

Keywords: *MCU, ARP, ISDP*

Retention: *Permanent*

**Sample Results from the Integrated Salt Disposition Program
Macrobatch 5 Tank 21H Qualification MST, ESS and PODD Samples**

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April 2012

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Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) performed experiments on qualification material for use in the Integrated Salt Disposition Program (ISDP) Batch 5 processing. This qualification material was a composite created from recent samples from Tank 21H and archived samples from Tank 49H to match the projected blend from these two tanks.

Additionally, samples of the composite were used in the Actinide Removal Process (ARP) and extraction-scrub-strip (ESS) tests. ARP and ESS test results met expectations. A sample from Tank 21H was also analyzed for the Performance Objectives Demonstration Document (PODD) requirements. SRNL was able to meet all of the requirements, including the desired detection limits for all the PODD analytes.

LIST OF ABBREVIATIONS

AA – Atomic Absorption
AD – Analytical Development
AMP – ammonium molybdophosphate
ARP – Actinide Removal Project
CVHg – Cold Vapor Mercury
ESS – extraction, scrub, strip
IC – Ion Chromatography
ICPES – Inductively Coupled Plasma Emission Spectroscopy
ICPMS – Inductively Coupled Plasma Mass Spectroscopy
ISDP – Integrated Salt Disposition Program
MST – monosodium titanate
PODD – Performance Objectives Demonstration Document
PuTTa – Plutonium thenoyl trifluoroacetone scintillation
SRNL – Savannah River National Laboratory
SRR – Savannah River Remediation
TIC-TOC – Total Inorganic Carbon-Total Organic Carbon
TTQAP - Task Technical and Quality Assurance Plan
TTR – Technical Task Request
WAC – Waste Acceptance Criteria
% RSD – percent relative standard deviation

1.0 Introduction

This report details the results of the Actinide Removal Process (ARP), Extraction-Scrub-Strip (ESS) and Performance Objectives Demonstration Document (PODD) samples of Macrobatch (Salt Batch) 5 of the Integrated Salt Disposition Program (ISDP).

Previous documents^{1,2} cover initial and subsequent characterization which include analytical results. This work was specified by Task Technical Request³ and by Task Technical and Quality Assurance Plan (TTQAP).⁴

Details for the work are contained in controlled laboratory notebooks.⁵

2.0 Experimental Procedure

Five Tank 21H samples (i.e., dip sample bottles HTF-21-11-114, HTF-21-11-115, HTF-21-11-116, HTF-21-11-117, and HTF-21-11-118) arrived at SRNL on October 13, 2011.

For this macrobatch, Tank 21H is used as the blend and preparation tank. This material will be transferred to Tank 49H where it will be combined with the heel from Macrobatch 4. In this qualification effort for Macrobatch 5, only samples from Tank 21H have been analyzed. In this campaign, the qualification and tank strategy⁶ indicates that analysis of Tank 49H is not needed as the material was qualified for Macrobatch 4. As long as the Tank 21H material is qualified, and the qualified Tank 49H material has not changed, then the blend of these two tanks will provide a usable composite. However, for the purposes of the Actinide Removal Process (ARP) and Extraction-Scrub-Strip (ESS) test, SRNL used a blend of Tank 21H and Tank 49H intended to mimic the contents of Tank 49H after transfer of waste from Tank 21H.

2.1 PODD Samples

Using 100 mL of the composite Tank 21H solution, the researchers acidified the sample with concentrated nitric acid, until the pH registered 1 or less. To this acidified solution, the researchers added ~1g of ammonium molybdophosphate (AMP). After stirring for a few minutes, the yellow AMP solids were removed by filtration. This procedure was repeated twice more with fresh quantities of AMP. The final filtrate from this procedure was sent in its entirety to AD for analysis. The dilution caused by the procedure was taken into account when reporting the results. A previous document indicates there is no effect of AMP on U and Pu.⁷ From a literature search, SRNL concludes that it is unlikely the other PODD elements would have an affinity for AMP.⁸

2.2 MST Sorption Test

For the MST Sorption Test, technicians generated ~400 mL of the ISDP5 composite. The composite was not filtered, nor was the turbidity measured; gross formation of solids was not observed. The composition of the composite is described in Table 1. The composition mimics the projected blend.

Table 1. Constituents of the ISDP5 Composite

Component	Mass Added (g)
Tank 49H	50.833
Tank 21H	486.48

Once prepared, the technicians measured the density of this composite to be 1.309 g/mL (25 °C). The analytical uncertainty is typically <1% for density measurements.

Technicians placed 200 mL of this solution into the experiment bottle, and the remainder (~200 mL) into the control bottle. Both bottles had magnetic stir bars added to provide mixing. Personnel added 0.4 g/L of MST solids (from an archived batch of material from Harrel Industries Lot 012808⁹) to the experiment bottle at time = 0 hours. Throughout the course of the test, the bottles were agitated using a magnetic stir plate and stir bars. Temperature control (to 25±3 °C) was provided by an actively controlled water bath.

During the experiment, personnel collected samples from each of the two bottles at 0, 2, 4, 6, 12, and 24 hours. For the sample at 0 hours, sampling occurred immediately prior to MST addition. Technicians filtered the samples using 0.45 µm Versapor™ syringe filters, removed the samples from the cells for analysis, and analyzed for plutonium (PuTTA), ⁹⁰Sr (beta scintillation), and ²³⁸U (ICPMS). Samples were sent to Analytical Development (AD) with moderate dilution, and those dilutions are accounted for in the results section. This test uses the same protocol as used in the previous Macrobatch testing.¹⁰

2.3 ESS Test Conditions

For the ESS Demonstration Test, material from the MST Sorption Test was used. For this test, the researchers used a nominal starting volume of 90 mL of aqueous feed and 30 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI).^r This test uses the same protocol as used in the previous Macrobatch testing.¹⁰

^r This batch of solvent was originally prepared with no extractant as S2-NOBOB-T-WI (see WSRC-NB-2005-00060). The extractant was added later (see WSRC-NB-2007-00054).

3.0 Results and Discussion

3.1 Performance Objectives Demonstration Document (PODD) Sample Analyses

The same stock of Tank 21H material was analyzed for the PODD radionuclide analysis.¹¹ In this case, a special sample preparation was followed to get the best detection limit possible. Table 2 contains the measured composition of the treated sample. The results are from single analyses, therefore values in parentheses are the analytical uncertainty.

Table 2. Nuclides Requiring Lower Detection Limits in Tank 21H for the PODD Analyses

Analyte	Result (pCi/mL)	Requested Detection Limit (pCi/mL)
⁵⁹ Ni	<2.55E+00	2.00E+01
⁹⁴ Nb	<02.77E-01	4.38E-01
¹³⁵ Cs	3.12E-01 (20%)	*
¹⁴⁴ Ce	<3.52E+00	1.03E+01
¹⁴⁴ Pr	<3.52E+00	1.03E+01
²²⁶ Ra	<1.16E+01	4.08E+01
²³² U	3.42E+00 (35.8%)	*
²³³ U	2.11E+01 (20%)	*
²⁴³ Am	<8.26E+00	*
²⁴⁴ Pu	<3.21E-03	**
²⁴⁵ Cm	<2.47E+01	**
²⁵¹ Cf	<2.94E+01	9.01E+01

* Measured above detection limits for Tank 50H.

** Detection limit should be as low as reasonably achievable.

Values in parentheses are percent relative standard deviation.

The ¹³⁵Cs is from ICPMS analysis, and we conservatively assume that all of mass-135 is due to ¹³⁵Cs, which is not necessarily true (as ¹³⁵Ba interferes). ¹⁴⁴Pr is calculated as equal to the ¹⁴⁴Ce result.

3.2 Results from the MST Sorption Test

For the MST Sorption Test, technicians used 200 mL of a composite made from Tank 21H and Tank 49H samples (see section 2.1). The composite was not filtered and no observation of gross formation of solids was made. The turbidity was not measured.

During the experiment, personnel collected samples from each of the two bottles at 0, 2, 4, 6, 12, and 24 hours. For the sample at 0 hours, sampling occurred immediately prior to

MST addition. Technicians filtered the samples using 0.45 μm Versapor™ syringe filters, removed the samples from the cells for analysis, and analyzed for plutonium (PuTTA), ^{90}Sr (beta scintillation), and ^{238}U (ICPMS). ^{237}Np and ^{243}Am were both observed to be below detection limits in the source material, and so these results are not reported. Samples were sent to Analytical Development (AD) with moderate dilution, and those dilutions are accounted for in the results section.

The four hour control sample results for all analytes were found to clearly be samples from the experiment and not the control as they were virtually the same result. We suppose that the technicians accidentally sampled from the experiment bottle rather than the control bottle for the 4 hour control samples. Therefore, we do not report the 4 hour control sample results.

3.2.1 Plutonium Results

Researchers analyzed the filtered samples for ^{238}Pu . Table 3 shows the plutonium results while Figure 1 shows the graphical results for ^{238}Pu . The ^{238}Pu data is more useful than the $^{239/40}\text{Pu}$ as the former is not limited by detection limit values. The relative standard deviation (shown in parentheses) in Table 3 is the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

Table 3. ^{238}Pu Concentrations in the MST Strike Filtrates

Time (hours)	Experiment	Control
	^{238}Pu (pCi/mL)	^{238}Pu (pCi/mL)
0*	1.50E+04 (4.66%)	1.50E+04 (4.66%)
2	1.47E+04 (5.29%)	1.42E+04 (4.21%)
4	3.73E+03 (6.33%)	compromised sample
6	3.60E+03 (7.12%)	1.39E+04 (4.40%)
12	3.09E+03 (7.05%)	1.52E+04 (4.61%)
24	2.61E+03 (5.80%)	1.46E+04 (4.61%)

*The time = 0 data are the same data point.

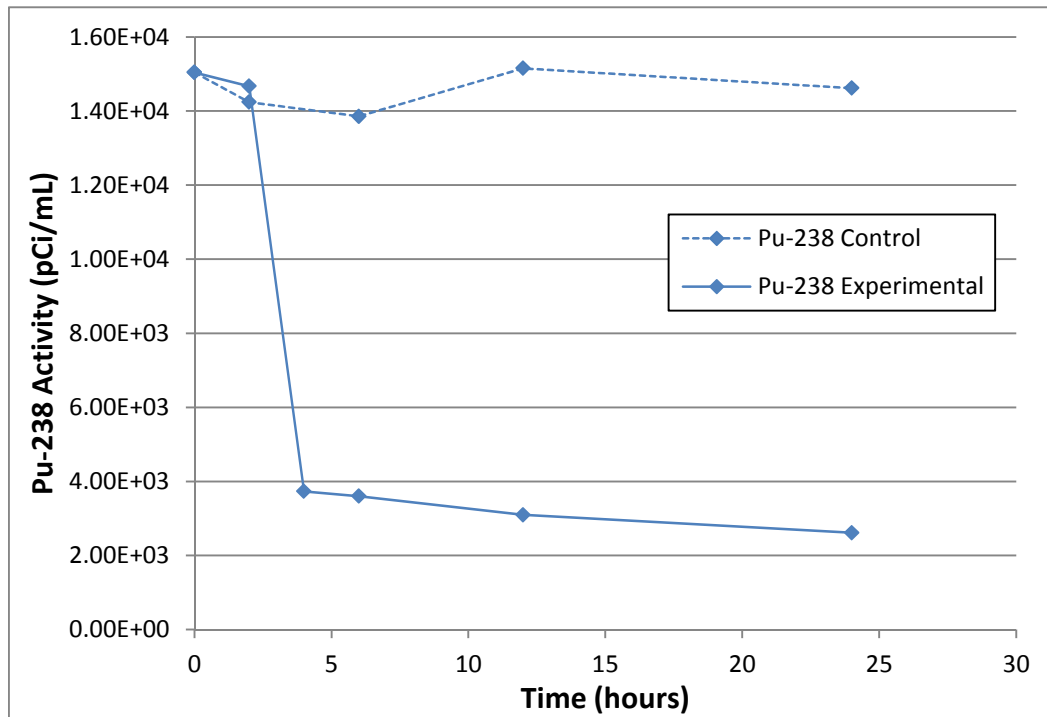
Figure 1. ^{238}Pu in Solution over Time for the MST Sorption Test

Table 4 lists the decontamination factors (DF) after the MST strike.

Table 4. ^{238}Pu Decontamination Factors (DF) over Time

Time (hours)	Experiment	Control
	DF	DF
2	1.03	1.06
4	4.03	N.A.
6	4.18	1.09
12	4.86	0.99
24	5.76	1.03

N.A. = not available

3.2.2 Strontium Results

Researchers analyzed the filtered samples for ^{90}Sr . Table 5 shows the strontium results while Figure 2 shows the graphical results for ^{90}Sr . The relative standard deviation (shown in parentheses) in Table 5 is the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental

and sampling methods. Note that the same sampling error as noted for Pu is also seen in the Sr analyses.

Table 5. ^{90}Sr Concentrations in the MST Strike Filtrates

Time (hours)	Experiment	Control
	^{90}Sr (pCi/mL)	^{90}Sr (pCi/mL)
0*	1.58E+05 (7.84%)	1.58E+05 (7.84%)
2	1.27E+05 (8.58%)	1.41E+05 (7.69%)
4	2.02E+03 (10.9%)	compromised sample
6	1.38E+03 (8.70%)	1.33E+05 (8.77%)
12	1.74E+03 (8.33%)	1.77E+05 (8.50%)
24	1.51E+03 (8.78%)	1.77E+05 (8.06%)

*The time = 0 data are the same data point.

Figure 2. ^{90}Sr in Solution over Time for the MST Sorption Test

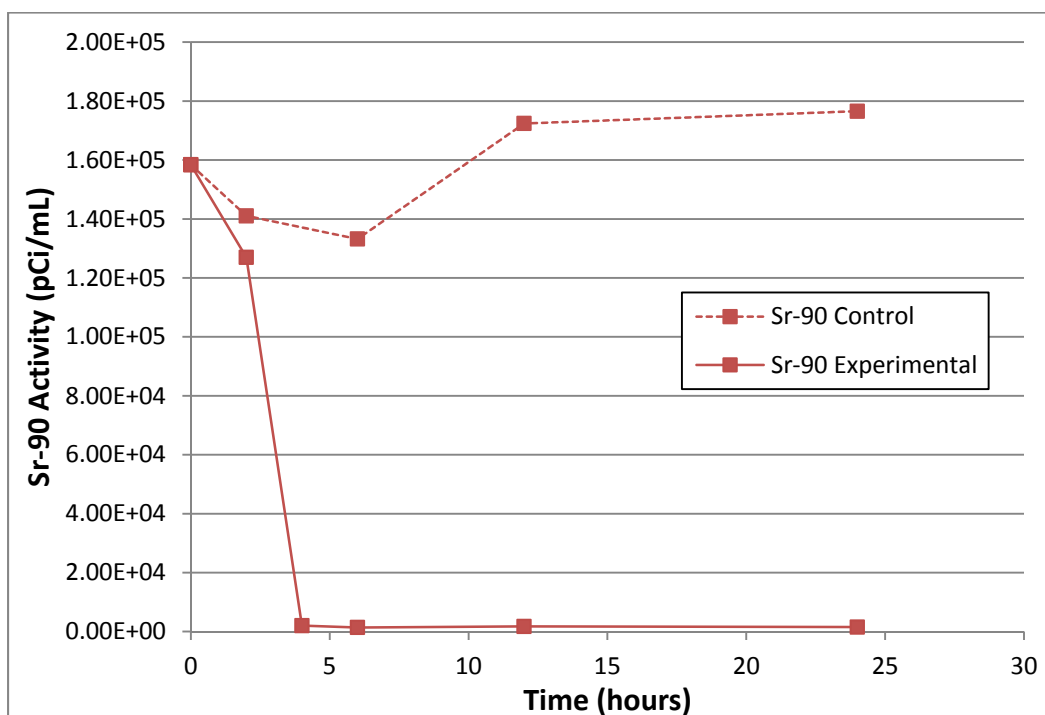


Table 6 lists the decontamination factors (DF) after the MST strike.

Table 6. ^{90}Sr Decontamination Factors (DF) over Time

Time (hours)	Experiment	Control
	DF	DF
2	1.24	1.12
4	78.49	N.A.
6	114.77	1.19
12	91.07	0.89
24	104.60	0.89

N.A. = not available

3.2.3 Uranium Results

Researchers analyzed the filtered samples for ^{238}U . Table 7 shows the uranium results while Figure 3 shows the graphical results for ^{238}U . The relative standard deviation (shown in parentheses) in Table 7 is the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

Table 7. ^{238}U Concentrations in the MST Strike Filtrates

Time (hours)	Experiment	Control
	^{238}U (pCi/mL)	^{238}U (pCi/mL)
0*	4.30E+00 (20%)	4.30E+00 (20%)
2	4.08E+00 (20%)	4.27E+00 (20%)
4	3.67E+00 (20%)	compromised sample
6	3.62E+00 (20%)	4.04E+00 (20%)
12	3.46E+00 (20%)	4.17E+00 (20%)
24	3.38E+00 (20%)	4.19E+00 (20%)

*The time = 0 data are the same data point.

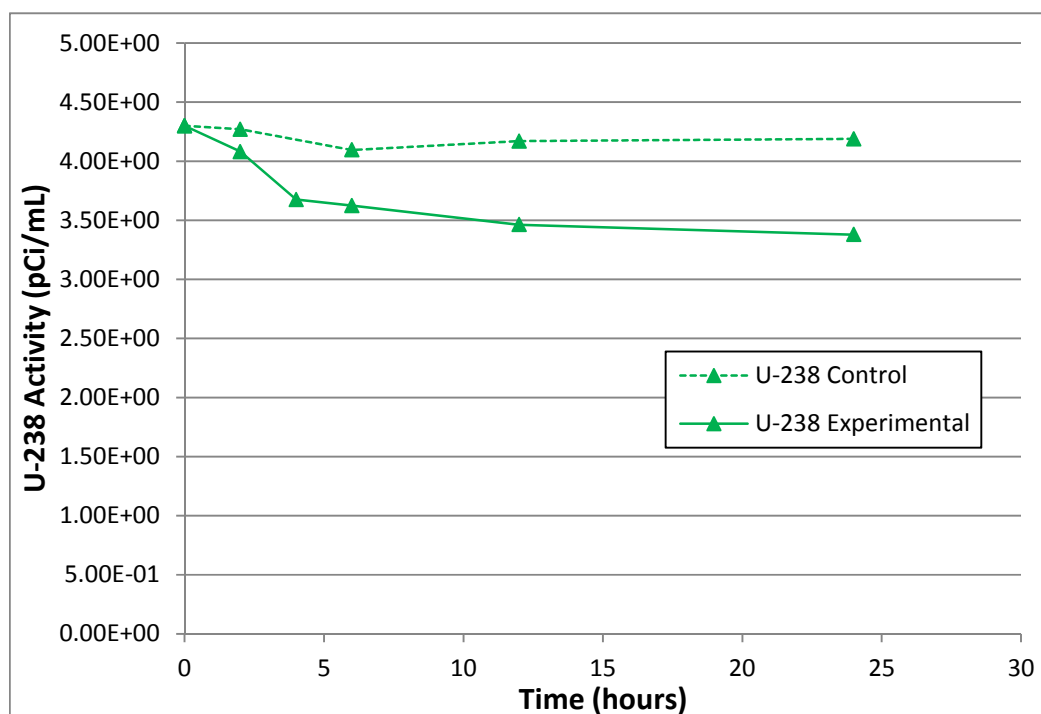
Figure 3. ^{238}U in Solution over Time for the MST Sorption Test

Table 8 lists the DF after the MST strike.

Table 8. ^{238}U Decontamination Factors (DF) Over Time

Time (hours)	Experiment	Control
	DF	DF
2	1.05	1.01
4	1.17	N.A.
6	1.19	1.05
12	1.24	1.03
24	1.27	1.03

N.A. = not available

3.2.4 Neptunium and Americium Results

There was insufficient ^{237}Np and ^{243}Am in the feed solution to determine any decontamination effects of MST. Both the ^{237}Np and ^{243}Am detection limits were 10 $\mu\text{g/L}$ for the control samples and 20 $\mu\text{g/L}$ for the experiment samples.

3.2.5 Consideration of DF Values

The DF values for plutonium and strontium are shown for all macrobatches to date.^{10,12,13,10} See Table 9.

Table 9. Pu and Sr 24-Hour DF Values for All 4 Macrobatches

Macrobatches	Pu DF	Sr DF
1	14.0	59.4
2	5.64	70.9
3	9.30	70.6
4	4.82	40.7
5	5.76	104.60

The current Macrobatches decontamination efficiency for Pu falls within the range of prior batches while the strontium removal is the highest seen within this series.

3.2.6 Analysis of MST Solids

After the MST test completed, personnel digested the retained MST solids (aqua regia/microwave) and sent them to AD for analysis. Table 10 shows the results of the titanium analysis.

Table 10. Tank 21H MST Solids Analyses Chemical Results

Analyte	Method	Result (mg/L)	% Uncertainty
Ti	ICPES	14000	10%

While in principle a known amount of MST (in this case, 571 mg) is added, it is uncertain the mass of the MST that is recovered at the end of the MST strike experiment; the mass is small to begin with and contains an unknown amount of interstitial salt solution. Due to this fact, the results of the MST solids analysis to the Ti result from the ICPES analyses must be reported. The MST solids were digested into a 0.05 L liquid sample, with a resulting density of ~1 g/mL. With a Ti result of 14000 mg/L, this means our sample had 280 mg of titanium (14,000 mg/L multiplied by 0.05 L digested sample volume).

For each of the relevant analytes (see Table 11), the AD results are converted into terms of “pCi analyte/g of Ti” value. Typically, this is done by taking the raw AD result (in pCi/mL) and multiplying by 50 mL to generate an analyte result in terms of pCi. The pCi result is then divided by the mass of Ti in grams, to get the final result of “pCi analyte/g of Ti”, which is reported in Table 11.

As there are no, or virtually no, sludge solids in the feed material, the solids digestion data reflects the MST solids, and whatever adsorbs to the MST, as well as entrained salt solution. Actinides and strontium adsorb to MST and the analysis of the MST provides relevant data for those species. However, the other results for materials that have no affinity for MST are a function of material in the feed solution. Results for these elements are from interstitial or entrained salt solution. As there is no experimental data for many of these analytes as to whether or not they adsorb to MST under our conditions, SRNL cannot conclusively determine if the real values for an analyte result is from MST sorption or interstitial liquid entrainment. Therefore, the values reported in Table 11 should all be considered upper bounds.

All results were single results as there was not enough material to analyze duplicates. Values in parentheses are the analytical uncertainty.

Table 11. Tank 49H MST Solids Radiological Results

Analyte	Result (pCi per gram of Ti)	Analyte	Result (pCi per gram of Ti)
²³³ U	<3.46E+00	¹⁵¹ Sm	<1.06E+05
²³⁴ U	<5.01E+00	¹³⁴ Cs	<2.31E+04
²³⁵ U	3.73E+01 (20.0%)	¹³⁷ Cs	1.14E+08 (5.00%)
⁹⁹ Tc	4.23E+04 (5.81%)	¹⁴⁴ Ce	<8.69E+03
²³⁷ Np	1.82E+01 (20.0%)	¹⁵⁴ Eu	2.72E+03 (12.0%)
²³⁸ Pu	4.04E+06 (5.73%)	¹⁵⁵ Eu	<5.08E+03
^{239/40} Pu	4.55E+05 (5.79%)	²²⁶ Ra	<2.82E+04
²⁴¹ Pu	1.34E+06 (15.1%)	²⁴¹ Am	2.50E+04 (12.0%)
²⁴² Pu	<3.46E+00	^{242m} Am	1.23E+02 (42.7%)
Total Alpha	<5.55E+06	²⁴³ Am	<1.02E+03
Total beta	2.58E+08 (10.0%)	²⁴² Cm	1.01E+02 (42.7%)
⁶⁰ Co	7.60E+02 (13.7%)	²⁴³ Cm	<7.38E+03
⁹⁰ Sr	4.29E+07 (7.35%)	²⁴⁴ Cm	1.12E+05 (14.8%)
⁹⁴ Nb	<6.14E+02	²⁴⁵ Cm	<6.06E+03
¹⁰⁶ Ru	<6.12E+03	²⁴⁷ Cm	<9.33E+03
¹²⁵ Sb	<4.25E+03	²⁴⁹ Cf	<9.74E+03
¹²⁶ Sb	<8.09E+02	²⁵¹ Cf	<6.81E+03
¹²⁶ Sn	<4.42E+03	¹⁴⁷ Pm	<7.85E+04

3.3 Results from the ESS Test

For the ESS Test, filtrate from the MST Sorption Test was used. For this test, the researchers used a nominal starting volume of 90 mL of aqueous feed and 30 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI).^r

Table 12 shows the results from the ESS Test, corrected to the normal process operating temperatures (i.e., 23 °C for extraction and 33 °C for scrubbing and stripping). As a comparison, the results from the previous macrobatch qualification ESS test (using the same solvent) in 2011 are displayed.¹⁰

Table 12. Cesium Distribution Values for the ESS Test

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
Acceptable Range	>8	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
S2-D1-YES BOB-T-WI, ISDP 4 (previous test)	12.33	1.69	0.892	0.0434	0.057	0.019
S2-D1-YES BOB-T-WI, ISDP 5 (current test)	15.96	1.57	0.953	0.0397	0.039	0.040

The current test shows acceptable values for all steps. From the bulk chemistry of the solution, an extraction DF of ~12.8 is predicted.¹⁴

3.6.1 Strip Effluent and DSS Results

During, and at the end of the ESS test, the gamma activity in the strip effluent and the decontaminated salt solution (DSS) was measured. The results are shown in Table 13.

Table 13. Strip Effluent and DSS Results

Sample	¹³⁷ Cs activity (pCi/mL)	pH
Strip Effluent #1	2.81E+08	3
Strip Effluent #2	1.78E+08	5
Strip Effluent #3	1.01E+08	5
DSS	6.19E+06	14

^r This batch of solvent was originally prepared with no extractant as S2-NOBOB-T-WI (see WSRC-NB-2005-00060). The extractant was added later (see WSRC-NB-2007-00054).

The analytical uncertainty on the ^{137}Cs activity is 10% and ± 1 pH unit for the pH measurement.

4.0 Conclusions

Analysis of the Tank 21H sample indicates that the material does not display any unusual characteristics. In conjunction with the previous reports,^{1,2} the Tank 21H material, when combined with the Tank 49H heel is acceptable for processing in the ISDP process.

This report also covers the MST sorption and ESS results for the ISDP Salt Batch 5 feed sample. The following observations are made from the work.

- A demonstration of the monosodium titanate removal of strontium and actinides provided acceptable 12 hour decontamination values for Pu and Sr of 4.86 and 91.07, respectively. These DF values are slightly lower than previous tests, but not enough to warrant a high degree of concern.
- A demonstration of cesium extraction, scrubbing and stripping cesium mass transfer – intended to partially mimic the MCU operations – yielded behavior within acceptable norms. The measured distribution values are: 15.96, 1.57, 0.953, 0.0397, 0.039, and 0.040 for Extraction, Scrub #1, Scrub #2, Strip #1, Strip #2, and Strip #3, respectively. The values indicate the cesium removal should be comparable to prior batches in MCU.
- SRNL was able to meet the desired detection limits for all the PODD analytes.

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