Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Analytical Results from the Salt Batch 10 Routine DSSHT and SEHT Monthly Samples

T. B. Peters

January 2019

SRNL-STI-2018-00708, Revision 0

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Prepared for
U.S. Department of Energy

Keywords: *MCU, NGS, DSS, SE*

Retention: *Permanent*

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REVIEWS AND APPROVALS

AUTHORS:

____

T. B. Peters, Advanced Characterization and Processing Date

TECHNICAL REVIEW:

____

L. E. Mathurin, Advanced Characterization and Processing,
Reviewed per E7 2.60 Date

APPROVAL:

____

B. J. Wiedenman, Manager Date
Advanced Characterization and Processing

____

S. D. Fink, Acting Director Date
Chemical Processing Technologies

____

E. A. Brass, Manager Date
MCU & Salt/Sludge Engineering

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") 10 have been analyzed for ^{238}Pu, ^{90}Sr, ^{137}Cs, elemental constituents (Inductively Coupled Plasma Emission Spectroscopy - ICPES), and in some cases anions (Ion Chromatography Anions - IC-A). The included tables contain the data.

These samples are from Salt Batch 10 material recently introduced into the Modular Caustic-Side Solvent Extraction Unit (MCU) system. The DSSHT results are typical for this type of sample, although the dilution due to processing is lower than expected. The August sample is known to have incorporated large amounts of rain water and cannot be compared to other DSSHT samples. The SEHT sample results are typical except for the May sample which is known to have rain water and Contactor Drain Tank (CDT) material in it.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARP	Actinide Removal Process
CDT	Contactor Drain Tank
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 Next Generation Solvent (NGS) 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 monosodium titanate (MST) effectiveness.[i] Even though MST is not currently used in the process, the analyses are still performed to provide other process monitoring data. A Task Technical and Quality Assurance Plan (TTQAP) was prepared to cover routine analyses.[ii] The cesium measurement is used to monitor cesium removal effectiveness while the ICPES and IC-A methods are used to monitor inorganic carryover.

A previous report provided the results of final sets of sample results from Macrobatch 9 operations.[iii] The sample results described in this report are from Macrobatch 10 operations.

2.0 Experimental Procedure

The samples were contained in 10-mL P-nut vials. The 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 elemental constituents (ICPES). The DSSHT samples were also analyzed for anion content (IC-A). The DSSHT samples were sent for analysis without dilution or filtration. The 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 electronic notebook ELN-A4571-00084-36.

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 collected at roughly monthly intervals. Values in parentheses are the one sigma analytical uncertainties as provided by Analytical Development (AD). The source material (Tank 49H) entries were derived from customer blend documents for Salt Batch 10 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-18-171/172/173	5/31/2018	7.43E+04 (6.9%)	6.57E+05 (14%)	9.98E+04 (5.0%)
MCU-18-227/228/229	6/19/2018	6.60E+04 (26%)	5.78E+05 (27%)	2.92E+05 (5.0%)
MCU-18-307/308/309	7/16/2018	7.24E+04 (6.7%)	7.49E+05 (16%)	4.57E+04 (5.0%)
MCU-18-364/365/366	8/21/2018	4.73E+04 (5.7%)	4.69E+05 (17%)	5.41E+05 (5.0%)
SEHT Samples				
MCU-18-177/178/179	5/31/2018	<1.67E+02	<1.54E+04	1.20E+09 (5.0%)
MCU-18-224/225/226	6/19/2018	<3.38E+02	<1.91E+04	7.05E+09 (5.0%)
MCU-18-301/302/303	7/16/2018	<2.10E+02	<1.53E+04	6.31E+09 (5.0%)
MCU-18-361/362/363	8/23/2018	1.22E+02 (55%)	<1.98E+04	2.42E+09 (5.0%)
Source Material (Salt Batch 10) [v]		6.77E+04	8.17E+05	3.95E+08

The August DSSHT sample is known to contain a large amount of rain water and therefore shows a decline in Pu and Sr concentrations. Variations in the Cs decontamination factors are likely masking the corresponding decline in Cs values in the DSSHT. Given that most of these samples were pulled during periods of operational issues with the strip effluent coalescer, where flushing was required in some instances to remove the media, comparisons to previous salt batch performances are not valid.

The meaningful (present in non-trace quantities) ICPES and IC-A results for the DSSHT 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 dilution into account. Of the reported analytes in Table 2, B, Cr, Na, nitrate, nitrite, and sulfate (shaded in the table) 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 shaded analytes percentage decline compared to the value of their concentration in Salt Batch 10 feed. For example, for the MCU-18-227/228/229 sample, the three analytes exhibit an average decline of 2.3% from their respective concentrations in the Salt Batch 10 feed. Typically, the DSSHT samples reflect this overall dilution, but in this case, only the August DSSHT sample (which is known to contain large quantities of rain water) shows a meaningful dilution. This lack of dilution cannot be easily explained. On average, the Salt Batch 9 material is ~0-10% higher in the six shaded analytes than for Salt Batch 10. Feeding from stratified SB9 material would then tend to bias the perceived dilution value low, to the same extent.

Table 2. ICPES and IC-A Results for the DSSHT Samples

Analyte		MCU-18-xxx Sample ID (mg/L)			
	Salt Batch 10[v]	171/172/173	227/228/229	307/308/309	364/365/366
Al	6260	5810	5150	5660	4870
B	55.9	51.1	55.4	61.2	45
Cr	59.2	61.9	63.8	67.1	54.6
K	491	392	424	480	345
Na	141000	133000	120000	127000	103000
Si	21.2	57	165	183	67.1
Zn	3.19	6.64	10.9	6.22	4.22
F	98.8	<100	<100	NM	<100
Formate	222	121	<100	NM	91.1
Cl	674	628	587	NM	520
Nitrite	33300	30600	30453	NM	26500
Nitrate	99800	116000	101542	NM	86400
Phosphate	389	329	429	NM	357
Sulfate	4940	5730	4991	NM	4330
Oxalate	410	404	349	NM	318
Avg % relative to feed concentration	NA	102%	97.7%	104%	83.3%

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

For the SEHT samples analysis (see Table 3), the comparison to Salt Batch 10 feed is for reference as the SEHT samples should bear no resemblance to the feed. The boron values are also ~85-100% of nominal value (108 mg/L), other than for the -177/178/179 sample which is due to incorporation of rain water and CDT material. The other analyte sample results are not unusual.

Table 3. ICPES Results for the SEHT Samples

Analyte		MCU-18-xxx Sample ID (mg/L)			
	Salt Batch 10[v]	177/178/179	224/225/226	301/302/303	361/362/363
Al	6260	<3.46	<5.08	<3.608	<3.05
B	55.9	17.1	89.5	89.7	107
K	491	<25.4	38.0	<26.5	<21.7
Na	141000	18.7	62.7	56.8	51.9

4.0 Conclusions

SEHT and DSSHT samples from several of the "microbatches" of ISDP Salt Batch ("Macrobatch") 10 have been analyzed for ^{238}Pu, ^{90}Sr, ^{137}Cs, elemental constituents (ICPES), and anions (IC-A).

These samples are from Salt Batch 10 material recently introduced into the MCU system. The DSSHT results are typical for this type of sample, although the dilution due to processing is lower than expected. The August sample is known to have incorporated large amounts of rain water and cannot be compared to other DSSHT samples. The SEHT sample results are typical except for the May sample which is known to have rain water and CDT material in it.

5.0 References

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Distribution:

timothy.brown@srnl.doe.gov
alex.cozzi@srnl.doe.gov
david.crowley@srnl.doe.gov
a.fellinger@srnl.doe.gov
samuel.fink@srnl.doe.gov
connie.herman@srnl.doe.gov
frank.pennebaker@srnl.doe.gov
william.ramsey@SRNL.DOE.gov
boyd.wiedenman@srnl.doe.gov
bill.wilmarth@srnl.doe.gov
charles.nash@srnl.doe.gov
jeffrey.crenshaw@srs.gov
james.folk@srs.gov
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patrick.jackson@srs.gov
tony.polk@srs.gov
jean.ridley@srs.gov
patricia.suggs@srs.gov
earl.brass@srs.gov
phoebe.fogelman@srs.gov
brent.gifford@srs.gov
vijay.jain@srs.gov
john.occhipinti@srs.gov
david.sherburne@srs.gov
keith.harp@srs.gov
christopher.weston@srs.gov
eric.freed@srs.gov
ryan.mcnew@srs.gov
Christina.Santos@srs.gov
Azadeh.Samadi-Dezfouli@srs.gov
kenneth.fernandez@srs.gov
michael.norton@srs.gov
Records Administration (EDWS)