



Savannah River
National Laboratory®

A U.S. DEPARTMENT OF ENERGY NATIONAL LAB • SAVANNAH RIVER SITE • AIKEN, SC • USA

Sludge Batch 11 Assembly: Tank 35

J. R. Dekarske

November 2023

SRNL-STI-2023-00366, Revision 0

DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

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

Keywords: *Tank Farm, Sludge Batch 11*

Retention: *Permanent*

Sludge Batch 11 Assembly: Tank 35

J. R. Dekarske

November 2023

Savannah River National Laboratory is operated by
Battelle Savannah River Alliance for the U.S. Department
of Energy under Contract No. 89303321CEM000080.



REVIEWS AND APPROVALS

AUTHORS:

J. R. Dekarske, Analytical and Tank Farm Characterization Date

TECHNICAL REVIEW:

J. M. Pareizs, Chemical Flowsheet Development, Reviewed per E7 2.60 Date

APPROVAL:

M. L. Whitehead, Manager Date
Analytical and Tank Farm Characterization

F. M. Pennebaker, Director, Nuclear and Chemical Processing Date

D. M. Yarbrough, SRMC Nuclear Safety & Engineering Integration Date

ACKNOWLEDGEMENTS

The author acknowledges assistance in the completion of sample transfers and handling tasks by the Shielded Cells Operations group particularly Teresa Baxter and the completion of sample analyses tasks by David DiPrete, the Nuclear Measurements group, the Analytical and Tank Farm Characterization group, and Catherine Housley.

EXECUTIVE SUMMARY

Savannah River Mission Completion Nuclear Safety and Engineering Integration (SRMC-E) has requested that Savannah River National Laboratory (SRNL) perform Tank 35 characterization analyses in support of Sludge Batch 11 (SB11) assembly. Two Tank 35 samples were delivered to SRNL and composited into a single sample in April 2023. The composite sample was analyzed for the following: density, weight percent solids, chemical composition, radionuclides, supernate corrosion control tests, and x-ray diffraction for burkeite, gibbsite, and boehmite.

- The slurry and supernate densities were observed to be 1.386 and 1.355 g/mL respectively.
- The weight percent solids analyses yielded a total solids (slurry basis) weight percent of 40.8%, soluble solids (slurry basis) weight percent of 37.6%, soluble solids (filtrate basis) weight percent of 38.8%, insoluble solids (slurry basis) weight percent of 3.2%, and a calcined solids (slurry basis) weight percent of 17.1%.
- The most prevalent radiochemical species, by weight percent of the total solids, were found to be Sr-90, I-129, Cs-137, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-238, Pu-239, Pu-240, and Am-241. The highest contributors of radioactivity, by Ci/gal, were Sr-90, Y-90, Cs-137, Ba-137m, Pu-238, and Pu-241.
- The most prevalent elements observed in the slurry, by mg/kg slurry, were Al, Fe, Hg, Mn, Na, K, and S which is typical of slurry. Additionally, the highest concentration elements seen in the supernate were Na, Al, S, K, P, Cr, B, Hg, and Mo. The highest level of anions found in the slurry and supernate were nitrate, nitrite, sulfate, carbonate, and free hydroxide.
- Through XRD, the phase composition of the unwashed slurry was determined to be trona, nitratine, sodium nitrite, boehmite, gibbsite, and hematite. The ratio of boehmite to gibbsite in the slurry was found to be approximately equal. No burkeite was observed in the sample.

TABLE OF CONTENTS

LIST OF TABLES	viii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS.....	ix
1.0 Introduction.....	1
2.0 Experimental Procedure.....	1
2.1 Quality Assurance	2
3.0 Results and Discussion	2
4.0 Conclusions.....	11
5.0 References.....	12

LIST OF TABLES

Table 3-1. Summary of Weight Percent Analyses of HTF-35-23-34/35	2
Table 3-2. Summary of Slurry Density Analyses of HTF-35-23-34/35	3
Table 3-3. Expedited slurry radionuclides and mercury methods, analytes, and digestions/dilutions	4
Table 3-4. Expedited slurry radionuclides and mercury results.....	5
Table 3-5. Slurry elemental analyses results.....	7
Table 3-6. Supernate elemental analyses results.....	8
Table 3-7. Supernate anions analyses results.....	9
Table 3-8. Sludge slurry anions analyses results	9
Table 3-9. Semi-quantitative percentages of minerals in the unwashed Tank 35 solids sample	10

LIST OF FIGURES

Figure 2-1. HTF-35-23-34 metal dip bottle container (left), HTF-35-23-35 sludge at bottom of dip bottle container (center), and HTF-35-23-34/35 composite sample (right).....	1
Figure 3-1. XRD of unwashed Tank 35 solids.....	10
Figure 3-2. XRD of washed Tank 35 solids.....	11

LIST OF ABBREVIATIONS

CST	Crystalline Silicotitanate
DMA	Direct Mercury Analyzer
MDA	Minimum Detectable Activity
MST	Monosodium Titanate
RSD	Relative Standard Deviation
SB11	Sludge Batch 11
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
XRD	X-ray Diffraction

1.0 Introduction

Savannah River Mission Completion Nuclear Safety and Engineering Integration (SRMC-E) has requested through a Technical Task Request (TTR) that Savannah River National Laboratory (SRNL) perform Tank 35 characterization analyses in support of Sludge Batch 11 (SB11) assembly.¹ Two Tank 35 samples were delivered to SRNL and composited into a single sample in April 2023. The composite sample was analyzed for the following as defined in the Task Technical and Quality Assurance Plan (TTQAP): density, weight percent solids, slurry elementals, chemical composition, radionuclides, supernate corrosion control tests, and x-ray diffraction for burkeite, gibbsite, and boehmite.²

2.0 Experimental Procedure

Two Tank 35 samples (HTF-35-23-34 and HTF-35-23-35) each containing approximately 200mL of slurry were delivered on April 11, 2023 to SRNL in metal dip bottle containers. Prior to pulling the samples, Tank 35 was mixed for 12 days. The samples were pulled without foil within 30 minutes of pump shutdown at 24 inches below the tank surface. Upon receipt, the samples were composited into a single sample (HTF-35-23-34/35). Each metal dip bottle was washed with decanted supernate and returned to the composite bottle in order to transfer all sludge into the composite bottle. Figure 2-1 shows the HTF-35-23-34 sample metal dip bottle container, the HTF-35-23-35 sample sludge present prior to washing with supernate, and the final composited sample bottle.

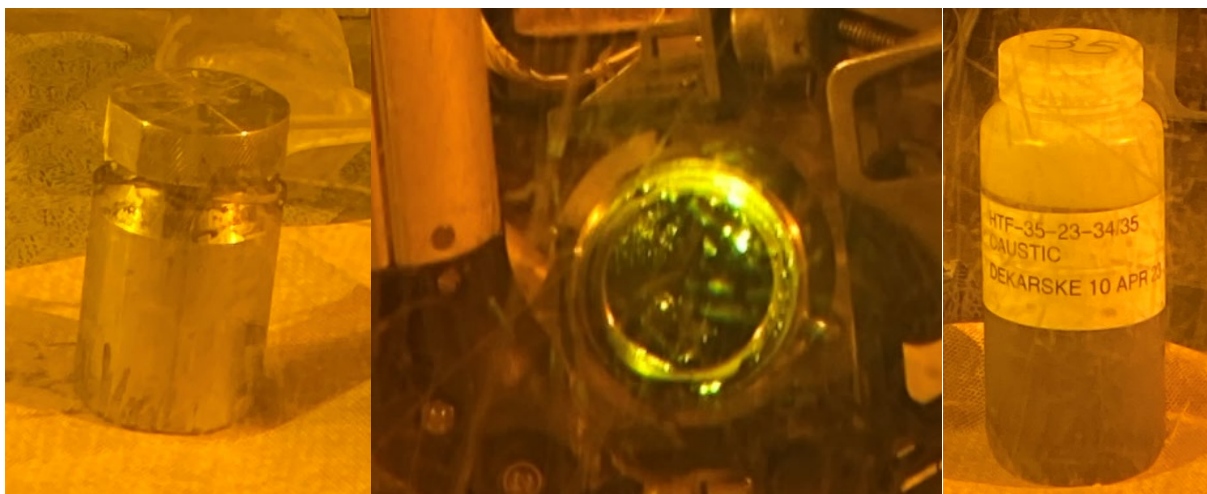


Figure 2-1. HTF-35-23-34 metal dip bottle container (left), HTF-35-23-35 sludge at bottom of dip bottle container (center), and HTF-35-23-34/35 composite sample (right).

After compositing the samples, the slurry density was measured gravimetrically by observing the weight of slurry at a known volume. Additionally, peroxide fusion digestion preparations utilizing sodium peroxide and nitric acid,³ aqua regia digestions (utilizing a sealed vessel) preparations,⁴ and slurry water dilutions (filtered and unfiltered) were performed for subsequent radiochemical and chemical slurry analyses. Supernate preparations were each diluted at a dilution factor of ~10 with deionized water and also submitted for radiochemical and chemical testing.

Aliquots of slurry were filtered and dried under vacuum on the filtration apparatus for three hours at ambient temperature. Approximately 200 mg of unwashed solid was transferred into a shielded bottle. The filtered solid was washed with 15mL of deionized water three times, and 100 mg of washed solids were transferred into a shielded bottle. These shielded bottles were submitted for x-ray diffraction (XRD) analysis for gibbsite, boehmite, and burkeite.

The weight percent solids were measured and determined for total solids (slurry basis), soluble solids (slurry and filtrate bases), insoluble solids (slurry basis), and calcined solids (slurry basis). The total solids were obtained through evaporation of liquid by heating quadruplicate sample preparations in an oven at 115 °C until there was no or minimal further loss of mass. To measure calcined solids, the sample preparations used for total solids were further placed into a furnace and heated at 1100 °C for 2 hours. The soluble solids (filtrate basis) were determined by filtering slurry through a 0.45 µm nylon filter membrane and analyzing four aliquots of the filtrate. The filtrate was heated at 115 °C until all liquid had evaporated and no further or minimal loss of mass. The insoluble solids (slurry basis) and soluble solids (slurry basis) were calculated from total solids (slurry basis) and soluble solids (filtrate basis).

The I-129 analysis required a separate preparation due to the I-129 being removed by peroxide fusion or aqua regia. These preparations required the use of monosodium titanate (MST), crystalline silicotitanate (CST), potassium iodide, nitric acid, and sodium hydroxide.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 2.60.⁵ SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.⁶ SRMC requested that a Functional Classification of Safety Class apply to this work. Additionally, the gibbsite, boehmite, and burkeite analyses are considered Production Support Functional Classification.²

3.0 Results and Discussion

Four memoranda were issued previously that reported weight percent solids and density (SRNL-L3120-2023-00011)⁷, expedited slurry radionuclides, slurry mercury, and weight percent calcined solids (SRNL-L3120-2023-00012)⁸, elemental analyses (SRNL-L3120-2023-00014)⁹, and gibbsite, boehmite, and burkeite (SRNL-L3130-2023-00003).¹⁰ These results are again stated in this report. The weight percent solids results that were consolidated into one table in SRNL-L3120-2023-00014 Revision 2 are reported here as Table 3-1. The slurry density results that were reported in SRNL-L3120-2023-00011 Revision 0 are reported here as Table 3-2.

Table 3-1. Summary of Weight Percent Analyses of HTF-35-23-34/35

Summary of Weight Percent Analyses of HTF-35-23-34/35		
Test	Average	%RSD (n)
Total Solids (slurry basis) (%)	40.8	0.4 (4)
Calculated Soluble Solids (slurry basis) (%)	37.6	N/A
Soluble Solids (filtrate basis) (%)	38.8	0.5 (4)
Calculated Insoluble Solids (slurry basis) (%)	3.2	N/A
Calcined Solids (slurry basis) (%)	17.1	5.8 (4)

Error propagation of the total solids (slurry basis) and soluble solids (filtrate basis) yielded a 95% confidence interval for the insoluble solids of approximately ±0.5 weight %. The confidence interval is derived using the inverse Student's t-distribution which requires probability and degrees of freedom, partial derivative of the weight percent of insoluble solids with respect to total solids, partial derivative of the weight percent of insoluble solids with respect to soluble solids, standard error of the weight percent total solids, and standard error of the weight percent soluble solids. Contributing factors to larger 95% confidence interval values include standard deviation of results (incorporated in standard error), number of replicates (incorporated in degrees of freedom), and average weight percent of soluble solids (incorporated in partial derivative of weight percent insoluble solids with respect to total solids and soluble solids). A higher

standard deviation of results, larger amount of weight percent soluble solids, and fewer number of replicates can lead to a larger 95% confidence interval value. The high soluble solids weight percent is not unexpected with Tank 35's involvement as a hub/source tank in Salt Batch preparations.

Table 3-2. Summary of Slurry Density Analyses of HTF-35-23-34/35

Summary of Slurry Density Analyses of HTF-35-23-34/35			
Replicate 1 (g/mL)	Replicate 2 (g/mL)	Average (g/mL)	%RSD
1.385	1.388	1.386	0.15

The supernate density which was reported in the corrosion control LIMS sample results report was observed to be 1.355 g/mL. The supernate density being lower than the slurry density is due to the insoluble solids not being present in the supernate density replicates. The high densities correspond with the larger total solids observed in the sample.

The methods, analytes, and digestions/dilutions for the expedited slurry radionuclides and slurry mercury and their results reported in SRNL-L3120-2023-00012 Revision 3 are displayed in Table 3-3 and Table 3-4 respectively. The total beta/gamma was calculated by summing the total beta results with the gamma emitting radionuclides.

Table 3-3. Expedited slurry radionuclides and mercury methods, analytes, and digestions/dilutions

Methods and Digestions for Determination of Slurry Radionuclides and Hg		
Method:	Digestion:	Analyte:
Liquid Scintillation Counting	Peroxide Fusion	Total Alpha, Total Beta
Gamma Scan Cs-removed	Peroxide Fusion	Co-60, Ru-106, Rh-106 (calculated, secular equilibrium with Ru-106), Ag-110m, Sb-125, Te-125m (calculated, secular equilibrium with Sb-125), Ce-144, Pr-144 (calculated, secular equilibrium with Ce-144), Pr-144m (calculated, secular equilibrium with Ce-144), Eu-152, Eu-154, Eu-155, Am-241
Gamma Scan	Peroxide Fusion	Cs-134, Cs-137, Ba-137m (calculated, secular equilibrium with Cs-137)
Pm-147/Sm-151	Peroxide Fusion	Pm-147, Sm-151
U_233_234_235_236	Peroxide Fusion	U-233, U-234, U-235, U-236, U-238
ICP-MS	Aqua Regia	Np-237
Pu238_Pu241	Peroxide Fusion	Pu-238, Pu-241
Pu_242_244	Peroxide Fusion	Pu-239, Pu-240
Tc_99	Aqua Regia	Tc-99
Am_Cm	Peroxide Fusion	Am-242m, Cm-244, Cm-245
Sr_90	Peroxide Fusion	Sr-90, Y-90 (calculated, secular equilibrium with Sr-90)
I_129_With_Separation	Special Preparation	I-129
Tritium	Water Dilution, Filtered	H-3
DMA	Aqua Regia	Total Hg

Table 3-4. Expedited slurry radionuclides and mercury results

Expedited Radiochemical and Hg Analyses					
Slurry Analyses	Average (wt% of total solids)	Average (Ci/g of total solids)	Average (Ci/gal of sludge slurry)	%RSD, n = 3	Uncertainty (1 sigma) (%)
Total Alpha	N/A	<2.69E-04	<5.74E-01	N/A	Upper Limit
Total Beta-gamma	N/A	1.01E-02	2.15E+01	N/A	N/A
H-3	<5.15E-11	<4.97E-09	<1.06E-05	N/A	MDA
Co-60	6.90E-09	7.80E-08	1.67E-04	2.30	5.00
Sr-90	3.49E-03	4.77E-03	1.02E+01	16.96	15.5-17.2
Y-90	8.76E-07	4.77E-03	1.02E+01	16.96	15.5-17.2
Tc-99	<1.36E-03	<2.30E-07	<4.92E-04	N/A	Upper Limit
Ru-106	<3.73E-09	<1.25E-07	<2.67E-04	N/A	MDA
Rh-106	<3.51E-15	<1.25E-07	<2.67E-04	N/A	MDA
Ag-110m	<6.19E-10	<2.94E-08	<6.29E-05	N/A	MDA
Sb-125	<8.57E-09	<8.84E-08	<1.89E-04	N/A	MDA
Te-125m	<4.91E-10	<8.84E-08	<1.89E-04	N/A	MDA
I-129	3.10E-04	5.47E-10	1.17E-06	4.17	3.83-4.47
Cs-134	<7.57E-08	<9.79E-07	<2.09E-03	N/A	MDA
Cs-137	6.67E-04	5.80E-04	1.24E+00	5.13	5.00
Ba-137m	1.02E-10	5.49E-04	1.17E+00	5.13	5.00
Ce-144	<1.27E-08	<4.06E-07	<8.68E-04	N/A	MDA
Pr-144m	<2.24E-13	<4.06E-07	<8.68E-04	N/A	MDA
Pr-144	<5.37E-13	<4.06E-07	<8.68E-04	N/A	MDA
Pm-147	<1.73E-06	<1.60E-05	<3.43E-02	N/A	Upper Limit
Sm-151	<7.14E-05	<1.88E-05	<4.02E-02	N/A	Upper Limit
Eu-152	<2.75E-08	<4.75E-08	<1.02E-04	N/A	MDA
Eu-154	2.88E-06	7.76E-06	1.66E-02	1.54	5.00
Eu-155	<2.76E-08	<1.28E-07	<2.74E-04	N/A	Upper Limit
U-233	4.11E-06	3.97E-10	8.50E-07	6.26	20
U-234	8.94E-04	5.59E-08	1.20E-04	5.04	20
U-235	8.40E-04	1.82E-11	3.88E-08	4.92	20
U-236	4.01E-04	2.60E-10	5.56E-07	5.43	20
U-238	5.83E-03	1.96E-11	4.19E-08	4.56	20
Np-237	1.50E-04	1.06E-09	2.26E-06	2.76	10
Pu-238	1.13E-03	1.94E-04	4.14E-01	10.74	5.54-6.39
Pu-239	3.19E-03	1.99E-06	4.25E-03	30.97	10.5-23.2
Pu-240	6.02E-04	1.37E-06	2.93E-03	30.10	10.6-23.2
Pu-241	5.46E-05	5.62E-05	1.20E-01	13.52	15.20-15.60
Pu-242	5.96E-05	2.28E-09	4.87E-06	29.4	12.3-23.8
Am-241	3.50E-04	1.20E-05	2.57E-02	5.92	5.00
Am-242m	5.63E-08	5.47E-09	1.17E-05	14.52	17.9-28.1
Cm-244	1.31E-07	1.06E-07	2.27E-04	5.35	10.6-11.3
Cm-245	<1.98E-05	<3.39E-08	<7.26E-05	N/A	MDA
Hg	0.606	N/A	N/A	4.07	10

Note: "<" results are not the average. They are the minimum of three replicate results.
MDA= minimum detectable amount

The most prevalent radiochemical species, by weight percent of the total solids, were found to be Sr-90, I-129, Cs-137, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-238, Pu-239, Pu-240, and Am-241. The highest contributors of radioactivity, by Ci/gal, were largely Sr-90 and Y-90 followed by Cs-137, Ba-137m, Pu-238, and Pu-241. These results agree with the total beta-gamma and total alpha observed.

The slurry elemental methods, analytes, and digestions for the expedited slurry elementals reported in SRNL-L3120-2023-00014 Revision 2 are displayed in Table 3-5. The most prevalent elements observed, by mg/kg slurry, were Al, Fe, Hg, Mn, Na, K, and S which is typical of slurry.

Table 3-5. Slurry elemental analyses results

Slurry Elemental Analysis								
Slurry	Average mg/kg Slurry	% RSD	Uncertainty (1 sigma) (%)	Number of Replicates	Digestion Method (s)	Analytical Method	Average mg/kg total solids	Average mg/kg calcined solids
Ag	<3.47E+00	N/A	N/A	3	Aqua Regia	ICP-ES	<8.51E+00	<2.03E+01
Al	1.41E+04	1.23	5	3	Peroxide Fusion	ICP-ES	3.46E+04	8.25E+04
B	6.49E+01	3.38	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	1.59E+02	3.80E+02
Ba	9.35E+01	1.62	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	2.29E+02	5.47E+02
Be	<7.5E-02	N/A	N/A	3	Aqua Regia	ICP-ES	<1.8E-01	<4.4E-01
Ca	2.82E+02	2.16	5	3	Aqua Regia	ICP-ES	6.93E+02	1.65E+03
Cd	2.28E+00	2.68	5	3	Aqua Regia	ICP-ES	5.60E+00	1.34E+01
Ce	1.17E+02	3.01	5	3	Aqua Regia	ICP-ES	2.86E+02	6.82E+02
Co	3.08E+00	3.39	10	3	Aqua Regia	ICP-MS	7.56E+00	1.80E+01
Cr	1.02E+02	6.39	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	2.50E+02	5.97E+02
Cu	6.15E+01	5.22	5	3	Aqua Regia	ICP-ES	1.51E+02	3.60E+02
Fe	9.73E+03	5.16	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	2.39E+04	5.69E+04
Gd	4.87E+00	N/A	10	3	Aqua Regia	ICP-MS	1.19E+01	2.85E+01
Hg	2.47E+03	4.07	10	3	Aqua Regia	DMA	6.06E+03	1.44E+04
K	1.06E+03	2.71	5	3	Aqua Regia	ICP-ES	2.61E+03	6.22E+03
La	5.79E+01	6.37	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	1.42E+02	3.38E+02
Li	<1.9E+01	N/A	N/A	3	Aqua Regia	ICP-ES	<4.6E+01	<1.1E+02
Mg	1.90E+02	3.77	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	4.67E+02	1.11E+03
Mn	2.04E+03	2.67	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	4.99E+03	1.19E+04
Mo	2.95E+01	2.20	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	7.25E+01	1.73E+02
Na	1.32E+05	2.62	2.5	3	Aqua Regia	ICP-ES	3.24E+05	7.72E+05
Ni	3.65E+02	2.12	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	8.96E+02	2.14E+03
P	2.31E+02	5.01	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	5.66E+02	1.35E+03
Pb	1.62E+01	N/A	10	3	Aqua Regia	ICP-MS	3.97E+01	9.47E+01
S	2.05E+03	1.71	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	5.02E+03	1.20E+04
Sb	2.01E-01	N/A	10	3	Aqua Regia	ICP-MS	4.9E-01	1.2E+00
Si	2.32E+02	7.23	5	3	Peroxide Fusion	ICP-ES	5.68E+02	1.35E+03
Sn	2.2E+00	N/A	10	3	Aqua Regia	ICP-MS	5.3E+00	1.3E+01
Sr	3.85E+01	2.77	5	6	Peroxide Fusion, Aqua Regia	ICP-ES	9.44E+01	2.25E+02
Th	2.0E+00	3.50	10	3	Aqua Regia	ICP-MS	4.9E+00	1.2E+01
Ti	7.42E+00	2.40	5	3	Aqua Regia	ICP-ES	1.82E+01	4.34E+01
U	3.25E+01	N/A	N/A	3	Peroxide Fusion	Calculated from U-233, U-234, U-235, U-236, U-238 activities	7.97E+01	1.90E+02
V	<1.8E+00	N/A	N/A	3	Aqua Regia	ICP-ES	<4.5E+00	<1.1E+01
Zn	2.59E+01	7.23	5	3	Aqua Regia	ICP-ES	6.35E+01	1.51E+02
Zr	1.65E+02	6.20	5	3	Aqua Regia	ICP-ES	4.04E+02	9.63E+02
Pu	2.06E+01	N/A	N/A	3	Peroxide Fusion	Calculated from Pu-238, Pu-239, Pu-240, Pu-241, Pu-242 activities	5.04E+01	1.20E+02

Note: "<" results are not the average. They are the minimum of the replicates analyzed.

A select number of supernate elementals and anions were analyzed as part of the corrosion control analyses. Therefore, the full elemental and anion suites were screened and are reported here in Table 3-6 and Table 3-7 respectively. Supernate total Hg was analyzed by DMA. The most prevalent elements seen in the supernate were Na, Al, S, K, P, Cr, B, Hg, and Mo which is typical of tank farm salt solution. The highest level of anions found in the slurry and supernate were nitrate, nitrite, sulfate, carbonate, and free hydroxide. Each of the anions were found to be in good agreement between the slurry and supernate with the differences being within method uncertainty. The cation and anion ratios for the slurry and supernate were both 1.0; the cation and anion ratios of the slurry and supernate each matched to 100%.

Table 3-6. Supernate elemental analyses results

Supernate Elemental Analysis by ICP-ES and DMA			
Element	Average (mg/L)	%RSD n = 2	Uncertainty (1 sigma) (%)
Ag	<4.64E+00	N/A	N/A
Al	9.84E+03	0.50	5
B	9.49E+01	1.42	5
Ba	<1.18E-01	N/A	N/A
Be	<1.0E-01	N/A	N/A
Ca	<5.29E-01	N/A	N/A
Cd	<1.53E-01	N/A	N/A
Ce	<2.71E+00	N/A	N/A
Co	<5.56E-01	N/A	N/A
Cr	1.18E+02	0.60	5
Cu	<1.04E+00	N/A	N/A
Fe	<4.28E-01	N/A	N/A
Gd	<4.49E-01	N/A	N/A
K	1.55E+03	0.00	5
La	<3.26E-01	N/A	N/A
Li	<7.72E+00	N/A	N/A
Mg	<5.71E-01	N/A	N/A
Mn	<6.07E-01	N/A	N/A
Mo	4.07E+01	1.04	5
Na	1.90E+05	0.00	2.5
Ni	<5.73E-01	N/A	N/A
P	2.63E+02	1.89	5
Pb	<3.66E+00	N/A	N/A
S	2.92E+03	0.48	5
Sb	<2.99E+00	N/A	N/A
Si	<1.36E+01	N/A	N/A
Sn	<8.21E+00	N/A	N/A
Sr	<1.0E-01	N/A	N/A
Th	<5.45E+00	N/A	N/A
Ti	<1.57E+00	N/A	N/A
U	<5.84E+00	N/A	N/A
V	<2.54E+00	N/A	N/A
Zn	7.38E+00	1.53	5
Zr	<5.11E-01	N/A	N/A
Hg	6.52E+01	11.43	10

Note: "<" results are not the average.
They are the minimum of the replicates analyzed.

Table 3-7. Supernate anions analyses results

Supernate Anions			
Anion	Average (mg/L)	%RSD, n = 2	Uncertainty (1 sigma) (%)
Free Hydroxide	2.85 (M)	0.50	5
Carbonate	3.31E+04	0.11	7
Formate	<9.77E+01	N/A	N/A
Nitrite	4.11E+04	7.05	10
Nitrate	1.77E+05	1.98	10
Sulfate	8.37E+03	1.22	10
Oxalate	1.58E+02	7.48	10
Phosphate	<9.97E+01	N/A	N/A
Chloride	2.63E+02	2.00	10
Aluminate	3.46E+04	0.50	5
Fluoride	<1.20E+02	N/A	N/A
Glycolate	<2.0E+01	N/A	N/A

Table 3-8. Sludge slurry anions analyses results

Sludge Slurry Anions			
Anion	Average (mg/L)	%RSD, n = 3	Uncertainty (1 sigma) (%)
Free Hydroxide	2.77 (M)	2.06	5
Formate	<2.65E+03	N/A	N/A
Nitrite	3.56E+04	1.17	10
Nitrate	1.79E+05	0.62	10
Sulfate	7.74E+03	0.66	10
Oxalate	<2.65E+03	N/A	N/A
Phosphate	<2.65E+03	N/A	N/A
Chloride	<2.65E+03	N/A	N/A
Fluoride	<2.65E+03	N/A	N/A
Carbonate	3.19E+04	4.56	7
Total Inorganic Carbon	6.37E+03	4.56	7
Total Organic Carbon	<3.12E+02	N/A	N/A

Note: For the "<" average, the smallest "<" result is reported.

The gibbsite, boehmite, and burkeite (or lack thereof) presence in the unwashed Tank 35 solids reported in SRNL-L3130-2023-00003 Revision 0 are displayed in Table 3-9. The percent of each phase was determined using a theoretical value to calculate the approximate percentage the phase contributed to the pattern. However, due to crystal orientation restrictions, the results in Table 3-9 are semi-quantitative. The XRD diffraction patterns also reported in SRNL-L3130-2023-00003 Revision 0 are shown here as Figures 3-1 and 3-2. By washing with water, the Tank 35 insoluble solids are shown to be hematite and the slightly water insoluble boehmite. The solids and salt residues washed away by water were trona, boehmite, nitratine, gibbsite, and sodium nitrite. The unwashed Tank 35 sample demonstrates that the amount of gibbsite compared to boehmite is approximately equal: 9.7% and 10.9% respectively. No burkeite was observed in the washed nor unwashed sample. This is further supported by the slurry anions having similar sulfate and carbonate concentrations as the supernate. If burkeite was present, the slurry would be expected to have higher sulfate and carbonate concentrations than the supernate.

Table 3-9. Semi-quantitative percentages of minerals in the unwashed Tank 35 solids sample

Phase	Percent
Hematite	5.8
Trona	35.2
Boehmite	10.9
Nitratine	26.4
Gibbsite	9.7
Sodium Nitrite	11.9

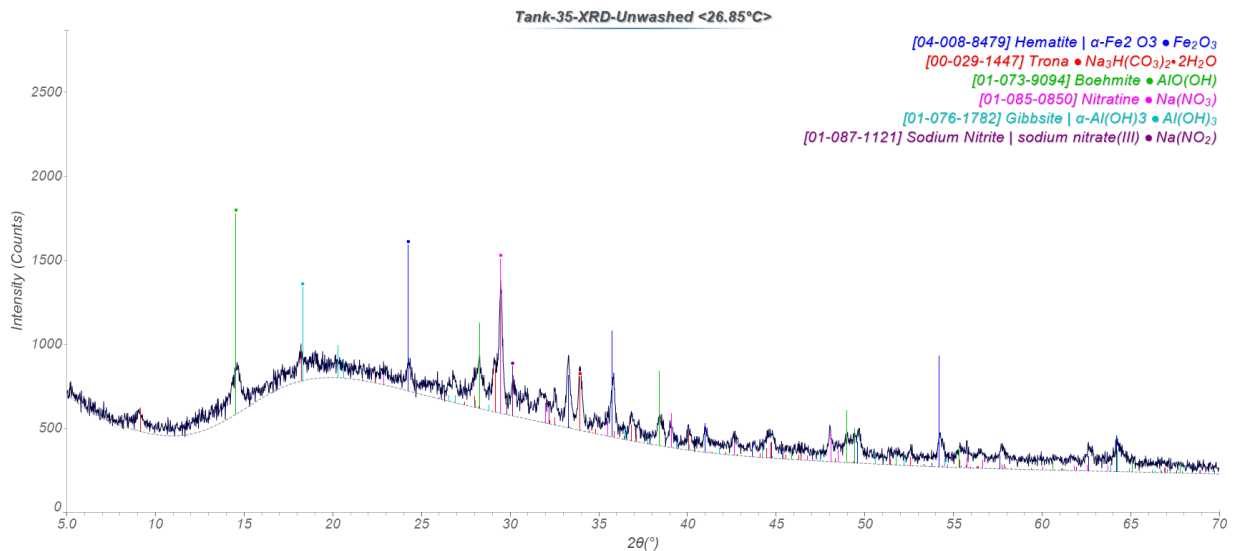


Figure 3-1. XRD of unwashed Tank 35 solids

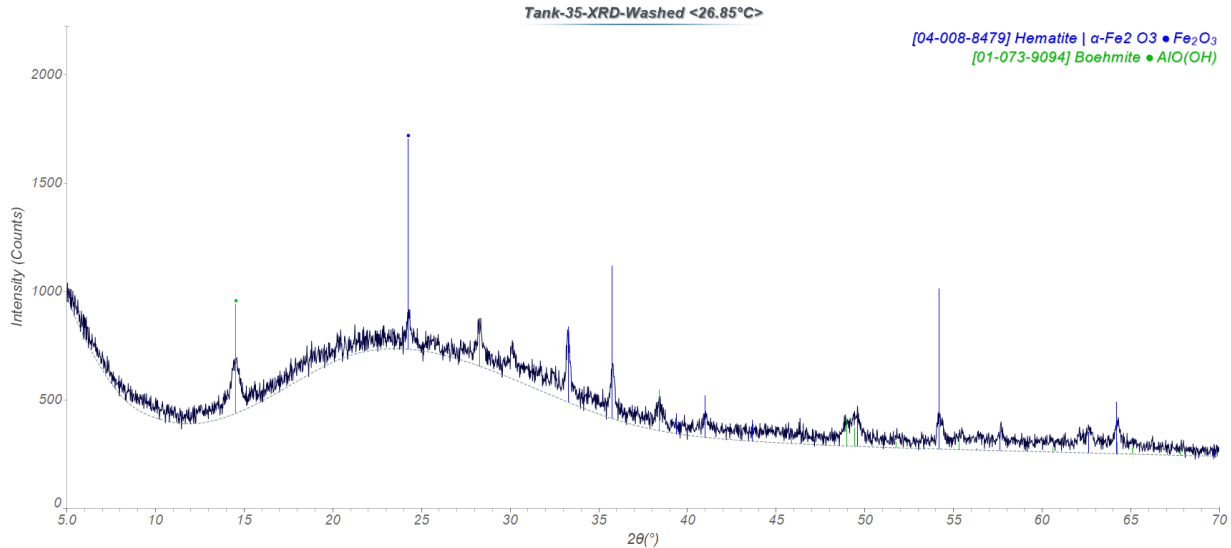


Figure 3-2. XRD of washed Tank 35 solids

4.0 Conclusions

SRNL performed Tank 35 characterization analyses in support of SB11 assembly. Two Tank 35 samples were delivered to SRNL and composited into a single sample in April 2023. The composite sample was analyzed for density, weight percent solids, chemical composition, radionuclides, supernate corrosion control tests, and x-ray diffraction for burkeite, gibbsite, and boehmite.

- The slurry and supernate densities were observed to be 1.386 and 1.355 g/mL respectively.
- The weight percent solids analyses yielded a total solids (slurry basis) weight percent of 40.8%, soluble solids (slurry basis) weight percent of 37.6%, soluble solids (filtrate basis) weight percent of 38.8%, insoluble solids (slurry basis) weight percent of 3.2%, and a calcined solids (slurry basis) weight percent of 17.1%.
- The most prevalent radiochemical species, by weight percent of the total solids, were found to be Sr-90, I-129, Cs-137, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-238, Pu-239, Pu-240, and Am-241. The highest contributors of radioactivity, by Ci/gal, were Sr-90, Y-90, Cs-137, Ba-137m, Pu-238, and Pu-241.
- The most prevalent elements observed in the slurry, by mg/kg slurry, were Al, Fe, Hg, Mn, Na, K, and S which is typical of slurry. Additionally, the highest concentration elements seen in the supernate were Na, Al, S, K, P, Cr, B, Hg, and Mo. The highest level of anions found in the slurry and supernate were nitrate, nitrite, sulfate, carbonate, and free hydroxide.
- Through XRD, the phase composition of the unwashed slurry was determined to be trona, nitratine, sodium nitrite, boehmite, gibbsite, and hematite. The ratio of boehmite to gibbsite in the slurry was found to be approximately equal. No burkeite was observed in the sample.

5.0 References

- ¹ Russell, K.J., *Sample Analysis for Sludge Batch 11 Sampling*, G-TTR-H-00019, Rev. 1, February 2023.
- ² Dekarske, J.R., *Task Technical and Quality Assurance Plan for Sample Analysis for Sludge Batch 11 Sampling*, SRNL-RP-2022-01039, Rev. 1, February 2023.
- ³ *Alkali Fusion Dissolutions of Sludge and Glass for Elemental and Anion Analysis (U)*, Manual L16.1, Procedure ADS-2502, Rev. 7, Savannah River National Laboratory, Aiken, SC.
- ⁴ *Aqua Regia Dissolution of Sludge for Elemental Analysis (U)*, Manual L16.1, Procedure ADS-2226, Rev. 10, Savannah River National Laboratory, Aiken, SC.
- ⁵ Savannah River Site *Conduct of Engineering*, Manual E7, Procedure 2.60, latest Revision, Savannah River Site, Aiken, SC.
- ⁶ “Savannah River National Laboratory Technical Report Design Check Guidelines”, Westinghouse Savannah River Company, WSRC-IM-2002-00011, Rev. 2, August 2004.
- ⁷ Dekarske, J.R., *Tank 35 (HTF-35-23-34/35) Expedited Analyses: Weight Percent Solids and Density*, SRNL-L3120-2023-00011, Rev. 0, May 2023.
- ⁸ Dekarske, J.R., *Tank 35 (HTF-35-23-34/35) Expedited Analyses: Radiochemical and Hg*, SRNL-L3120-2023-00012, Rev. 3, November 2023.
- ⁹ Dekarske, J.R., *Tank 35 (HTF-35-23-34/35) Elemental Analyses*, SRNL-L3120-2023-00014, Rev. 2, November 2023.
- ¹⁰ Dekarske, J.R., *Tank 35 (HTF-35-23-34/35) Gibbsite, Boehmite, and Burkeite*, SRNL-L3130-2023-00003, Rev. 0, August 2023.

Distribution:

cj.bannochie@srnl.doe.gov
William.bates@srnl.doe.gov
marion.cofer@srnl.doe.gov
alex.cozzi@srnl.doe.gov
connie.herman@srnl.doe.gov
brady.lee@srnl.doe.gov
Joseph.Manna@srnl.doe.gov
Gregg.Morgan@srnl.doe.gov
Mary.Whitehead@srnl.doe.gov
Brandi.clark@srnl.doe.gov
Patrick.Westover@srnl.doe.gov
frank.pennebaker@srnl.doe.gov
William.Ramsey@srnl.doe.gov
eric.skidmore@srnl.doe.gov
michael.stone@srnl.doe.gov
william.swift@srnl.doe.gov
Boyd.Wiedenman@srnl.doe.gov
bill.clark@srs.gov
jeffrey.crenshaw@srs.gov
james.folk@srs.gov
Curtis.Gardner@srs.gov
timothy.littleton@srs.gov
Anna.Murphy@srs.gov
tony.polk@srs.gov
Anthony.Robinson@srs.gov
mark-a.smith@srs.gov
patricia.suggs@srs.gov
thomas.temple@srs.gov
celia.aponte@srs.gov
timothy.baughman@srs.gov
Andrew.Marvel@srs.gov
Keisha.Martin@srs.gov
Donna.Yarbrough@srs.gov
phillip.norris@srs.gov
Christine.Ridgeway@srs.gov
Azadeh.Samadi-Dezfouli@srs.gov
Vijay.Jain@srs.gov
Bruce.wiersma@srnl.doe.gov
arthur.wiggins@srs.gov
Kirk.russell@srs.gov
John.pareizs@srnl.doe.gov
Chris.martino@srnl.doe.gov
Matthew.siegfried@srnl.doe.gov
John.dekarske@srnl.doe.gov
Crystal.currie@srnl.doe.gov
Seth.campbell@srs.gov
Mason.clark@srs.gov
Nicholas.shaub@srs.gov
Spencer.isom@srs.gov
Records Administration (EDWS)