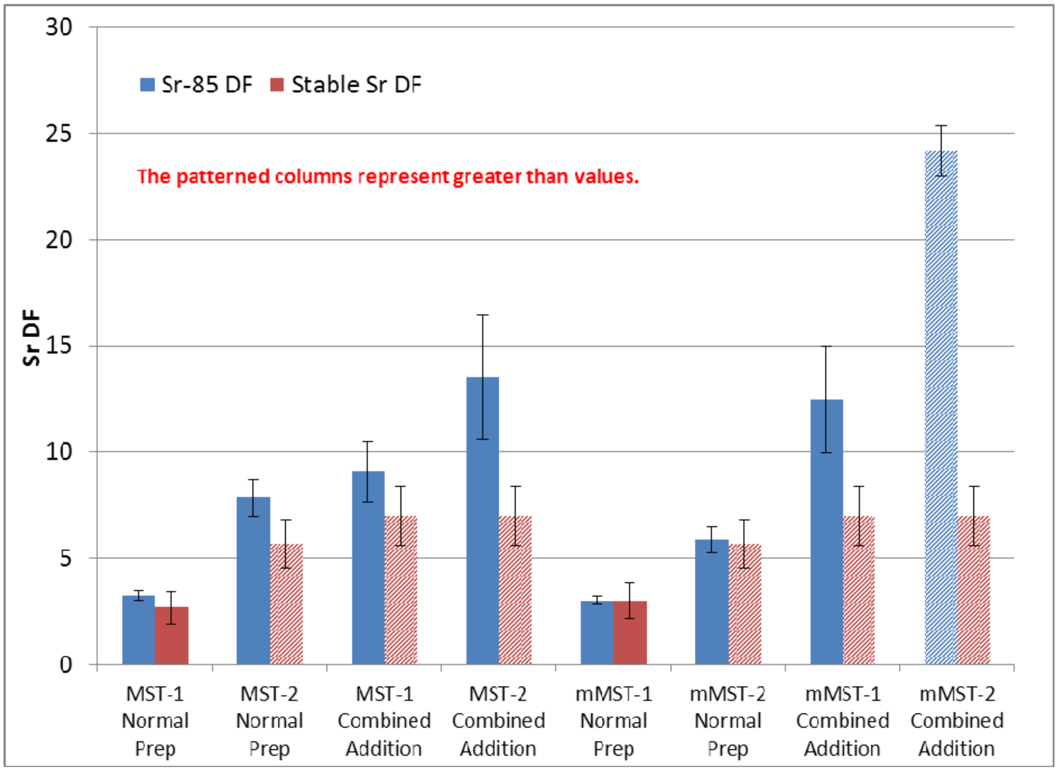

Figure 3-1. Comparison of 24 hour DFs for Test Set 2.

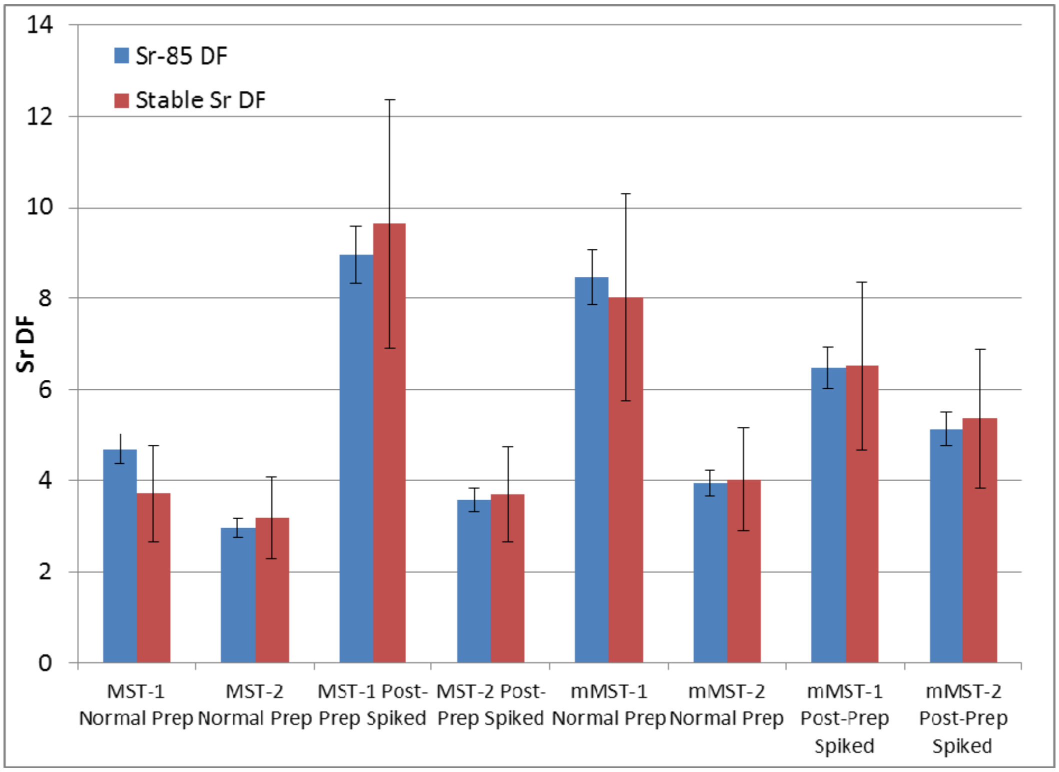

Figure 3-2. Comparison of 24 hour DFs for Test Set 3.

4.0 Conclusions

Based on these results, the discrepancies seen in the SCIX testing between the stable Sr and ^{85}Sr DFs was not due to the method of preparation of the simulant. The original hypothesis was that the difference in uptake may be due to the stable Sr and ^{85}Sr radiotracer being in different chemical forms in the simulant due to the methods of addition. However, results from this testing indicate that the normal method used to prepare the simulant, adding the stable Sr and ^{85}Sr separately, but still while the simulant is in an acidic form, does not lead to a difference in uptake. In addition, the practice of spiking simulants with additional ^{85}Sr after preparation is complete, also does not cause a difference in the uptake of the two forms of Sr.

5.0 References

1. K. M. L. Taylor-Pashow, D. T. Hobbs, and M. R. Poirier "Strontium and Actinide Sorption by MST and mMST Under Conditions Relevant to the Small Column Ion-Exchange (SCIX) Process", SRNL-STI-2011-00215, Rev. 0, April 2011.
2. M. Nyman and D. T. Hobbs, "A Family of Peroxo-titanate Materials Tailored for Optimal Strontium and Actinide Sorption." *Chem. Mater*. **2006**, *18*, 6425.

Appendix

Table A-1. Concentrations of sorbates in test set 1 after 24 hours of contact of SWS-2-2011 A (Normal Prep) and B (Combined Addition) with MST (0.4 g/L) or mMST (0.2 g/L).

Test ID	**Simulant A – Control**	**Simulant A – MST-1**	**Simulant A – MST-2**	**Simulant A – mMST-1**	**Simulant A – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	3820	< 120	< 113	< 119	< 121
^{237}Np (mg/L)	376	444	288	445	< 373
^{137}Cs (dpm/mL)	300,000	306,000	292,000	294,000	308,000
PuTTA					
Total Pu (µg/L)	207	63.4	59.2	7.13	8.54
ICP-MS					
Total Sr (µg/L)	508	< 151	< 151	< 151	< 151
^{237}Np (µg/L)	< 276	< 276	454	< 276	514
^{239}Pu (µg/L)	220	< 150	< 150	< 150	< 150
^{238}U (µg/L)	10,100	8,360	8,240	10,100	9,780
Test ID	**Simulant B – Control**	**Simulant B – MST-1**	**Simulant B – MST-2**	**Simulant B – mMST-1**	**Simulant B – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	3950	< 125	< 97.2	< 126	< 118
^{237}Np (mg/L)	343	308	453	< 437	369
^{137}Cs (dpm/mL)	300,000	306,000	292,000	294,000	308,000
PuTTA					
Total Pu (µg/L)	225	62.5	60.4	6.32	7.70
ICP-MS					
Total Sr (µg/L)	467	< 151	< 151	< 151	< 151
^{237}Np (µg/L)	< 276	< 276	< 276	< 276	< 276
^{239}Pu (µg/L)	252	< 150	< 150	< 150	< 150
^{238}U (µg/L)	10,300	8,320	8,500	10,400	10,500

Table A-2. Concentrations of sorbates in test set 2 after 24 hours of contact of SWS-2-2011 A (Normal Prep) and B (Combined Addition) with MST (0.1 g/L) or mMST (0.05 g/L).

Test ID	**Simulant A – Control**	**Simulant A – MST-1**	**Simulant A – MST-2**	**Simulant A – mMST-1**	**Simulant A – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	2,870	881	365	949	488
^{237}Np (mg/L)	376	584	468	346	343
^{137}Cs (dpm/mL)	306,000	308,000	306,000	308,000	306,000
ICP-MS					
Total Sr (µg/L)	690	257	< 121	229	< 121
^{237}Np (µg/L)	608	470	510	462	358
^{239}Pu (µg/L)	276	181	202	126	108
^{238}U (µg/L)	10,200	10,100	9,780	10,200	10,200
Test ID	**Simulant B – Control**	**Simulant B – MST-1**	**Simulant B – MST-2**	**Simulant B – mMST-1**	**Simulant B – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	2,590	285	191	208	< 107
^{237}Np (mg/L)	501	< 322	419	330	281
^{137}Cs (dpm/mL)	302,000	306,000	300,000	304,000	296,000
ICP-MS					
Total Sr (µg/L)	637	< 90.8	< 90.8	< 90.8	< 90.8
^{237}Np (µg/L)	462	464	474	400	382
^{239}Pu (µg/L)	214	130	95.4	76.6	< 75.0
^{238}U (µg/L)	10,400	9,940	9,740	10,400	9,900

Table A-3. Concentrations of sorbates in test set 3 after 24 hours of contact of SWS-5-2011 A (Normal Prep) and B (Post-prep Spiked) with MST (0.1 g/L) or mMST (0.05 g/L).

Test ID	**Simulant A – Control**	**Simulant A – MST-1**	**Simulant A – MST-2**	**Simulant A – mMST-1**	**Simulant A – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	23,400	4,980	7,910	2,760	5,930
^{237}Np (mg/L)	< 346	358	< 424	573	431
^{137}Cs (dpm/mL)	272,000	272,000	280,000	274,000	280,000
PuTTA					
Total Pu (µg/L)	525	305	331	138	188
ICP-MS					
Total Sr (µg/L)	973	262	305	121	242
^{237}Np (µg/L)	444	426	408	350	402
^{239}Pu (µg/L)	484	278	314	125	176
^{238}U (µg/L)	9,580	9,260	9.340	9,580	9,220
Test ID	**Simulant B – Control**	**Simulant B – MST-1**	**Simulant B – MST-2**	**Simulant B – mMST-1**	**Simulant B – mMST-2**
Gamma					
^{85}Sr (dpm/mL)	23,100	2,580	6,450	3,570	4,510
^{237}Np (mg/L)	591	361	415	249	445
^{137}Cs (dpm/mL)	320,000	322,000	322,000	322,000	322,000
PuTTA					
Total Pu (µg/L)	659	308	436	199	228
ICP-MS					
Total Sr (µg/L)	1,220	126	329	187	227
^{237}Np (µg/L)	458	384	388	352	368
^{239}Pu (µg/L)	576	240	356	181	185
^{238}U (µg/L)	10,800	10,500	10,600	10,900	11,300

Distribution:

A. B. Barnes, 999-W
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
K. M. L. Taylor-Pashow, 773-A
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F. F. Fondeur, 773-A
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P. R. Jackson, 703-46A
K. H. Subramanian, 766-H

M. A. Rios-Armstrong, 773-66A
T. H. Huff, 773-66A
R. E. Edwards, 773-67A
M. W. Geeting, 241-152H
K. L. Lang, 704-27S
B. A. Oard, 704-61H