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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Macrobatch (Salt Batch) 7 for the Interim Salt Disposition Program (ISDP). An ARP and several ESS tests were also performed. This document reports characterization data on the samples of Tank 21H as well as simulated performance of ARP/MCU. No issues with the projected Salt Batch 7 strategy are identified, other than the presence of visible quantities of dark colored solids.

A demonstration of the monosodium titanate (0.2 g/L) removal of strontium and actinides provided acceptable 4 hour average decontamination factors for Pu and Sr of 3.22 and 18.4, respectively. The Four ESS tests also showed acceptable behavior with distribution ratios ($D_{(Cs)}$) values of 15.96, 57.1, 58.6, and 65.6 for the MCU, cold blend, hot blend, and Next Generation Solvent (NGS), respectively. The predicted value for the MCU solvent was 13.2. Currently, there are no models that would allow a prediction of extraction behavior for the other three solvents. SRNL recommends that a model for predicting extraction behavior for cesium removal for the blended solvent and NGS be developed.

While no outstanding issues were noted, the presence of solids in the samples should be investigated in future work. It is possible that the solids may represent a potential reservoir of material (such as potassium) that could have an impact on MCU performance if they were to dissolve back into the feed solution.

This salt batch is intended to be the first batch to be processed through MCU entirely using the new NGS-MCU solvent.

LIST OF ABBREVIATIONS

AA	Atomic Absoption
AD	Analytical Development
AMP	ammonium molybdophosphate
ARP	Actinide Removal Project
CVHg	Cold Vapor Mercury
$D_{(Cs)}$	distribution ratio for cesium
DF	decontamination factor
ESS	extraction, scrub, strip
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectroscopy
ICPMS	Inductively Coupled Plasma Mass Spectroscopy
ISDP	Interim Salt Disposition Program
MCU	Modular CSSX Unit
MST	monosodium titanate
NGS	Next Generation Solvent
PuTTa	plutonium thenoyl trifluoroacetone scintillation
RSD	relative standard deviation (percent)
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

1.0 Introduction

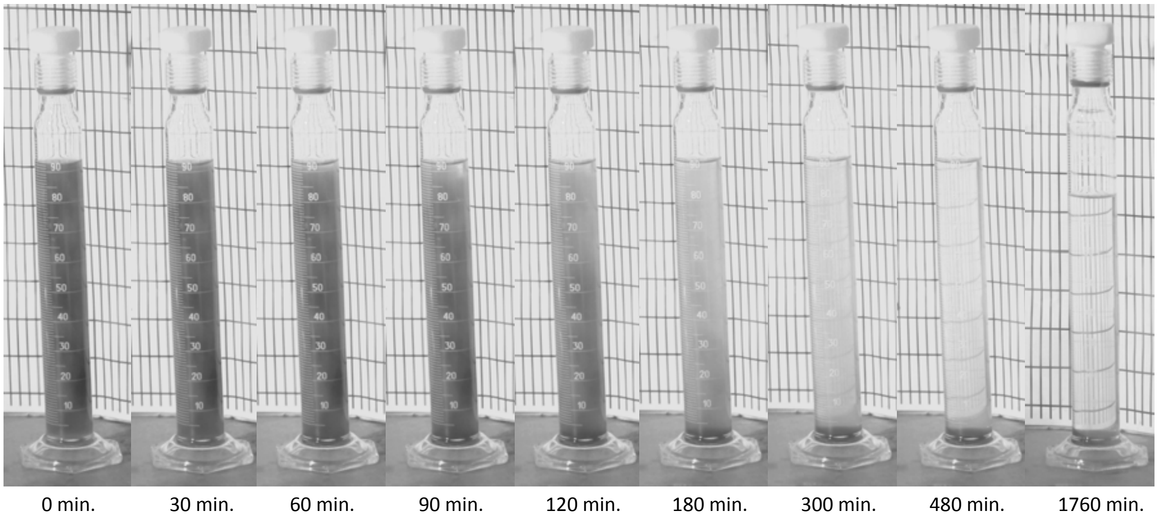
This report covers the Tank 21H qualification sample results for Macrobatch (Salt Batch) 7 of the Interim Salt Disposition Program (ISDP). A previous document covers initial characterization which includes results for a number of non-radiological analytes.[1] This work was specified by Technical Task Request [2] and by Task Technical and Quality Assurance Plan (TTQAP).[3]

Details for the work are contained in controlled laboratory notebooks.[4]

2.0 Experimental Procedure

Three Tank 21H samples (i.e., 80 mL dip sample bottles HTF-21-13-79 and HTF-21-13-80, and a 2 L sample HTF-21-13-81) were pulled on May 16 and arrived at SRNL on May 16, 2013. The samples each contained visible quantities of fine dark colored solids, which is not typical. 10 mL well-mixed samples from each of the three sample bottles were then removed for archival purposes. A well-mixed portion of material from HTF-21-13-81 was placed in a 100 mL graduated cylinder. Over a period of 29 hours, the contents were allowed to settle and multiple pictures were taken. See Figure 1.

Figure 1. Pictures of Settling Tank 21H Sample


0 min. 30 min. 60 min. 90 min. 120 min. 180 min. 300 min. 480 min. 1760 min.

Within 2 hours, there was visible stratification in the sample. After 8 hours, the sample was mostly settled, and by 29 hours, the sample had visibly clarified. Please note that the missing supernate in the last picture (t=1760) is due to a sample having been removed for analytical purposes.[1] The length of settling was designed to conservatively bound the time the feed tank would settle, accounting for the different settling heights.[5]

The density of filtered samples (using a 0.45 µm syringe filter) from each sample were measured and tabulated. The samples of the clarified material were also measured for density, without any filtration. The results of the density measurements are listed in Table 1.

With customer concurrence, the contents of the three sample bottles (HTF-21-13-79, 80, 81) 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", samples of the clarified material were sent forward for analyses.

2.1 MST Sorption Test

The Tank 21H composite material had visible quantities of dark colored solids. These solids were allowed to settle for 29 hours before the supernate was removed for use.

For the MST Sorption Test, approximately 475 mL of the ISDP7 Tank 21H material was obtained for processing. The composite salt solution was previously measured with a density of 1.272 g/mL at (25 °C). 200 mL each (totaling 400 mL) of the salt solution was placed into the first and second experiment bottles, while the remainder (~75 mL) was placed into the control bottle. Two experiment bottles were used in order to provide enough solution for the later ESS tests.

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 15.8 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 (25±3 ºC) were provided.

During the experiment, samples were collected from each of the three bottles at 0, 4 and 8 hours. For the sample at 0 hours, sampling occurred immediately prior to MST addition solely from the control bottle. For the sample at 4 and 8 hours, sampling occurred immediately at the 4 or 8 hour mark preventing additional MST sorption. Personnel 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). Samples were sent to AD with moderate dilution, and those dilutions are accounted for in the presented data. This test uses the same protocol as used in the previous Macrobatch testing.[6]

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

2.2 ESS Demonstration

For the ESS Demonstrations, filtrate from the MST Sorption Demonstration was used. Using this material, the researchers performed four ESS tests. All of the tests used the same general protocol as used in the previous Macrobatch testing.[6] The first test used a nominal starting volume of 90 mL of aqueous feed and 30 mL of fresh, unused MCU solvent for extraction.[♣] In the second test, the NGS solvent was used.[♦] In the third ESS test, the NGS-MCU solvent blend [♥] was used, where the MCU solvent was radiologically clean (unused, “Cold blend”). In the fourth ESS test, the NGS-MCU solvent blend (“Hot blend”) was used, where the MCU solvent blend was prepared from previously processed solvent from MCU. The second, third and fourth tests all used 80 mL of aqueous feed and 20 mL of solvent for extraction. For the first test, the scrub and strip solutions were 0.05M and 0.001 M nitric acid, respectively. For the other three tests, the scrub and strip solutions were 0.025 M NaOH and 0.01 M boric acid, respectively. In order to prevent cross-contamination of the different scrub and strip solutions, the first test was performed in a separate physical location than the other three tests. Confirmation by pH strip was also performed.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 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 were measured and tabulated. The samples of the clarified material were also measured for density, without any filtration. The results of the density measurements are listed in Table 1.

[♣] MCU solvent is composed of four components; 0.007 M BOBCalixC6 (Calix[4]arene-*bis*(*t*-octylbenzo-crown-6), 0.75M Cs-7SB Modifier (1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol), 0.003 trioctylamine (TOA), and the balance Isopar ™ L. This particular batch was from a prepared lot S2-D1-YESBOB-T-WI.

[♦] 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 give 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. In the case of the cold blend solvent, the MCU solvent was from batch S2-D1-YESBOB-T-WI. In the case of the hot blend solvent, the MCU solvent was from a composite of samples MCU-13-143/144/145/146/147/148.

Table 1. Sample Density Measurements (25 ºC)

Sample	Measured Density (g/mL)
HTF-21-13-79 (settled)	1.293
HTF-21-13-80 (settled)	1.260
HTF-21-13-81 (settled)	1.262
HTF-21-13-79 (filtered)	1.248
HTF-21-13-80 (filtered)	1.229
HTF-21-13-81 (filtered)	1.256
Average, settled (%RSD)	1.272 (1.45%)
Average, filtered (%RSD)	1.244 (1.11%)

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

The slight differences in the settled and filtered values are not statistically significant. In a previous document,[1] density, Inductively Coupled Plasma Emission Spectroscopy (ICPES), Ion Chromatography (IC) 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 IC results is 10%. The analytical uncertainty for the TIC/TOC results is 10%. The analytical uncertainty for the Free Hydroxide result 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 0.299 M. The free hydroxide converts to a pH of 14. The nickel (Ni) result converted into a concentration of $Ni(OH)_2$ is <3.27 mg/L.

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

Table 2. Previous Results

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.12	Sb	<34.5
Al	3320 (0.21%)	Si	74.2 (0.76%)
B	56.6 (0.13%)	Sn	<11.8
Ba	<0.62	Sr	<0.05
Be	<0.12	Th	<5.12
Ca	1.18 (0.60%)	Ti	<0.58
Cd	<0.84	U	<28.2
Ce	<6.45	V	<0.63
Cr	38.0 (0.19%)	Zn	4.90 (0.29%)
Cu	<0.98	Zr	<0.49
Fe	1.51 (5.62%)	F^-	<100
Gd	<1.38	Cl^-	264 (0.81%)
K	288 (1.47%)	Br^-	<1000
La	<1.26	Formate	649 (0.55%)
Li	21.9 (0.00%)	Nitrite	33000 (0.86%)
Mg	0.183 (5.81%)	Nitrate	148,000 (1.44%)
Mn	<0.16	Phosphate	556 (0.13%)
Mo	<5.99	Sulfate	9080 (0.23%)
Na	137,000 (0.52%)	Oxalate	392 (0.54%)
Ni	<2.07	TIC	3590 (0.59%)
P	212 (1.00%)	TOC	327 (0.43%)
Pb	<8.18	Free Hydroxide	1.93 (1.10%) M
S	3140 (1.58%)		

Values in parentheses are the RSD.

The bulk chemical characteristics of this batch are roughly similar to that of Salt batch 6.

3.1 Tank 21H Qualification Analyses

The tank samples were analyzed by Analytical Development (AD) by the listed non-radiological methods (Table 3) and radiological (Table 4) methods. Analyses were performed in duplicate and reported in Tables 5 and 6, respectively. Averages of the individual results, with the percent relative standard deviation (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
AA-As	As
AA-Se	Se
CV-Hg	Hg
HPLC	tetraphenylborate, phenol
SVOA	tributylphosphate
VOA	isopropanol, butanol, isobutanol

Table 4. Radiological Analyses

Method	Analyte
Tritium	^{3}H
^{14}C	^{14}C
gamma scan, Cs-removed	^{60}Co, ^{94}Nb, ^{106}Ru, ^{125}Sb, ^{126}Sn, ^{144}Ce, ^{154}Eu, ^{155}Eu, ^{241}Am, ^{226}Ra
^{90}Sr	^{90}Sr
^{129}I	^{129}I
gamma scan	^{134}Cs, ^{137}Cs
^{232}U	^{232}U
$^{238\text{-}241}Pu$ (filtered and unfiltered) (Plutonium thenoyl trifluoroacetone scintillation)	^{238}Pu, $^{239/40}Pu$, ^{241}Pu
Am/Cm	^{241}Am, ^{243}Am, ^{244}Cm, ^{245}Cm
$^{59/63}Ni$	$^{59/63}Ni$
^{99}Tc	^{99}Tc
$^{147}Pr/^{151}Sm$	$^{147}Pr/^{151}Sm$
ICPMS (Inductively Coupled Plasma Mass Spectroscopy)	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, tributyl phosphate, 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 results. Results shaded in green are calculated results. Values in parentheses are RSD.

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

Analyte	Result
ammonium	<100
wt % insoluble solids	0.0147 (53.6%) wt%
phenol	<10
tetraphenylborate	<10
tributylphosphate	<0.25
isopropanol	<0.25
butanol	<0.25
isobutanol	<0.25
methanol	< 125
As	0.181 (32.8%)
Hg	79.0 (19.5%)
Se	0.308 (0.00%)

Methanol is a calculated value.
Values in parentheses are the RSD.

SRNL was unable to meet the requested detection limit for ammonium and tetraphenylborate due to the high salt content of the samples.

The oxalate concentration is 392 mg/L, and the formate result is 649 mg/L. The oxalate result is converted to the equivalent carbon result of 173 mg/L. The formate result is converted to the equivalent carbon result of 107 mg/L. Subtracting these results from the TOC result gives a remainder of 47 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 125 mg/L. This methanol result should be considered an upper bound as no direct analytical method for methanol exists.

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 are 20%. Other analytical methods have varying uncertainties, typically 5-10% and are noted for single sample results.

^{90}Y is calculated as equal to the ^{90}Sr result. ^{106}Rh is calculated as equal to the ^{106}Ru result. The ^{125m}Te is calculated as equal to the ^{125}Sb result.^{137m}Ba is calculated as 94.7% of the ^{137}Cs result (as seen in Table 5).[7] ^{144}Pr is calculated as equal to the ^{144}Ce result. The ^{135}Cs result assumes that all of mass 135 from the ICPMS result is ^{135}Cs. The ^{232}Th result assumes that all of mass 232 from the ICPMS result is ^{232}Th. Total gamma is calculated as the sum of the ^{137}Cs, ^{134}Cs, ^{135}Cs, ^{60}Co, ^{94}Nb, ^{106}Ru, ^{125}Sb, ^{126}Sn, ^{144}Ce, ^{144}Pr, ^{154}Eu,

^{155}Eu, and ^{226}Ra. The ^{238}Pu, $^{239/40}$Pu, and ^{241}Pu results are from radio-counting, while the other Pu results are from ICPMS. The $^{239/40}$Pu value is not tainted by detection limits as the ICPMS ^{240}Pu, although care must be taken into assuming a correct 239/240 isotopic breakdown.

Table 6. Radiological Results of Tank 21H Analyses for Macrobatch 7

Analyte	Average Result	Analyte	Average Result
^{3}H	*5.28E+02 (14.1%)*	^{155}Eu	<4.73E+01
^{14}C	7.40E+02 (30.3%)	^{226}Ra	<6.71E+01
^{59}Ni	<2.03E+00	^{232}U	2.32E+00 (26.0%)
^{63}Ni	<3.15E+01	^{233}U	<9.68E+01
^{60}Co	<1.88E+00	^{234}U	9.66E+01 (18.8%)
^{90}Sr	2.61E+05 (7.33%)	^{235}U	4.19E-01 (0.00%)
^{90}Y	2.61E+05 (7.33%)	^{236}U	1.09E+00 (20.6%)
^{94}Nb	<1.03E+01	^{238}U	9.16E+00 (1.82%)
^{99}Tc	1.67E+04 (1.71%)	^{237}Np	<7.05E+00
^{106}Ru	<1.41E+02	^{238}Pu (unfiltered)	1.15E+04 (1.38%)
^{106}Rh	<1.41E+02	^{238}Pu (filtered)	1.20E+04 (3.18%)
^{125}Sb	<1.07E+02	^{239}Pu	*1.57E+03 (20%)*
^{125m}Te	<1.07E+02	^{240}Pu	<2.28E+03
^{126}Sn	1.32E+02 (9.88%)	$^{239/40}$Pu	6.62E+02 (4.81%)
^{129}I	1.21E+01 (6.86%)	^{241}Pu	2.21E+03 (3.02%)
^{134}Cs	<4.07E+03	^{242}Pu	<3.82E+01
^{135}Cs	2.47E+02 (12.9%)	^{244}Pu	<1.77E-01
^{137}Cs	4.61E+07 (2.07%)	^{241}Am	<2.53E+00
^{137m}Ba	4.37E+07 (2.07%)	^{243}Am	<7.16E-01
^{144}Ce	<1.16E+02	^{244}Cm	2.34E+00 (43.1%)
^{144}Pr	<1.16E+02	^{245}Cm	<1.87E+00
^{147}Pm	<2.71E+01	Total Alpha	<1.01E+05
^{151}Sm	<1.23E+01	Total Beta	9.18E+07 (0.69%)
^{154}Eu	<7.11E+00	Total Gamma	4.61E+07

Shaded results are calculated values. Values in parentheses are the RSD unless only a single result, then the value is the analytical uncertainty. Values in italics are single results.

Results given in italics indicate that one of the sample results was either below detection or quantification limits, 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 Strike

During the experiment, personnel collected samples from each of the two bottles at 0, 4, and 8 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. Additionally, the anion (IC Anions) and metal (ICPES) concentrations were tracked after the first scrub and first strip to better understand the chemistry occurring between these steps in the NGS, NGS-MCU blends, and MCU solvents. Samples were sent to Analytical Development (AD) with moderate dilution, and those dilutions are accounted for in the results section.

Compared to the MST test in Salt Batch 6,[8] the current test delivered better Pu removal, but worse 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 2 shows the graphical results for ^{238}Pu. The ^{238}Pu data is more useful than the $^{239/40}$Pu as the former is not limited by detection limit values. 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#1	**Experiment#2**	**Control**
0*	1.23E+04 (5.56%)	1.23E+04 (5.56%)	1.23E+04 (5.56%)
4	3.62E+03 (6.00%)	4.16E+03 (5.07%)	1.26E+04 (5.42%)
8	3.48E+03 (5.64%)	2.78E+03 (4.96%)	1.77E+04 (9.34%)

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

The elevated 8 hour control point is likely due to cross-contamination.

Figure 2. ^{238}Pu in Solution over Time for the MST Sorption Test

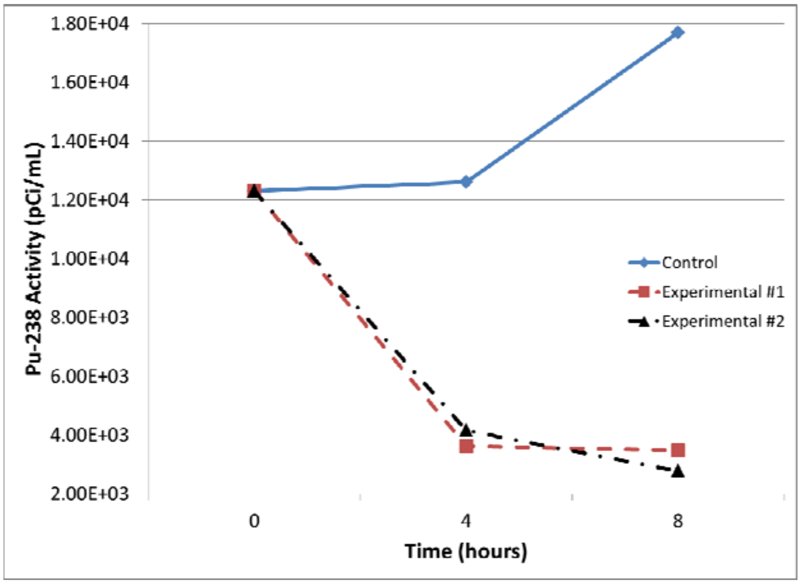


Table 8 lists the decontamination factors (DF) after the MST strike.⊕

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

Time (hours)	DF		
	Experiment#1	**Experiment#2**	**Control**
4	3.40	2.96	0.98
8	3.53	4.42	0.69

While there is only a limited amount of data related to Pu removal under the experimental conditions, the results 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 3 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.

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

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

Time (hours)	^{90}Sr (pCi/mL)		
	Experiment#1	**Experiment#2**	**Control**
0*	2.23E+05 (10.5%)	2.23E+05 (10.5%)	2.23E+05 (10.5%)
4	1.23E+04 (10.7%)	1.21E+04 (10.6%)	2.23E+05 (10.7%)
8	1.49E+04 (11.3%)	1.27E+04 (9.88%)	2.23E+05 (10.8%)

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

Figure 3. ^{90}Sr in Solution over Time for the MST Sorption Test

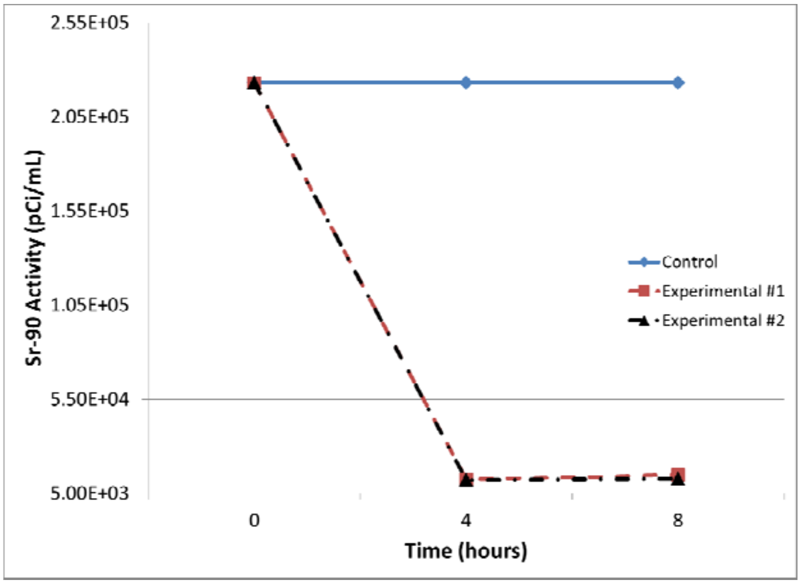


Table 10 lists the decontamination factors (DF) after the MST strike.

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

Time (hours)	DF		
	Experiment#1	**Experiment#2**	**Control**
4	18.2	18.5	1.00
8	15.0	17.5	1.00

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

3.4.3 Uranium Results

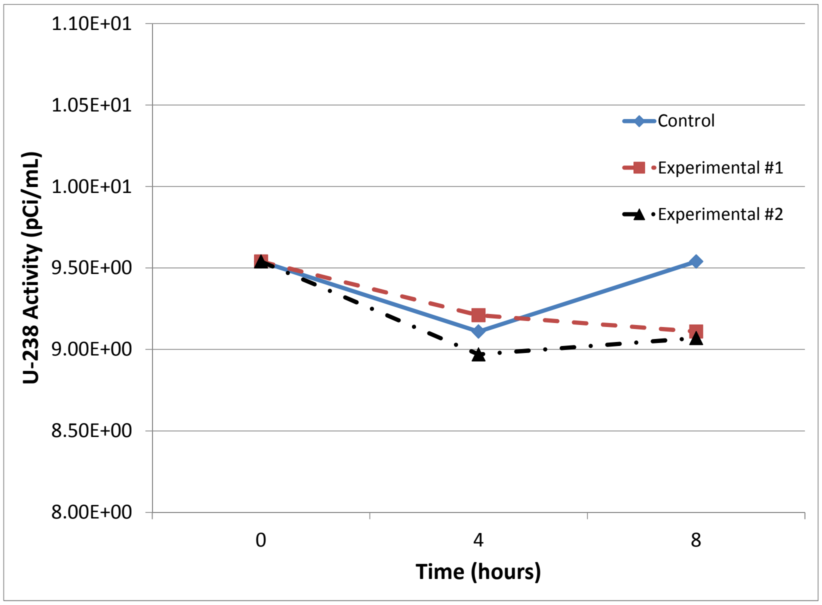
Researchers analyzed the filtered samples for ^{238}U. Table 11 shows the uranium results while Figure 4 shows the graphical results for ^{238}U. The values in parentheses in Table 11 are the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

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

Time (hours)	^{238}U (pCi/mL)		
	Experiment#1	**Experiment#2**	**Control**
0*	9.54E+00 (20%)	9.54E+00 (20%)	9.54E+00 (20%)
4	9.21E+00 (20%)	8.97E+00 (20%)	9.11E+00 (20%)
8	9.11E+00 (20%)	9.07E+00 (20%)	9.54E+00 (20%)

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

Figure 4. ^{238}U in Solution over Time for the MST Sorption Test



The small dip in the U graph is within the analytical uncertainty of the measurement and is not indicative of a drop in performance.

Table 12 lists the DF after the MST strike.

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

Time (hours)	DF		
	Experiment#1	**Experiment#2**	**Control**
4	1.00	1.03	1.02
8	1.01	1.02	1.00

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 was insufficient ^{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 the MCU solvent test, the temperature correction factors for the BOBCalix solvent formulation were used. For the other three 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 22.3 ºC to 26.0 ºC with an average temperature of 24.8 ºC. As a comparison, the results from the previous macrobatch qualification ESS test (using the same solvent) are displayed.[6]

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

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
Acceptable Range for BOBCalix Solvent	>8	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
S2-D1-YES BOB-T-WI, ISDP 6 (previous test)	9.14	2.72	0.800	0.0396	0.0184	0.0247
MCU Solvent[ϒ]	15.96	1.25	0.662	0.0365	0.0220	0.0204
Cold Blend (NGS)	57.1	4.45	1.74	0.0237	0.00053	0.00741
Hot Blend (NGS)	58.6	2.32	2.58	0.00057	0.00257	0.0111
NGS	65.6	4.05	1.82	0.00020	0.00103	0.00995

[ϒ] 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 four current tests show the expected behaviors, with good overall performance. For the MCU solvent test, the extraction, scrub and strip values are all comparable to a range of previous test results. For the other three tests, other than some differences in the strip results, the results were very similar. For example, strip#1 for the Cold Blend is somewhat higher than other strip#1 results for the NGS type experiments. This may be related to the elevated levels of sodium, nitrite and nitrite in the strip#1 solution (see Table 17), although the precise reason for this is unclear. On the other hand, the strip#2 result for the Cold Blend test is the best of all three NGS type tests. Despite the variations between the three NGS type strip results, all three NGS type tests give acceptable results.

From the bulk chemistry of the solution, an extraction $D_{(Cs)}$ of 13.2 is predicted for the MCU solvent.[9] The bulk chemical characteristics of this batch are roughly similar to that of Salt batch 6.[∉] The largest chemical differences that could affect the cesium distributions values are the potassium and nitrate. Salt Batch 6 had a higher potassium concentration (460 vs 288 mg/L) and a lower nitrate concentration (138500 vs. 148000 mg/L).

At this time we do not have a simple model to predict the extraction behavior of any solvent containing NGS or a blend containing NGS.

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 Results

Sample	**^{137}Cs activity (pCi/mL)**			
	MCU	Cold Blend	Hot Blend	NGS
Strip Effluent #1	2.01E+08	3.91E+08	4.77E+08	5.00E+08
Strip Effluent #2	6.85E+07	9.14E+07	2.86E+06	2.03E+06
Strip Effluent #3	1.58E+07	1.30E+06	2.62E+05	1.09E+05
DSS	6.71E+06	3.22E+06	3.12E+06	2.68E+06

The analytical uncertainty on the ^{137}Cs activity is 5%.

[∉] As a point of comparison, Salt Batch 6 had a predicted extraction distribution value of 13.7.

Table 15. Strip Effluent and DSS Results

Sample	pH			
	MCU	Cold Blend	Hot Blend	NGS
Strip Effluent #1	4	8	7	8
Strip Effluent #2	4	7	7	7
Strip Effluent #3	4	7	8	8
DSS	14	14	14	14

The analytical uncertainty is ±1 pH unit for the pH measurement. The differences in the strip pH values between the MCU solvent and the other three tests are due to the different scrub and strip solutions.

3.5.2 Anion and Metal Results

Researchers analyzed the aqueous portion of the first scrub and first strip samples using Ion chromatography for anions and inductively coupled plasma electron spectroscopy for metal concentrations. The intent lies in investigating any patterns or trends that appear in each test either in the presence or absence of an ion or metal that can be related to the efficacy of the test. The scrub#1 results are shown in Table 16 and the strip#1 results in Table 17.

Table 16. Anion and Metal Results for Scrub#1 Samples (mg/L)

Analyte	MCU Scrub #1	Cold Blend Scrub #1	Hot Blend Scrub #1	NGS Scrub #1
Nitrite	61	258	239	218
Nitrate	2090	1380	1310	1260
Sulfate	<10	37	35	26
Al	0.409	17.9	19.1	17.2
B	<0.08	0.292	0.236	<0.159
Ca	0.174	0.336	0.28	0.317
K	173	398	319	470
Li	0.132	0.205	0.24	0.146
Mg	0.0234	0.0174	0.0288	<0.008
Na	723	1940	1770	2290
Si	8.59	11.6	14.8	10.2

The analytical uncertainty is 10%.

There are some noticeable differences between the MCU solvent results (0.05 M nitric acid scrub) and the other three tests (0.025 M NaOH scrub). Nitrite is much lower in the

MCU solvent test. Nitrate in the MCU is elevated, undoubtedly due to the nitric acid scrub used in that test, although one would predict a nitrate concentration of 3100 mg/L (0.05 M).

Aluminum and sulfate are much higher in the non-MCU tests, although the reason for this is not known.

Potassium and sodium are elevated in the non-MCU tests, which must be due to the higher concentration of extractant, which is known to uptake these elements, and therefore, these results are not surprising.

Table 17. Anion and Metal Results for Strip#1 Samples (mg/L)

Analyte	MCU Strip #1	Cold Blend Strip #1	Hot Blend Strip #1	NGS Strip #1
Nitrite	<10	100	3	1
Nitrate	173	285	19	<10
Sulfate	<10	28	<10	<10
Al	<0.315	3.85	<1.23	<0.315
B	<0.08	105	103	104
Ca	0.298	0.34	0.54	0.178
K	<0.952	<4.16	<10.4	2.57
Li	<0.028	<0.076	<0.19	0.052
Mg	0.03	0.0444	0.101	0.0506
Na	4.51	477	36.1	17.4
Si	3.16	4.28	3.15	4.45

The analytical uncertainty is 10%.

Other than the boron values (from the strip boric acid), there are no obvious trends in the strip acid data. The sodium value in the cold blend is higher than expected, given the other results, but the reason is not known at this time.

4.0 Conclusions

Results of the analyses of the Tank 21H samples from this report in conjunction with the findings of the previous report,[1] indicates that the material does not display any unusual characteristics nor pose any concerns for processing or the batching strategy.

This report also covers the MST sorption and ESS results for the ISDP Salt Batch 7 feed sample. The following observations are made from the work.

- A demonstration of the monosodium titanate (0.2 g/L) removal of strontium and actinides provided acceptable 4 hour average decontamination factor for Pu and Sr of 3.22 and 18.4, respectively.

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

While no outstanding issues were noted, the presence of solids in the samples should be investigated in future work. It is possible that the solids may represent a potential reservoir of material (such as potassium) that could have an impact on MCU performance if they were to dissolve back into the feed solution.

In addition, SRNL recommends that a model for predicting extraction behavior for cesium removal for the blended solvent and NGS be developed.

Appendix A. Temperature Correction Factors for the ESS Tests

The actual MCU facility uses active temperature control to keep the extraction and scrub steps to 23 °C, and the strip steps to 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 18 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 18. Temperature Coefficients

Step	**MCU** [10]	**NGS** [11]
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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