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Results of Initial Analyses of the Macrobatch 7 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 Salt (Macro) Batch 7 for the Interim Salt Disposition Program (ISDP) through ARP/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 Batch 7 strategy are identified, other than the presence of visible quantities of dark colored solids. Based upon a SRNL settling test, the solids should settle well within the months-long settling period to be employed in Tank 21H. However, SRNL recommends analyzing the solids to provide input to OLI modeling in order to evaluate the impacts of these solids to present and future salt batches.

LIST OF ABBREVIATIONS

AD	Analytical Development
ARP	Actinide Removal Process
ESS	Extraction Scrub Strip
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 describes the laboratory results of Salt (Macro) Batch 7 preliminary samples from Tank 21H. These results will be used by SRR for their blend calculations. This work was specified by Technical Task Request (TTR)[1] and by Task Technical and Quality Assurance Plan (TTQAP).[2]

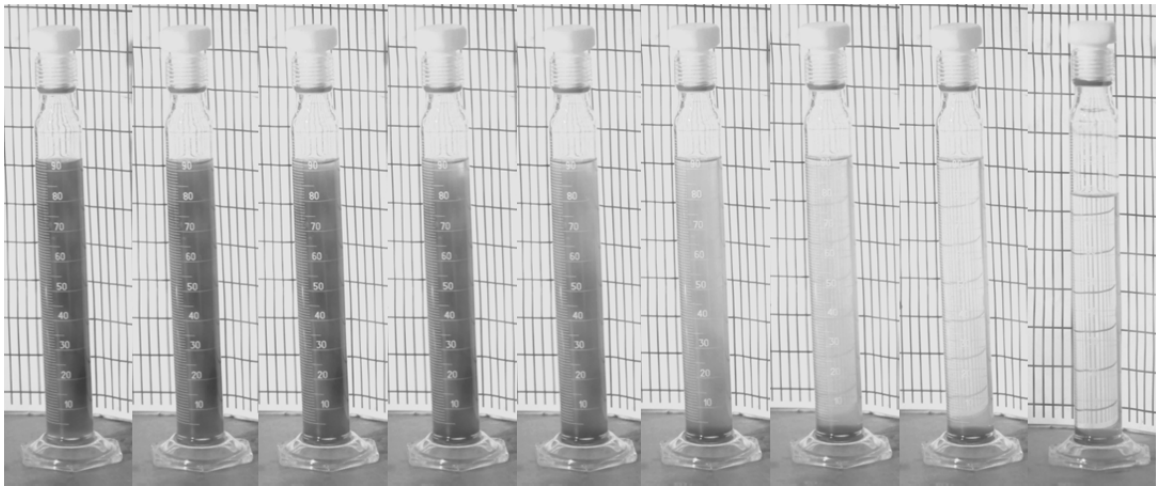
Details for the work are contained in controlled laboratory notebooks.[3]

Further analytical results, as well as results from the Actinide Removal Process (ARP) and Extraction-Scrub-Strip (ESS) testing will be described in future reports. From all of these results, SRNL will evaluate the impacts on the solvent and determine whether or not the feed is acceptable for use at MCU.

2.0 Experimental Procedure

Three Tank 21H samples (i.e., dip sample bottles HTF-21-13-79 and HTF-21-13-80, and a 2 L sample HTF-21-13-81) arrived at SRNL on May 16, 2013. The samples each contained visible quantities of dark colored solids, which is not typical. The density of filtered samples (using a 0.45 μm syringe filter) from each sample were measured and reported in Table 1. The next day, samples of the clarified material were measured for density, with settling and filtration used to remove the solids. These results are reported in Table 1. 10 mL well-mixed samples from each of the three sample bottles were then removed for archival purposes. A well-mixed portion of material from HTF-21-13-81 was placed in a 100 mL graduated cylinder. Over a period of 29 hours, the contents were allowed to settle and multiple pictures were taken. See Figure 1.

Figure 1. Pictures of Settling Tank 21H Sample



Within 2 hours, there was visible stratification in the sample. After 8 hours, the sample was mostly settled, and by 29 hours, the sample had visibly completely clarified. Please note that the missing supernate in the last picture (t=1760) is due to a sample having been removed for analytical purposes. Sampling after 29 hours sufficiently represents the projected settling in Tank 21 based on an evaluation performed by SRR.[4]

With SRR concurrence, the contents of the three sample bottles (HTF-21-13-79, 80, 81) 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. 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 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.[5]

3.0 Results and Discussion

The results of the density measurements are listed in Table 1.

Table 1. Sample Density Measurements (25 ºC)

Sample	Measured Density (g/mL)
HTF-21-13-79 (settled)	1.293
HTF-21-13-80 (settled)	1.260
HTF-21-13-81 (settled)	1.262
HTF-21-13-79 (filtered)	1.248
HTF-21-13-80 (filtered)	1.229
HTF-21-13-81 (filtered)	1.256
Average, settled (%RSD)	1.272 (1.45%)
Average, filtered (%RSD)	1.244 (1.11%)

The analytical uncertainty is typically <1% for density measurements.

The results of the ICPES analysis are listed in Table 2.

Table 2. ICPES Results

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.12	Mo	<5.99
Al	3320 (0.21%)	Na	137,000 (0.52%)
B	56.6 (0.13%)	Ni	<2.07
Ba	<0.62	P	212 (1.00%)
Be	<0.12	Pb	<8.18
Ca	1.18 (0.60%)	S	3140 (1.58%)
Cd	<0.84	Sb	<34.5
Ce	<6.45	Si	74.2 (0.76%)
Cr	38.0 (0.19%)	Sn	<11.8
Cu	<0.98	Sr	<0.05
Fe	1.51 (5.62%)	Th	<5.12
Gd	<1.38	Ti	<0.58
K	288 (1.47%)	U	<28.2
La	<1.26	V	<0.63
Li	21.9 (0.00%)	Zn	4.90 (0.29%)
Mg	0.183 (5.81%)	Zr	<0.49
Mn	<0.16		

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

Results from the IC Anions, Free Hydroxide, and TIC/TOC are listed in Table 3.

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

Analyte	Result (mg/L)
F^-	<100
Cl^-	264 (0.81%)
Br^-	<1000
Formate	649 (0.55%)
Nitrite	33000 (0.86%)
Nitrate	148,000 (1.44%)
Phosphate	556 (0.13%)
Sulfate	9080 (0.23%)
Oxalate	392 (0.54%)
TIC	3590 (0.59%)
TOC	327 (0.43%)
Free Hydroxide	1.93 M (1.10%)

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

The TIC and TOC results are in terms of mg/L of carbon. If we assume that the entire TIC result is carbonate, this translates to a carbonate concentration of 0.299 M.

4.0 Conclusions

Analysis of the Tank 21H sample indicates that the material does not display any unusual characteristics, other than the presence of the dark colored solids. The solids were found to settle cleanly in a short time period (~1 day or less), and were not disturbed by decanting. Further sample results will be reported in a future document.

SRNL recommends analyzing the solids to determine the physical makeup. While the solids themselves would be filtered off at ARP, there remains the possibility that the solids represent a feedstock of material that could be leached or dissolved in Salt Batch 7 or future salt batches, and thus provide an upset to the MCU operations.

5.0 References

[1] S. E. Campbell, TTR "Technical Task Request – Qualification of ISDP Salt Batch 7", HLW-DWPF-TTR-2013-0043, Rev. 0, May 8, 2013.

[2] T. B. Peters and A. L. Washington, II. "Task Technical and Quality Assurance Plan for ISDP Salt Batch 7 Sample Qualification", SRNL-RP-2013-00283, Rev.0, May, 2013.

[3] T. B. Peters, "ISDP7", SRNL-NB-2013-00040, June 11, 2013.

[4] S. E. Campbell, "Solids Settling in Tank 21H Sample", SRR-LWE-2013-00105, May 29, 2013.

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