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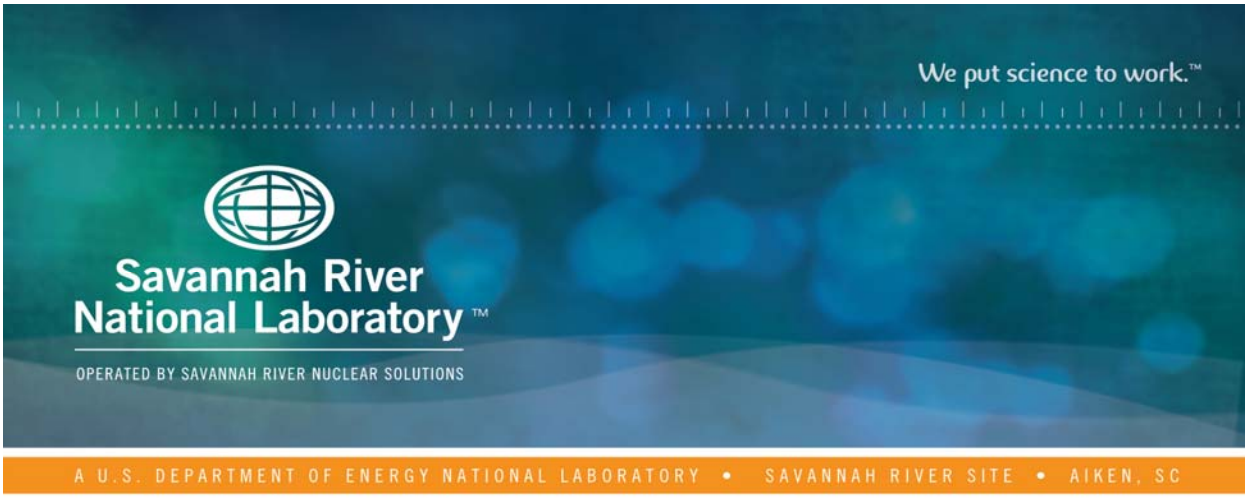

Characterization of the SRNL-Washed Tank 51 Sludge Batch 9 Qualification Sample

J. M. Pareizs

January 2016

SRNL-STI-2015-00693, Revision 0

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) personnel have been requested to qualify the next sludge batch (Sludge Batch 9 – SB9) for processing at the Defense Waste Processing Facility (DWPF). To accomplish this task, Savannah River Remediation (SRR) sent SRNL a 3-L sample of Tank 51H slurry to be characterized, washed, and then used in a lab-scale demonstration of the DWPF flowsheet (after combining with Tank 40H sludge). SRNL has washed the Tank 51H sample per the Tank Farm washing strategy as of October 20, 2015. 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 is in progress and will be documented in a separate report. The analytical results of this characterization are comparable to the Tank Farm projections. Therefore, it is recommended that SRNL uses this washed slurry for the ongoing SB9 qualification activities.

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LIST OF ABBREVIATIONS

AAAS	atomic absorption - As
AASE	atomic absorption – Se
AF	alkali fusion
AR	aqua regia
CVHG	SRNL-AD method – cold vapor atomic absorption for Hg
DWPF	Defense Waste Processing Facility
IC	ion chromatography
ICPES	SRNL-AD method RADICPES (inductively coupled plasma – electron spectroscopy)
ICPES-S	ICPES special method for sulfur
ICPMS	SRNL-AD method RAD ICPMS (inductively coupled plasma – mass spectroscopy)
n	number of replicates
RSD	relative standard deviation
SB9	Sludge Batch 9
SRNL	Savannah River National Laboratory
SRNL-AD	Savannah River National Laboratory Analytical Development
SRR	Savannah River Remediation
SVOA	semivolatile organic analysis
TBP	tributyl phosphate
TIC	total inorganic carbon
Titr	SRNL-AD 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 9 – SB9) for processing at the Defense Waste Processing Facility (DWPF).[1] To accomplish this task, Savannah River Remediation (SRR) sent SRNL a 3-L sample of Tank 51H slurry (Tank Farm sample ID HTF-51-15-81) to be characterized, washed, and then used in a lab-scale demonstration of the DWPF flowsheet (after combining with Tank 40H sludge). SRNL has washed the Tank 51H sample per the Tank Farm washing strategy as of October 20, 2015. 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 is in progress and 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

SRNL received the Tank 51H SB9 qualification sample on July 23, 2015. The sample was transferred from the Tank Farm sampler to a one gallon glass bottle. The slurry was subsampled and characterized.[3] Subsequently, the slurry was washed, mimicking Tank Farm washing; washing and decanting were volumetrically scaled to Tank Farm planned amounts. Details of SRNL washing will be included in the final SB9 qualification report. A subsample of the SRNL-washed slurry was then used for slurry and solids characterization. A portion of SRNL's final decant was utilized for supernatant analyses.

2.1 Weight Percent (wt%) Solids and Density

Aliquots of slurry and supernatant were dried to a constant weight at 110 °C for wt% total solids and wt% dissolved solids, respectively. 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).

2.2 Sample Preparations for Supernatant Characterization

The required results of supernatant characterization include various anions, free hydroxide, and several elemental constituents. Decanted supernatant was diluted by approximately 26X with deionized water to reduce personnel radioactivity exposure prior to submission to Savanah River National Laboratory-Analytical Development (SRNL-AD). Supernatant was diluted in quadruplicate, and the water used in the dilutions was submitted as a blank.

2.3 Sample Preparations for Oxalate Analysis (Slurry Dilutions)

Sodium oxalate was determined from a water dilution of slurry and by an "acid strike". For the water dilution, slurry was diluted with water by ~45X. Sodium oxalate solubility increases significantly as aqueous sodium concentration decreases.[4] Thus, a dilution of slurry to reduce the sodium concentration to less than 0.1 M should result in the dissolution of the majority of existing sodium oxalate.

The "acid strike" is a room temperature acid dissolution of slurry – 1 g of slurry is mixed with 2 mL concentrated HCl and 2 mL concentrated HNO_3 and diluted to 250 mL. This method dissolves oxalate present as calcium oxalate. A disadvantage of this method is the fact that the acids can destroy oxalate and samples must be analyzed as quickly as possible after sample preps to minimize analytical bias associated with the destruction of oxalate from the acids.

2.4 Sample Preparations for Total Solids Characterization

To characterize the solids of the Tank 51H sample, aliquots of slurry were digested and submitted to SRNL-AD 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 aqua regia 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-AD for analysis. For the AF digestions, aliquots of slurry were dried and fused at 675 °C with sodium peroxide. The fusions were then dissolved with nitric acid and water. The resulting liquids were diluted to 100 mL with water. The SRNL-AD results were then converted from a slurry basis to a wt% total solids basis using the measured wt% total solids. In general, AR results have lower detection limits compared to AF; AF utilizes a larger dilution prior to analysis due to the sodium used in the sample preparation. The AF is a more rigorous digestion, and is better for some forms of aluminum (e.g., boehmite) and silicon. In addition to slurry samples, reagent blanks and digested glass of known composition were processed. Results of these samples were used in evaluating AR and AF slurry digestion effectiveness.

2.5 Slurry Inorganic and Organic Analyses

Slurry samples diluted with water by a factor of ~45X were submitted for Total Inorganic Carbon (TIC), Total Organic Carbon (TOC), and Volatile Organic Analysis (VOA). VOA is designed to quantify organic materials boiling below 150 °C, and include the specific analytes benzene, toluene, isopropanol, and butanol.

Slurry samples were also extracted with methylene chloride to measure semivolatile organic (SVOA) compounds via SRNL-AD method SVOA. Compounds quantified by this method generally include those organic materials boiling above 150 °C. Diluted slurry aliquots were mixed with a basic solution and extracted. The extractant from this process yields the process chemicals tributyl phosphate (TBP), Isopar, and Norpar. A second set of diluted slurry aliquots was mixed with a buffer at pH 7. The extractant from this process yields n-paraffin, and phenol. Slurry was diluted by approximately 10x to reduce insoluble solids concentration; insoluble solids make it difficult to distinguish the aqueous and organic layers in the cells. 5 mL of diluted slurry was mixed with 5 mL of buffer and extracted with 10 mL of methylene chloride.

2.6 SRNL-AD Methods

Given in Table 2–1 are the types of samples (described above) and the SRNL-AD methods used for characterization. Table 2–2 lists the SRNL-AD methods and the analytes reported from these methods.

Table 2–1. Analyses Performed by SRNL-AD

SRNL-AD Method	Supernatant Dilutions	Slurry Dilutions	Slurry Dilutions (Aqueous Phase)	Aqua Regia Digestions	Alkali Fusion Digestions	Methylene Chloride Extraction
RAD Inductively Coupled Plasma – Electron Spectroscopy (ICPES) LEEMAN	X	–	-	X	X	-
RAD ICPES SULFUR (ICPES-S) AXIAL	X	–	-	X	–	-
Cold Vapor Atomic Absorption (CVHG)		–	-	X	–	-
CVHG DIGESTED	X	–	-	–	–	-
Atomic Absorption for As and Se (AAAS and AASE)			-	X		-
Ion Chromatography (IC) ANIONS	X	-	X	–	–	-
T BASE/OH/OTHER BASE EXC CO_3^{2-}	X	–	-	–	–	-
TIC/TOC	X	–	X	–	–	-
RAD Inductively Coupled Plasma – Mass Spectroscopy (ICPMS)	–	–	-	X	–	-
VOA	-	X	-	–	-	-
SVOA	–	–	-	–	-	X

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

Table 2–2. Analytes from SRNL-AD Methods

SRNL-AD Method	Expected Results to Satisfy Technical Task Request (TTR)[1] Requirements
RAD ICPES LEEMAN	Ag, Al, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, Gd, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Si, Sn, Sr, Th, Ti, U, V, Zn, Zr
RAD ICPES SULFUR AXIAL	S
CVHG and CVHG DIGESTED	Hg
AAAS and AASE	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)
RAD ICPMS	Isotopic results are used to calculate Pd, Ru, Rh, Nd, Pd, Th, and U
VOA	benzene, toluene, isopropanol, and butanol.
SVOA	TBP, Isopar, Norpar, n-paraffin, and phenol.

2.7 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 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 2.1, 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 and dissolved solids.[5] Wt% calcined solids were determined by heating dried slurry to 1100 °C, cooling, and weighing.

Table 3–1. Densities and Wt% Solids

Property	Result	RSD, n*
Slurry Density (g/mL) T = 18 °C	1.15	5%, 4
Supernatant Density (g/mL) T = 22 °C	1.04	1%, 3
Wt% Total Solids (Slurry Basis)	19.6	0.6%, 4
Wt% Dissolved Solids (supernatant basis)	5.8	0.7%, 4
Wt% Insoluble Solids (Slurry Basis)	14.6	NA
Wt% Soluble Solids (Slurry Basis)	5.0	NA
Wt% Calcined Solids (Slurry Basis)	15.3	0.7%, 4

* 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 supernatant results. These results were determined from analysis of water-diluted supernatant (nominally 26X). 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. Mercury was determined by a digestion technique performed by SRNL-AD followed by cold vapor atomic absorption (SRNL-AD method CVHG). The remaining elements were quantified by ICPES. The analytical methods for the various analytes are reiterated in the table.

As has been observed in previous Tank Farm supernatant samples, sulfate measured by IC is approximately 80% of the total sulfur as measured by ICPES-S. This difference is likely due to non-sulfate species in the sludge slurry. Therefore, it is recommended that sulfur be projected and tracked with sulfur via ICPE-S instead of by IC, particularly if sulfur content approaches DWPF limits in the final glass waste form.

Table 3–2. Supernatant Results

Analyte	Analytical Method*	Units	Result	RSD, n†
Bromide	IC	M	< 2E-02	NA
Chloride	IC	M	< 7E-03	NA
Fluoride	IC	M	< 1E-02	NA
Formate	IC	M	< 6E-03	NA
Nitrate	IC	M	1.38E-01	0.4%, 4
Nitrite	IC	M	2.96E-01	1%, 4
Oxalate	IC	M	5.43E-02	0.3%, 4
Phosphate	IC	M	< 3E-03	NA
Sulfate	IC	M	6.70E-03	3%, 4
Carbonate	TIC	M	9.87E-02	11%, 4
Free OH	Titr.	M	2.47E-01	3%, 4
Hg	CVHG	mg/L	8.78E+01	2%, 4
Sodium	ICPES	M	9.94E-01	0.4%, 4
Aluminum	ICPES	M	4.89E-02	0.4%, 4
Potassium	ICPES	M	2.99E-03	3%, 4
Sulfur	ICPES-S	M	8.49E-03	3%, 4

*IC = ion chromatography; TIC = total inorganic carbon (all inorganic carbon is assumed to be carbonate); Titr. = titration; ICPES = inductively coupled plasma-electron spectroscopy; ICPES-S = ICPES for sulfur: CVHG = cold vapor atomic absorption for mercury

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

Presented in Table 3–3 is a comparison between projections from October 20, 2015[6] and measurements of the SRNL washed Tank 51 sample. When washing, SRNL targeted sodium, weight percent total solids, and weight percent insoluble solids; washing consisted only of adding water and decanting, therefore, other constituents such as nitrite, nitrate, and free hydroxide were not intentionally adjusted. As can be seen in the table, SRNL was well within 10% of projections for sodium and weight percent total and insoluble solids. Nitrite and nitrate were also close to projections. Other analytes, particularly free hydroxide, carbonate, sulfur, oxalate, and Hg, deviated from projections to a greater degree. For carbonate, the higher measured concentration (as compared to the projection) is likely due to absorbed carbon dioxide from air, as the projections are based on simple dilution calculations which do not capture potential changes associated with vapor phase/liquid phase equilibrium chemistry. For free hydroxide, the

lower measured concentration (as compared to the projection) could be due to analytical uncertainly, as SRNL-AD's one sigma uncertainty is 10%, leading to a 95% confidence interval of approximately +/- 20% - and/or it could be due to consumption of free hydroxide that occurred as the absorbed carbon dioxide reacted with sodium hydroxide to form sodium carbonate. Aluminum, oxalate, and mercury are partially soluble in supernatant; solubility is related to free hydroxide (and carbonate, as discussed above) concentration, making projections difficult. The difference in sulfur measurement and projection could be due to insoluble sulfur bearing compounds that partially dissolved during washing.

Table 3–3. Comparison Between Projections and Measurements

Analysis	Units	Projection	Measurement	% Difference*
Wt% Insol. Solids	wt%	13.74	14.6	6
Wt% Total Solids	wt%	18.97	19.6	3
Supernatant Density	g/mL	1.049	1.04	-1
Sodium	M	1.006	0.994	-1
Nitrite	M	0.320	0.296	-8
Nitrate	M	0.139	0.138	-1
Free OH	M	0.300	0.247	-18
Chloride	M	0.001	<0.007	NA
Sulfur	M	0.007	0.00849	21
Fluoride	M	0.001	<0.01	NA
Carbonate	M	0.033	0.0987	199
Aluminum	M	0.059	0.0489	-17
Oxalate	M	0.048	0.0543	13
Phosphate	M	0.000	<0.003	NA
Potassium	M	0.003	0.00299	-0.3
Hg	mg/L	52	87.8	69

* Difference = [(Measurement – Projection) / Projection] x 100

3.3 Slurry Oxalate

Oxalate in slurry was determined using both a water dilution and an "acid strike", with both preparations followed by IC. Dilution by water decreases the sodium concentration, which increases the oxalate solubility, allowing sodium oxalate present in the slurry to dissolve. The "acid strike" method dissolves any calcium oxalate present in the slurry. However, the acids used in the method can destroy oxalate. SRNL analyzes these samples as quickly as possible to minimize analytical bias associated with acid-caused oxalate destruction. Results are presented in Table 3–4. The soluble oxalate in the supernatant (reported in Table 3–2 above) corresponds to 3,900 mg oxalate per kg slurry, suggesting all of the oxalate in the washed slurry was soluble.

Table 3–4. Slurry Oxalate Concentration

Sample Prep	Oxalate Concentration (mg/kg slurry)	RSD, n†
Water dilution	3,880	5%, 4
Acid dilution	3,660	1%, 3

† RSD = relative standard deviation with n = number of measurements.

3.4 Analysis of Total Solids

Presented in Table 3–5 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 RAD ICPES; AR digestions were submitted for CVHG, ICPMS, and AAAS and AASE. In addition to the slurry samples, reagent blanks and digestions of a reference glass of known composition were analyzed by RAD ICPES.[7] The results of the blanks and reference glass digestions were used in evaluating the slurry results (as discussed below).

- The element Hg was determined from aqua regia digestions and the SRNL-AD CVHG method.
- For the elements Al, Ba, Cr, Fe, Gd, Mn, Ni, and Zn, both RAD ICPES measurements from aqua regia and alkali fusions were used because there was no significant difference between respective analytical results.
- For Ca, results from the aqua regia digestion were used solely because the alkali fusion results were high in the reference glass digestion (likely due to a Ca impurity in the reagent chemicals).
- For Ce, the alkali fusion results were used. The results from the alkali fusions were seven times higher than the result from the aqua regia results. No Ce was detected in the reagent blank or the reference glass, thus, this high result, compared to the aqua regia result, is likely not due to reagent impurities. It is possible that the aqua regia digestion did not completely digest the Ce in the sludge.
- For Na and Zr, the aqua regia digestion results were used; alkali fusions utilize Na as a reagent and they are performed in Zr crucibles.
- For Si, results from the alkali fusion digestion were used because the aqua regia results were low in the reference glass digestion.
- Several elements were determined from RAD ICPMS results from aqua regia digestions. Nd was calculated from the sum of masses 143-146, 148, and 150. Note that Nd may be biased low; 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.[8] Rh was determined from mass 103. Ru was calculated from the sum of masses 101, 102, and 104. Th was determined from mass 232. U was calculated from the sum of masses 233-236 and 238.
- Aqua regia digestions were submitted for As and Se measurements by atomic absorption (AA). These elements were not detected.
- Supernatant results (Table 3–2) were used for Cl and F. The detection limits in the supernatant were placed on a total solids basis by utilizing the supernatant density, the slurry wt% insoluble solids, and the slurry wt% total solids. This calculation assumes that Cl and F are soluble in the slurry.
- For the remaining elements, results (or detection limits for elements not detected) were obtained from measurements performed on aqua regia digestions.

All replicates for Fe are reported in Table 3–6 in the event that replicates are needed for fissile uncertainty analyses.

Table 3–5. Elemental Composition of Total (Dried) Solids

Element	Dig, Analytical Method*	wt% of Total Solids	RSD, n‡
Ag	AR, ES	1.11E-02	1%, 4
Al	AR/AF, ES	6.95E+00	4%, 8
As	AR, AA	< 1E-03	NA
B	AR, ES	3.64E-02	6%, 4
Ba	AR/AF, ES	6.03E-02	3%, 8
Be	AR, ES	2.20E-03	1%, 4
Ca	AR, ES	1.03E+00	0.4%, 4
Cd	AR, ES	1.73E-02	3%, 4
Ce	AF, ES	1.86E-01	5%, 4
Cl	SUP, IC	< 1E-01	NA
Co	AR, ES	8.37E-03	3%, 4
Cr	AR/AF, ES	1.20E-01	2%, 7
Cu	AR, ES	4.12E-02	0.5%, 4
F	SUP, IC	< 1E-01	NA
Fe	AR/AF, ES	1.80E+01	3%, 8
Gd	AR/AF, ES	7.32E-02	5%, 8
Hg	AR, CVHG	3.12E+00	1%, 4
K	AR, ES	1.69E-01	10%, 4
La	AR, ES	2.30E-02	1%, 4
Li	AR, ES	7.32E-02	0.4%, 4
Mg	AR, ES	2.30E-01	0.5% 4
Mn	AR/AF, ES	6.03E+00	3%, 8
Mo	AR, ES	1.42E-02	11%, 4
Na	AR, ES	1.22E+01	0.8%, 4
Nd	AR, MS†	1.04E-01	0.7%, 4
Ni	AR/AF, ES	7.68E-01	4%, 8
P	AR, ES	2.03E-01	9%, 4
Pb	AR, MS†	2.85E-02	0.9%, 4
Pd	AR, MS†	2.02E-03	2%, 4
Rh	AR, MS†	8.87E-03	0.2%, 4
Ru	AR, MS†	4.60E-02	3%, 4
S	AR, ES-S	1.76E-01	11%, 4
Sb	AR, ES	< 7E-02	NA
Se	AR, AA	< 2E-03	NA
Si	AF, ES	1.73E+00	2%, 4
Sn	AR, ES	< 4E-02	NA
Sr	AR, ES	2.21E-02	0.3%, 4
Th	AR, MS†	8.01E-01	0.7%, 4
Ti	AR, ES	3.26E-02	0.5%, 4
U	AR, MS†	3.09E+00	1%, 4
V	AR, ES	< 2E-03	NA
Zn	AR/AF, ES	3.10E-02	1%, 8
Zr	AR, ES	5.87E-02	32%, 4

* Dig, Analytical Method: AR=Aqua Regia; AF=Alkali Fusion; ES= ICPES; ES-S=ICPES-S; MS=ICPMS; CVHG=cold vapor atomic absorption for mercury; AA=atomic absorption. For Cl and F, supernatant (SUP) 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; Th is determined from mass 232; and U is calculated from the sum of masses 233-236 and 238.

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

Table 3–6. Iron Replicates

Digestion	Wt% of Total Solids
Aqua Regia	18.1 18.4 18.5 18.5
Alkali Fusion	17.3 17.4 17.7 18.1

3.5 Carbon Analysis

Presented in Table 3–7 are results of various carbon measurements. Inorganic and organic carbon was detected in the slurry. No specific volatile or semivolatile compounds were identified.

Table 3–7. Carbon Analysis

Analysis	Result (mg/kg slurry)	RSD, n[‡]
Total Inorganic Carbon	1.04E+03	4%, 4
Total Organic Carbon	9.31E+02	14%, 4
Volatile Organics Analysis	< 4E+01	NA
Semivolatile Organics Analysis	< 3E+01	NA

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

4.0 Recommendations

The major supernatant components and the weight percent solids of the SRNL-washed sample were compared to the Tank Farm projections from Tank Farm planning spreadsheet SB9_102015.xlsm[6] in Table 4–1. With the exception of free hydroxide, differences are less than 10% (the free hydroxide discrepancy may be due to analytical uncertainty). Therefore, it is recommended that SRNL proceed with SB9 qualification using this sample.

Table 4–1. Comparison Between Tank Farm Projections and Measurements for the Tank 51 SB9 Qualification Sample

	Projection	Measurement	Difference
Wt% Insoluble Solids	13.7	14.6	6%
Wt% Total Solids	19.0	19.6	3%
Supernatant Density (g/mL)	1.05	1.04	-1%
Sodium (M)	1.006	0.994	-1%
Nitrite (M)	0.320	0.296	-8%
Nitrate (M)	0.139	0.138	-1%
Free Hydroxide (M)	0.300	0.247	-18%

Difference = (Measurement – Projection) / Projection x 100

5.0 References

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