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Analytical Results from final Salt Batch 9 Routine DSSHT and SEHT Monthly Samples

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EXECUTIVE SUMMARY

Strip Effluent Hold Tank (SEHT) and Decontaminated Salt Solution Hold Tank (DSSHT) samples from several of the “microbatches” of Integrated Salt Disposition Project (ISDP) Salt Batch (“Macrobatch”) 9 have been analyzed for ^{238}Pu, ^{90}Sr, ^{137}Cs, cations (Inductively Coupled Plasma Emission Spectroscopy - ICPES), and in some cases anions (Ion Chromatography Anions - IC-A).

These three samples are from Salt Batch 9 material in the Modular Caustic-Side Solvent Extraction Unit (MCU) system, and prior to actual Salt Batch 10 processing. These samples are known to contain contactor cleaning material and should not be considered typical for samples under operating conditions.

While the DSSHT sample is typical of this type of sample, the SEHT samples indicate a gross inclusion of nitric acid, likely from contactor cleaning solutions.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARP	Actinide Removal Process
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
IC-A	Ion chromatography - anions
ICPES	Inductively-coupled plasma emission spectroscopy
ISDP	Interim Salt Disposition Project
MCU	Modular Caustic-Side Solvent Extraction Unit
MST	Monosodium titanate
NGS	Next Generation Solvent
SE	Strip Effluent
SEHT	Strip Effluent Hold Tank
SRNL	Savannah River National Laboratory
TTQAP	Task Technical and Quality Assurance Plan

1.0 Introduction

During operation of the ISDP, quantities of salt waste are processed through the Actinide Removal Process (ARP) and MCU in batches of ~3,800 gallons. MCU uses solvent extraction technology to extract cesium from salt waste and concentrate cesium in an acidic aqueous stream (Strip Effluent – SE), leaving a decontaminated caustic salt aqueous stream (Decontaminated Salt Solution – DSS). Sampling occurs in the DSSHT and SEHT in the MCU process. The MCU sample plan requires that batches be sampled and analyzed on a quarterly frequency for plutonium and strontium content by the Savannah River National Laboratory (SRNL) to determine MST effectiveness.[i] A Task Technical and Quality Assurance Plan (TTQAP) were prepared to cover routine analyses.[ii] The cesium measurement is used to monitor cesium removal effectiveness while the ICPES and IC-A are used to monitor inorganic carryover.

A previous report provided the results of several sets of sample results from, and ending Macrobatch 8B operations.[iii] The sample results described in this report are from post Macrobatch 9 operations.

2.0 Experimental Procedure

The samples were contained in 10-mL P-nut vials. SEHT samples were delivered in doorstops for shielding purposes, while the DSSHT samples were delivered in “thief” holders. Samples of the same type were each composited into a single bottle. The SEHT samples were analyzed for ^{137}Cs, ^{238}Pu, and ^{90}Sr content, as well as for cation content (ICPES). The DSSHT samples were also analyzed for anion content (IC-A). The DSSHT samples were sent for analysis without dilution or filtration. SEHT samples were sent for analysis with dilution using deionized water only when necessary, but without filtration.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. For SRNL documents, the extent and type of review using the SRNL Technical Report Design Checklist is outlined in WSRC-IM-2002-00011, Rev. 2.[iv] Records for this work are contained in an electronic notebook ELN-A4571-00084-29.

3.0 Results and Discussion

3.1 Results from DSSHT and SEHT Samples

The ^{137}Cs, ^{90}Sr, and ^{238}Pu results from the DSSHT and SEHT radiochemical analyses are listed in Table 1. These samples were roughly monthly samples. Values in parentheses are the 1 sigma analytical uncertainties as provided by Analytical Development (AD). The source material (Tank 49H) entries were derived from customer blend documents for Salt Batch 9, and are used for comparison.[v]

Table 1. Radiochemical Results for the DSSHT and SEHT Samples

Sample ID	Sample Date	^{238}Pu (dpm/mL)	^{90}Sr (dpm/mL)	^{137}Cs (dpm/mL)
DSSHT Samples				
MCU-17-165/166/167	12/21/2017	2.78E+04 (6.2%)	6.90E+05 (20%)	2.40E+06 (5.0%)
SEHT Samples				
MCU-17-168/169/170	12/21/2017	3.83E+04 (7.3%)	1.36E+05 (20%)	6.11E+08 (5.0%)
MCU-18-7/8/9	1/25/2018	1.12E+04 (13%)	2.04E+05 (21%)	5.16E+08 (5.0%)
Source Material (Salt Batch 9) [v]		9.63E+04	9.70E+05	5.22E+08

Given that the December and January samples do not reflect typical batch processing, no comparison of Pu, Sr or Cs removal is made.

The meaningful (present in non-trace quantities) ICPES (B, Cr, Na) and IC-A (nitrite, nitrate, sulfate) results for the DSSHT and SEHT samples are listed in Table 2.

The material from Tank 49H undergoes a ~13 vol % dilution in ARP and MCU while no MST is in use.[vi] Therefore, direct comparisons between the source material and the DSSHT sample results should take this into account. Of the reported analytes in Table 2, B, Cr and Na are the analytes that are only subject to dilution effects in the ARP/MCU system – they are not affected by the solvent extraction, nor are they subject to solubility changes. These analytes are shaded in Table 2. In Table 2, the "% decline from feed concentration" row is the average of the three shaded analytes percentage decline compared to the value of their concentration in Salt Batch 9 feed. For example, for the MCU-17-165/166/167 sample, the three analytes are an average of 83% of their respective concentrations in the Salt Batch 9 feed. This is not atypical of DSSHT samples from past history. For the SEHT samples, the comparison to Salt Batch 9 feed is for reference as the SEHT samples should bear no resemblance to the feed. However, both SEHT samples show high Na, which is very atypical. Whenever SEHT samples have sodium higher than 50 ppm or so, this indicates some inflow of other material into the SEHT. The boron values are also ~15% of nominal value. There are two other data points associated with the SEHT samples that indicate this cross contamination. First, both SEHT samples show a pH of 1 by pH paper (0.01 M boric acid has an ideal pH of ~5.5). Second, the December SEHT sample was analyzed via IC-A, which gave all less-than detectable amounts of anions, except for nitrate, which measured 24,111 ppm (0.39 M). This points to a large inflow of nitric acid, which must be from contactor cleaning solution.

Table 2. ICPES Results for the DSSHT and SEHT Samples

Analyte	MCU-17/18-xxx Sample ID (mg/L)			
	Salt Batch 9[v]	MCU-17-165/166/167	MCU-17-168/169/170	MCU-18-7/8/9
Al	5860	4970	54.5	59.5
B	52.3	49.7	18.8	11.4
Cr	67.5	52	<2.86	<7.30
K	566	399	<53.6	166
Na	144000	109000	487	614
Si	21.0	68.7	9.12	<23.3
Zn	12.2	3.48	12.2	5.21
F	99.4	NM	<171	NM
Formate	189	NM	<171	NM
Cl	638	NM	<171	NM
Nitrite	32700	NM	<171	NM
Nitrate	109000	NM	24110	NM
Phosphate	469	NM	<171	NM
Sulfate	5630	NM	<171	NM
oxalate	407	NM	<171	NM
% decline from feed concentration	NA	17%	NM	NM

The analytical uncertainty for the ICPES and IC-A analyses is 10%.
NM indicated the analyte was not measured.

4.0 Conclusions

SEHT and DSSHT samples from several of the “microbatches” of ISDP Salt Batch (“Macrobatch”) 9 have been analyzed for ^{238}Pu, ^{90}Sr, ^{137}Cs, cations (ICPES), and anions (IC-A).

These three samples are from residual Salt Batch 9 material in the MCU system. Furthermore, these samples are known to contain contactor cleaning material and should not be considered typical for samples under operating conditions.

While the DSSHT sample is typical of this type of sample, the SEHT samples indicate a gross inclusion of nitric acid, likely from contactor cleaning solutions.

5.0 References

[i] M. W. Geeting, "Interim Salt Disposition Project (ISDP) Sample Plan", U-ESR-H-00068, Rev. 5, April 28, 2009.

[ii] T. B. Peters, A. L. Washington II, F. F. Fondeur, "Task Technical and Quality Assurance Plan for Routine Samples in Support of ARP and MCU", SRNL-RP-2013-00536, Rev. 1, May 2014.

[iii] T. B. Peters, "Analytical Results from Salt Batch 9 Routine DSSHT and SEHT Monthly Samples", SRNL-STI-2017-00330, Rev.0, June 2017.

[iv] Savannah River National Laboratory, "Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Rev. 2.

[v] C. I. Aponte, "Blend Evaluation for Tank 49 Feed for ISDP Salt Batch 9", X-ESR-H-00844, Rev.1, October 11, 2016.

[vi] A. G. Garrison, "Evaluation of Interim Salt Disposition Project (ISDP) Macrobatch Dilution Bases Experienced at Actinide Removal Process and Modular Caustic Solvent Extraction Unit (ARP/MCU)", X-ESR-H-00724, Rev. 1, May 28, 2015.

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