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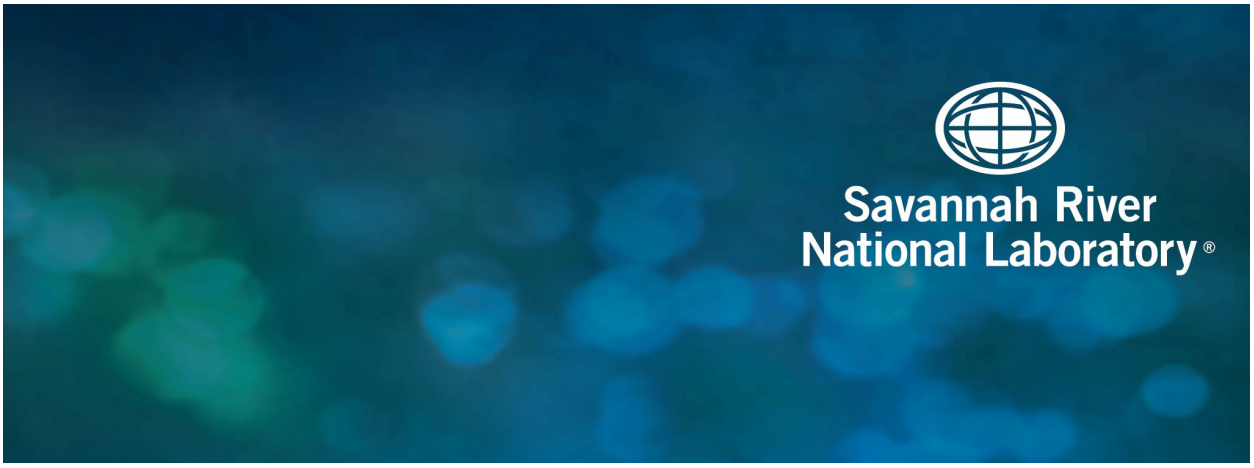

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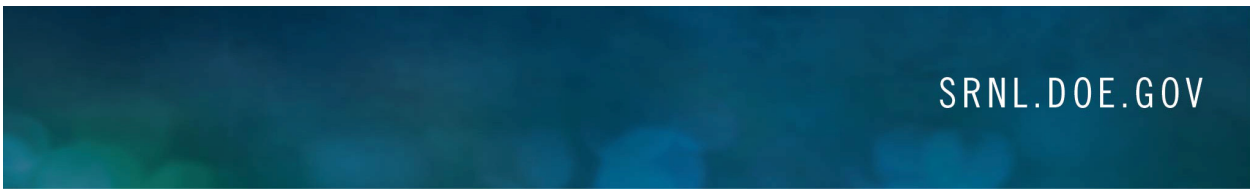
Characterization of the SRNL-Washed Tank 51 Sludge Batch 10 Qualification Sample

J. M. Pareizs

February 2022

SRNL-STI-2021-00449, Revision 0



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REVIEWS AND APPROVALS

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) personnel have been requested to qualify the next sludge batch (Sludge Batch 10 – SB10) for processing at the Defense Waste Processing Facility (DWPF). To accomplish this task, Savannah River Remediation (SRR) sent SRNL two 3-L samples of Tank 51H slurry to be characterized, washed, and then used in a lab-scale demonstration of the DWPF flowsheet. Sample HTF-51-19-114 was received on January 28, 2020, and HTF-51-20-15 was received on February 4, 2020. SRNL washed the Tank 51H sample per the Tank Farm washing strategy. During washing, material from H Canyon Tanks 16.3 and 16.4 was also added to the Tank 51 samples to simulate canyon discharges subsequent to sampling. A part of the qualification process is extensive radionuclide and chemical characterization of the SRNL-washed Tank 51H slurry. This report documents the chemical characterization of the washed slurry; radiological characterization will be documented in a separate report.

The major supernatant components, elements on a weight percent calcined basis, and the weight percent solids of the SRNL-washed sample were comparable to the Tank Farm projections, with the exception of free hydroxide and carbonate. Therefore, this SRNL-washed sample is suitable for further SB10 qualification activities and SRR planning for SB10.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF Figures

LIST OF ABBREVIATIONS

AF	alkali fusion
AR	aqua regia
CPC	Chemical Process Cell
DMA	SRNL-SaM method – direct mercury analysis
DWPF	Defense Waste Processing Facility
IC	ion chromatography
ICPES	SRNL-SaM method RADICPES (inductively coupled plasma – electron spectroscopy)
ICPMS	SRNL-SaM method RAD ICPMS (inductively coupled plasma – mass spectroscopy)
n	number of replicates
NA	not applicable
ND	not detected
RSD	relative standard deviation
SB10	Sludge Batch 10
SB9	Sludge Batch 9
SRNL	Savannah River National Laboratory
SRNL-SaM	Savannah River National Laboratory Sensing and Metrology
SRR	Savannah River Remediation
TIC	total inorganic carbon
Titr	SRNL-SaM method T BASE/OH/OTHER BASE EXC CO_3^{2-}
TOC	total organic carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VOA	volatile organic analysis
wt%	weight percent

1.0 Introduction

Savannah River National Laboratory (SRNL) personnel have been requested to qualify the next sludge batch (Sludge Batch 10 – SB10) for processing at the Defense Waste Processing Facility (DWPF).[1] To accomplish this task, Savannah River Remediation (SRR) sent SRNL two 3-L samples of Tank 51H slurry to be characterized, washed, and then used in a lab-scale demonstration of the DWPF flowsheet. Sample HTF-51-19-114 was received on January 28, 2020, and HTF-51-20-15 was received on February 4, 2020. SRNL has washed the Tank 51H sample per the Tank Farm washing strategy as of August 2020, and revised February 2021. During washing, material from H Canyon Tanks 16.3 and 16.4 was also added. A part of the qualification process is extensive radionuclide and chemical characterization of the SRNL-washed Tank 51H slurry. This report documents the chemical characterization of the washed slurry; radiological characterization will be documented in a separate report. This task is governed by a Task Technical and Quality Assurance Plan (TTQAP).[2]

2.0 Experimental Procedure

The two SB10 Tank 51 samples were combined into an 8-L beaker equipped with an overhead mixer and the ability to subsample while maintaining mixing. The slurry was subsampled and characterized.[3] See Figure 2-1 for a photograph of the washing vessel and qualification sample.

Figure 2-1. Photo of the Washing Vessel with Qualification Sample

2.1 Tank 51H Washing

Following the initial sample analysis, the slurry was washed. Initially, SRNL followed the washing plans of 9/24/20 (see Appendix A). During washing and following discussions between SRNL and SRR, washing plans changed, specifically the endpoint. The changed endpoint is shown in Appendix B. While SRNL targeted the same amount of H Canyon addition and the Tank Farm wash endpoint, SRNL accomplished this through fewer wash cycles (larger washes and decants). An outline of SRNL's washing follows. Note that more details, including analytical results during washing will be reported in the overall SB10 qualification report.

The as-received, combined Tank 51H material was allowed to settle, and a decant was completed (designated as Decant K). Washing was paused from March 2020 to September 2020 as plans for the addition of high aluminum-low uranium and sodium reactor experiment (SRE) solutions from H Canyon Tanks 16.3 and 16.4[4] were finalized.

In September, washing was resumed with the addition of wash water, settling and a decant (designated as Decant L). Mn (as $Mn(NO_3)_2$) and NaOH (50wt%) were added to H Canyon samples from Tanks 16.3 and 16.4 per H Canyon plans.[4] $Mn(NO_3)_2$ was added to Tank 16.3 and 16.4 material to attain a target of 64:1 Mn:$^{235}U_{eqslu}$ ratio. $^{235}U_{eqslu}$ is defined in the DWPF Waste Acceptance Criteria document.[5] NaOH was added to attain 1.2 M excess free hydroxide. Actual amounts added to the SRNL qualification sample are given in Table 2–1. The resulting mixtures were added to the Tank 51 sample. The amount of material added was determined by ratioing SRNL volumes to Tank Farm volumes. Following the H Canyon additions, an analysis of the supernatant showed a higher than projected concentration of sodium, indicating loss of water due to evaporation since the washing vessel is not leak tight. The subsequent wash water addition was adjusted to account for this loss. See Table 2–1 for addition amount.

Wash water was added and the sludge allowed to settle for Decant N. Wash water was added and the sludge allowed to settle for Decant Q. Wash water and 40 wt% $NaNO_2$ to target a NO_2^-/NO_3^- ratio of 1.8 was added, followed by settling and a decant (Decant R). Wash water was added and the sludge allowed to settle for Decant S.

Decant S was analyzed and the NO_2^-/NO_3^- was 2.3, significantly higher than the target of 1.8. Therefore, a portion of Decant N was added with the final wash to lower the ratio. The sludge was allowed to settle and then decanted. The resulting slurry, the SRNL washed Tank 51H SB10 material, was sampled and characterized.

A summary of SB10 washing is presented in Table 2–1. This should not be considered a rigorous mass balance. For example, the evaporation amount (on which the targeted amount of water added to account for evaporation was based) was determined by intermediate sample analysis. All decants were submitted for analysis, and several intermediate slurry samples were taken throughout washing. Wash amounts were adjusted based on analyses, particularly near the end of washing. A more detailed description of washing, along with the analytical results of the decants and intermediate slurry samples, will be included in the final SB10 qualification report.

Table 2–1. Summary of SRNL SB10-Tank 51 Washing

Initial Mass of SB10-Tank 51 Qualification Sample in Washing Vessel	6,540 g slurry
Decant K	–1,028 g supernatant
Wash Water Addition	+564 g wash water
Decant L	–362 g supernatant
Tank 16.4 Addition	+23.66 g Tank 16.4 +19.59 g 50% $Mn(NO_3)_2$ +22.12 g 50% NaOH +48.8 g water
Tank 16.3 Addition	+52.81 g Tank 16.3 +32.60 g 50% $Mn(NO_3)_2$ +45.61 g 50% NaOH +56.94 g water
An analysis after the 16.4 and 16.3 additions showed higher than expected Na and anion concentrations. This was attributed to evaporation. Wash water and NaOH was added as a wash and to account for the evaporation and readjust the Na to anion ratios.	+391 g 50 wt% NaOH +2,444 g water
Decant N	–2,656 g supernatant
Wash Water addition	+2,040 g wash water
Decant Q (small slurry sample taken after Decant Q)	–2,078 g supernatant
Wash water and $NaNO_2$ addition, targeting a NO_2/NO_3 ratio of 1.8	+152 g $NaNO_2$ +1,745 g wash water
Decant R	–1232 g supernatant
Wash water addition	+2,050 Wash Water
Decant S (small slurry sample taken after Decant S)	–1,918 g
Wash water and material from N addition to adjust NO_2/NO_3 ratio	+1,200 g of Decant N +1,000 g wash water
Decant	–1,826 g supernatant
Wash Water Addition	+1,147 g wash water
Final Decant	–1,552 g

2.2 Weight Percent (wt%) Solids and Density

Aliquots of slurry and supernatant were dried to a constant weight (change between subsequent weights of <0.005 g or weight increase) at 110 °C for wt% total dried solids and wt% dissolved solids, respectively. Supernatant was obtained by decanting; the washing final decant was sampled for supernatant analyses. Wt% insoluble and soluble solids were calculated from the total and dissolved solids measurements. Dried slurry samples were heated to 1100 °C, held at that temperature, and then cooled and weighed to determine wt% calcined solids.

Slurry and supernatant densities were determined gravimetrically from sample weights in vessels of known volume (plastic test tubes of nominally 8 mL capacity). As stated above, supernatant from SRNL's final decant was used for the density measurements.

2.3 Sample Preparations for Supernatant Characterization

The required results of supernatant characterization include various anions, free hydroxide, and elemental constituents. Decanted (unfiltered) supernatant from the final decant (see above) was submitted directly to Savanah River National Laboratory-Sensing and Metrology (SRNL-SaM) in quadruplicate.

2.4 Sample Preparations for Slurry Analysis

Slurry samples were diluted nominally 30× with water. Unfiltered aliquots were submitted to SRNL-SaM for organic carbon, inorganic carbon, volatile organic carbon and total base (base to reach pH of 7). After pulling unfiltered aliquots, the dilutions were allowed to settle and the aqueous portion was sampled and submitted for anions. The anions and total base are predominantly needed for the Chemical Processing Cell (CPC) demonstration in the SRNL Shielded Cells.

2.5 Sample Preparations for Total Dried Solids Characterization

To characterize the solids of the Tank 51H sample, aliquots of slurry were digested and submitted to SRNL-SaM for analysis. Slurry samples were digested by two methods, aqua regia (AR) and alkali fusion (AF). For the AR digestions, aliquots of slurry were mixed with AR and heated in closed vessels for several hours at ~110 °C. The resulting liquids were diluted to 100 mL with water and submitted to SRNL-SaM for analysis. For the AF digestions, aliquots of slurry were dried (~110 °C) and fused at 675 °C with sodium peroxide. The flux was then dissolved with nitric acid and diluted to 100 mL with water. Details of the digestion methods can be found in Procedure L16.1-ADS-2226 (AR) and Procedure L16.1-ADS-2502 (AF).[6] The SRNL-SaM results were then converted from a slurry basis to a wt% total solids basis using the measured wt% total solids. Reagent blanks and digested glass of known composition were processed and submitted for analysis at the same time as process samples. Results of these samples were used in evaluating AR and AF slurry digestion effectiveness and evaluation of contamination in the samples from the Shielded Cells.

2.6 Summary of Sample Preparations and SRNL-SaM Methods

Given in Table 2–2 are the types of samples (described above) and the SRNL-SaM methods used for characterization. Table 2–3 lists the SRNL-SaM methods and the analytes reported from these methods.

Table 2–2. Analyses Performed by SRNL-SaM

SRNL-SaM Method	Supernatant	Slurry Dilutions	Slurry Dilutions (Aqueous Phase)	Aqua Regia Digestions	Alkali Fusion Digestions
RAD ICPES	X	–	–	X	X
AA_DMA_TOTAL_MERCURY	X	–	–	X	–
RAD_ICPMS_AS and RAD_ICPMS_SE	–	–	–	X	–
IC ANIONS	X	–	X	–	–
T BASE/OH/OTHER BASE EXC CO_3^{2-} (Titr)	X	X	–	–	–
TIC/TOC	X	X	–	–	–
RAD ICPMS	–	–	–	X	–
VOA	–	X	–	–	–

X: Sample prep submitted to SRNL-SaM; –: Sample prep not submitted to SRNL-SaM for this analysis.

Table 2–3. Analytes from SRNL-SaM Methods

SRNL-SaM Method	Expected Results to Satisfy Technical Task Request Requirements
RAD ICPES	Ag, Al, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, Gd, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Si, Sn, Sr, Th, Ti, U, V, Zn, Zr
AA_DMA_TOTAL_MERCURY	Hg
RAD_ICPMS_AS and RAD_ICPMS_SE	As, and Se
IC ANIONS	Bromide, Chloride, Fluoride, Formate, Nitrate, Nitrite, Oxalate, Phosphate, Sulfate
T BASE/OH/OTHER BASE EXC CO_3^{2-} (Titr)	Free OH^-
TIC/TOC	Total inorganic carbon (CO_3^{2-} is calculated from the TIC result), and total organic carbon
RAD ICPMS	Isotopic results are used to calculate Pd, Ru, Rh, Nd, and Pb
VOA	benzene, toluene, isopropanol, and butanol.

2.7 Quality Assurance

This work was performed under a TTR.[1] The analysis herein satisfies the chemical characterization portion of Task 1, Item 3 of the TTQAP associated with this TTR. The TTR identifies these results as waste form affecting and complies with requirements of RW-0333P. The TTR also identifies the Functional Classification as Safety Class. Thus, this document was reviewed by Design Verification by Document Review. The requirements for performing reviews of technical reports and the extent of review are established in manual E7 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.

3.0 Results and Discussion

3.1 Density and Wt% Solids

Presented in Table 3–1 are the density and wt% solids results. As stated in Section 2.2, decanted supernatant was used for supernatant density and wt% dissolved solids measurements.

The wt% insoluble solids and soluble solids are calculated from the measured wt% total dried solids and wt.% dissolved solids.[7] Wt% calcined solids were determined by heating dried slurry to 1100 °C, cooling, and weighing.

Table 3–1. Densities and Wt% Solids of the SRNL-Washed SB10 Tank 51 Qualification Sample

Property	Result	RSD, n*
Slurry Density (g/mL) T = 18 °C	1.10	1.0%, 4
Supernatant Density (g/mL) T = 22 °C	1.06	0.4%, 4
Wt% Total Dried Solids (Slurry Basis)	15.0	0.8%, 4
Wt% Dissolved Solids (Supernatant basis)	7.4	3%, 3
Wt% Insoluble Solids (Slurry Basis)	8.2	NA
Wt% Soluble Solids (Slurry Basis)	6.8	NA
Wt% Calcined Solids (Slurry Basis)	11.3	0.1%, 3

* RSD = relative standard deviation, with n equal to the number of measurements. NA = not applicable, as result is calculated.

3.2 Supernatant Analytical Results

Presented in Table 3–2 are anion, carbonate, and free OH supernatant results. These results were determined from analysis of decanted (unfiltered) supernatant – the final decant. Anions (with the exception of carbonate and hydroxide) were determined by IC. Carbonate was determined from a TIC analysis by assuming all TIC was carbonate. Free hydroxide was determined from a titration (Procedure L16.1-ADS-1207).[6]

Elements quantified by ICPES and Direct Mercury Analysis (DMA) are reported in Table 3–3. As can be seen, only Al, Cr, Hg, Na, and S were detected in the supernatant.

Table 3–2. Supernatant Anion Results

Analyte	Analytical Method*	Units	Result	RSD, n=4†
Bromide	IC	M	<6.26E-04	NA
Chloride	IC	M	1.06E-03	0.3%
Fluoride	IC	M	<5.26E-03	NA
Formate	IC	M	<2.22E-04	NA
Nitrate	IC	M	1.46E-01	1%
Nitrite	IC	M	2.34E-01	1%
Oxalate	IC	M	7.11E-03	0.1%
Phosphate	IC	M	2.37E-04	3%
Sulfate	IC	M	2.12E-02	0.4%
Carbonate	TIC	M	2.24E-01	1%
Free OH	Titr.	M	2.40E-01	5%

*IC = ion chromatography; TIC = total inorganic carbon (all inorganic carbon is assumed to be carbonate); Titr. = titration.
† RSD = relative standard deviation, with n equal to the number of measurements.

Table 3–3. Supernatant Elemental Results

Element	mg/L	M	RSD, n=4†
Ag	<2.4E-01	<2.2E-06	NA
Al	1.21E+03	4.48E-02	3%
B	<2.1E+01	<2.0E-03	NA
Ba	<1.5E-01	<1.1E-06	NA
Be	<1.1E-01	<1.3E-05	NA
Ca	<4.3E+00	<1.1E-04	NA
Cd	<3.0E-01	<2.7E-06	NA
Ce	<1.3E+00	<9.4E-06	NA
Co	<7.1E-01	<1.2E-05	NA
Cr	5.35E+01	1.03E-03	3%
Cu	<1.7E+00	<2.7E-05	NA
Fe	<1.3E+00	<2.4E-05	NA
Gd	<5.3E-01	<3.4E-06	NA
Hg	1.79E+02	8.92E-04	35%
K	<5.5E+01	<1.4E-03	NA
La	<3.6E-01	<2.6E-06	NA
Li	<2.0E+00	<2.9E-04	NA
Mg	<3.2E-01	<1.3E-05	NA

Element	mg/L	M	RSD, n=4†
Mn	<8.1E-01	<1.5E-05	NA
Mo	<4.1E+00	<4.3E-05	NA
Na	2.37E+04	1.03E+00	4%
Ni	<3.8E+00	<6.4E-05	NA
P	<6.9E+00	<2.2E-04	NA
Pb	<1.5E+00	<7.2E-06	NA
S	6.38E+02	1.99E-02	3%
Sb	<7.4E+00	<6.0E-05	NA
Si	<1.0E+01	<3.6E-04	NA
Sn	<6.1E+00	<5.2E-05	NA
Sr	<6.9E-01	<7.9E-06	NA
Th	<3.2E+00	<1.4E-05	NA
Ti	<1.4E+00	<2.8E-05	NA
U	<6.8E+00	<2.9E-05	NA
V	<1.4E+00	<2.8E-05	NA
Zn	<1.0E+00	<1.5E-05	NA
Zr	<2.1E-01	<2.3E-06	NA

† RSD = relative standard deviation, with n equal to the number of measurements.

Presented in Table 3–4 is a comparison between Tank Farm targets from projections from February 16, 2021 and measurements of the SRNL washed Tank 51 sample. See Appendix B for an excerpt from the Tank Farm planning spreadsheet. When washing, SRNL targeted sodium and weight percent insoluble solids. That is, adjustments were not made to adjust free OH and carbonate, the two analytes that are significantly different between targets and measured results. This difference (lower hydroxide and higher carbonate) was also seen with the Sludge Batch 9 sample.[8]

The SRNL sample is slightly less washed than tank farm projections, resulting in a higher wt% total dried solids. SRNL completed the final decant after the sludge stopped settling, and as much supernatant was decanted as possible without disturbing the sludge. That is, SRNL obtained the highest reasonably attainable wt% insoluble solids in this configuration (8-L beaker) with this sludge.

Table 3–4. Comparison Between Tank Farm Projections and Measurements

Analysis	Units	Projection	Measurement
Wt% Insol. Solids	wt%	8.85	8.2*
Wt% Total Dried Solids	wt%	13.85	15.0*
Supernatant Density	g/mL	1.04	1.06*
Sodium	M	0.940	1.03‡
Nitrite	M	0.209	0.234†
Nitrate	M	0.143	0.146†
Free OH	M	0.425	0.240†
Chloride	M	0.001	0.00106†
Sulfur	M	0.0175	0.0199‡
Fluoride	M	0.005	<5.3E-03†
Carbonate	M	0.036	0.224†
Aluminum	M	0.042	0.0448‡
Oxalate	M	0.006	0.00711†

* From Table 3–1. † From Table 3–2. ‡From Table 3–3

3.3 Slurry Analytical Results

Presented in Table 3–5 are analytical results from slurry dilutions. The analytical methods for each analyte are also presented.

Table 3–5. Slurry Dilution Results

Analyte	Analytical Method*	Units	Result	RSD, n=4†
Bromide	IC	mg/kg	<1.6E+03	NA
Chloride	IC	mg/kg	<3.1E+02	NA
Fluoride	IC	mg/kg	<3.1E+02	NA
Formate	IC	mg/kg	<3.1E+02	NA
Nitrate	IC	mg/kg	7.67E+03	3%
Nitrite	IC	mg/kg	9.27E+03	3%
Oxalate	IC	mg/kg	5.66E+02	5%
Phosphate	IC	mg/kg	<3.1E+02	NA
Sulfate	IC	mg/kg	1.69E+03	5%
Base to pH 7	Titr.	M	6.50E-01	3%
Inorganic Carbon	TIC	mg/kg	2.10E+03	7%
Organic Carbon	TOC	mg/kg	<6.3E+02	NA
Volatile organic carbon	VOA	mg/kg	<6.3E+00	NA

* IC = ion chromatography; TIC = total inorganic carbon; TOC = total organic carbon; VOA = volatile organic analysis; Titr. = titration

† RSD = relative standard deviation, with n equal to the number of measurements.

3.4 Analysis of Total Dried Solids

Presented in Table 3–6 are elemental analyses of the total dried solids of the SRNL-washed Tank 51H sample. As described above, slurry material was digested by both AR and AF. Both digestions were submitted for ICPES; AR digestions were submitted for DMA and ICPMS. In addition to the slurry

samples, reagent blanks and digestions of a reference glass of known composition[9] were analyzed by ICPES. The results of the blanks and reference glass digestions were used in evaluating the slurry results (as discussed below).

- The element Hg (total Hg) was determined from AR digestions and the SRNL-SaM DMA method. A portion of Hg would be volatilized in the AF digestion, therefore, AF digestions are not submitted for Hg analysis.
- For Al, the alkali fusion result was used. The result from AF was significantly higher than the result from AR and it is known that some mineral forms of Al are not dissolved by AR.
- For the elements Ba, Ce, Cr, Cu, Fe, Gd, Mg, Mn, Ni, Sr, Th, U, and Zn, both ICPES measurements from AR and AFs were used because there was no significant difference between respective analytical results.
- For Ca, results from the AR digestion were used because Ca was detected in the AF blank, likely due to a Ca impurity in the reagent chemicals.
- For Na, S, and Zr, the AR digestion results were used. AFs utilize Na as a reagent and they are performed in Zr crucibles, thus these elements cannot be determined from AF. Some S may volatilize in the AF process, therefore, only the AR digestion is used for this element.
- For Si, results from the AF digestion were used because the AR results were low in the reference glass digestion.
- For La, the AR by ES result was used. There was not good agreement between the two digestion results. The result from AR was nearly 50% higher than the result from AF.
- Several elements were determined from RAD ICPMS results from AR digestions. Nd was calculated from the sum of masses 143-146, 148, and 150. Note that Nd may be biased low since mass 142 is not included in the calculation because both Ce and Nd contribute to mass 142. Pb was calculated from the sum of masses 206 to 208. Pd was calculated from mass 105 and fission yield values from masses 105-108 and 110.[10] Rh was determined from mass 103. Ru was calculated from the sum of masses 101, 102, and 104.
- AR digestions were submitted for As and Se measurements by ICPMS.
- Slurry dilution results (Table 3–4) were used for Cl and F. The detection limits in the slurry dilutions were placed on a total solids basis by utilizing the slurry wt% total solids. This calculation assumes that Cl and F are soluble in the slurry.
- For the elements not detected in either digestion, the lower of the two methods is reported. For B, Cd, Co, Li, P, Sb, Sn, Ti, and V, the detection limits from AF are reported. For Ag, Be, K, and Mo, the detection limits from AR are reported.

Samples were digested by each method in quadruplicate. Thus, if only one digestion method is reported, number of samples (n) would equal 4. If both digestions are used, n=8. All replicates for Fe are reported in Table 3–7, as requested in the TTR.[1]

Table 3–6. Elemental Composition of Total Dried Solids

Element	Dig, Analytical Method*	wt% of Total Dried Solids	RSD, n‡
Ag	AR, ES	<8.7E-03	NA
Al	AF, ES	1.24E+01	5%, 4
As §	AR, MS	3.28E-03	4%, 4
B	AF, ES	<8.3E-02	NA
Ba	AR/AF, ES	4.59E-02	5%, 8
Be	AR, ES	<2.4E-03	NA
Ca	AR, ES	5.30E-01	3%, 4
Cd	AF, ES	<4.0E-03	NA
Ce	AR/AF, ES	9.72E-02	9%, 8
Cl	SL, IC	<2.1E-01	NA
Co	AF, ES	<4.7E-03	NA
Cr	AR/AF, ES	1.61E-01	3%, 8
Cu	AR/AF, ES	3.52E-02	4%, 8
F	SL, IC	<2.1E-01	NA
Fe	AR/AF, ES	8.51E+00	3%, 8
Gd	AR/AF, ES	4.44E-02	5%, 8
Hg	AR, DMA	2.85E+00	6%, 4
K	AR, ES	<7.6E-02	NA
La	AR, ES	3.38E-02	3%, 4
Li	AF, ES	<6.3E-03	NA
Mg	AR/AF, ES	2.09E-01	3%, 8
Mn	AR/AF, ES	2.66E+00	3%, 8
Mo	AR, ES	<7.7E-03	NA
Na	AR, ES	1.66E+01	1%, 4
Nd	AR, MS†	1.27E-01	NA
Ni	AR/AF, ES	2.74E-01	5%, 8
P	AF, ES	<9.3E-02	NA
Pb	AR, MS†	9.32E-03	NA
Pd	AR, MS†	1.45E-03	NA
Rh	AR, MS†	8.70E-03	NA
Ru	AR, MS†	4.24E-02	NA
S	AR, ES	3.79E-01	2%, 4
Sb	AF, ES	<1.7E-02	NA
Se	AR, MS	<1.0E-04	NA
Si	AF, ES	4.24E-01	2%, 4
Sn	AF, ES	<2.5E-02	NA
Sr	AR/AF, ES	1.63E-02	5%, 8
Th	AR/AF, ES	1.73E+00	3%, 8
Ti	AF, ES	<1.4E-02	NA
U	AR/AF, ES	2.22E+00	5%, 8
V	AF, ES	<3.4E-03	NA
Zn	AR/AF, ES	1.75E-02	5%, 8
Zr	AR, ES	1.13E-01	3%, 4

* Dig, Analytical Method: AR=Aqua Regia; AF=Alkali Fusion; ES= ICPES; MS=ICPMS; DMA=direct mercury analysis. For Cl and F, slurry dilution (SL) IC results were used; it is assumed that any F or Cl present in the slurry would be soluble.

† For the elements quantified by ICPMS: Nd is calculated from the sum of masses 143-146, 148, and 150; Pb is calculated from the sum of masses 206 to 208; Rh is determined from mass 103; Ru is calculated by summing masses 101, 102, and 104; Pd is calculated from mass 105 and fission yields from masses 105-108 and 110; As and Se were direct ICP-MS measurements.

‡ RSD = relative standard deviation; n = number of replicates.

§ The As result may be biased high due to possible interferences with double-charged lanthanides. See Jones (2018)[11] for a discussion.

Table 3–7. Iron Replicates

Digestion	Wt% of Total Dried Solids
Aqua Regia	8.93E+00 8.33E+00 8.60E+00 8.47E+00
Alkali Fusion	8.33E+00 8.20E+00 8.67E+00 8.53E+00

SRNL was requested to compare the elemental composition of the washed sample calcined solids to the Tank Farm projections. Per email from SRR, their projected elemental composition is a normalized composition. Therefore, SRNL normalized results for comparison. Elements that were detected by SRNL and listed by SRR were converted to oxides using oxide and element molecular weights. The oxides were then summed. Each oxide was then divided by the sum. Finally, the oxide was converted from oxide to element. SRR's projected composition is presented in Appendix C. SRNL's calculation is presented in Appendix D. A comparison between SRR's projection and SRNL results is presented in Table 3–8. As can be seen, with the exception of the low concentration elements, there is good agreement between the Tank Farm projections and SRNL's results for elements on a calcined solids basis; percent difference is less than 4% for elements greater than 1% in the calcined solids. The agreement between Tank Farm and SRNL Hg is greater than 10%. Hg is not on a calcined solids basis as it is not present in the final glass waste form.

Table 3–8. Comparison Between SRR Projections and SRNL Results (Elements on a wt% of Calcined Solids Basis)

Element	Tank Farm Projection	SRNL Result†	% Difference *
Al	17.53	17.7	-1.0
B	0.003	ND	NA
Ba	0.06	0.0655	-8.8
Ca	0.74	0.756	-2.1
Ce	0.12	0.139	-14.7
Cr	0.23	0.230	0.0
Cu	0.05	0.0502	-0.4
Fe	11.91	12.1	-1.6
K	0.08	ND	NA
La	0.03	0.0482	-46.5
Li	0.02	ND	NA
Mg	0.29	0.298	-2.7
Mn	3.72	3.79	-1.9
Na	24.44	23.7	3.1
Ni	0.38	0.391	-2.9
Pb	0.02	0.0133	40.2
S	0.54	0.540	0.0
Si	0.59	0.605	-2.5
Th	2.42	2.47	-2.0
Ti	0.02	ND	NA
U	3.08	3.17	-2.9
Zn	0.02	0.03	-22.2
Zr	0.16	0.161	-0.6
Hg (wt% of total dried solids)	3.23	2.85	12.4

† ND=not detected in the total dried solids and thus not shown here.

* $\%Difference = \frac{Tank\ Farm\ Projection - SRNL\ Result}{Average(Tank\ Farm\ Projection, SRNL\ Result)} \times 100$

3.5 Carbon Analysis

Presented in Table 3–9 are results of various carbon measurements. Only inorganic carbon was detected in the slurry. No volatile compounds were detected. Note that semivolatile analyses are not yet complete and will be reported in the final SB10 qualification report.

Table 3–9. Carbon Analysis

Analysis	Result (mg/kg slurry)	RSD, n‡
Total Inorganic Carbon	2.10E+03	7%, 4
Total Organic Carbon	<6.3E+02	NA
Volatile Organics Analysis	<6.3E+00	NA

‡ RSD = relative standard deviation; n = number of replicates.

4.0 Conclusion and Recommendation

The major supernatant components, elements on a weight percent calcined basis, and the weight percent solids of the SRNL-washed sample were comparable to the Tank Farm projections, with the exception of free hydroxide and carbonate. Therefore, this SRNL-washed sample is suitable for further SB10 qualification activities and SRR planning for SB10.

5.0 References

1. K.J. Russell, "Technical Task Request: Sludge Batch 10 Qualification, Confirmatory, and Waste Acceptance Product Specifications Samples," Savannah River Remediation, Aiken, SC, X-TTR-S-00075, Rev. 3, 2021.

2. J.M. Pareizs and C.J. Martino, "Task Technical and Quality Assurance Plan for Sludge Batch 10 Sample Analysis and Qualification," Savannah River National Laboratory, Aiken, SC, SRNL-RP-2019-00658, Rev. 2, 2021.

3. J.M. Pareizs, "Analytical Results of the Tank 51H Sludge Batch 10 Qualification Sample," Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2020-00008, Rev. 0, 2020.

4. W.M. Bennett, "Waste Characterization Report for High Aluminum-Low Uranium and SRE Solutions in Tanks 16.3 and 16.4 for Disposal to Sludge Batch 10," SRNS-E1120-2020-00008, Rev. 3, 2020.

5. J.W. Ray, "Waste Acceptance Criteria for Raw Salt Solution, Sludge and SWPF Salt Streams Transfers to DWPF (U)," Savannah River Remediation, Aiken, SC, X-SD-S-00001, Rev. 1, 2019.

6. "Analytical Operating Procedures," Savannah River National Laboratory, Aiken, SC, Manual L16.1, Latest Revision.

7. J.C. Marek, "Correction Factor for Soluble and Insoluble Solids (U)," Savannah River Site, Aiken, SC, SRTC-PTD-92-0040, Rev. 0, 1992.

8. J.M. Pareizs, "Characterization of the SRNL-Washed Tank 51 Sludge Batch 9 Qualification Sample," Savannah River National Laboratory, Aiken, SC, SRNL-STI-2015-00693, Rev. 0, 2016.

9. G.L. Smith, "Characterization of Analytical Reference Glass – 1 (Arg-1)," Pacific Northwest National Laboratory, Richland, WA, PNL-8992, 1993.

10. N.E. Bibler, "Measuring and Predicting Fission Product Noble Metals in Savannah River Site High Level Waste Sludges," Savannah River National Laboratory, Aiken, SC, WSRC-TR-2005-00098, Rev. 0, 2005.

11. M.A. Jones, N. Wyeth, T.L. White, M.L. Whitehead, and C.M. Gregory, "Inductively Coupled Plasma Mass Spectrometry (Icpms) and Emission Spectroscopy (Icpes) to Replace Atomic Absorption (Aa) for as and Se Measurements," Savannah River National Laboratory, Aiken, SC, SRNL-L4130-2018-00006, Rev. 0, 2018.

Appendix A. SRR Wash Sheet - 092420

	A	DJ	DK	DL	DM	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW
1				2/29/2020					8/17/2020						
2	Tank 51	Tank 51 after Burkeite Dissolution	Tank 51 with As-Received Qual Sample Results	Decant K to Tank 32	Tank 51 after Decant	Add IW	Tank 51 with IW	Adjust Oxalate	Decant L to Tank 37	Tank 51 after Decant	Added IW	Added 50 Wt% NaOH	Tank 51 with IW and Caustic	Add SRE Stream from Tank 16.4	Add SRE Stream from Tank 16.3
3	***Initial tank Level (in)***	**261.90**	261.90		**227.81**		**255.10**	255.10		**240.20**			240.20		
4	***liquid volume (gal)***	877752	902958	119656	758097	95788	853884	854339	**52299**	802040	0	0	802040	**16199**	**12882**
5	***solids volume (gal)***	41517	16311		41517		41517	41062		41062			41062	258	215
6	***settled sludge level (in)***	194.09						186.19							
7	***kg insol. solids***	330685	246205		246205		246205	242183		242183			242183	**3176**	**2652**
8	***wt% insol solids***	7.88	**5.8**		6.85			6.12		6.49					
9	***decanted level***	219.09						**112.10**							
10						OH/Al =	7.13				OH/Al =		7.13		
11															
12	***Mn***														
13	***SpG***	1.163	**1.17**	1.167	1.167	1	1.148	1.149	1.148	1.149	1	1.530	1.149	1.150	1.175
14	***Na***	**3.720**	**3.610**	3.665	3.665	0.021	3.256	3.273	3.256	3.273	0.021	19.125	3.273	3.869	4.551
15	***NO2***	**0.428**	**0.358**	0.393	0.393	0.011	0.350	0.350	0.350	0.350	0.011		0.350	**0.002**	**0.003**
16	***NO3***	**0.702**	**0.589**	0.646	0.646		0.573	0.573	0.573	0.573			0.573	**2.339**	**3.016**
17	***OH***	**1.740**	**1.530**	1.635	1.635	0.01	1.453	1.452	1.453	1.452	0.01	19.125	1.452	**1.280**	**1.280**
18	***Cl***	**0.0057**	**<0.01**	0.006	0.006		0.005	0.005	0.005	0.005			0.005	**0.003**	**0.005**
19	***SO4 or S***	**0.1004**	**0.0955**	0.098	0.098		0.087	0.087	0.087	0.087			0.087	**0.001**	**0.002**
20	***F***	**0.003**	**<0.03**	0.003	0.003		0.003	0.003	0.003	0.003			0.003	**0.010**	**0.015**
21	***CO3-2***	**0.188**	**0.231**	0.210	0.210		0.186	0.186	0.186	0.186			0.186	**0.000**	**0.000**
22	***AlO2***	**0.237**	**0.222**	0.230	0.230		0.204	0.204	0.204	0.204			0.204	**0.232**	**0.229**
23	***C2O4-2***	**0.01250**	**0.01250**	0.0125	0.0125		0.011	0.02037	0.011	0.020			0.020		
24	***PO4-3***	0.0012	**<0.005**	0.0012	0.0012		0.001	0.001	0.001	0.001			0.001		
25	***K***	**0.00882**	**0.00854**	0.0087	0.0087		0.008	0.008	0.008	0.008			0.008	**0.011**	**0.015**
26	***slurry spg***		**1.22**		1.187		1.167			**1.169**			1.1685419		
27	***Na2C2O4 Solubility***	0.01710						0.02053							
28	***solid Na2C2O4, kg***	7878			7878		7878	3856		3856					
29	***Mass TS, kg***	1019450	997553	99565	897988	420	898408	898408	40171	858237	0	0	858237	19755	18659
30	***wt% TS***		**23.5**												

	A	DX	DY	DZ	EA	EB	EC	ED	EE	EF	EG	EH	EI	EJ	EK
1						3/13/2021									4/6/2021
2	Tank 51	Add SRE Stream from Tank 16.3	SRE Post-Flush	Tank 51 with Additions	Adjust Oxalate	Decant M to Tank 32	Tank 51 after Decant	Add 50 Wt% NaOH	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.4	SRE Post-Flush	Tank 51 with SRE Stream & IW	Adjust Oxalate	Decant N to Tank 30
3	***Initial tank Level (in)***			252.78	252.78		207.91			260.84			260.84	260.84	
4	***liquid volume (gal)***	12882	1500	845503	845541	**157500**	688041	23447	162358	873846	**0**	0	873846	874143	**157500**
5	***solids volume (gal)***	215		41750	41712		41712			41712	0		41712	41415	
6	***settled sludge level (in)***				172.52									190.97	
7	***kg insol. solids***	2652		250663	250327		250327			250327	**0**		250327	247697	
8	***wt% insol solids***				6.37		7.72							6.20	
9	***decanted level***				197.52									215.97	
10				7.05					OH/Al =	10.25					
11															
12	***Mn***									0.49217					
13	***SpG***	1.175	1.033	1.149	1.149	1.149	1.149	1.530	1	1.132	1.243	1.033	1.132	1.132	1.132
14	***Na***	4.551	1.200	3.320	3.321	3.320	3.321	19.125	0.021	3.132	6.423	1.200	3.132	3.143	3.132
15	***NO2***	0.003		0.332	0.332	0.332	0.332		0.011	0.264	**0.004**		0.264	0.263	0.264
16	***NO3***	3.016		0.680	0.680	0.680	0.680			0.535	**4.339**		0.535	0.535	0.535
17	***OH***	1.280	1.200	1.443	1.443	1.443	1.443	19.125	0.01	1.651	**1.620**	1.200	1.651	1.651	1.651
18	***Cl***	0.005		0.005	0.005	0.005	0.005			0.004	**0.006**		0.004	0.004	0.004
19	***SO4 or S***	0.002		0.083	0.083	0.083	0.083			0.065	**0.003**		0.065	0.065	0.065
20	***F***	0.015		0.003	0.003	0.003	0.003			0.003	**0.019**		0.003	0.003	0.003
21	***CO3-2***	0.000		0.176	0.176	0.176	0.176			0.139	**0.000**		0.139	0.139	0.139
22	***AlO2***	0.229		0.205	0.205	0.205	0.205			0.161	**0.430**		0.161	0.161	0.161
23	***C2O4-2***			0.019	0.02011	0.019	0.020			0.016			0.016	0.02176	0.016
24	***PO4-3***			0.001	0.001	0.001	0.001			0.001			0.001	0.001	0.001
25	***K***	0.015		0.008	0.008	0.008	0.008			0.006	**0.020**		0.006	0.006	0.006
26	***slurry spg***														
27	***Na2C2O4 Solubility***				0.02011									0.02176	
28	***solid Na2C2O4, kg***			3856	3520		3520			3520			3520	890	
29	***Mass TS, kg***	18659	273	915583	915583	123918	791665	67891	712	860268	0	0	860268	860268	110371
30	***wt% TS***														

	A	EL	EM	EN	EO	EP	EQ	ER	ES	ET	EU	EV	EW	EX	EY	EZ	FA
1										5/16/2021							
2	Tank 51	Tank 51 after Decant	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.3	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Adjust Oxalate	Decant O to Tank 32	Tank 51 after Decant	Add 40 Wt% NaNO2	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.3	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	215.97		247.57				247.57	247.57		201.42			251.62			
4	***liquid volume (gal)***	716643	110916	827559	**0**	~~**0**~~	0	827559	827659	**162000**	665659	0	176223	841882	**0**	~~**0**~~	0
5	***solids volume (gal)***	41415		41415	0	~~0~~		41415	41315		41315			41315	0	~~0~~	
6	***settled sludge level (in)***								176.42								
7	***kg insol. solids***	247697		247697	**0**	~~**0**~~		247697	246807		246807			246807	**0**	~~**0**~~	
8	***wt% insol solids***	7.46							6.60		8.08						
9	***decanted level***								201.42								
10																	
11																	
12	***Mn***																
13	***SpG***	1.132	1	1.114	1.245	~~1.262~~	1.033	1.114	1.115	1.115	1.115	1.320	1	1.091	1.245	~~1.243~~	1.033
14	***Na***	3.143	0.021	2.724	6.488	~~6.956~~	1.200	2.724	2.728	2.728	2.728	7.652	0.021	2.162	6.488	~~6.423~~	1.200
15	***NO2***	0.263	0.011	0.230	**0.004**	~~**0.004**~~		0.230	0.230	0.230	0.230	7.652	0.011	0.184	**0.004**	~~**0.004**~~	
16	***NO3***	0.535		0.464	**4.452**	~~**4.661**~~		0.464	0.463	0.463	0.463			0.366	**4.452**	~~**4.339**~~	
17	***OH***	1.651	0.01	1.431	**1.660**	~~**1.870**~~	1.200	1.431	1.430	1.430	1.430		0.01	1.133	**1.660**	~~**1.620**~~	1.200
18	***Cl***	0.004		0.003	**0.007**	~~**0.006**~~		0.003	0.003	0.003	0.003			0.003	**0.007**	~~**0.006**~~	
19	***SO4 or S***	0.065		0.056	**0.003**	~~**0.003**~~		0.056	0.056	0.056	0.056			0.044	**0.003**	~~**0.003**~~	
20	***F***	0.003		0.002	**0.022**	~~**0.018**~~		0.002	0.002	0.002	0.002			0.002	**0.022**	~~**0.019**~~	
21	***CO3-2***	0.139		0.120	**0.000**	~~**0.000**~~		0.120	0.120	0.120	0.120			0.095	**0.000**	~~**0.000**~~	
22	***AlO2***	0.161		0.139	**0.337**	~~**0.393**~~		0.139	0.139	0.139	0.139			0.110	**0.337**	~~**0.430**~~	
23	***C2O4-2***	0.022		0.019				0.019	0.02096	0.021	0.021			0.017			
24	***PO4-3***	0.001		0.001				0.001	0.001	0.001	0.001			0.001			
25	***K***	0.006		0.005	**0.023**	~~**0.018**~~		0.005	0.005	0.005	0.005			0.004	**0.023**	~~**0.020**~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***								0.02665								
28	***solid Na2C2O4, kg***							890	0								
29	***Mass TS, kg***	749897	487	750384	0	~~0~~	0	750384	750384	98566	651817	0	773	652591	0	~~0~~	0
30	***wt% TS***																

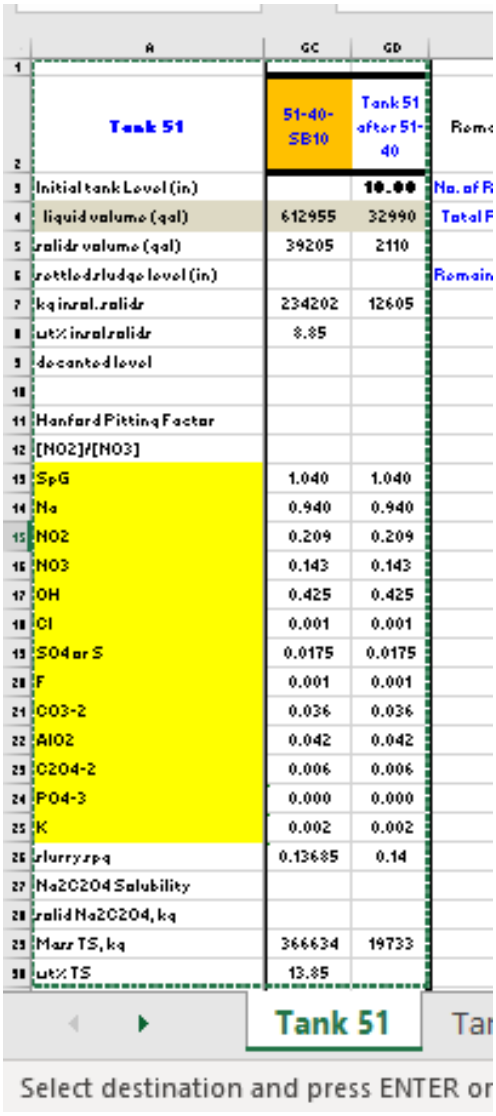
	A	FB	FC	FD	FE	FF	FG	FH	FI	FJ	FK	FL	FM	FN	FO	FP	FQ
1			8/13/2021								9/6/2021						
2	Tank 51	Tank 51 with SRE Stream & IW	Decant P to Tank 32	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant Q to Tank 32	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	251.62		205.47						220.96		204.58					
4	***liquid volume (gal)***	841882	162000	679882	54360	0	~~0~~	~~0~~	0	734242	57497	676745	180484	38626	~~0~~	~~0~~	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315			~~0~~	~~0~~	
6	***settled sludge level (in)***	180.47								179.58							
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807			~~0~~	~~0~~	
8	***wt% insol solids***			8.08								8.16					
9	***decanted level***	205.47								204.58							
10																	NO2/NO3=
11																	
12	***Mn***																
13	***SpG***	1.091	1.091	1.091	1	1.320	~~1.260~~	~~1.262~~	1.033	1.084	1.084	1.084	1	1.320	~~1.260~~	~~1.262~~	1.033
14	***Na***	2.162	2.162	2.162	0.021	7.652	~~6.900~~	~~6.956~~	1.200	2.003	2.003	2.003	0.021	7.652	~~6.900~~	~~6.956~~	1.200
15	***NO2***	0.184	0.184	0.184	0.011	7.652	~~0.004~~	~~0.004~~		0.171	0.171	0.171	0.011	7.652	~~0.004~~	~~0.004~~	
16	***NO3***	0.366	0.366	0.366			~~4.694~~	~~4.661~~		0.339	0.339	0.339			~~4.694~~	~~4.661~~	
17	***OH***	1.133	1.133	1.133	0.01		~~1.860~~	~~1.870~~	1.200	1.050	1.050	1.050	0.01		~~1.860~~	~~1.870~~	1.200
18	***Cl***	0.003	0.003	0.003			~~0.006~~	~~0.006~~		0.002	0.002	0.002			~~0.006~~	~~0.006~~	
19	***SO4 or S***	0.044	0.044	0.044			~~0.003~~	~~0.003~~		0.041	0.041	0.041			~~0.003~~	~~0.003~~	
20	***F***	0.002	0.002	0.002			~~0.020~~	~~0.018~~		0.002	0.002	0.002			~~0.020~~	~~0.018~~	
21	***CO3-2***	0.095	0.095	0.095			~~0.000~~	~~0.000~~		0.088	0.088	0.088			~~0.000~~	~~0.000~~	
22	***AlO2***	0.110	0.110	0.110			~~0.310~~	~~0.393~~		0.102	0.102	0.102			~~0.310~~	~~0.393~~	
23	***C2O4-2***	0.017	0.017	0.017						0.015	0.015	0.015					
24	***PO4-3***	0.001	0.001	0.001						0.001	0.001	0.001					
25	***K***	0.004	0.004	0.004			~~0.021~~	~~0.018~~		0.004	0.004	0.004			~~0.021~~	~~0.018~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	652591	78083	574507	238	0	~~0~~	~~0~~	0	574746	25680	549065	792	77194	~~0~~	~~0~~	0
30	***wt% TS***																

	A	FR	FS	FT	FU	FV	FW	FX	FY	FZ	GA	GB	GC	GD	GE	GF	GG
1			10/6/2021								11/19/2021						
2	Tank 51	Tank 51 with SRE Stream & IW	Decant R to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant S to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	267.00		207.00						239.58		203.14					
4	***liquid volume (gal)***	895855	210592	685263	114353	0	~~0~~	~~0~~	0	799616	127911	671704	160337	0	~~0~~	~~0~~	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315			~~0~~	~~0~~	
6	***settled sludge level (in)***	182.00								178.14							
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807			~~0~~	~~0~~	
8	***wt% insol solids***			8.12								8.35					
9	***decanted level***	207.00								203.14							
10		1.800							NO2/NO3=	1.807							NO2/NO3=
11																	
12	***Mn***																
13	***SpG***	1.077	1.077	1.077	1	1.320	~~1.256~~	~~1.259~~	1.033	1.066	1.066	1.066	1	1.320	~~1.256~~	~~1.259~~	1.033
14	***Na***	1.847	1.847	1.847	0.021	7.652	~~6.787~~	~~6.877~~	1.200	1.586	1.586	1.586	0.021	7.652	~~6.787~~	~~6.877~~	1.200
15	***NO2***	0.461	0.461	0.461	0.011	7.652	~~0.004~~	~~0.004~~		0.397	0.397	0.397	0.011	7.652	~~0.004~~	~~0.004~~	
16	***NO3***	0.256	0.256	0.256			~~4.629~~	~~4.548~~		0.220	0.220	0.220			~~4.629~~	~~4.548~~	
17	***OH***	0.795	0.795	0.795	0.01		~~1.800~~	~~1.900~~	1.200	0.683	0.683	0.683	0.01		~~1.800~~	~~1.900~~	1.200
18	***Cl***	0.002	0.002	0.002			~~0.007~~	~~0.006~~		0.002	0.002	0.002			~~0.007~~	~~0.006~~	
19	***SO4 or S***	0.031	0.031	0.031			~~0.003~~	~~0.003~~		0.027	0.027	0.027			~~0.003~~	~~0.003~~	
20	***F***	0.001	0.001	0.001			~~0.021~~	~~0.018~~		0.001	0.001	0.001			~~0.021~~	~~0.018~~	
21	***CO3-2***	0.066	0.066	0.066			~~0.000~~	~~0.000~~		0.057	0.057	0.057			~~0.000~~	~~0.000~~	
22	***AlO2***	0.077	0.077	0.077			~~0.321~~	~~0.397~~		0.066	0.066	0.066			~~0.321~~	~~0.397~~	
23	***C2O4-2***	0.012	0.012	0.012						0.010	0.010	0.010					
24	***PO4-3***	0.000	0.000	0.000						0.000	0.000	0.000					
25	***K***	0.003	0.003	0.003			~~0.022~~	~~0.018~~		0.003	0.003	0.003			~~0.022~~	~~0.018~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	627051	89386	537666	502	0	~~0~~	~~0~~	0	538168	46608	491560	703	0	~~0~~	~~0~~	0
30	***wt% TS***																

	A	GH	GI	GJ	GK	GL	GM	GN	GO	GP	GQ	GR	GS	GT	GU	GV	GW
1			12/21/2021								1/28/2022				2/28/2022		
2	Tank 51	Tank 51 with SRE Stream & IW	Decant T to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 18.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant U to Tank 37	Tank 51 after Decant	Add IW	Tank 51 with IW	Decant V to Tank 37	Tank 51 after Decant	Add IW
3	***Initial tank Level (in)***	248.82		209.08						262.37		216.21		230.83		207.35	
4	***liquid volume (gal)***	832042	139500	692542	187052	0	~~0~~	~~0~~	0	879593	162000	717593	51290	768883	82403	686480	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315		41315		41315	
6	***settled sludge level (in)***	184.08								191.21				182.35			
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807		246807		246807	
8	***wt% insol solids***			8.20								8.02				8.37	
9	***decanted level***	209.08								216.21				207.35			
10		1.819							NO2/NO3=	1.836			OH/AL=	10.44			OH/AL=
11																	
12	***Mn***																
13	***SpG***	1.053	1.053	1.053	1	1.320	~~1.256~~	~~1.259~~	1.033	1.042	1.042	1.042	1	1.039	1.039	1.039	1
14	***Na***	1.285	1.285	1.285	0.021	7.652	~~6.787~~	~~6.877~~	1.200	1.016	1.016	1.016	0.021	0.950	0.950	0.950	0.021
15	***NO2***	0.323	0.323	0.323	0.011	7.652	~~0.004~~	~~0.004~~		0.256	0.256	0.256	0.011	0.240	0.240	0.240	0.011
16	***NO3***	0.177	0.177	0.177			~~4.629~~	~~4.548~~		0.140	0.140	0.140		0.130	0.130	0.130	
17	***OH***	0.553	0.553	0.553	0.01		~~1.800~~	~~1.900~~	1.200	0.438	0.438	0.438	0.01	0.409	0.409	0.409	0.01
18	***Cl***	0.001	0.001	0.001			~~0.007~~	~~0.006~~		0.001	0.001	0.001		0.001	0.001	0.001	
19	***SO4 or S***	0.022	0.022	0.022			~~0.003~~	~~0.003~~		0.017	0.017	0.017		0.016	0.016	0.016	
20	***F***	0.001	0.001	0.001			~~0.021~~	~~0.018~~		0.001	0.001	0.001		0.001	0.001	0.001	
21	***CO3-2***	0.046	0.046	0.046			~~0.000~~	~~0.000~~		0.036	0.036	0.036		0.034	0.034	0.034	
22	***AlO2***	0.053	0.053	0.053			~~0.321~~	~~0.397~~		0.042	0.042	0.042		0.039	0.039	0.039	
23	***C2O4-2***	0.008	0.008	0.008						0.006	0.006	0.006		0.006	0.006	0.006	
24	***PO4-3***	0.000	0.000	0.000						0.000	0.000	0.000		0.000	0.000	0.000	
25	***K***	0.002	0.002	0.002			~~0.022~~	~~0.018~~		0.002	0.002	0.002		0.002	0.002	0.002	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	492263	41153	451110	821	0	~~0~~	~~0~~	0	451931	37779	414152	225	414377	17959	396418	0
30	***wt% TS***																

	A	GX	GY	GZ	HA	HB	HC	HD	HE
1			4/7/2022						
2	Tank 51	Tank 51 with IW	Decant W to Tank 37	Tank 51 after Decant	Add IW	Tank 51 with IW	Decant X to Tank 37	Tank 51 after Decant	51-40-SB10
3	***Initial tank Level (in)***	207.35		181.08		181.08		181.08	
4	***liquid volume (gal)***	686480	92207	594273	0	594273	0	594273	561455
5	***solids volume (gal)***	41315		41315		41315		41315	39033
6	***settled sludge level (in)***	168.08				165.28			
7	***kg insol. solids***	246807		246807		246807		246807	233178
8	***wt% insol solids***			9.55				9.55	9.55
9	***decanted level***	181.08				190.28			
10		10.44			OH/AL=	10.44			
11									
12	***Mn***								
13	***SpG***	1.039	1.039	1.039	1	1.039	1.039	1.039	1.039
14	***Na***	0.950	0.950	0.950	0.021	0.950	0.950	0.950	0.950
15	***NO2***	0.240	0.240	0.240	0.011	0.240	0.240	0.240	0.240
16	***NO3***	0.130	0.130	0.130		0.130	0.130	0.130	0.130
17	***OH***	0.409	0.409	0.409	0.01	0.409	0.409	0.409	0.409
18	***Cl***	0.001	0.001	0.001		0.001	0.001	0.001	0.001
19	***SO4 or S***	0.0176	0.0176	0.0176		0.0176	0.0176	0.0176	0.0176
20	***F***	0.001	0.001	0.001		0.001	0.001	0.001	0.001
21	***CO3-2***	0.034	0.034	0.034		0.034	0.034	0.034	0.034
22	***AlO2***	0.039	0.039	0.039		0.039	0.039	0.039	0.039
23	***C2O4-2***	0.006	0.006	0.006		0.006	0.006	0.006	0.006
24	***PO4-3***	0.000	0.000	0.000		0.000	0.000	0.000	0.000
25	***K***	0.002	0.002	0.002		0.002	0.002	0.002	0.002
26	***slurry spg***								
27	***Na2C2O4 Solubility***								
28	***solid Na2C2O4, kg***								
29	***Mass TS, kg***	396418	20095	376322	0	376322	0	376322	355540
30	***wt% TS***								14.56

Appendix B. SRR Wash Endpoint - 021621



	A	GC	GD	
1				
2	Tank 51	51-40-SB10	Tank 51 after 51-40	Rema
3	Initial tank Level (in)		10.00	No. of R
4	liquid volume (gal)	612955	32990	Total F
5	solids volume (gal)	39205	2110	
6	settled sludge level (in)			Remain
7	kg inrol. solids	234202	12605	
8	wt% in rol. solids	8.85		
9	decanted level			
10				
11	Hanford Pitting Factor			
12	[NO2]/[NO3]			
13	SpG	1.040	1.040	
14	Na	0.940	0.940	
15	NO2	0.209	0.209	
16	NO3	0.143	0.143	
17	OH	0.425	0.425	
18	Cl	0.001	0.001	
19	SO4 or S	0.0175	0.0175	
20	F	0.001	0.001	
21	CO3-2	0.036	0.036	
22	AlO2	0.042	0.042	
23	C2O4-2	0.006	0.006	
24	PO4-3	0.000	0.000	
25	K	0.002	0.002	
26	slurry sp g	0.13685	0.14	
27	Na2C2O4 Solubility			
28	solid Na2C2O4, kg			
29	Mass TS, kg	366634	19733	
30	wt% TS	13.85		

Tank 51 Tar

Select destination and press ENTER or

Appendix C. SRR Projected Elemental Composition of SB10 Washed Tank 51

SRR provided SRNL with the projected elemental composition of SB10 Washed Tank 51 via email. An excerpt from the email and the applicable excerpt from the spreadsheet attached to the email, for comparison to SRNL results, is given here.

John Pareizs

From: Hasmukh Shah
Sent: Tuesday, February 15, 2022 8:35 PM
To: John Pareizs; Chris Martino
Cc: Gregg Morgan
Subject: FW: SB10 Batch Projection using TK51 washed qual Sample Results for MARS Assessment
Attachments: SB10 Batch Compositions at 1 and 0.9M Na with MST and wo MST from TK51 SRNL Washed Qual Sample.xlsx

John

What I provided here is in reference to your request for me to provide batch compositions for the comparison.
I believe your work would fit item # 1 in the email below. They are on calcine solids basis.

Hasmukh B. Shah,
Manager, Sludge and Salt Planning
Savannah River Remediation, Contractor to
US Department of Energy, Savannah River Office
Office 803 208 3756, Cell 803 507 5888, Pager 14586
hasmukh.shah@srs.gov

From: Hasmukh Shah
Sent: Thursday, May 13, 2021 9:45 PM
To: Fabienne Johnson <Fabienne.Johnson@srnl.doe.gov>; Matthew02 Williams <Matthew02.Williams@srnl.doe.gov>; Chris Martino <chris.martino@srnl.doe.gov>; John Pareizs <john.pareizs@srnl.doe.gov>
Cc: Terri Fellinger <Terri.Fellinger@srs.gov>; Bill Holtzscheiter <bill.holtzscheiter@srs.gov>; Ryan Mcnew <ryan.mcnew@srs.gov>; Azadeh Samadi-Dezfouli <Azadeh.Samadi-Dezfouli@srs.gov>; Jeff Ray <jeff.ray@srs.gov>; Kirk Russell <Kirk.Russell@srs.gov>; Helen Boyd <Helen.Boyd@srs.gov>; Spencer Isom <Spencer.Isom@srs.gov>; Frank Pennebaker <frank.pennebaker@srnl.doe.gov>; Dan Lambert <dan.lambert@srnl.doe.gov>; Gregg Morgan <Gregg.Morgan@srnl.doe.gov>; Alex Cozzi <alex.cozzi@srnl.doe.gov>; Thuy Le <thuy.le@srs.gov>; Peter Hill <peter.hill@srs.gov>; William Barnes <william.barnes@srs.gov>
Subject: SB10 Batch Projection using TK51 washed qual Sample Results for MARS Assessment

Fabienne

Please refer to attached file for the SB10 projected compositions (calculations performed by Thuy and reviewed by Spencer and me) that are based on TK51 washed qualification sample results provided by John Pareizs.
We have provided you the batch compositions at 0.9M and 1.0M Na end point. Washing SS was updated based on sample results to determine the batch projections.
The file has four Tabs:

1. SB10 TK51 and 40_Na_1M with MST
2. SB10 TK51 and 40_Na_1M without MST
3. SB10 TK51 and 40_Na_0.9M with MST
4. SB10 TK51 and 40_Na_0.9M without MST

Elemental composition are wt% of calcined solids.

SB10 Projection using TK51 Washed Sample Results

SB10 TK51_40_Na 1M_with MST

STATION	SB9 Tk 40	SB10.prep	SB10.Start
DESCRIPTION	Tk 40 SB9 at WASP samples	Tank 51 SB10 end of washing	Tk40, SB-10 Blend READY
Calcine Solids Mass, kg	208,067	279,640	390,740
wt%, Al	8.54	17.53	13.32
B	0.04	0.00	0.01
Ba	0.10	0.06	0.06
Ca	1.45	0.74	0.74
Ce	0.31	0.12	0.13
Cr	0.11	0.23	0.17
Cu	0.04	0.05	0.04
Fe	22.18	11.91	11.63
K	0.11	0.08	0.08
La	0.06	0.03	0.03
Li	0.06	0.02	0.02
Mg	0.30	0.29	0.25
Mn	7.21	3.72	3.68
Na	19.58	24.44	25.74
Ni	1.46	0.38	0.49
Pb	0.06	0.02	0.02
S	0.35	0.54	0.53
Si	1.75	0.59	0.68
Th	1.13	2.42	1.82
Ti	0.03	0.02	4.45
U	4.08	3.08	2.95
Zn	0.04	0.02	0.02
Zr	0.04	0.16	0.11
Hg (total solids basis)	2.22	3.23	2.78

Appendix D. Calculation of Elements on a wt% of Calcined Solids Basis

- Elements not detected by SRNL are not used.
- Only elements given by SRR (see Appendix C) are considered in this calculation.
- Hg is not considered in the calcine calculation as it is volatilized in the calcine process, and is not present in the final glass waste form.
- wt% of total dried solids is taken from Table 3–6 above.
- Oxide to element conversion factor is:

$$Ox\ El\ Conv\ Fact = \frac{MW_{oxide}}{MW_{El} \times N}$$

Where
MW_{oxide} = molecular weight of oxide
MW_{El} = molecular weight of element
N = number of moles of element in the oxide

- Oxide is the oxide form of the element in the final glass waste form.
- Oxide (% of total solids) is the elemental wt% of total dried solids times the oxide to element conversion factor, essentially the amount of oxide one would expect if one calcined 100 g of dried solids.
- Normalized Oxide (wt%) is the calculated percent oxides in the material if it were calcined (nothing but elements and oxides).
- Normalized Element (wt%) is the normalized oxide (wt%) divided by the oxide to element conversion factor.

Element	wt% of total dried solids	Oxide to Element Conversion Factor	Oxide	Oxide (% of total solids)	Normalized Oxide (wt%)	Normalized Element (wt%)
Al	1.24E+01	1.8895	Al2O3	2.34E+01	33.41	17.7
B	<8.3E-02	3.2199	B2O3	NA	NA	NA
Ba	4.59E-02	1.1165	BaO	5.12E-02	0.07	0.0655
Ca	5.30E-01	1.3992	CaO	7.42E-01	1.06	0.756
Ce	9.72E-02	1.2284	CeO2	1.19E-01	0.17	0.139
Cr	1.61E-01	1.4616	Cr2O3	2.35E-01	0.34	0.230
Cu	3.52E-02	1.2518	CuO	4.41E-02	0.06	0.0502
Fe	8.51E+00	1.4297	Fe2O3	1.22E+01	17.35	12.1
K	<7.6E-02	1.2046	K2O	NA	NA	NA
La	3.38E-02	1.1728	La2O3	3.96E-02	0.06	0.0482
Li	<6.3E-03	4.3051	Li2O	NA	NA	NA
Mg	2.09E-01	1.6583	MgO	3.47E-01	0.49	0.298
Mn	2.66E+00	1.2912	MnO	3.43E+00	4.90	3.79
Na	1.66E+01	1.3480	Na2O	2.24E+01	31.91	23.7
Ni	2.74E-01	1.2726	NiO	3.49E-01	0.50	0.391
Pb	9.32E-03	1.0772	PbO	1.00E-02	0.01	0.0133
S	3.79E-01	2.9958	SO4	1.14E+00	1.62	0.540
Si	4.24E-01	2.1393	SiO2	9.07E-01	1.29	0.605
Th	1.73E+00	1.1379	ThO2	1.97E+00	2.81	2.47
Ti	<1.4E-02	1.6683	TiO2	NA	NA	NA
U	2.22E+00	1.1792	U3O8	2.62E+00	3.73	3.17
Zn	1.75E-02	1.2447	ZnO	2.18E-02	0.03	0.0250
Zr	1.13E-01	1.1754	ZrO	1.33E-01	0.19	0.161
Hg	2.85E+00	NA	NA	NA	NA	NA
			Total	7.01E+01	100.00	

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