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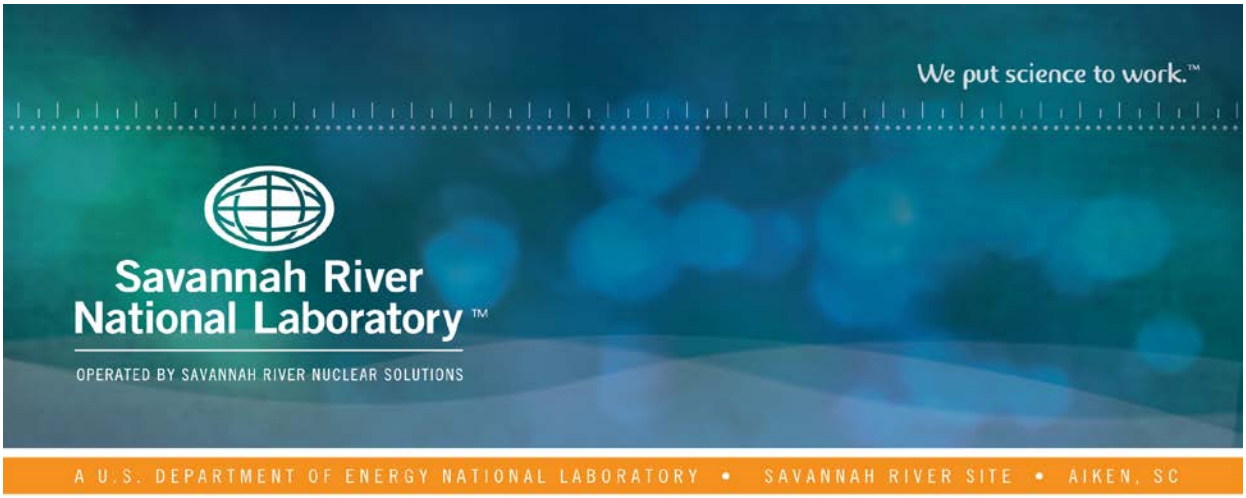

Results of Initial Analyses of the Salt (Macro) Batch 10 Tank 21H Qualification Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Interim Salt Disposition Project (ISDP) Salt (Macro) Batch 10 for processing through the Actinide Removal Process (ARP) and the Modular Caustic-Side Solvent Extraction Unit (MCU). This document reports the initial results of the analyses of samples of Tank 21H. Analysis of the Tank 21H Salt (Macro) Batch 10 composite sample indicates that the material does not display any unusual characteristics or observations, such as floating solids, the presence of large amount of solids, or unusual colors. Further sample results will be reported in a future document. This memo satisfies part of Deliverable 3 of the Technical Task Request (TTR).

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARP	Actinide Removal Process
IC	Ion Chromatography
ICPES	Inductively-Coupled Plasma Emission Spectroscopy
ISDP	Interim Salt Disposition Project
MCU	Modular Caustic-Side Solvent Extraction Unit
MST	Monosodium titanate
%RSD	Percent Relative Standard Deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance Plan
WAC	Waste Acceptance Criteria

1.0 Introduction

This report provides initial analytical laboratory results of Salt (Macro) Batch 10 samples from Tank 21H. These results will be used by Savannah River Remediation (SRR) to (1) perform salt batch OLI model simulations (OLI Systems, Inc., Cedar Knolls, NJ) to determine if additional caustic needs to be added to the batch and (2) determine if a Monosodium Titanate (MST) strike is required for this salt batch to meet downstream Waste Acceptance Criteria (WAC). This work was specified by a TTR[i] and Task Technical and Quality Assurance Plan (TTQAP).[ii] Details for the work are contained in controlled laboratory notebooks.[iii]

2.0 Experimental Procedure

Two 200 mL Tank 21H samples (HTF-21-16-104 and -105) and a single 1L Tank 21H sample (HTF-21-16-106) were pulled and delivered to SRNL on November 21, 2016. The two 200 mL samples were surface samples and the 1L sample was a variable depth sample obtained approximately 62" from the bottom of the tank (transfer pump suction). Tank 21H was mixed at full speed for approximately 15 hours with one pump before the samples were pulled; the samples were pulled approximately 28 hours after pump shutdown. All the samples had the same visual appearance, clear solutions with no apparent solids.

The density of filtered solution (using a 0.45 μm syringe filter) from each sample was measured twice and reported in Table 1. With SRR concurrence, the contents of the three sample bottles were then combined (without filtering) and mixed. After compositing and allowing the contents of the composite bottle to sit for 10 days, it was found that a very fine layer of fine off-white solids had settled to the bottom of the composite bottle. Duplicate filtered samples (using a 0.45 μm syringe filter) were sent to Analytical Development (AD) for analysis. The one exception is the Hg (unfiltered) result in Table 4. In this case, a well-mixed sample from the composite bottle was removed for analysis with no filtration. None of the samples were diluted before delivery to AD.

2.1 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. Results from this report are not RW-0333P as per the TTR.

3.0 Results and Discussion

The results of the density measurements are listed in Table 1. Values in parentheses are the percent relative standard deviation (%RSD) values.

Table 1. Sample Density Measurements (20.0 ºC)

Sample	Measured Density (g/mL)
HTF-21-16-104	1.253 (0.73%)
HTF-21-16-105	1.257 (0.17%)
HTF-21-16-106	1.253 (0.34%)
Average (%RSD)	1.254 (0.20%)

The analytical uncertainty is typically <1% for density measurements. The results are typical for dissolved saltcake of this type. For comparison, the average density of the Salt Batch 9 solution was 1.250 g/mL (24.9 °C).[iv]

The results of the Inductively-Coupled Plasma Emission Spectroscopy (ICPES) analysis are listed in Table 2. The values in the parentheses are the %RSD.

Table 2. ICPES Results[ɣ]

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.53	Mo	13.6 (1.0%)
Al	6770 (0.84%)	Na	140000 (0.51%)
B	60.5 (0.23%)	Ni	<6.04
Ba	<0.116	P	134 (1.1%)
Be	<0.0483	Pb	<20.7
Ca	<1.24	S	1690 (0.42%)
Cd	<1.5	Sb	<42.7
Ce	<4.66	Si	21.7 (4.3%)
Cr	52.0 (0.27%)	Sn	<13.1
Cu	<5.46	Sr	<0.0422
Fe	<2.36	Th	<9.93
Gd	<1.15	Ti	<8.96
K	425 (4.7%)	U	<43.9
La	<1.17	V	<0.7
Li	11.8 (1.2%)	Zn	1.79 (3.6%)
Mg	<0.283	Zr	<0.707
Mn	<0.21		

ICPES analytical uncertainty is 10%.

[ɣ] While many of the analytes listed in Table 2 were not requested in the TTR, they have been included for the sake of completeness.

The ICPES results do not vary greatly from historical data. The sodium concentration of 140000 mg/L (6.09 M) is within past operating concentrations.

Results from the Ion Chromatography (IC) Anions, Free Hydroxide Titration, and Total Inorganic Carbon (TIC)/Total Organic Carbon (TOC) analysis are listed in Table 3. The values in the parentheses are the %RSD.

Table 3. IC Anions, Free Hydroxide Titration, and TIC/TOC Results ♦

Analyte	Result (mg/L)
F^-	<100
Cl^-	722 (0.29%)
Br^-	<500
Formate	258 (0.27%)
Nitrite	34600 (3.7%)
Nitrate	92200 (2.0%)
Phosphate	315 (1.4%)
Sulfate	4350 (2.3%)
Oxalate	420 (0.51%)
TIC	3400 (0.0%)
TOC	238 (3.6%)
Free Hydroxide	2.71 (0.26%) M

The analytical uncertainty for IC, TIC/TOC, and Free Hydroxide results are 10%.

The oxalate value of 420 mg/L is typical of the salt batches prepared to date.

The TIC and TOC results are in terms of mg/L of carbon. Assuming the entire TIC result is carbonate, this translates to a carbonate concentration of 0.283 M.

As per the latest revision of the TTR, the ^{137}Cs, ^{238}Pu, $^{239/40}Pu$, ^{241}Pu, ^{90}Sr, total alpha, and Hg results have been added to this initial set of results. These are reported in Table 4. In the case of the Hg analyses, there are two results – one for filtered samples, and one for unfiltered samples. The total alpha result is from samples that had the cesium removed in order to provide for superior detection limits. The values in the parentheses are the %RSD.

♦ While the bromide analyte listed in Table 3 was not requested in the TTR, it has been included for the sake of completeness.

Table 4. Radiochemistry and Hg Results

Analyte	Result
^{137}Cs	1.24E+08 pCi/mL (2.3%)
^{238}Pu	1.83E+04 pCi/mL (19%)
$^{239/40}$Pu	8.37E+02 pCi/mL (11%)
^{241}Pu	5.63E+03 pCi/mL (17%)
^{90}Sr	3.06E+05 pCi/mL (13%)
Total alpha [⊗]	<2.36E+04 pCi/mL
Hg (filtered)	101 mg/L (2.8%)
Hg (unfiltered)	112 mg/L (2.5%)

The ^{137}Cs result is lower than Salt Batch 9 (2.44 E+08 pCi/mL). The ^{137}Cs result for Salt Batch 10 converts to 0.47 Ci Cs/gal. Other radiochemical results are typical of previous salt batches.

The trivial difference between the Hg filtered and unfiltered results indicates that the presence of the solids does not statistically differentiate the filtered and unfiltered Hg results.

4.0 Conclusions

Analysis of the Tank 21H Salt (Macro) Batch 10 composite sample indicates that the material does not display any unusual characteristics or observations, such as floating solids, the presence of large amount of solids, or unusual colors. Further sample results will be reported in a future document. This memo satisfies part of Deliverable 3 of the Technical Task Request (TTR).

[⊗] In this case, the value is the measurement with ^{137}Cs having been removed.

5.0 References

[i] A. Samadi-Dezfouli, "Salt Batch Qualification for Feed to the Interim Salt Disposition Project (ISDP)", X-TTR-H-00068, Rev. 0, October 26, 2016.

[ii] T. B. Peters and D. H. Jones, "Task Technical and Quality Assurance Plan for Qualification of Salt Batches for Feed to ISDP", SRNL-RP-2015-00704, Rev. 1, November 2016.

[iii] T. B. Peters, "Salt Batch 10 Qualification", ELN, A4571-00084-27.

[iv] T. B. Peters, "Results of Initial Analyses of the Salt (Macro) Batch 9 Tank 21H Qualification Samples", SRNL-STI-2015-00513, October 2015.

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