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Disclaimer:

Sample Results from the Interim Salt Disposition Program Macrobatch 8 Tank 21H Qualification Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Macrobatch (Salt Batch) 8 for the Interim Salt Disposition Program (ISDP). An Actinide Removal Process (ARP) and several Extraction-Scrub-Strip (ESS) tests were also performed. This document reports characterization data on the samples of Tank 21H as well as simulated performance of ARP and the Modular Caustic Side Solvent Extraction (CSSX) Unit (MCU). No issues with the projected Salt Batch 8 strategy are identified.

A demonstration of the monosodium titanate (MST) (0.2 g/L) removal of strontium and actinides provided acceptable average decontamination factors for plutonium of 2.62 (4 hour) and 2.90 (8 hour); and average strontium decontamination factors of 21.7 (4 hour) and 21.3 (8 hour). These values are consistent with results from previous salt batch ARP tests.

The two ESS tests also showed acceptable performance with extraction distribution ratios ($D_{(Cs)}$) values of 52.5 and 50.4 for the Next Generation Solvent (NGS) blend (from MCU) and NGS (lab prepared), respectively. These values are consistent with results from previous salt batch ESS tests. Even though the performance is acceptable, SRNL recommends that a model for predicting extraction behavior for cesium removal for the blended solvent and NGS be developed in order to improve our predictive capabilities for the ESS tests.

LIST OF ABBREVIATIONS

AA	Atomic Absorption
AD	Analytical Development
ARP	Actinide Removal Process
CSSX	Caustic Side Solvent Extraction
CV-Hg	Cold Vapor Mercury
$D_{(Cs)}$	distribution ratio for cesium
DF	decontamination factor
DSS	Decontaminated Salt Solution
ESS	extraction, scrub, strip
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectroscopy
ICPMS	Inductively Coupled Plasma Mass Spectrometry
ISDP	Interim Salt Disposition Program
MCU	Modular CSSX Unit
MST	monosodium titanate
M&TE	Measurement and Test Equipment
NGS	Next Generation Solvent
PuTTa	plutonium thenoyl trifluoroacetone
RSD	relative standard deviation (percent)
SRNL	Savannah River National Laboratory
SVOA	Semi Volatile Organic Analysis
TIC-TOC	Total Inorganic Carbon-Total Organic Carbon
TTQAP	Task Technical and Quality Assurance Plan
VOA	Volatile Organic Analysis

List of Figures

List of Tables

1.0 Introduction

This report provides the Tank 21H qualification sample results for ISDP Macrobatch (Salt Batch) 8. A previous document covered initial characterization which includes results for a number of non-radiological analytes.[1] This work was specified in a Technical Task Request[2] and in a Task Technical and Quality Assurance Plan (TTQAP).[3] Details of the work are contained in controlled laboratory notebooks.[4]

2.0 Experimental Procedure

Six 200 mL Tank 21H samples (i.e., sample bottles HTF-21-14-126, -127, -128, -130, -131, -132) arrived at SRNL on September 18, 2014. Four of these samples were surface samples and two were obtained approximately 62" from the bottom of the tank (transfer pump suction height). The slurry pumps were run for approximately 22 hours, and the samples were pulled approximately 48 hours after the slurry pumps were shutdown. The samples contained no visible quantities of solids, which is different than what was observed in the Salt Batch 7 qualification samples.[5] The Salt Batch 7 qualification samples were obtained immediately after the slurry pumps were shut down; it was anticipated that solids may have been present in the samples. The density of the contents of each dip sample bottle was measured (at an ambient temperature of 23 ºC), using filtered samples (0.45 μm syringe filter). The results of the density measurements are listed in Table 1.

Table 1. Sample Density Measurements (23 ºC)

Sample	Measured Density (g/mL)
HTF-21-14-126	1.244
HTF-21-14-127	1.262
HTF-21-14-128	1.257
HTF-21-14-130	1.254
HTF-21-14-131	1.263
HTF-21-14-132	1.261
Average (%RSD)	1.257 (0.56%)

The analytical uncertainty is typically <1% for density measurements. The value in parentheses is the percent relative standard deviation (%RSD).[℘]

[℘] RSD is defined as the standard deviation of the array, divided by the average of the array, expressed in % terms.

With customer concurrence, the contents of the six sample bottles were then combined and mixed. After combining, duplicate filtered samples (using a 0.45 μm syringe filter) were sent to Analytical Development (AD) for analysis without dilution. For the weight% solids[∇] and "total Pu", well mixed (unfiltered) samples of the composite material were sent forward for analyses.

2.1 MST Sorption Test

For the MST Sorption Test, approximately 500 mL of the ISDP Salt Batch 8 Tank 21H composite material was obtained for testing. Due to the inability to clearly determine if solids were present or not, and whether they would settle, the 500 mL was filtered through a 0.45 μm filter cup before use. Normally, the solution is decanted, not filtered.

The composite salt solution was previously measured with a density of 1.257 g/mL at 23 °C. 200 mL each (totaling 400 mL) of the salt solution was placed into the first and second experiment bottles, while the remainder (100 mL) was placed into the control bottle. Two experiment bottles were used in order to provide enough solution for the later ESS tests and enough MST solids for future analysis. All three bottles had magnetic stir bars added to provide sufficient mixing for batch contact tests. The target concentration for MST was 0.2 g/L. Personnel added 0.253 g of MST solids in a 16.4 wt% solution from Blue Grass Chemical Specialties MST-2723 to each experiment bottle. This material was an archived batch that has been utilized on all recent salt batches by SRNL. The time was recorded and designated as time 0. Throughout the course of the MST test, agitation and temperature control (via water bath, 25±3 ºC) were provided.

During the experiment, a single sample was collected from the control bottle at time = 0 immediately prior to MST addition to the experiment bottles. For the samples at 4 and 8 hours, sampling and subsequent filtering occurred immediately at the 4 or 8 hour mark preventing additional MST sorption in each of the control and two experiment bottles. Personnel filtered the samples using 0.45 μm Versapor ™ syringe filters, removed the samples from the cells for analysis, and analyzed for ^{238}Pu and $^{239/40}$Pu (Plutonium Thenoyl Trifluoroacetone - PuTTa), ^{90}Sr (beta scintillation), and ^{238}U (Inductively Coupled Plasma Mass Spectrometry - ICPMS). Samples were sent for characterization without dilution. This test uses the same protocol as used in the previous Macrobatch testing.[5]

When the filtrate results were received, it was found that the decontamination factor (DF) values for experiment #1 were far lower than experiment #2 or historical results. Given that the two tests were run at the same time at the same conditions, SRNL hypothesized

[∇] The wt% solids used Measurement & Test Equipment (M&TE) balances in their sample preparation. Other analyses used M&TE where appropriate.

that experiment #1 inadvertently did not receive the full amount of MST. A third experiment and concurrent control was run, using the same material, under the same conditions (except run at a smaller scale as no material was needed for further tests), and this third test gave results similar to experiment #2 as well as historical precedent. Therefore, the results from experiment #2 and experiment #3 are used. As there were two sets of control data, both control sets are plotted with their respective experiments.

2.2 ESS Demonstration

For the ESS Demonstrations, filtrate from both MST Sorption Tests was used. Using this material, the researchers performed two ESS tests. Both of the tests used the same general protocol as used in previous Macrobatch testing.[5] The first test used a nominal starting volume of 80 mL of aqueous feed and 20 mL of freshly prepared, NGS solvent.♦ The second test used a nominal starting volume of 80 mL of aqueous feed and 20 mL of used NGS blend solvent from MCU. As-received, this solvent was low in suppressor concentration; therefore SRNL trimmed up the material to generate a solvent that was up to specifications.♥ For both tests, the scrub and strip solutions were 0.025 M NaOH and 0.01 M boric acid, respectively.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

The density of filtered samples (using a 0.45 μm syringe filter) from each sample was measured and tabulated. The results of the density measurements are listed in Table 1. In a previous document,[1] density, Inductively Coupled Plasma Emission Spectroscopy (ICPES), Ion Chromatography (IC), Total Inorganic Carbon/Total Organic Carbon (TIC/TOC), and Free Hydroxide results were reported for the Tank 21H composite. These results are also reported here for completeness (Table 2). The analytical uncertainty for the ICPES, the IC, the TIC/TOC, and the Free Hydroxide results is 10%. The values in the parentheses are the %RSD. The TIC and TOC results are in terms of mg/L of carbon. If we assume that the entire TIC result is carbonate, this translates to a carbonate concentration of 0.275 M. The free hydroxide converts to a pH of 14. The nickel (Ni) result converted into a concentration of $Ni(OH)_2$ is <7.80 mg/L.

♦ NGS solvent is composed of four components: 0.05 M MaxCalix (1,3-*alt*-25,27-bis(3,7-dimethyloctyl-1-oxy)calix[4]arene-benzocrown-6), 0.5 M Cs-7SB Modifier (1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol), 0.003 M TiDG (N,N',N"-tris (3,7-dimethyloctyl) guanidine), and the balance Isopar ™ L.

♥ The NGS-MCU blend (either hot or cold) is a 50/50 volume % blend of MCU solvent and a prepared mixture of compounds, that once mixed, gives a nominal composition as follows: 0.0035 M BOBCalixC6, 0.5M Cs-7SB Modifier, 0.0015 M (TOA), 0.003 M TiDG, 0.0465 M MaxCalix, and the balance Isopar ™ L.

The bulk chemical characteristics of this batch are roughly similar to that of Salt Batches 6-D and 7-B. Of note is the potassium result, which at 643 mg/L is slightly higher than the next highest potassium value of 580 mg/L (from Salt Batch 3). This concentration of potassium is less than the MCU processing requirement of 1950 mg/L, and is unlikely to interfere with the solvent performance given past testing with high potassium simulants.

Table 2. Previously Reported Results

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.94	Sb	<41.1
Al	5410 (0.0%)	Si	57.4 (1.2%)
B	70.8 (0.7%)	Sn	<93.1
Ba	<1.00	Sr	<12.8
Be	<0.14	Th	<11.6
Ca	1.25 (0.0%)	Ti	<0.93
Cd	<1.27	U	<69.9
Ce	<11.2	V	<0.69
Cr	72.4 (0.7%)	Zn	5.13 (1.2%)
Cu	<3.54	Zr	<0.62
Fe	<1.39	F^-	<100
Gd	<4.44	Cl^-	401 (0.4%)
K	643 (7.5%)	Br^-	<500
La	<1.97	Formate	468 (0.8%)
Li	19.5 (3.3%)	Nitrite	38400 (6.1%)
Mg	<8.61	Nitrate	122000 (0.5%)
Mn	<0.8	Phosphate	545 (5.6%)
Mo	24.9 (1.4%)	Sulfate	5270 (3.4%)
Na	142000 (0.5%)	Oxalate	187 (8.0%)
Ni	<4.94	TIC	3300 (0.6%)
P	231 (0.9%)	TOC	216 (0.0%)
Pb	<130	Free Hydroxide	2.24 M (0.6%)
S	2490 (0.9%)		

Values in parentheses are the %RSD.

3.1 Tank 21H Qualification Analyses

The tank samples were analyzed by AD using listed non-radiological methods (Table 3) and radiological methods (Table 4). Analyses were performed in duplicate and reported in Tables 5 and 6, respectively. Averages of the individual results, with the RSD in parentheses, are reported.

Table 3. Non-Radiological Analyses

Method	Analyte
IC Cations	NH_4^+
IC Anions	F, Cl, Br, formate, nitrite, nitrate, sulfate, phosphate, oxalate
TIC	total inorganic carbon
TOC	total organic carbon
Atomic Absorption (AA)-As	As
AA-Se	Se
Cold Vapor (CV)-Hg	Hg
High Performance Liquid Chromatography (HPLC)	tetraphenylborate
Semi Volatile Organic Analysis (SVOA)	tributylphosphate, phenol
Volatile Organic Analysis (VOA)	isopropanol, butanol, isobutanol

Table 4. Radiological Analyses

Method	Analyte
Tritium	^{3}H
^{14}C	^{14}C
gamma scan, Cs-removed	^{60}Co, ^{106}Ru, ^{125}Sb, ^{126}Sb, ^{126}Sn, ^{144}Ce, ^{154}Eu, ^{155}Eu
^{90}Sr, ^{94}Nb, ^{129}I, ^{99}Tc , ^{135}Cs, ^{226}Ra	Individual radio count method
gamma scan	^{134}Cs, ^{137}Cs
^{232}U	^{232}U
$^{238-241}Pu$ (filtered and unfiltered) (Plutonium thenoyl trifluoroacetone scintillation)	^{238}Pu, $^{239/40}Pu$, ^{241}Pu
Am/Cm	^{241}Am, ^{243}Am, ^{244}Cm, ^{245}Cm
$^{59/63}Ni$	$^{59/63}Ni$
$^{147}Pr/^{151}Sm$	$^{147}Pr/^{151}Sm$
ICPMS	isotopes from mass number 81 to 209 and 230 to 252, incl. ^{233}U and above, ^{237}Np, ^{230}Th, ^{232}Th
Liquid Scintillation Counting	total alpha, total beta

3.2 Tank 21H Qualification Results (non-radiological analytes)

The results for the IC-Cations, weight percent insoluble solids, phenol, tetraphenylborate, tributylphosphate, isopropanol, methanol, isobutanol, butanol, arsenic, mercury, and selenium are listed in Table 5. The analytical uncertainty for all listed analyses is 20%, except for the IC-Cations and wt% insoluble solids, which are 10%. Shaded results are calculated values. Values in parentheses are %RSD.

Table 5. Miscellaneous Results (mg/L unless otherwise noted)

Analyte	Result
ammonium	<10
wt % insoluble solids	*0.014 wt%*
phenol	<100
tetraphenylborate	<5
tributylphosphate	<1
isopropanol	<0.25
butanol	<0.75
isobutanol	<0.75
methanol	<107
As	0.229 (6.5%)
Hg	129 (9.9%)
Se	<0.205

The wt% solids result is a single result, with the duplicate analysis giving a less-than value (<0.002). In this case, there is no %RSD, and the analytical uncertainty is 10% as previously stated. This low value is not atypical of salt batch feeds.

SRNL also notes the higher than usual Hg value, compared to the Salt Batch 7 value of 79 mg/L, and the previous highest concentration of 88.2 mg/L in Salt Batch 5.

Per Table 2, the oxalate concentration is 187 mg/L, and the formate concentration is 468 mg/L. The oxalate result is converted to the equivalent carbon result of 50.9 mg/L. The formate result is converted to the equivalent carbon result of 125 mg/L. Subtracting these results from the TOC result gives a remainder of 40.1 mg/L of carbon. If we assume all of this remainder carbon is in the form of methanol, this gives a calculated methanol result of 107 mg/L. This methanol result should be considered an upper bound as no direct analytical method for methanol is available.

3.3 Tank 21H Qualification Results (radiological analytes)

The results of the radiological analysis in pCi/mL are listed in Table 6. The analytical uncertainty for ICPMS samples is 20%. Other analytical methods have varying uncertainties, typically 5-10%, and are noted for single sample results (^{14}C).

Table 6. Radiological Results of Tank 21H Analyses for Macrobatch 8

Analyte	Average Result	Analyte	Average Result
^{3}H	<2.01E+03	^{155}Eu	<7.02E+01
^{14}C	*6.48E+02 (6.5%)*	^{226}Ra	<5.76E+01
^{59}Ni	<3.56E+01	^{232}U	3.24E+00 (3.9%)
^{63}Ni	<1.81E+01	^{233}U	<2.83E+01
^{60}Co	<6.17E+00	^{234}U	1.18E+02 (4.8%)
^{90}Sr	4.91E+05 (6.4%)	^{235}U	4.18E-01 (0.5%)
^{90}Y	4.91E+05 (6.4%)	^{236}U	1.57E+00 (6.1%)
^{94}Nb	<1.54E+01	^{238}U	8.42E+00 (0.3%)
^{99}Tc	5.76E+04 (5.5%)	^{237}Np	<7.05E+00
^{106}Ru	<6.35E+01	^{238}Pu (unfiltered)	5.24E+04 (3.0%)
^{106}Rh	<6.35E+01	^{238}Pu (filtered)	5.63E+04 (9.1%)
^{125}Sb	<4.27E+01	^{239}Pu	9.89E+02 (0.9%)
^{125m}Te	<4.27E+01	^{240}Pu	<2.28E+03
^{126}Sn	5.72E+02 (31%)	$^{239/40}$Pu	1.35E+03 (12%)
^{129}I	4.53E+01 (8.8%)	^{241}Pu	2.23E+04 (8.9%)
^{134}Cs	<4.50E+04	^{242}Pu	<3.82E+01
^{135}Cs	8.24E+02 (2.8%)	^{244}Pu	<1.77E-01
^{137}Cs	2.13E+08 (2.2%)	^{241}Am	<5.85E+00
^{137m}Ba	2.01E+08 (2.2%)	^{243}Am	<7.07E+00
^{144}Ce	<1.36E+02	^{244}Cm	1.99E+00 (91%)
^{144}Pr	<1.36E+02	^{245}Cm	<1.83E+01
^{147}Pm	<7.56E+01	Total Alpha ♣	<4.33E+04
^{151}Sm	<3.68E+01	Total Beta (Cs removed)	9.45E+05 (2.7%)
^{154}Eu	<1.98E+01	Total Beta	2.73E+08 (0.23%)
		Total Gamma	2.01E+08

Shaded values are calculated results. ^{90}Y is calculated as equal to the ^{90}Sr result. ^{106}Rh is calculated as equal to the ^{106}Ru result. ^{125m}Te is calculated as equal to the ^{125}Sb result. ^{137m}Ba is calculated as 94.7% of the ^{137}Cs result.♦ ^{144}Pr is calculated as equal to the ^{144}Ce result. Total gamma is calculated as the sum of the ^{60}Co, ^{94}Nb, ^{106}Rh, ^{125}Sb, ^{125m}Te,

♣ The total alpha value was measured on a cesium removed sample to eliminate the spillover caused by cesium.

♦ While the ^{137m}Ba result is calculated from the analytically provided ^{137}Cs result, in actuality the gamma of the ^{137m}Ba is measured and the ^{137}Cs is determined from that.

^{126}Sn, ^{134}Cs, ^{137m}Ba, ^{144}Ce, ^{144}Pr, ^{154}Eu, ^{155}Eu, ^{226}Ra, ^{235}U, ^{237}Np, ^{241}Am, ^{243}Am, and ^{245}Cm. The ^{238}Pu, $^{239/40}Pu$, and ^{241}Pu results are from radio-counting, while the ^{242}Pu and ^{244}Pu results are from ICPMS. The $^{239/40}Pu$ value is a sum of the ^{239}Pu and ^{240}Pu. If care is taken into assuming a correct 239/240 isotopic breakdown (from facility history), the individual ^{239}Pu and ^{240}Pu values can be calculated. The total alpha result is an upper limit result (a signal was detected but not quantified) from a sample with the cesium removed before analysis. The total alpha and total beta methods are generally less precise than the individual method results. The total beta result was measured both with and without cesium being present and both results are presented.

Values in parentheses are the RSD unless only a single result is available, then the value in parentheses is the analytical uncertainty. Values in italics are single results (^{14}C). Results given in italics indicate that one of the sample results was either below the detection limit or the quantification limit, in which case the value in the parentheses is the analytical uncertainty. Only quantifiable measured values are reported when available.

3.4 Results of the MST Sorption Test

During the experiment, personnel collected samples from each of the three bottles at 0, 4, and 8 hours (time 0 sample was only taken from the control bottle). 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 ^{238}Pu and $^{239/40}Pu$ (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) without dilution.

As previously mentioned, DF results for experiment #1 were far lower than experiment #2 or historical results; therefore, a third experiment and concurrent control was run. Results from the third test gave results similar to experiment #2 as well as historical precedent. Therefore, the results from experiment #2 and experiment #3 are used. Experiment #1 and associated control DFs are provided as reference.

Compared to the MST test in Salt Batch 7,[5] the current test delivered slightly poorer Pu removal, but slightly better Sr removal. However, given the variability between the tests, these differences are not significant. Based on the data, the MST test results provide for adequate Pu and Sr removal for the salt batch.

3.4.1 *Plutonium Results*

Researchers analyzed the filtered samples for ^{238}Pu. Table 7 shows the plutonium results while Figure 1 shows the graphical results for ^{238}Pu. The ^{238}Pu data is more useful than the $^{239/40}Pu$ as the former has a higher specific activity, is easier to measure, and tends to have superior analytical uncertainty. The values in parentheses in Table 7 are the

analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

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

Time (hours)	^{238}Pu (pCi/mL)			
	Experiment #2	**Control #2**	**Experiment #3**	**Control #3**
0*	6.22E+04 (7.2%)	6.22E+04 (7.2%)	5.23E+04 (5.2%)	5.23E+04 (5.2%)
4	2.64E+04 (5.8%)	5.41E+04 (5.7%)	2.15E+04 (5.1%)	5.59E+04 (5.5%)
8	2.53E+04 (5.5%)	5.27E+04 (5.8%)	1.86E+04 (5.6%)	5.41E+04 (8.8%)

*The time = 0 data are the same data points for each set.

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

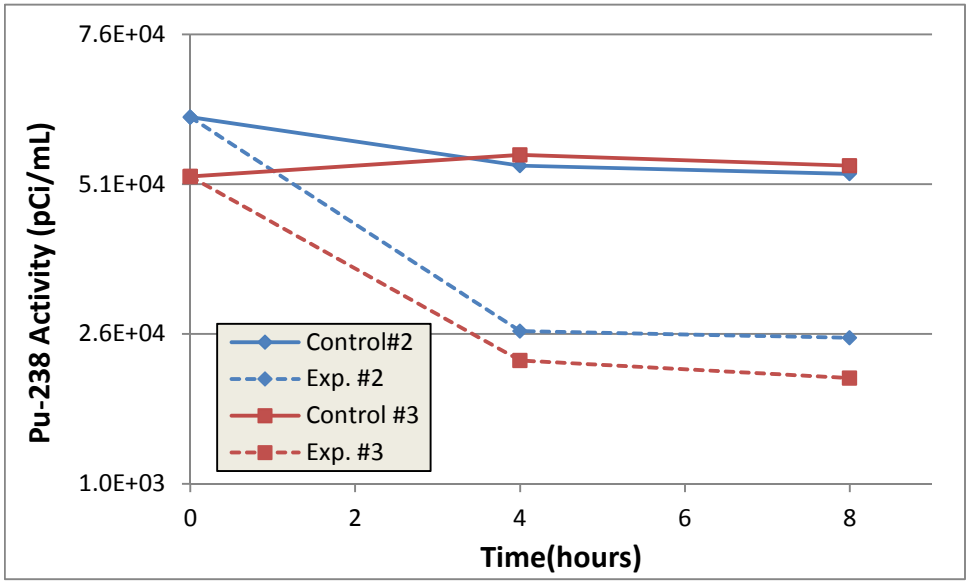

Table 8 lists the DFs after the MST strike.[⊕] The experiment #1 and control data points are provided as reference.

[⊕] DF is defined as the analyte concentration before decontamination (time = 0), divided by the analyte concentration after decontamination.

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

Data Point	DF	
	Time = 4h	**Time = 8h**
Experiment #1	1.23	1.30
Control #1	1.15	1.18
Experiment #2	2.35	2.46
Control #2	1.15	1.18
Experiment #3	2.89	3.34
Control #3	1.11	1.15

While there is only a limited amount of data related to Pu removal under the experimental conditions, the results of experiments #2 and #3 are within general expectations.

3.4.2 *Strontium Results*

Researchers analyzed the filtered samples for ^{90}Sr. Table 9 shows the strontium results while Figure 2 shows the graphical results for ^{90}Sr. The values in parentheses in Table 9 are the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

Table 9. ^{90}Sr Concentrations in the MST Strike Filtrates

Time (hours)	^{90}Sr (pCi/mL)			
	Experiment #2	**Control #2**	**Experiment #3**	**Control #3**
0*	4.59E+05 (14%)	4.59E+05 (14%)	3.78E+05 (16%)	3.78E+05 (16%)
4	2.00E+04 (14%)	4.95E+05 (14%)	2.26E+04 (29%)	4.95E+05 (19%)
8	1.76E+04 (14%)	5.23E+05 (14%)	2.81E+04 (24%)	4.95E+05 (23%)

*The time = 0 data are the same data point

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

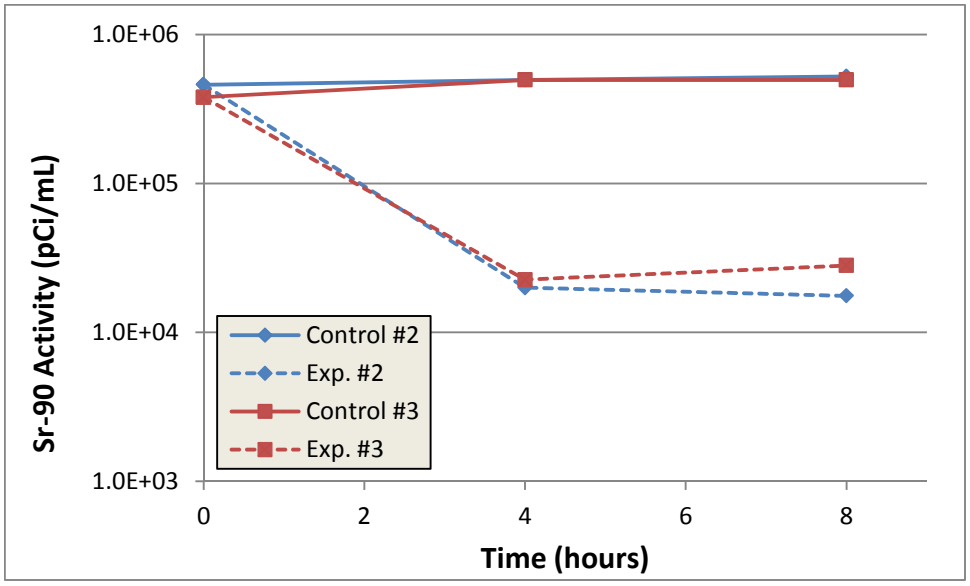

Table 10 lists the DFs after the MST strike. The experiment #1 and control data points are provided as reference.

Table 10. ^{90}Sr Decontamination Factors (DF) Over Time

Data Point	DF	
	Time = 4h	Time = 8h
Experiment #1	1.22	1.94
Control #1	0.93	0.88
Experiment #2	23.0	26.2
Control #2	0.93	0.88
Experiment #3	20.4	16.4
Control #3	0.93	0.93

While there is only a limited amount of data related to Sr removal under the experimental conditions, the results of experiment #2 and #3 are within general expectations.

3.4.3 *Uranium Results*

Researchers analyzed the filtered samples for ^{238}U. Table 11 shows the uranium results while Figure 3 shows the graphical results for ^{238}U. The analytical uncertainty for each sample is 20%.

Table 11. ^{238}U Concentrations in the MST Strike Filtrates

Time (hours)	^{238}U (pCi/mL)			
	Experiment #2	**Control #2**	**Experiment #3**	**Control #3**
0*	8.27	8.27	8.37	8.37
4	7.76	8.30	7.39	8.30
8	7.76	8.64	7.29	8.43

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

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

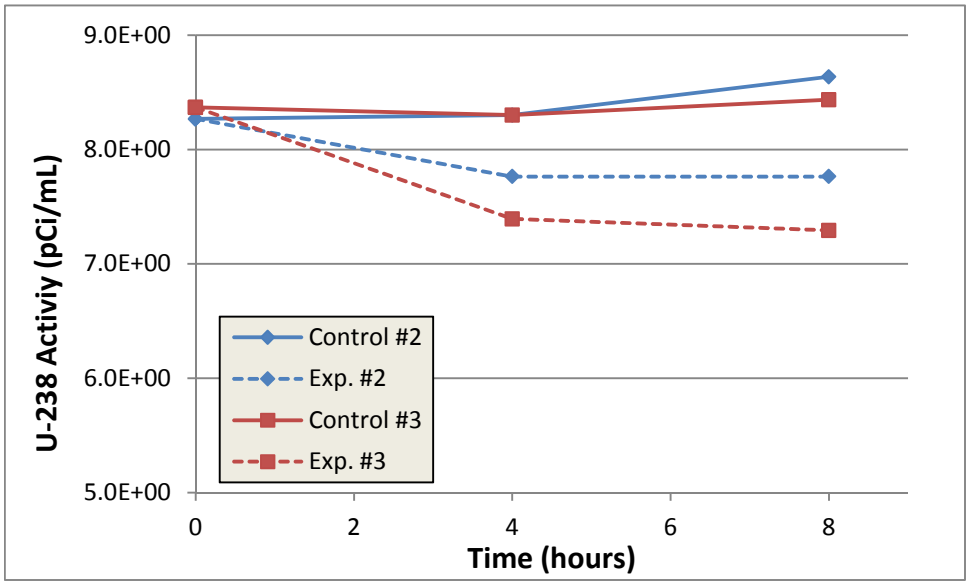

Table 12 lists the DFs after the MST strike. The experiment #1 and control data points are provided as reference.

Table 12. ^{238}U Decontamination Factors (DF) Over Time

Data Point	DF	
	Time = 4h	Time = 8h
Experiment #1	1.01	1.04
Control #1	1.00	0.96
Experiment #2	1.06	1.06
Control #2	1.00	0.96
Experiment #3	1.12	1.13
Control #3	1.00	0.98

Given the small concentration of MST used and the small effect that MST has on uranium, the uranium removal results are within expectations.

3.4.4 *Neptunium and Americium Results*

There were insufficient concentrations of ^{237}Np and ^{243}Am in the feed solution to determine any decontamination effects of MST.

3.5 Results of the ESS Test

For the ESS Tests, filtrate from the MST Sorption Test was used. Table 13 shows the results from the ESS tests, corrected to the normal process operating temperatures (i.e., 23 ºC for extraction and 33 ºC for scrubbing and stripping). For these tests, the temperature correction factors for the NGS solvent were used (see Appendix A).

The temperature in the Shielded Cells during the ESS test ranged from 20.5 ºC to 24.9 ºC with an average temperature of 23.4 ºC. As a comparison, the results from the previous macrobatch qualification ESS test (using the same solvent) are displayed.[5]

Table 13. Cesium Distribution Ratios ($D_{(Cs)}$) for the ESS Tests

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
SB 7 Hot NGS Blend	58.6	2.32	2.58	0.00057	0.00257	0.0111
SB 8 Hot NGS Blend	52.5	7.39	2.67	0.00226	0.00106	0.00671
SB 8 Cold NGS	50.4	3.30	1.64	0.000608	0.000553	0.00491

The current tests show the expected behaviors, with good overall performance. While the extraction steps are slightly lower than Salt Batch 7, the strip steps are better than typical. The variation in the scrub results is not unusual and should not be a concern. At this time we do not have a simple model to predict the extraction D_{Cs} of any solvent containing NGS or a blend containing NGS, but the values from the two tests are consistent with previous results.

3.5.1 *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) for a single extraction was measured. The results are shown in Table 14. The pH measurements of the same are shown in Table 15.

Table 14. Strip Effluent and DSS ^{137}Cs Results

Sample	^{137}Cs activity (dpm/mL)	
	Cold NGS	Hot Blend
Strip Effluent #1	6.34E+09	6.77E+09
Strip Effluent #2	7.27E+07	2.66E+08
Strip Effluent #3	7.96E+05	4.83E+06
DSS	2.47E+07	2.43E+07

The analytical uncertainty on the ^{137}Cs activity is 5%. The ^{137}Cs results from both tests are typical, given the large increase in ^{137}Cs activity in the feed.

Table 15. Strip Effluent and DSS pH Results

Sample	pH	
	Cold NGS	Hot Blend
Strip Effluent #1	7	7
Strip Effluent #2	7	7
Strip Effluent #3	6	6
DSS	14	14

The analytical uncertainty is ±1 pH unit for the pH measurement.

The pH results from both tests are typical.

4.0 Conclusions

Results of the analyses of the Tank 21H samples from this report indicate that the material does not display any unusual characteristics nor pose any concerns for processing.

This report also covers the MST sorption and ESS results for the ISDP Salt Batch 8 feed sample. The following observations are made from the work.

- A demonstration of the MST (0.2 g/L) removal of strontium and actinides provided acceptable average decontamination factors for plutonium of 2.62 (4 hour) and 2.90 (8 hour); and average strontium decontamination factors of 21.7 (4 hour) and 21.3 (8 hour). These values are consistent with results from previous salt batch ARP tests.

- A set of two demonstrations of cesium extraction, scrubbing, and stripping cesium mass transfer – intended to mimic any possible solvent system – yielded expected behavior.

- Even though the ESS performance is acceptable, SRNL recommends that a model for predicting extraction behavior for cesium removal for the blended solvent and NGS be developed in order to improve our predictive capabilities for the ESS tests.

Appendix A. Temperature Correction Factors for the ESS Tests

The actual MCU facility uses active temperature control to keep the extraction and scrub steps at 23 °C, and the strip steps at 33 °C. However, the ESS tests do not have active temperature control. During each step of an ESS test, the calculated distribution values must be corrected for temperature. The general formula for temperature correction is as follows:

correction factor = EXP((**COEF**/0.0083144)*((1/**TEMP**)-(1/(**STEP**))))

where "COEF" is the particular temperature coefficient for the step in question, the "TEMP" is the ambient temperature, in Kelvin, and "STEP" is 296.15 for extraction and scrub and 306.15 for strip steps. There is one set of coefficients for the MCU solvent, and one set of coefficients for use in NGS type solvents with MaxCalix (NGS, cold blend, hot blend).

Table 16 lists the temperature coefficients for each step in an ESS test. The coefficients for the NGS solvent are derived from the van't Hoff formalism in equation 1 of the applicable reference.

Table 16. Temperature Coefficients

Step	MCU [6]	NGS [7]
Extraction	-47.95	-90.12
Scrub#1	-86.82	-115.5
Scrub#2	-74.24	-91.40
Strip#1	-79.36	-80.18
Strip#2	-82.94	-143.4
Strip#3	-82.49	-65.63

5.0 References

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[3] T. B. Peters and A. L. Washington, II. “Task Technical and Quality Assurance Plan for ISDP Salt Batch 8 Sample Qualification”, SRNL-RP-2014-00839, Rev. 0, September 2014.

[4] T. B. Peters, “Salt Batch 8 Qualification”, ELN A4571-00084-13.

[5] T. B. Peters, A. L. Washington II, “Sample Results from the Interim Salt Disposition Program Macrobatch 7 Tank 21H Qualification Samples”, SRNL-STI-2013-00437, August 2013.

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Distribution

S. L. Marra, 773-A
T. B. Brown, 773-A
D. H. McGuire, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)
A. Samadi-Dezfouli, 704-27S
C. J. Scherman, 241-152H
R. T. McNew, 241-152H
J. K. Fortenberry, 766-H
T. A. Le, 766-H
S. A. Utlak, 704-27S
M. C. Clark, 704-25S
R. E. Edwards, 766-H
M. A. Rios-Armstrong, 766-H
J. M. Bricker, 704-S
J. S. Contardi, 704-56H
T. L. Fellinger, 766-H
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J. F. Iaukea, 704-27S
J. W. Ray, 704-27S
H. B. Shah, 766-H
C. B. Sudduth, 707-7E
V. Jain, 766-H
J. N. Leita, 704-56H
T. E. Smith, 241-152H
E. A. Brass, 241-121H
C. K. Chiu, 704-30S
A. G. Garrison, 241-121H
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A. R. Shafer, 766-H
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M. A. Broome, 704-29S
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R. D. Thames, 241-152H