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Results of Initial Analyses of the Salt (Macro) Batch 9 Tank 21H Qualification Samples

T. B. Peters

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REVIEWS AND APPROVALS

AUTHORS:

T. B. Peters, Author, Advanced Characterization and Processing Date

TECHNICAL REVIEW:

C. A. Nash, Technical Reviewer, Advanced Characterization and Processing Date
Reviewed per E7 2.60

APPROVAL:

F. M. Pennebaker, Manager Date
Advanced Characterization and Processing

A. P. Fellinger, Manager Date
Environmental & Chemical Process Technology Research Programs

E. J. Freed, Manager Date
DWPF and Saltstone Facility Engineering

J. S. Contardi, Manager Date
Tank Farm Facility Engineering

R. E. Edwards, Manager Date
Nuclear Safety and Engineering Integration

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 9 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 9 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 results on the chemistry and other tests will be issued in the future.

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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 9 samples from Tank 21H. These results will be used by Savannah River Remediation (SRR) to (1) perform salt batch OLI model simulations 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 Technical Task Request (TTR)[i] and Task Technical and Quality Assurance Plan (TTQAP).[ii] Details for the work are contained in controlled laboratory notebooks.[iii]

Revision 1 documents the analyses needed to determine if an MST strike is required for this salt batch; these analyses were not available at the time Revision 0 of this report was issued.

2.0 Experimental Procedure

Two 200 mL Tank 21H samples (HTF-21-15-106 and -107) and a single 1L Tank 21H sample (HTF-21-15-108) were pulled and delivered to SRNL on September 1, 2015. 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 and mixed. After compositing and allowing the contents of the composite bottle to sit for several 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 (24.9 ºC)

Sample	Measured Density (g/mL)
HTF-21-15-106	1.251 (0.25%)
HTF-21-15-107	1.246 (0.99%)
HTF-21-15-108	1.254 (0.17%)
Average (%RSD)	1.250 (0.32%)

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 8 solution was 1.257 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	<1.17	Mo	25.7 (0.83%)
Al	6010 (0.47%)	Na	144000 (0.49%)
B	48.7 (0.15%)	Ni	<4.18
Ba	0.12 (0.0%)	P	198 (1.4%)
Be	<0.106	Pb	<17.1
Ca	1.04 (20%)	S	1930 (0.73%)
Cd	<1.60	Sb	<34.8
Ce	<4.61	Si	12.9 (1.1%)
Cr	67.2 (0.11%)	Sn	<22.1
Cu	<1.17	Sr	<0.094
Fe	4.21 (3.0%)	Th	<6.27
Gd	<1.44	Ti	<9.35
K	558 (1.7%)	U	<51.3
La	1.12 (8.2%)	V	1.19 (1.8%)
Li	<8.86	Zn	4.56 (0.62%)
Mg	<0.129	Zr	<0.412
Mn	<0.419		

ICPES analytical uncertainty is 10%. The values in the parentheses are the %RSD.

The ICPES results are largely as expected, with the concentrations of the various analytes not varying greatly from historical precedent. The sodium concentration of 144000 mg/L (6.24 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^-	696 (0.9%)
Br^-	<500
Formate	127 (0.6%)
Nitrite	31700 (1.3%)
Nitrate	106000 (1.3%)
Phosphate	455 (0.9%)
Sulfate	5660 (3.5%)
Oxalate	453 (9.2%)
TIC	2920 (0.24%)
TOC	184 (1.2%)
Free Hydroxide	2.52 (0.0%) M

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

The phosphate result from IC (455 mg/L) does not corroborate particularly well with the phosphorus result from the ICPES (198 mg/L – the phosphorus value should theoretically be ~1/3 of the phosphate), but this is not unusual in salt solutions and has been encountered before. The oxalate value of 453 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 0.243 M.

Upon customer request, 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.

Table 4. Radiochemistry and Hg Results

Analyte	Result
^{137}Cs	2.44E+08 (0.3%) pCi/mL
^{238}Pu	4.16E+04 (13%) pCi/mL
$^{239/40}$Pu	8.72E+02 (7.7%) pCi/mL
^{241}Pu	<1.70E+04 pCi/mL
^{90}Sr	4.31E+05 (1.4%) pCi/mL
Total alpha ⊗	<3.43E+04 pCi/mL
Hg (filtered)	62.8 (3.0%) mg/L
Hg (unfiltered)	58.5 (12%) mg/L

The ^{137}Cs result is slightly higher than the previous salt batch (2.13E+08 pCi/mL). Other radiochemical results are typical of previous salt batches. The total alpha result is lower than the ^{238}Pu alone. The total alpha method is generally less precise than the individual method results.

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 9 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.

⊗ In this case, the ^{238}Pu result plus the %RSD is a better measure of the total alpha.

5.0 References

[i] M. A. Rios-Armstrong, TTR "Technical Task Request – Salt Batch Qualification for Feed to the Interim Salt Disposition Project (ISDP)", X-TTR-H-00059, Rev. 0, August 24, 2015.

[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. 0, September 2015.

[iii] T. B. Peters, "Salt Batch 9 Qualification", ELN, A4571-00084-23.

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