



Results of Initial Analyses of the Salt (Macro) Batch 8 Tank 21H Qualification Samples

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October 2014

SRNL-STI-2014-00472, Revision 0



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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *MCU, ARP, Cesium*

Retention: *Permanent*

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Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Salt (Macro) Batch 8 for the Interim Salt Disposition Program (ISDP) 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. Further results on the chemistry and other tests will be issued in the future. No issues with the projected Salt (Macro) Batch 8 strategy are identified.

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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 Program
MCU	Modular Caustic Side Solvent Extraction Unit
%RSD	Percent Relative Standard Deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon
TTR	Task Technical Request
TTQAP	Task Technical And Quality Assurance Plan

1.0 Introduction

This report provides initial analytical laboratory results of Salt (Macro) Batch 8 samples from Tank 21H. These results will be used by Savannah River Remediation (SRR) to perform salt batch OLI model simulations. This work was specified by Technical Task Request (TTR)ⁱ and by Task Technical and Quality Assurance Plan (TTQAP).ⁱⁱ Details for the work are contained in controlled laboratory notebooks.ⁱⁱⁱ

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). The slurry pumps were run for approximately 22 hours, and the samples were pulled approximately 48 hours after the slurry pumps were shutdown. All the samples had the same visual appearance, clear solutions with no solids. While the previous salt batch samples contained solids, apparently the mixing program and timing for this set of samples precluded capturing these solids if they were present at all.

The density of filtered solution (using a 0.45 μm syringe filter) from each sample was measured and reported in Table 1. With SRR 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. The samples were not 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.

Table 1. Sample Density Measurements (23° C)

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 results are typical for dissolved saltcake of this type. For comparison, the average density of the Salt Batch 7 solution (settled) was 1.272 g/mL.

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

Table 2. ICPES Results

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.94	Mo	24.9 (1.42%)
Al	5410 (0.00%)	Na	142000 (0.50%)
B	70.8 (0.70%)	Ni	<4.94
Ba	<1.00	P	231 (0.92%)
Be	<0.14	Pb	<130
Ca	1.25 (0.00%)	S	2490 (0.85%)
Cd	<1.27	Sb	<41.1
Ce	<11.2	Si	57.4 (1.23%)
Cr	72.4 (0.68%)	Sn	<93.1
Cu	<3.54	Sr	<12.8
Fe	<1.39	Th	<11.6
Gd	<4.44	Ti	<0.93
K	643 (7.48%)	U	<69.9
La	<1.97	V	<0.69
Li	19.5 (3.27%)	Zn	5.13 (1.24%)
Mg	<8.61	Zr	<0.62
Mn	<0.8		

ICPES analytical uncertainty is 10%. The values in the parentheses are the percent relative standard deviation (%RSD).

The ICPES results are largely as expected, with the concentrations of the various analytes not varying greatly from historical precedent. 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.^{iv} However, it is possible that a slight decline in the cesium distribution factor [$D_{(Cs)}$] may occur, as this value is outside the historical precedent with real waste. The sodium concentration of 142000 mg/L (6.17 M) is within past operating concentrations.

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

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

Analyte	Result (mg/L)
F ⁻	<100
Cl ⁻	401 (0.35%)
Br ⁻	<500
Formate	468 (0.76%)
Nitrite	38400 (6.06%)
Nitrate	122000 (0.53%)
Phosphate	545 (5.58%)
Sulfate	5270 (3.36%)
Oxalate	187 (7.96%)
TIC	3300 (0.64%)
TOC	216 (0.00%)
Free Hydroxide	2.24 M (0.63%)

The analytical uncertainty for IC, TIC/TOC, and Free Hydroxide results is 10%. The values in the parentheses are the %RSD.

The sulfate result from IC (5270 mg/L) does not corroborate particularly well with the sulfur result from the ICPES (2490 mg/L – the sulfur value should theoretically be 1/3 of the sulfate), but this is typical in salt solutions and has been encountered before. The oxalate value of 187 mg/L is one of the lowest values in 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 0.275 M.

Upon customer request, the ^{137}Cs value is reported here. The ^{137}Cs was measured to be $2.13\text{E}+08$ pCi/mL with a %RSD of 2.24%. This value is approximately 4.6 times higher than the previous salt batch (Salt Batch 7 feed was analyzed to be $4.61\text{E}+07$ pCi/mL), and was an expected result of increased ^{137}Cs in the batch recipe.

4.0 Conclusions

Analysis of the Tank 21H Salt (Macro) Batch 8 composite sample indicates that the material does not display any unusual characteristics. Further sample results will be reported in a future document.

5.0 References

- ⁱ A. R. Shafer, TTR “Technical Task Request – Qualification of ISDP Salt Batch 8”, X-TTR-H-00047, Rev. 0, June 30, 2014.
- ⁱⁱ 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.
- ⁱⁱⁱ T. B. Peters, “Salt Batch 8 Qualification”, ELN, A4571-00084-13.
- ^{iv} T. B. Peters, A. L. Washington II, S. D. Fink, “Results of the Extraction-Scrub-Strip Testing Using an Improved Solvent Formulation and Salt Waste Processing Facility Simulated Waste”, SRNL-STI-2011-00689, January 2012.

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