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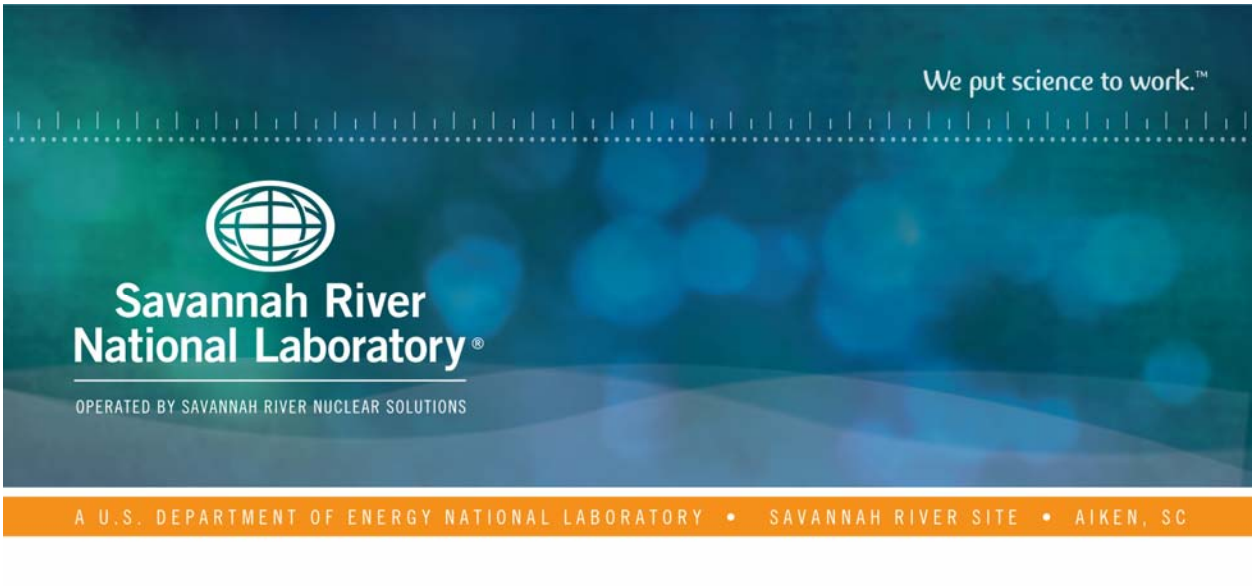

Results of Initial Analyses of the Salt Waste Processing Facility (SWPF) Batch 2 Tank 21H Qualification Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Salt Waste Processing Facility (SWPF) Batch 2 for processing. This document reports the initial expedited results of the analyses of the Tank 21H qualification sample. Analysis of the Tank 21H SWPF Batch 2 sample indicates that the material does not display any unusual characteristics or observations, such as floating solids, the presence of large amounts of solids, or unusual color. Further sample results will be reported in a future document. This memo satisfies part of Deliverable 3 of the Technical Task Request (TTR).[i]

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LIST OF ABBREVIATIONS

IC	Ion Chromatography
ICPES	Inductively-Coupled Plasma Emission Spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
%RSD	Percent Relative Standard Deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SWPF	Salt Waste Processing Facility
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 SWPF Batch 2 samples from Tank 21H. These results will be used by Savannah River Remediation (SRR) to determine if this salt batch meets downstream Waste Acceptance Criteria (WAC).[ii] This work was specified by a TTR [i] and Task Technical and Quality Assurance Plan (TTQAP).[iii] Details for the work are contained in controlled laboratory notebooks.[iv]

This document provides the rapid turnaround results (Table 4-1 in the TTQAP). A future document will report the longer turnaround time results listed in Table 4-2 of the TTQAP.

2.0 Experimental Procedure

A 3L sample (HTF-21-19-86) was pulled and delivered to SRNL on September 10, 2019. The 3L sample was a variable depth sample obtained approximately 62" from the bottom of the tank (transfer pump suction). Tank 21H was mixed for approximately 8.3 hours with two pumps on 30 August 2019 before the sample was pulled; the sample was pulled 11 days after pump shutdown. The sample was visually a clear solution with no apparent solids.

The density of the filtered solution (using a 0.45 μm syringe filter) from the sample was measured twice and reported in Table 1. A well-mixed, but unfiltered sample was also measured for density and reported in Table 1.

For the total mercury and methyl mercury (results to be reported in later document) measurements, ~1.5 mL of unfiltered and filtered samples were diluted into 39 mL of ultrapure water in glass vials with Teflon caps with almost no headspace; this follows the recommended process for preparing these types of samples.[v]

For the wt % Insoluble Solids measurements, approximately 300 mL of the well mixed salt solution (weighed on an electronic balance) was processed through a pre-weighed 0.2 micron porosity nylon filter to collect the insoluble solids. The solids and filter were washed with several portions (~50 mL total) of de-ionized water to remove the soluble salts and then dried to constant weight.[∇]

For other unfiltered samples, well-mixed solution was removed from the cells without dilution. For other filtered samples, filtration was provided by using a 0.45 μm syringe filter and the filtrate removed without dilution.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60. This is Safety Class work. SRNL documents the extent and type of review for Safety Class work using the SRNL Technical Report Design Checklist (design verification) contained in WSRC-IM-2002-00011, Rev. 2. The work performed, all analyses, and

[∇] To compare, the previously used method used much smaller volumes and did not explicitly rinse the captured solids.

the review process for this report complies with those requirements. Results from this report are not RW-0333P (enhanced quality assurance requirements) 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

Sample	Measured Density (g/mL)	Temperature (◦C)
Filtered	1.281 (2.1%)	23.9
Unfiltered	1.310 (0.24%)	25

The analytical uncertainty is typically <3% (1-sigma) for density measurements. The results are typical for dissolved saltcake of this type. For comparison, the average density of the Salt Batch 11 (SWPF Batch 1) solution (filtered) was 1.269 g/mL (27.0 ◦C).[vi]

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

Table 2. ICPES Results[r]

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.74	Mo	20.7 (0.68%)
Al	6920 (0.31%)	Na	6.22 (0.99%) M
B	57.1 (0.12%)	Ni	<15.8
Ba	<0.995	P	152 (2.8%)
Be	<0.253	Pb	<19.6
Ca	<1.98	S	2420 (1.2%)
Cd	<2.46	Sb	<35.4
Ce	<27.4	Si	13.2 (2.1%)
Cr	74.9 (0.47%)	Sn	<113
Cu	<5.17	Sr	<0.232
Fe	<6.31	Th	<14.3
Gd	<3.76	Ti	<3.04
K	440 (0.6%)	U	<97
La	<1.92	V	<1.63
Li	<6.91	Zn	6.25 (2.0%)
Mg	0.220 (0.0%)	Zr	<1.15
Mn	<0.454		

[r] While many of the analytes listed in Table 2 were not requested in the TTR, they have been included for the sake of completeness. In particular, the Uranium value in this table should be disregarded in favor of the sum of the U isotopes given in Table 4.

The ICPES 1-sigma analytical uncertainty is 10%.

The ICPES results do not vary greatly from historical data. The sodium concentration of 143000 mg/L (6.22 M) is within past Modular Caustic-Side Solvent Extraction Unit (MCU) salt batch concentrations. The potassium continues to be low compared to the WAC limit of 2240 mg/L and is well within the ranges of previous salt batches.[ii]

Results from the Ion Chromatography (IC) Anions, Free Hydroxide Titration, Total Inorganic Carbon (TIC)/Total Organic Carbon (TOC), Total Mercury, and wt % Insoluble Solids analyses are listed in Table 3. These were filtered samples, except for the Total Hg and Insoluble solids. The values in the parentheses are the %RSD.

Table 3. Further Non-Radiochemical Results ♦

Analyte	Result (mg/L)
F^-	<100
Cl^-	319 (0.9%)
Br^-	<100
Formate	113 (1.3%)
Nitrite	33600 (1.7%)
Nitrate	121000 (0.48%)
Phosphate	365 (8.5%)
Sulfate	6900 (0.63%)
Oxalate	361 (4.1%)
TIC	4280 (1.3%)
TOC	180 (2.0%)
Free Hydroxide	44800 (0.27%)
Total Hg	35.5 (3.9%)
Insoluble Solids	39.3 (0.0%) ↑

The 1-sigma analytical uncertainty for all listed samples is 10%, except for the Insoluble Solids, which is 20%.

The anion results in Table 3 are from two sets of analyses of the sample and are averaged from all four results. Two of the formate results were <100 mg/L (detection limit). Two of the formate results were real number values, and are the only values used for the formate average.

♦ While several anion analytes listed in Table 3 were not requested in the TTR, they have been included for the sake of completeness.
↑ The results were the same for the replicates.

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 21400 mg/L.

The radiochemical results are reported in Table 4. The values in the parentheses are the %RSD.

Table 4. Radiochemistry Results

Analyte	Filtered Result (pCi/mL)	Unfiltered Result (pCi/mL)
^{137}Cs	1.81E+08 (1.8%)	1.78E+08 (0.71%)
^{137m}Ba/total gamma	1.71E+08 (1.8%)	1.69E+08 (0.71%)
^{238}Pu	2.86E+04 (1.7%)	2.81E+04 (2.4%)
^{239}Pu	1.42E+03 (0.62%)	1.15E+03 (1.3%)
^{240}Pu	6.20E+02 (1.0%)	5.00E+02 (1.3%)
^{242}Pu	<3.82E+00	<7.92E+01
^{244}Pu	<1.77E-02	<3.67E-01
^{90}Sr	2.85E+05 (71%)	3.93E+05 (9.2%)
^{90}Y	2.85E+05 (71%)	3.93E+05 (9.2%)
Total alpha (w/Cs)	<2.39E+06	<1.86E+06
Total alpha (wo/Cs)	<4.55E+04	<4.23E+04
Total Beta (w/Cs)	1.92E+08 (2.0%)	1.95E+08 (5.4%)
Total beta (wo/Cs)	8.87E+05 (0.0%)	8.67E+05 (1.5%)
^{233}U	1.91E+01 (0.36%)	<2.01E+02
^{234}U	1.12E+02 (4.3%)	<1.30E+02
^{235}U	5.71E-01 (0.80%)	5.46E-01 (1.1%)
^{236}U	2.90E+00 (0.47%)	2.82E+00 (2.1%)
^{238}U	3.45E+00 (0.69%)	3.20E+00 (0.19%)
Total U (233+234+235+236+238)	10.6 mg/L	<9.85 mg/L

The 1-sigma analytical uncertainly is 10% for t4he total alpha and total beta results, 20% for the uranium, ^{239}Pu and ^{240}Pu results, 5% for the cesium and barium results, 5-20% (sample specific) for the plutonium results, and 19-25% (sample specific) for the strontium results.

For the filtered ^{239}Pu and ^{240}Pu values, the ICPMS analyses provided real numbers, which are reported. The %RSD from the ICPMS results is also used. In the case of the unfiltered samples, the ^{239}Pu/^{240}Pu mass ratio (8.42:1) from the ICPMS filtered results are used to calculate the individual ^{239}Pu and ^{240}Pu values from the PuTTa result for the $^{239/40}$Pu. In this case, the %RSD is from the PuTTa analysis.

The ^{90}Y is calculated as equal to the ^{90}Sr result. ^{137m}Ba is calculated as 94.7% of the ^{137}Cs result.

The ^{137}Cs result for the filtered SWPF Batch 2 converts to 0.684 Ci Cs/gal.

The radiochemical results are typical of previous salt batches. The total uranium value is the sum of the ^{233}U, ^{234}U, ^{235}U, ^{236}U and ^{238}U and is reported in mg/L.

In general, the filtered and unfiltered samples show good agreement. The differences in the $^{90}Sr/^{90}Y$ are likely due to the large %RSD in the filtered sample duplicate measurements. The differences in the $^{239/240}Pu$ values are likely due to a large analytical uncertainty in both the ICPMS and PuTTa measurements for that analyte.

4.0 Conclusions

Analysis of the Tank 21H SWPF Batch 2 composite sample indicates that the material does not display any unusual characteristics or observations, such as floating solids, the presence of large amounts of solids (confirmed by the low wt % insoluble solids measurement), or unusual color. Further sample results will be reported in a future document. This memo satisfies part of Deliverable 3 of the TTR.

5.0 References

[i] A. Samadi-Dezfouli, "Salt Batch Qualification for Feed to Salt Waste Processing Facility", X-TTR-H-00090, Rev. 0, August 27, 2019.

[ii] M. R. Norton, "Salt Waste Processing Feed Waste Acceptance Criteria", X-ESR-J-00001, August 5, 2019.

[iii] T. B. Peters, "Task Technical and Quality Assurance Plan for SWPF Feed Batch Qualification Testing", SRNL-RP-2019-00582, Rev. 0, September 2019.

[iv] T. B. Peters, "SWPF 2 Batch Qualification", ELN, A4571-00084-43.

[v] W. R. Wilmarth, "Best Handling Practices for Elemental Mercury, Organo-Mercury, and Inorganic Mercury Compounds", SRNL-TR-2019-00243, Rev.0, August 29, 2019.

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