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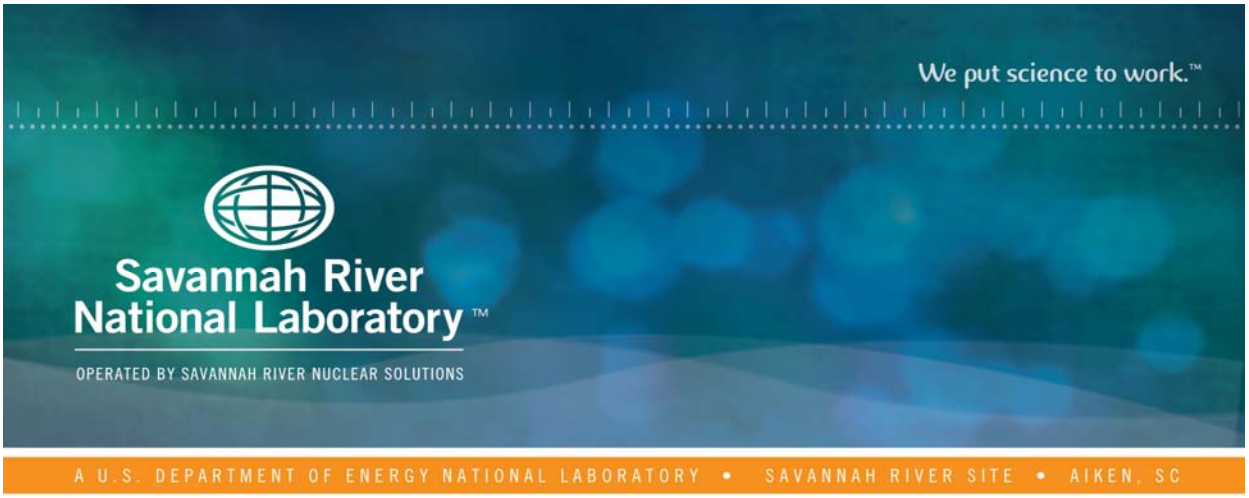

Results of the Supplementary Analyses of Salt Batch 10 and 11 Samples

T. B. Peters

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EXECUTIVE SUMMARY

Supplementary analyses of material from the Interim Salt Disposition Project (ISDP) Salt Batch (SB) 10 and Salt Batch 11 (SB11) samples were analyzed beyond what is described in previous reports.[i,ii] These additional analyses are required to verify these materials meet Salt Waste Processing Facility (SWPF) Waste Acceptance Criteria (WAC) feed requirements.

The stored SB10 and SB11 samples have been analyzed without filtering and the results found to be very similar to the previous filtered analyses performed for SB10 and SB11. The only result of significant difference is the wt% insoluble solids result for the SB11 sample. It would appear the newer method of analyzing larger volumes gives more accurate measurements for solutions with low quantities of solids.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ISDP	Interim Salt Disposition Project
%RSD	% Relative Standard Deviation
SAA	Satellite Accumulation Area
SB	Salt Batch
SRNL	Savannah River National Laboratory
SWPF	Salt Waste Processing Facility
TTQAP	Task Technical and Quality Assurance Plan
TTR	Task Technical Request
WAC	Waste Acceptance Criteria

1.0 Introduction

This report provides supplementary analytical laboratory results of ISDP SB 10 and SB11 samples from Tank 21H. These analyses are required to verify these materials meet SWPF Waste Acceptance Criteria (WAC).

This work follows a customer Task Technical Request (TTR)[iii] , and its corresponding Task Technical and Quality Assurance Plan (TTQAP)[iv] was written.

2.0 Experimental Procedure

Samples of SB10 (derived from HTF-21-16-104/105/106) and SB11 (derived from HTF-21-17-70/71/72) had been stored in a Satellite Accumulation Area (SAA) for future use. At the time of placement in the SAA, both samples showed very little visual evidence of solids other than a light dusting of solids. The visual observations were the same when the samples were retrieved for the supplementary analyses.

For the unfiltered analyses, well-mixed material from each of the composite bottles were removed for analysis with no filtration. None of the unfiltered samples were diluted before delivery to Analytical Development (AD), although AD performed an aqua-regia digestion on the samples.

The weight % insoluble solids measurement for the recent SB11 sample was obtained by filtering approximately 300 mL of the well mixed salt solution (weighed on an electronic balance) through a pre-weighed 0.2 micron porosity nylon filter to collect the insoluble solids. The solids and filter were washed with several portions of de-ionized water to remove the soluble salts and then dried to constant weight.[♣] The Wt % insoluble solids measurements were redone for SB11 since a larger sample of material was used to improve the analytical method results. The weight % insoluble solids were calculated from the weight of the insoluble solids and the total weight of salt solution filtered. This method using larger samples volumes proved more accurate than previous methods.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60 (Design Check). For Savannah River National Laboratory (SRNL) documents, the extent and type of review using the SRNL Technical Report Design Checklist is outlined in WSRC-IM-2002-00011, Rev. 2.[v] Records for this work are contained in electronic notebook ELN-A4571-00084-36.

3.0 Results and Discussion

The averaged results of each type of analyses are given in Table 1 (SB10) and Table 2 (SB11). The value in parentheses are the % relative standard deviation (%RSD) of the duplicate results.

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

As a comparison, the previous (filtered) measured values are given. The "Total U" term is the sum of the uranium isotope values listed in each table, respectively. The mg/L term for the $^{239/240}$Pu term is derived using a weighted average of 0.0769 Ci/g for the specific activity.[vi,♦] Both the Total Alpha and Total Beta measurements are with the ^{137}Cs removed.

Table 1. Relevant Results from Analysis of the SB10 Sample

Analyte	Previous (Filtered) Results [i]	Current (Unfiltered) Results
^{90}Sr	3.06E+05 (13%) pCi/mL	3.34E+05 (18%) pCi/mL
^{233}U	2.35E-03 (2.2%) mg/L	<2.34E-02 mg/L
^{234}U	1.49E-02 (3.5%) mg/L	1.47E-02 (3.6%) mg/L
^{235}U	3.17E-01 (3.4%) mg/L	3.23E-01 (2.5%) mg/L
^{236}U	5.20E-02 (3.7%) mg/L	5.32E-02 (0.5%) mg/L
^{238}U	1.21E+01 (5.7%) mg/L	1.24E+01 (1.2%) mg/L
Total U	1.24E+01 mg/L	1.28E+01 mg/L
^{238}Pu+$^{239/40}$Pu	1.29E-02 mg/L	1.39E-02 mg/L
Total Alpha	<2.36E+04 pCi/mL	<2.23E+04 pCi/mL
Total Beta	7.79E+05 (1.2%) pCi/mL	9.29E+05 (1.3%) pCi/mL

Table 2. Relevant Results from Analysis of the SB11 Sample

Analyte	Previous (Filtered) Results [ii]	Current (Unfiltered) Results
^{90}Sr	2.61E+05 (11%) pCi/mL	3.01E+05 pCi/mL (5.4%)
^{233}U	2.62E-03 (1.8%) mg/L	<2.23E-02 mg/L
^{234}U	1.80E-02 (0.3%) mg/L	1.54E-02 (6.4%) mg/L
^{235}U	2.30E-01 (0.6%) mg/L	2.12E-01 (4.2%) mg/L
^{236}U	3.68E-02 (0.1%) mg/L	3.40E-02 (12%) mg/L
^{238}U	1.45E+01 (0.0%) mg/L	1.28E+01 (5.4%) mg/L
Total U	1.48E+01 mg/L	1.31E+01 mg/L
^{238}Pu+$^{239/40}$Pu	1.69E-02 mg/L	1.38E-02 mg/L
Total Alpha	<3.88E+04 pCi/mL	<2.86E+04 pCi/mL
Total Beta	8.62E+05 (0.4%) pCi/mL	8.63E+05 (4.7%) pCi/mL
Wt% Insoluble Solids	5584 (68%) mg/L	25.4 (25%) mg/L

♦ This value is derived from the measured isotopic distribution of plutonium of 239 (91.2%) and 240 Pu (8.8%) in Sludge batch 9 solids.

The 1-sigma analytical uncertainty for the uranium results is 20%. The 1-sigma analytical uncertainty for the total beta measurements is 10%. The 1-sigma analytical uncertainty for the ^{90}Sr measurements ranged from 18.3 to 21.4%. The 1-sigma analytical uncertainty for the ^{238}Pu measurements ranged from 4.90 to 10.0%. The 1-sigma analytical uncertainty for the $^{239/40}Pu$ measurements ranged from 6.23 to 10.8%.

In the case of the wt% insoluble solids for the recent SB11 results, one measurement gave a less-than value, and one measurement gave an actual value. The actual value is used with the value in parentheses being the 1-sigma analytical uncertainty. This variation is due to the fact that even when using large volumes of sample, the very low insoluble solids concentration is difficult to measure.

The values between the old and recent analyses are very similar, indicating that the filtration used in the old samples did not appear to measurably affect the results, which indicates the new method for wt% insoluble solids is superior.

4.0 Conclusions

The current SB10 and SB11 samples have been analyzed and were found to give very similar results to the previous (filtered) analyses done as part of SB qualification. The only result of significant difference is the wt% insoluble solids result for the SB11 sample. While the physical observations of this material have not changed, it would appear the newer method of analyzing larger volumes gives more accurate measurements for solutions with low quantities of solids. SRNL recommends using this method for future analyses of this type.

5.0 References

[i] T. B. Peters, C. J. Bannochie, "Results from the Interim Salt Disposition Program Macrobatch 10 Tank 21H Qualification Samples", SRNL-STI-2017-00055, Rev. 0, February 2017.

[ii] T. B. Peters, C. J. Bannochie, "Results from the Interim Salt Disposition Program Macrobatch 11 Tank 21H Acceptance Samples", SRNL-STI-2017-00698, Rev. 1, October 2018

[iii] A. Samadi-Dezfouli, TTR "Additional SWPF Feed Batch 1 Qualification Testing", X-TTR-H-00089, Rev. 0, July 2, 1029.

[iv] T. B. Peters, M. S. Hay, "Task Technical and Quality Assurance Plan for Additional SWPF Feed Batch 1 Qualification Testing", SRNL-RP-2019-00494, Rev.0, July 2019.

[v] Savannah River National Laboratory, "Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Rev. 2, August 2004.

[vi] C. L. Trivelpiece, W. L. Kublius, D. P. DiPrete, "Determination of Reportable Radionuclides for Defense Waste Processing Facility (DWPF) Sludge Batch 9 (Macrobatch 11)", SRNL-STI-2018-00680, Rev. 0, May 2019.

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