SRNL-STI-2009-00544
Revision 0

TANK 4 CHARACTERIZATION, SETTLING, AND WASHING STUDIES

C. J. Bannochie
J. M. Pareizs
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September 2009

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under
Contract Number DE-AC09-08SR22470

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EXECUTIVE SUMMARY

A sample of PUREX sludge from Tank 4 was characterized, and subsequently combined with a Tank 51 sample (Tank 51-E1) received following Al dissolution, but prior to a supernate decant by the Tank Farm, to perform a settling and washing study to support Sludge Batch 6 preparation. The sludge source for the majority of the Tank 51-E1 sample is Tank 12 HM sludge. The Tank 51-E1 sample was decanted by SRNL prior to use in the settling and washing study. The Tank 4 sample was analyzed for chemical composition including noble metals. The characterization of the Tank 51-E1 sample, used here in combination with the Tank 4 sample, was reported previously[1].

SRNL analyses on Tank 4 were requested by Liquid Waste Engineering (LWE) via Technical Task Request (TTR) HLE-TTR-2009-103[2]. The sample preparation work is governed by Task Technical and Quality Assurance Plan (TTQAP)[3], and analyses were controlled by an Analytical Study Plan[4] and modifications received via customer communications. Additional scope included a request for a settling study of decanted Tank 51-E1 and a blend of decanted Tank 51-E1 and Tank 4, as well as a washing study to look into the fate of undissolved sulfur observed during the Tank 4 characterization. The chemistry of the Tank 4 sample was modeled with OLI Systems, Inc. StreamAnalyzer to determine the likelihood that sulfate could exist in this sample as insoluble Burkeite ($2Na_2SO_4 \cdot Na_2CO_3$). The OLI model was also used to predict the composition of the blended tank materials for the washing study.

The following conclusions were drawn from the Tank 4 analytical results reported here:

- Any projected blend of Tank 4 and the current Tank 51 contents will produce a SB6 composition that is lower in Ca and U than the current SB5 composition being processed by DWPF.

- Unwashed Tank 4 has a relatively large initial S concentration of 3.68 wt% on a total solids basis, and approximately 10% of the total S is present as an insoluble or undissolved form.

- There is 19% more S than can be accounted for by IC sulfate measurement. This additional soluble S is detected by ICP-AES analysis of the supernate.

- Total supernate and slurry sulfur by ICP-AES should be monitored during washing in addition to supernate sulfate in order to avoid under estimating the amount of sulfur species removed or remaining in the supernate.

- OLI simulation calculations show that the presence of undissolved Burkeite in the Tank 4 sample is reasonable, assuming a small difference in the Na concentration that is well within the analytical uncertainties of the reported value.

The following conclusions were drawn from the blend studies of Tank 4 and decanted Tank 51-E1:

- The addition of Tank 4 slurry to a decanted Tank 51-E1 sample significantly improved the degree and time for settling.

- The addition of Tank 4 slurry to a decanted Tank 51-E1 sample significantly improved the plastic viscosity and yield stress.

- The SRNL washing test, where nearly all of the wash solution was decanted from the solids, indicates that approximately 96% or more of the total S was removed from the blend in these tests, and the removal of the sulfur tracks closely with that of Na. Insoluble (undissolved) S

remaining in the washed sludge was calculated from an estimate of the final slurry liquid fraction, the S result in the slurry digestion, and the S in the final decant (which was very close to the method detection limit). Based on this calculated result, about 4% of the initial total S remained after these washes; this amount is equivalent to about 18% of the initially undissolved S.

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[1] Pickenheim, B. R., Bannochie, C. J., Pareizs, J. M., and Click, D. R. *Results of the Analysis of Tank 51 E-1 Sample (HTF-51-09-74 & -75) and Tank 12 Post-Aluminum Dissolution Rheology*, SRNL-L3100-2009-00190, Savannah River Site, Aiken, SC 29808 (2009).

[2] Martin, K. B. *Tank 4F Characterization in Support of Bulk Waste Removal and Sludge Batch Preparation*, HLE-TTR-2009-103, Rev. 0, Savannah River Site, Aiken, SC 29808 (2009).

[3] Bannochie, C. J., Pareizs, J. M. *Qualification of DWPF Sludge Batch 6 and Characterization of Tank 4 Samples in the SRNL Shielded Cells: Task Technical and Quality Assurance Plan*, SRNL-RP-2009-00473, Rev. 0, Savannah River Site, Aiken, SC 29808 (2009).

[4] Bannochie, C. J., Pareizs, J. M. *Qualification of DWPF Sludge Batch 6 and Characterization of Tank 4 in the SRNL Shielded Cells: Analytical Study Plan*, SRNL-RP-2009-00474, Rev. 0, Savannah River Site, Aiken, SC 29808 (2009).

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LIST OF ACRONYMS/ABBREVIATIONS

AD	Analytical Development
ARG – 1	Analytical Reference Glass – 1
ASP	Analytical Study Plan
CV-AA	Cold Vapor – Atomic Absorption Spectroscopy
DWPF	Defense Waste Processing Facility
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
HM	H-Area Modified PUREX
L	Liter
LWE	Liquid Waste Engineering
M	Molar
NA	Not Available (e.g. Not Measured)
NIST	National Institute of Standards and Testing
PUREX	Plutonium Uranium Redox Extraction
RSD	Relative Standard Deviation
SB5	Sludge Batch 5
SB6	Sludge Batch 6
SME	Slurry Mix Evaporator
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
Std. Dev.	Standard Deviation
TS	Total Solids
TTR	Technical Task Request
WAPS	Waste Acceptance Product Specifications
Wt %	Weight Percent

1.0 INTRODUCTION AND BACKGROUND

A sample of PUREX sludge from Tank 4 was characterized, and subsequently combined with a Tank 51 sample (Tank 51-E1) received following Al dissolution, but prior to a supernate decant by the Tank Farm, to perform a settling and washing study to support Sludge Batch 6 (SB6) preparation. The sludge source for the majority of the Tank 51-E1 sample is Tank 12 HM sludge. The Tank 51-E1 sample was decanted by SRNL prior to use in the settling and washing study. The Tank 4 sample was analyzed for chemical composition including noble metals. The characterization of the Tank 51-E1 sample, used here in combination with the Tank 4 sample, was reported previously[1].

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One 1-L sample of Tank 4 was pulled on July 20, 2009 following slurry operations. The sample was designated FTF-04-09-32 by F-Tank Farm Operations. The sample was sent to SRNL on July 21, 2009.

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2.0 APPROACH AND RESULTS

2.1 Analytical Methods

At the Savannah River National Laboratory (SRNL), the 1-L Tank 4 sample was transferred from the shipping container into a 2-L high density polyethylene bottle and solids allowed to settle overnight. Supernate was then siphoned off and circulated through the shipping container to complete the transfer of the sample of 1598 g. Following thorough mixing of the 1-L sample, a 233 g sub-sample was removed. This sub-sample was then utilized for all subsequent analytical samples.

Eight separate aliquots of the slurry were digested, four with HNO_3/HCl (aqua regia[5]) in sealed Teflon® vessels and four in Na_2O_2 (alkali or peroxide fusion[6]) using Zr crucibles. Due to the use of Zr crucibles and Na in the peroxide fusions, Na and Zr cannot be determined from this preparation. Additionally, other alkali metals, such as Li and K that may be contaminants in the Na_2O_2 are not determined from this preparation. Three Analytical Reference Glass – 1[7] (ARG-1) standards were digested along with a blank for each preparation. The ARG-1 glass allows for an assessment of the completeness of each digestion. Each aqua regia digestion and blank was diluted to 1:100 mL with deionized water and submitted to Analytical Development (AD) for inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analysis, inductively coupled plasma – mass spectrometry (ICP-MS) analysis of masses 81-209 and 230-252, and cold vapor atomic absorption (CV-AA) analysis for Hg. Equivalent dilutions of the peroxide fusion digestions and blank were submitted to AD for ICP-AES analysis.

Tank 4 supernate was collected with a 0.45 μm filter cup from a mixed slurry sample in the SRNL Shielded Cells and submitted to AD for ICP-AES, ion chromatography (IC), and total base analyses.

2.2 Analytical Results

Table 2-1 presents the measured Tank 4 density and weight percent solids data[8]. A calcine factor was also calculated by taking the ratio of the weight percent calcined solids and the weight percent total solids. The Tank 4 Sample has a value of 0.60 grams of calcined solids per gram of dried solids. Due to the high dissolved solids a new technique was employed to measure the calcine value. A known amount of frit was added to the dried total solids, and the mixture calcined at 1100 ºC. The addition of frit eliminated the continual drift in mass measurements seen for previous high Na (unwashed) tank samples during calcining.

Table 2-1. Weight Percent Solids and Density for Tank 4 Samples [Number of Samples Included in Average]

Property	Tank 4 (% RSD)
Slurry Density	1.35 (0.8) [4]
Supernate Density	1.33 (0.4) [4]
Wt % Total Solids (Slurry Basis)	37.3 (0.3) [4]
Wt % Calcined Solids (Slurry Basis)	22.2 (2.1) [4]
Wt % Dissolved Solids[a] (Supernate Basis)	35.0 (0.2) [4]
Wt % Insoluble Solids (Slurry Basis)	3.62 (2.9)[c]
Wt % Soluble Solids[b] (Slurry Basis)	33.7 (0.2)[c]

[a] Also known as Uncorrected Soluble Solids
[b] Also known as Corrected Soluble Solids
[c] %RSD here is more correctly defined as % standard error for these calculated values.

Table 2-2 provides the anion results for the Tank 4 sample. The supernate sulfur result is calculated from total sulfur detected in the supernate by ICP-AES. The Al, B, Ca, Cr, K, Mn, Mo, Na, and P values also shown in this table were calculated from the ICP-AES data for the supernate and converted to a slurry basis using the insoluble solids content from Table 2-1. Other supernate elements measured were below the ICP-AES detection limits.

Table 2-2. Supernate Analyses for Tank 4 Samples [Number of Samples Included in Average]

Analyte	Tank 4 (%RSD) Molar	Tank 4 (%RSD) soluble mg/ kg slurry	Method
NO_3^-	1.37 (0.5) [4]	61600 (0.5) [4]	IC
NO_2^-	1.25 (0.5) [4]	41900 (0.5) [4]	IC
SO_4^{2-}	0.427 (0.7) [4]	29800 (0.7) [4]	IC
PO_4^{3-}	<0.010	<720	IC
Br^-	<0.012	<720	IC
Cl^-	<0.028	<720	IC
CHO_2^-	<0.022	<720	IC
$C_2O_4^{2-}$	<0.011	<720	IC
F^-	<0.052	<720	IC
Al	0.202 (0.3) [4]	3960 (0.3) [4]	ICP-AES
B	0.00170 (0.7) [4]	13.4 (0.7) [4]	ICP-AES
Ca	0.000140 (5.5) [3]	4.09 (5.5) [3]	ICP-AES
Cr	0.0131 (0.2) [4]	495 (0.2) [4]	ICP-AES
K	0.0296 (8.7) [4]	840 (8.7) [4]	ICP-AES
Mn	0.0000982 (4.0) [4]	3.92 (4.0) [4]	ICP-AES
Mo	0.00123 (1.1) [4]	85.9 (1.1) [4]	ICP-AES
Na	7.25 (1.0) [4]	121000 (1.0) [4]	ICP-AES
P	0.0116 (0.7)[4]	262 (0.7) [4]	ICP-AES
S	0.526 (1.0) [4]	12300 (1.0) [4]	ICP-AES

Table 2-3 provides the base measurements made on the Tank 4 sample. Total base represents the value determined from an inflection endpoint titration to pH 7. Free OH^- represents the value determined after precipitation of carbonate with $BaCl_2$ and titration to the first inflection endpoint between pH 11 and 8. Further titration of this treated sample to pH 7 yields the value for other base.

Table 2-3. Base Analysis for Tank 4 Samples [Number of Samples Included in Average]

Analyte	Tank 4 (%RSD) Molar
Total Base	2.71 (3.7) [4]
Free OH^-	1.5 (1.8) [4]
Other Base	0.3 (1.5) [4]

The elemental concentrations determined by ICP-AES, ICP-MS, and CV-AA analyses are presented in Table 2-4. For the Tank 4 sample, results from both digestions have been combined where appropriate.

Due to the use of Zr crucibles and Na_2O_2 in the alkali fusions, Zr and Na values, as well as other alkali metals, were determined from the aqua regia digestion. Mercury is also reported form the aqua regia digestion due to its volatility. In the case of Be, Li, Sb, Sn, and V, both preparations yielded values below the detection limits, hence the lowest detection limit value was selected. Alkali fusion data was used to report values for Al and Si for the Tank 4 sample since the aqua regia preparation fails to dissolve all forms of these elements. The alkali fusion value for Ca is generally reported as well, but in these preps, the replicates were inconsistent, so the aqua regia digestion value is reported. The aqua regia prep values for K, P, Ti and Zn are reported because the alkali fusion preps gave values below the ICP-AES's quantification limits. Sulfur is also reported from only the aqua regia preps due to generally improved detection limits. ICP-MS analysis of the aqua regia digestion was also used to determine the concentrations of Cd, Ce, Gd, La, Pb, and U. In the case of Ce the distribution of isotopes was not natural but rather the result of fission product yields from U-235. Hence the sum of the respective isotopic masses was used to determine the reported concentrations for Ce and U. The U value reported here from ICP-MS compares to a value determined by ICP-AES of 1.02 wt% of total solids. For Cd, Gd, and Pb, the reported value was determined from all measured values calculated using the various isotopes' natural abundance. In the case of La-139 a single isotope has 100% natural abundance and was used to calculate the value given in the table.

Table 2-4. Elemental Concentrations* in Tank 4 Samples in Wt % of Total Solids (%RSD) [Number of Samples Included in Average]

Element	Tank 4	Element	Tank 4
Al	1.22 (0.9) [4]	Mn	0.171 (1.2) [8]
B	0.0106 (5.9) [3]	Mo	0.0234 (7.0) [8]
Ba	0.0187 (1.3) [8]	Na	31.5 (0.6) [4]
Be	<0.0024	Ni	0.827 (1.5) [8]
Ca	0.125 (0.7) [4]	P	0.110 (2.0) [4]
Cd‡	0.000679 (7.1) [2]	Pb‡	0.00679 (7.2) [4]
Ce‡‡	0.0144 (2.8) [4]	S	3.68 (0.6) [4]
Cr	0.141 (2.3) [8]	Sb	<0.011
Cu	0.0107 (7.2) [8]	Si	0.170 (7.6) [4]
Fe	3.44 (0.8) [8]	Sn	<0.013
Gd‡	0.00159 (7.0) [4]	Sr	0.00686 (3.3) [8]
Hg^	0.00274 (11) [4]	Ti	0.00262 (4.7) [4]
K	0.258 (6.8) [4]	U‡‡	0.988 (1.1) [4]
La‡	0.0133 (2.1) [4]	V	<0.0049
Li	<0.012	Zn	0.00349 (1.1) [4]
Mg	0.0137 (6.1) [4]	Zr	0.0411 (0.9) [4]

* ICP-AES data unless specified otherwise
‡ Calculated from MS data for Cd-112; La-139; Gd-157; and Pb: Pb-206, Pb-207, Pb-208, respectively
‡‡ Calculated from the sum of MS data for U: U-234, U-235, U-236 and U-238; Ce: Ce-140, Ce-142
^ Calculated from CV-AA data

The fission product noble metal and silver concentrations are given in Table 2-5. The values were calculated from ICP-MS data using an Excel spreadsheet. This spreadsheet uses the fission yield for each isotope to account for the mass contribution from isotopes in the tank that could not be measured because

isotopes of natural Cd interfere at this mass. An example of this is the measurement at mass 110, which is comprised of Pd-110 and Cd-110. The uncertainties were analyzed using statistical techniques appropriate for replicate measurements of non-highly correlated data.

Table 2-5. Noble Metal Fission Products and Silver Concentrations in Tank 4 in Wt % of Total Solids (%RSD)

Element	Tank 4
Ag (-107, -109)	0.00161 (2.0)
Pd (-105, -106, -107, -108, -110)	0.00293 (2.0)
Rh (-103)	0.00577 (2.7)
Ru (-101, -102, -104)	0.0237 (0.4)

2.3 Rheology Methods

Rheological properties of radioactive samples are determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. Heating/cooling of the cup/sample/bob is through the holder that holds the cup. The shear stress is determined from the torque measurement and is independent of the rheological properties. Conditions that impact the measured torque are; slip (material does not properly adhere to the rotor or cup), phase separation (buildup of a liquid layer on the rotor), sedimentation (particles settling out of the shearing zone), homogeneous sample (void of air), lack of sample (gap not filled), excess sample (primarily impacts rheologically thin fluids), completely filling up the void below the bob (air buffer that is now filled with fluid) and Taylor vortices. The first five items yield lower stresses and the last three add additional stresses. The shear rate is geometrically determined using the equations of change (continuity and motion) and is that for a Newtonian fluid. This assumption also presupposes that the flow field is fully developed and the flow is laminar. The shear rate can be calculated for a non-Newtonian fluid using the measured data and fitting this data to the rheological model or corrected as recommended by Darby[9]. In either case, for shear thinning non-Newtonian fluids typical of Savannah River Site (SRS) sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a thinner fluid. Correcting the flow curves was not performed in this task; therefore, the results are biased high.

The bob typically used for measuring tank sludge is the MV I rotor. The shape, dimensions, and geometric constants for the MV I rotor is provided in Table 2-6.

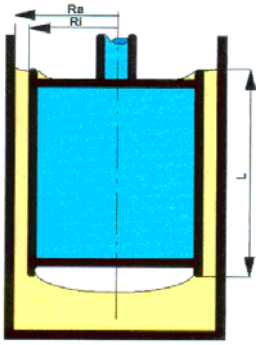
Prior to performing the measurements, the rotors and cups were inspected for physical damage. The torque/speed sensors and temperature bath verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard, using the MV I rotor. The resulting flow curves were then fitted as a Newtonian fluid and this calculated viscosity must be within ± 10% of the reported NIST viscosity at a given temperature for the system to be considered functionally operable. A N10 oil standard was used to verify system operability prior to the sludge measurements.

The flow curves for the sludge are fitted to the down curves using the Bingham Plastic rheological model, Equation (1), where τ is the measured stress (Pa), τ_o is the Bingham Plastic yield stress (Pa), μ_∞ is

the plastic viscosity (Pa·sec), and $\dot{\gamma}$ is the measured shear rate (sec^{-1}). During all these measurements, the sample remained in the cup for the 2nd measurement, due to the limited sample availability.

$$\tau = \tau_o + \mu_\infty \dot{\gamma} \quad (1)$$

Table 2-6. MV I Rotor Specifications and Flow Curve Program

Rotor Design	Dimensions and Flow Curve Program	
	Rotor Type	MV I
	Rotor radius - R_i (mm)	20.04
	Cup Radius - R_a (mm)	21.0
	Height of rotor -L (mm)	60
	Sample Volume (cm^3) minimum	40
	A factor (Pa/%torque)	3.22
	M factor (s^{-1}/%RPM)	11.7
	Shear rate range (s^{-1})	0 – 600
	Ramp up time (min)	5
	Hold time (min)	1
	Ramp down time (min)	5

2.4 Rheology Results

Figure 2-1 through Figure 2-6 provide the shear stress versus shear rate flow curves for Tank 4, decanted Tank 51-E1, and the blend of Tank 4 with the decanted Tank 51-E1 sample. Refer to Section 2.5 for details on the blend ratio. The resulting plastic viscosities in cP and yield stresses in Pa are summarized in Table 2-7. The Tank 4 sample had virtually no yield stress, so this value is reported as zero.

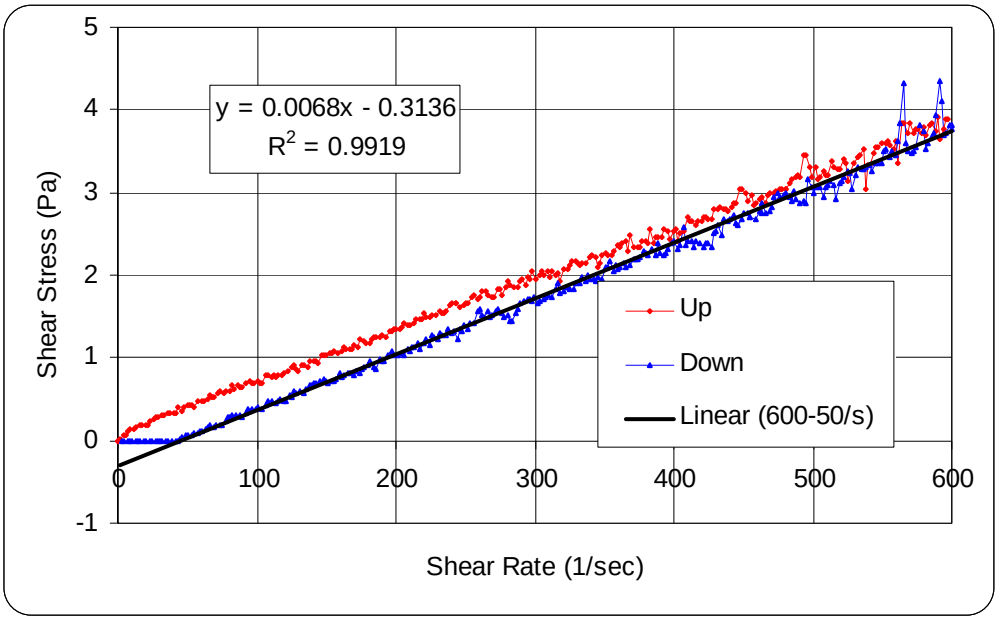


Figure 2-1. Tank 4 Shear Stress vs. Shear Rate Replicate 1

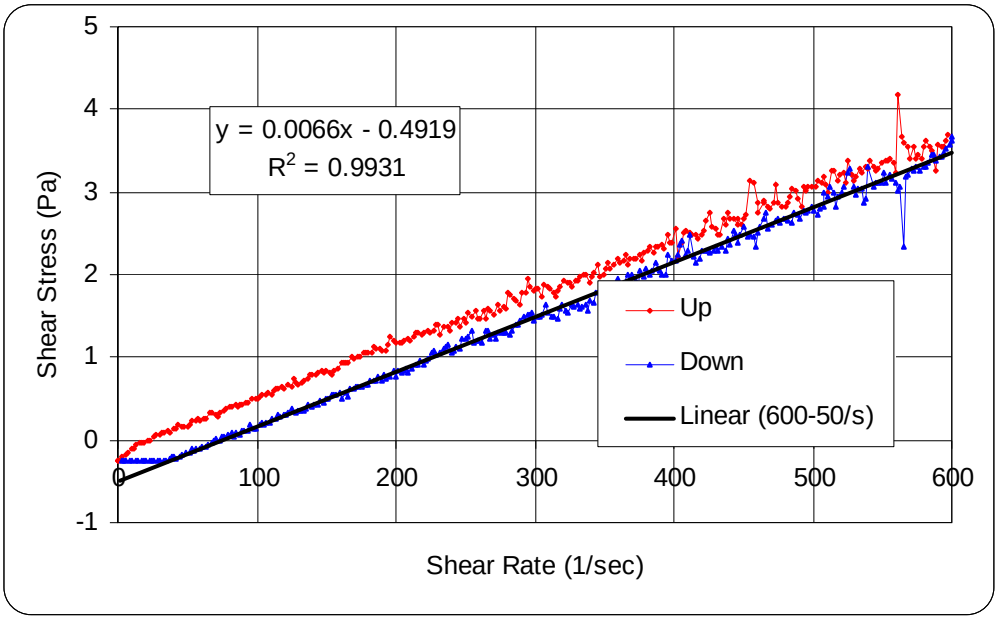


Figure 2-2. Tank 4 Shear Stress vs. Shear Rate Replicate 2

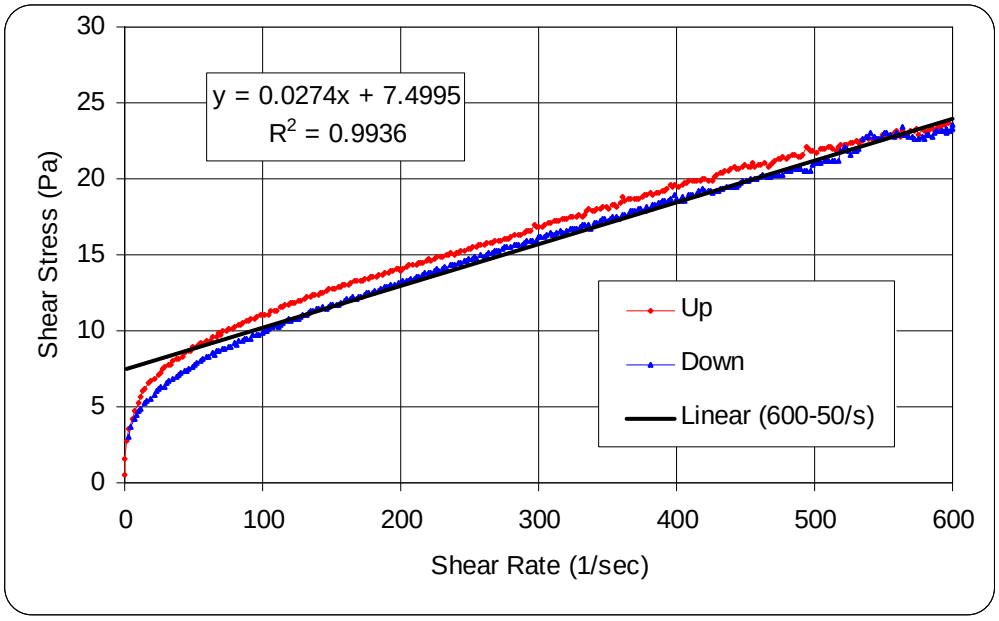


Figure 2-3. Decanted Tank 51-E1 (43% of total volume removed) Shear Stress vs. Shear Rate Replicate 1

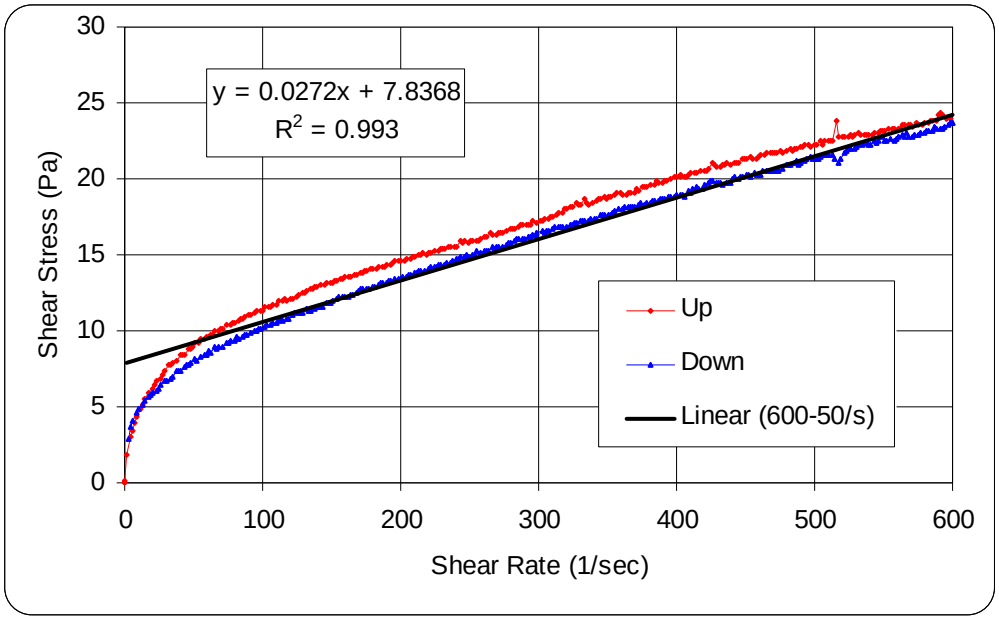


Figure 2-4. Decanted Tank 51-E1 (43% of total volume removed) Shear Stress vs. Shear Rate Replicate 2

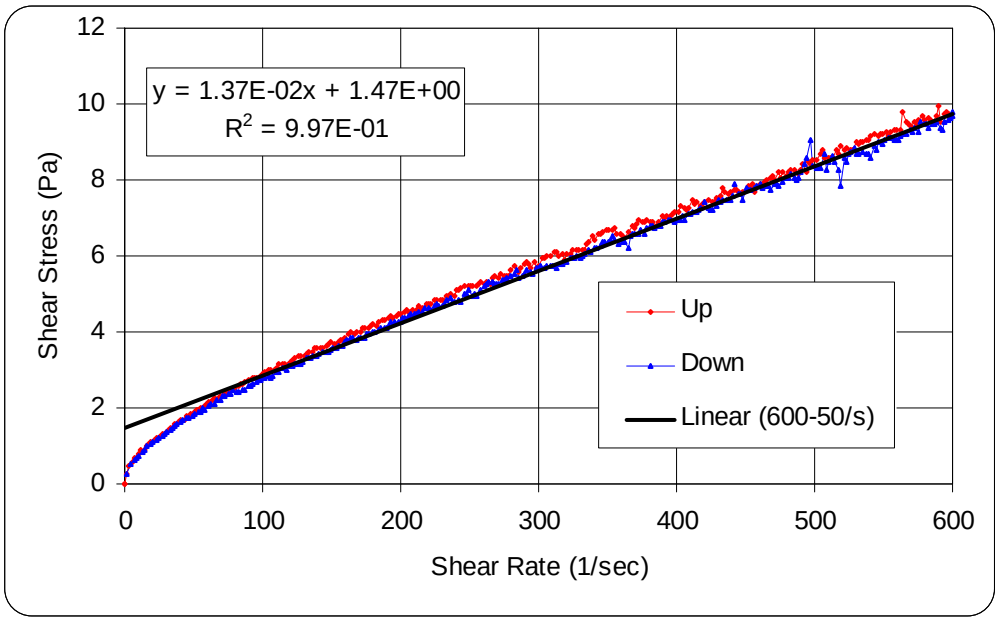


Figure 2-5. Tank 4/Decanted Tank 51-E1 Blend Shear Stress vs. Shear Rate Replicate 1

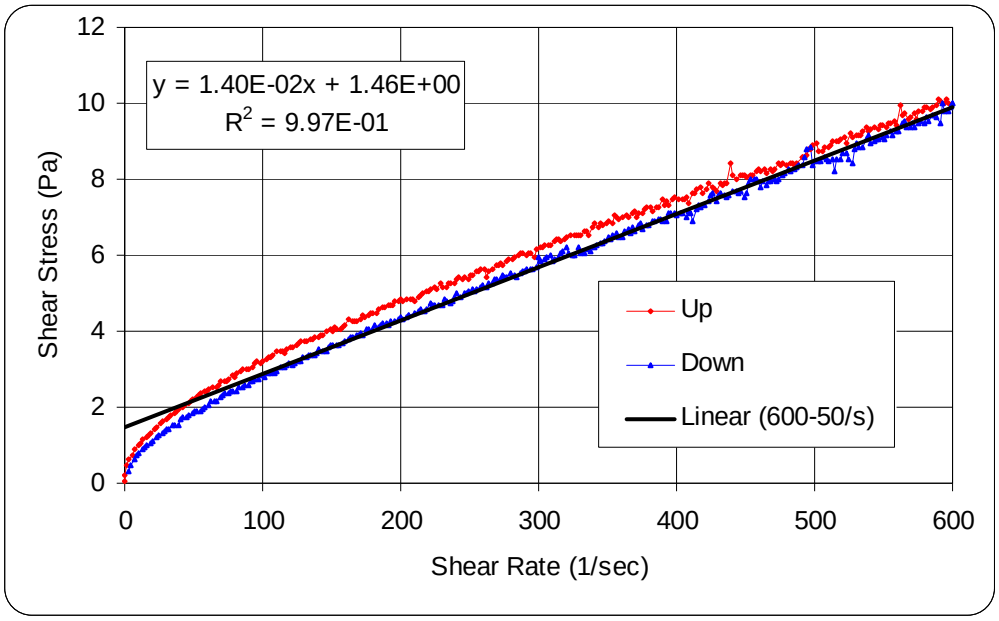


Figure 2-6. Tank 4/Decanted Tank 51-E1 Blend Shear Stress vs. Shear Rate Replicate 2

Table 2-7. Rheology Summary for Tank 4, Decanted Tank 51-E1, and Tank 4/Decanted Tank 51-E1 Blend.

Tank – Replicate	Plastic Viscosity (cP)	Yield Stress (Pa)
Tank 4 – 1	6.8	0*
Tank 4 – 2	6.6	0*
Tank 4 – average	*6.7*	*0*
Tank 51-E1 Decanted – 1	27.2	7.5
Tank 51-E1 Decanted – 2	27.4	7.8
Tank 51-E1 Decanted – average	*27.3*	*7.7*
Tank 4/51-E1 Blend – 1	13.7	1.5
Tank 4/51-E1 Blend – 2	14.0	1.5
Tank 4/51-E1 Blend – average	*13.9*	*1.5*

* The sample has essentially no yield stress, so this is reported as zero.

2.5 Settling Study Methods

A comparison of settling between a decanted sample of Tank 51 and Tank 4 was requested by LWE. For the Tank 51 portion, the Tank 51-E1 sample was decanted. The target decant amount, per LWE, was 45% of the total volume. However, only 43% of the total volume could be decanted without disturbing the settled solids. It should be noted that the sample had been undisturbed for several weeks prior to this decant. Two 50 mL graduated cylinders were used as the settling vessels. The decanted Tank 51 slurry was added to the 50 mL mark in the first cylinder. The decanted Tank 51 slurry was added to the 29 mL mark in the second cylinder followed by 21 mL of Tank 4 slurry. A ratio of 2.1 to 1 of Tank 51 insoluble

solids to Tank 4 insoluble solids was targeted. Both cylinders were mixed by shaking and left undisturbed. The volumetric sludge level was recorded at least daily during working days.

2.6 Settling Study Results

Figure 2-7 provided the results of the settling study for both the decanted Tank 51-E1 sample and a blend of this sample with Tank 4 slurry. Settled volume was recorded in mL and plotted versus time in days.

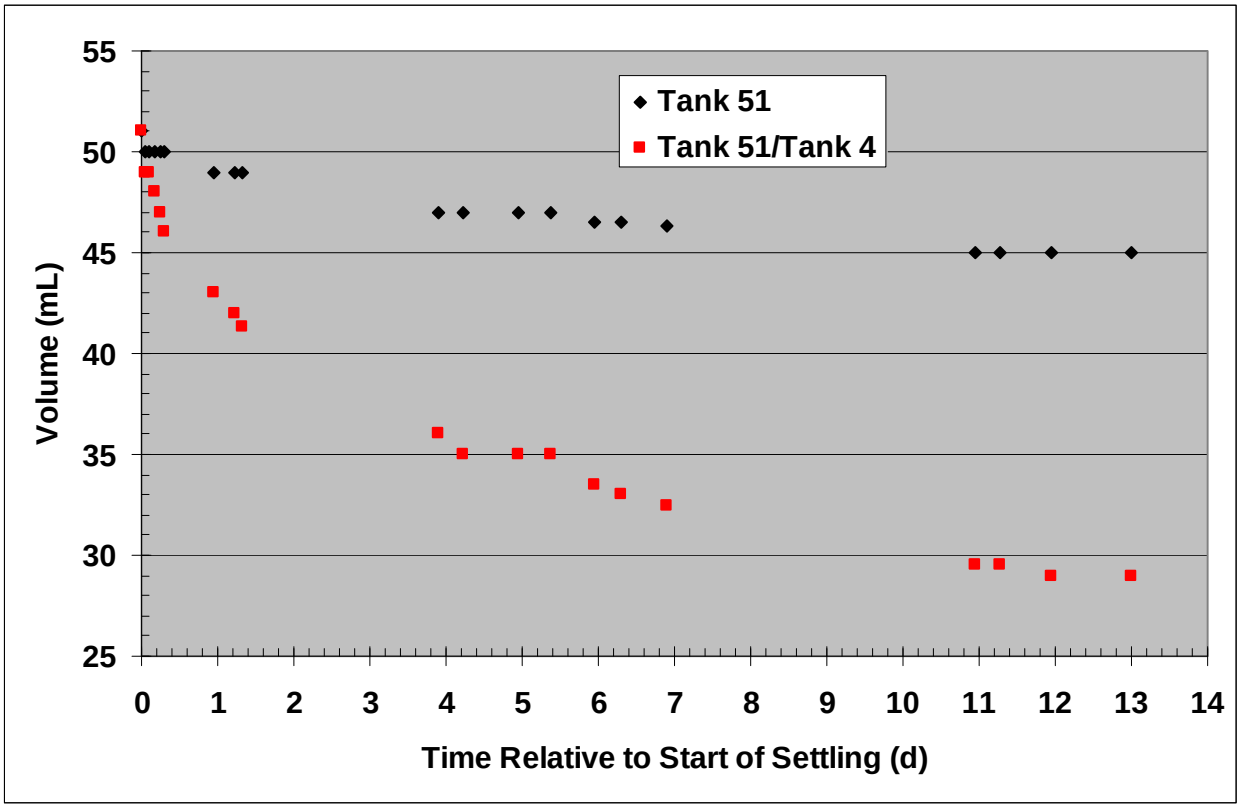


Figure 2-7. Comparison of settled volume (mL) vs. time (days) for a decanted Tank 51-E1 sample and a blend of Tank 51-E1 and Tank 4.

2.7 Modeling Study Methods

The OLI (OLI Systems, Inc.) StreamAnalyzer was used to model the chemistry of the Tank 4 sample to determine the likelihood that sulfate could exist in this sample as insoluble Burkeite ($2Na_2SO_4 \cdot Na_2CO_3$). StreamAnalyzer was used with the Public, Corrosion, Geochemisty, and WTPBase thermodynamic databases. The Tank 4 sample supernate and total slurry species compositions were used to generate an overall composition vector. The original slurry (wt% of solids) measurements were converted to a per kg slurry basis. The values used are shown in Table 2-8. These values were converted to the OLI inputs in mmol/kg slurry shown in the last two columns. Note that these values do not necessarily match the final reported values in this report; the OLI calculations were performed in parallel with the final data validation, so preliminary values had to be used.

Table 2-8. Input Concentrations for OLI StreamAnalyzer Model of Tank 4 Slurry

Species	Slurry (wt% of total dried solids)	Total Slurry (mg/kg slurry)	Supernate (mg/kg slurry)	Total Slurry (mmol/kg slurry)	Supernate (mmol/kg slurry)	Undissolved Solids (mmol/kg slurry)
Al	1.22	4551	3964	169	147	22
B	0.0106	39.5	13.4	3.66	1.24	2.42
Ba	0.0187	69.8		0.508		0.508
Ca	0.125	466	4.56	11.6	0.114	11.5
Cd	0.000679	2.53		0.0225		0.0225
Ce	0.0144	53.7		0.383		0.383
Cr	0.141	526	495	10.1	9.52	0.58
Cu	0.0107	39.9		0.628		0.628
Fe	3.44	12831	21.5	230	0.385	230
Gd	0.00159	5.93		0.0377		0.0377
Hg	0.00274	10.2		0.0510		0.0510
K	0.258	962	841	24.6	21.5	3.1
La	0.0133	49.6		0.357		0.357
Mg	0.0137	51.1		2.10		2.10
Mn	0.171	638	3.92	11.6	0.0714	11.5
Mo	0.0234	87.3	85.9	0.910	0.895	0.015
Na(Original)	31.5	117495		5111		
Na(Adjusted)	(to supernate value)	121205	121205	5272	5272	0
Ni	0.827	3085		52.6		52.6
P	0.11	410	262	13.2	8.46	5.7
Pb	0.00679	25.3		0.122		0.122
S	3.68	13726	12277	428	383	45
Si	0.17	634		22.6		22.6
Sr	0.00689	25.7		0.293		0.293
Ti	0.00262	9.77		0.204		0.204
U	0.988	3685		15.5		15.5
Zn	0.00349	13.0		0.199		0.199
Zr	0.0411	153		1.68		1.68
	Supernate (mol/L)					
NO_3^-	1.42	NA	64051	NA	1033	0
NO_2^-	1.30	NA	43508	NA	946	0
SO_4^{2-}	0.526	NA	36780	NA	383	0
PO_4^{3-}	0.0116	NA	803	NA	8.46	0
CO_3^{2-} calc.	0.869	NA	37925	NA	632	0
OH^-	1.50	NA	18558	NA	1091	0
Cl^-	0.0170	NA	438	NA	12.4	0
$C_2O_4^{2-}$	0.00410	NA	263	NA	2.98	NM
F^-	0.00500	NA	69.1	NA	3.64	0

NA ≡ not applicable, NM ≡ not measured, NC ≡ not calculated

Table 2-9. Input Parameters for OLI StreamAnalyzer Model of Tank 4 Slurry

Slurry Density kg/L	1.350
Supernate Density kg/L	1.326
Total Solids wt%	37.30%
Soluble Solids wt%	33.76%
Insoluble Solids wt%	3.54%
Dissolved Solids wt%	35.00%

2.8 Modeling Study Results

The Na amount measured in the slurry was lower than the supernate measurement, so the supernate value was assumed for the slurry. The carbonate value was calculated to balance the charge in the supernate. The Na values were also adjusted for formation of Burkeite. The sulfur concentration in the total slurry was 13726 mg/kg slurry and the supernate value was 12277 mg/kg slurry, suggesting that about 1449 mg/kg slurry of undissolved sulfur existed in the slurry. Because Tank 4 was a Burkeite-containing tank, this undissolved sulfur was assumed to be Burkeite; the only other sulfate compound predicted to be insoluble was $BaSO_4$ and there is insufficient Ba to account for the amount of undissolved sulfate measured. Some of the assumed carbonate (1351 mg/kg) was precipitated in the model to form this Burkeite. The total carbonate was not adjusted for the charge balance because the amount precipitated was relatively small (1351 of 37925 mg/kg) and OLI closes the charge balance by adjusting the dissolved species. The composition of the Tank 4 sample was simulated in OLI at 35°C because of a databank limitation that prevents the formation of Burkeite below 31°C.

The results of the OLI model are compared to the measured concentrations in Table 2-10 and Table 2-11. The modeling results demonstrate that the existence of Burkeite in the Tank 4 sample is possible.

Table 2-10. Predicted Tank 4 Composition

Species	Measured Total Slurry (mmol/kg slurry)	Measured Supernate (mmol/kg slurry)	Measured Undissolved Solids (mmol/kg slurry)	OLI Predicted Total Slurry (mmol/kg slurry)	OLI Predicted Supernate (mmol/kg slurry)	OLI Predicted Undissolved Solids (mmol/kg slurry)
Al	169	147	22	169	162	7
B	3.66	1.24	2.42	3.65	3.65	0
Ba	0.508		0.508	0.507	0	0.507
Ca	11.6	0.114	11.5	11.6	0	11.6
Cd	0.0225		0.0225	0.0225	0.0225	0
Ce	0.383		0.383	0.382	0	0.382
Cr	10.1	9.52	0.58	10.1	9.50	0.59
Cu	0.628		0.628	0.627	0.0436	0.583
Fe	230	0.385	230	230	0	230
Gd	0.0377		0.0377	0.0376	0.0376	0
Hg	0.0510		0.0510	0.0509	0.0509	0
K	24.6	21.5	3.1	24.6	24.6	0
La	0.357		0.357	0.356	0	0.356
Mg	2.10		2.10	2.10	0	2.10
Mn	11.6	0.0714	11.5	11.6	0.696	10.9
Mo	0.910	0.895	0.015	0.908	0.908	0
Na	5272	5272	0	5379	5243	136
Ni	52.6		52.6	52.5	0	52.5
P	13.2	8.46	5.7	13.2	10.4	2.74
Pb	0.122		0.122	0.122	0.122	0
S	428	383	45	427	383	44.2
Si	22.6		22.6	22.6	14.9	7.67
Sr	0.293		0.293	0.292	0.00168	0.291
Ti	0.204		0.204	0.204	0	0.203
U	15.5		15.5	15.5	0.00372	15.5
Zn	0.199		0.199	0.199	0	0.199
Zr	1.68		1.68	1.68	0	1.68
NO_3^-	1033	1033	0	1031	1031	0
NO_2^-	946	946	0	944	944	0
SO_4^{2-}	428	383	45	427	383	44.2
PO_4^{3-}	13.2	8.46	4.74	13.2	10.4	2.74
CO_3^{2-} calc.	632	632	0	631	609	22.2
OH^-		1091		NC	NC	NC
Cl^-	12.4	12.4	0	12.4	12.4	0
$C_2O_4^{2}$	NM	2.98	NM	2.97	2.97	0
F^-	3.64	3.64	0	3.63	2.72	0.913

NM ≡ not measured, NC ≡ not calculated

Table 2-11. Predicted Tank 4 Properties

Parameter	Measured	Predicted
Slurry Density kg/L	1.350	1.334
Supernate Density kg/L	1.326	1.294
Total Solids wt%	37.30	36.25
Soluble Solids wt%	33.76	32.40
Insoluble Solids wt%	3.54	3.85
Dissolved Solids wt%	35.00	33.70

2.9 Washing Study Methods

The blend of decanted Tank 51 and Tank 4 used for the settling study was used in a washing study, as requested by LWE in light of their concern about the S concentration in Tank 4 and specific evidence indicating that some of this S is insoluble or undissolved. The sludge was divided equally into two centrifuge tubes. The samples were then centrifuged for approximately 15 – 35 minutes, or until the solids were below the 5 mL mark on the centrifuge tube, then as much supernate as possible was removed without disturbing the solids. Water was then added and a vortex mixer was used to mix the decanted slurry and wash water. Again supernate was decanted. This process was repeated two more times for a total of three washes and four decants. Approximately, 3x dilutions were targeted for each wash – 10 to 12 mL of decanted slurry diluted to 35 to 40 mL with deionized water. Initial slurry masses, decant masses, and wash water masses are given in Table 2-12.

Table 2-12. Sulfate Washing Slurry Masses, Decant Masses, and Wash Water Masses

Operation	Replicate 1 (g)	Replicate 2 (g)
Initial Slurry Mass	29.25	30.68
Initial Decant Mass	10.37	12.21
Wash A (water) Added	24.31	24.46
Decant A Removed	32.64	32.80
Wash B (water) Added	26.20	27.17
Decant B Removed	26.93	27.74
Wash C (Water) Added	20.77	21.45
Decant C Removed	23.79	23.75
Remaining Slurry Mass	6.80	7.26

The decanted supernates were then diluted and submitted to SRNL AD for anion analysis by IC and elementals by ICP-AES. Also, the densities of the decanted supernates were measured, but it was not possible to measure the densities or weight percent solids of the intermediate or final slurry samples. The slurry remaining after the final decant was digested with a modified aqua regia digestion and submitted to SRNL-AD for elemental analysis by ICP-AES. There were trace solids left following the digestion which were not analyzed due to time constraints, but in the past these solids have generally been found to be undissolved Al (Boehmite).

2.10 Washing Study Results

The initial slurry composition was not measured because of the small amount of sample and time constraints. However, both Tank 4 and Tank 51 (prior to decanting) were characterized separately. This data was used to calculate a slurry composition for the mixture using the same blend ratio as noted in Section 2.5. The OLI model assumptions employed were 1) that Fe and U concentrations in the supernate are zero, and 2) nitrate and nitrite concentrations in the insoluble species are zero.

Table 2-13. Major Elements (>1,000 mg/kg), Nitrite and Nitrate in the Decanted-Tank 51/Tank 4 Blend Prior to Washing Calculated by OLI From Analyses for Tank 51 and Tank 4

Species	Concentration (mg/kg)
Al (total)	19000
Al (soluble)	12400
Fe (total)	10500
Na (total)	133000
S (total)	6230
S (soluble)	5450
U (total)	2360
NO_2^-	42000
NO_3^-	55700

The decant analytical results for each replicate are given in Table 2-14. Density measurements are also provided.

Table 2-14. Analytical Results for the Decants from Washing the Decanted-Tank 51/Tank 4 Sludge Blend

Species (Method)	Initial Decant (mg/kg supernate)	Decant A (mg/kg supernate)	Decant B (mg/kg supernate)	Decant C (mg/kg supernate)
Replicate 1				
NO_2^- (IC)	43200	17200	6320	1010
NO_3^- (IC)	61400	25700	8960	1430
SO_4^{2-} (IC)	20600	7020	3010	423
Na (ICP-AES)	136000	54000	20200	3150
S (ICP-AES)	7060	2700	1090	183
Al (ICP-AES)	11500	4590	1710	254
Replicate 2				
NO_2^- (IC)	45100	16100	6190	825
NO_3^- (IC)	63800	24000	8800	1160
SO_4^{2-} (IC)	21300	6580	2920	330
Na (ICP-AES)	138000	50700	20000	2550
S (ICP-AES)	6990	2490	1060	152
Al (ICP-AES)	11700	4380	1700	207
Density (g/mL)	1.34	1.12	1.04	1.00

The elemental results for the digestion of the slurry remaining after Decant C are given in Table 2-15. A mass balance on Al gives a result that is four times the measured result.

Table 2-15. Major Elements (>1,000 mg/kg) Detected by ICP-AES in the Washed Decanted-Tank 51/Tank 4 Sludge Blend

Species	Replicate 1 (mg/kg slurry)	Replicate 2 (mg/kg slurry)
Al	7860	7700
Ca	2020	1950
Fe	46700	45300
Mn	11100	10800
Na	20700	20600
Ni	8580	8330
S	1100	948
U	11200	10900

3.0 DISCUSSION

3.1 Sulfur

The conversion of the total supernate sulfur values, as shown in Table 2-2, from molar to wt% of total solids, yields 3.28 wt% S for the Tank 4 sample. Comparing this value with the total slurry sulfur value in Table 2-4, 3.68 wt% S, indicates that 0.40 wt% of the total sulfur on a total dried solids basis is insoluble, or possibly better described as undissolved. By comparison, 0.127 wt% of total sulfur in the current SB5 material being processed in DWPF is "undissolved"[10]. This latter sample was the first time that a major fraction of the sulfur had been found in the insoluble solids fraction of a DWPF sludge batch. The degree to which this "undissolved" sulfur would be removed during washing of SB6 in Tank 51 has been a matter of some concern. The scope of the original TTR[2] for this work was expanded by the customer to help address questions about the impact of washing on total sulfur levels in SB6. This is discussed further in the washing discussion below.

When the Tank 4 supernate sulfur value by ICP-AES (Table 2-2) is put on a slurry sulfate basis, the result is 36,800 mg sulfate/kg slurry. This is higher than the sulfate value measured in the supernate by IC of 29,800 mg/kg slurry, indicating that some 19% of the soluble sulfur is present as a species other than sulfate and hence not detected by ion chromatography. Therefore total supernate sulfur by ICP-AES should be monitored during washing in addition to supernate sulfate in order to avoid under estimating the amount of sulfur species removed or remaining in the supernate.

3.2 Elemental Ratios

A comparison of the elemental ratios for the major insoluble solids species using data from Table 2-4 and References 1 and 10 is given in Table 3-1. These ratios should remain constant, with the exception of Al for these unwashed samples, unless an addition of material containing one or more elements of interest is made. The Al/Fe ratio is misleading because a significant portion of the soluble Al in the unwashed, undecanted Tank 51-E1 sample will be removed. That aside, the Ca/Fe and U/Fe ratios for the Tank 4 and Tank 51-E1 components of SB6 are lower than the current ratios for these elements in SB5. The final Mn/Fe ratio will depend on the relative contributions of Tank 4 and Tank 51 to the final SB6 composition, so at this time one cannot predict if the ratio will be higher or lower than in SB5.

Table 3-1. Comparison of Elemental Ratios for Major Elements in the Tank 4, Tank 51-E1, and Current SB5 Samples

Element Ratio	Tank 4 (Unwashed)	Tank 51 – E1[1] (Unwashed)	Tank 40 – SB5 WAPS[10]
Al/Fe	0.36	4.8	0.60
Ca/Fe	0.036	0.058	0.092
Mn/Fe	0.050	0.47	0.23
U/Fe	0.29	0.18	0.33

1. Pickenheim, B. R., Bannochie, C. J., Pareizs, J. M., and Click, D. R. *Results of the Analysis of Tank 51 E-1 Sample (HTF-51-09-74 & -75) and Tank 12 Post-Aluminum Dissolution Rheology*, SRNL-L3100-2009-00190, Savannah River Site, Aiken, SC 29808 (2009). 10. Bannochie, C. J., Click, D. R. *Tank 40 Final SB5 Chemical Characterization Results Prior to Np Addition*, SRNL-STI-2009-00060, Rev. 1, Savannah River Site, Aiken, SC 29808 (2009).

3.3 Noble Metal Ratios

A comparison of the fission yield ratios for Ru:Rh, Ru:Pd, and Ru:Ag with those measured for the Tank 4 sample is provided in Table 3-2. The Tank 51-E1 sample and Tank 40 SB5 WAPS sample results are also provided for comparison. The ratios are based upon Ru due to its relatively high concentration in the sludge as compared with the other noble metals. The Ru:Rh ratio agrees reasonably well for all three samples, while the Ru:Ag ratios differ significantly from the fission yield ratios. This lack of agreement for the Ag ratios is not unexpected since the majority of the Ag is natural Ag originating from Ag saddles used in the dissolvers to scavenge radioactive iodine, while the noble metals are fission products of U-235. Consequently the relative concentration of Ag is not expected to be in proportion to the fission yields of its two isotopes. The Ru:Pd ratio for Tank 4 agrees reasonable well with that predicted by the fission yield, but it differs significantly for the unwashed Tank 51-E1 and current SB5 material. A possible explanation for this is that a portion of the Pd is soluble and hence has fractioned off into the salt waste, thus increasing the ratio of Ru to Pd in the sludge waste.

Table 3-2. Fission Yield Ratios and Measured Noble Metal Ratios for Tank 4, Tank 51-E1, and the SB5 WAPS Samples

Ratio	Fission Yield	Tank 4	Tank 51 – E1[1]	Tank 40 – SB5 WAPS[10]
Ru:Rh	3.7	4.1	4.8	4.6
Ru:Pd	6.9	8.1	21	29
Ru:Ag	342	15	5.0	7.2

1. Pickenheim, B. R., Bannochie, C. J., Pareizs, J. M., and Click, D. R. *Results of the Analysis of Tank 51 E-1 Sample (HTF-51-09-74 & -75) and Tank 12 Post-Aluminum Dissolution Rheology*, SRNL-L3100-2009-00190, Savannah River Site, Aiken, SC 29808 (2009).
10. Bannochie, C. J., Click, D. R. *Tank 40 Final SB5 Chemical Characterization Results Prior to Np Addition*, SRNL-STI-2009-00060, Rev. 1, Savannah River Site, Aiken, SC 29808 (2009).

3.4 Rheology Conclusions

Three main conclusions can be drawn from the rheology results. First, the Tank 4 sample appears, at this level of insoluble solids concentration, to behave as a Newtonian liquid with no measurable yield stress as shown by the data reported in Table 2-7. Second, the decanted Tank 51-E1 material exhibited yield stress along with much higher viscosity than measured for Tank 4. And finally, the blend of Tank 4 and decanted Tank 51-E1 showed that a significant reduction in the rheological properties of the decanted Tank 51-E1 material can be gained by the addition of Tank 4 material.

3.5 Settling Study Observations

The settling curves shown previously in Figure 2-7 indicate that the settling behavior of the decanted Tank 51-E1 sample (Al dissolution treated HM sludge) is very slow and essentially stops after 10 days. The addition of Tank 4 material (PUREX sludge) significantly improves the settling behavior of the decanted Tank 51-E1 sludge. The blended material had rapid settling over the first five days, but continued to settle slowly even after 10 days.

3.6 Modeling Observations

The results of the OLI simulation calculations show that the presence of undissolved Burkeite in the Tank 4 sample is reasonable given the experimentally determined composition and assuming a small difference in the Na concentration that is well within the analytical uncertainty.

The overall composition predictions from the OLI model match the measured values very closely. The major differences are:

1. The predicted undissolved Al is about 4 mol% of the total Al compared to the measured 13 mol%. The OLI software does not predict Al solubility well unless the actual form of the undissolved Al is known.
2. The predicted soluble phosphate is higher than measured.
3. About 3.5 mol% of the carbonate was calculated to be associated with Burkeite whereas none was calculated for the sample analysis.
4. The Na content had to be increased from 5272 to 5379 mmol/kg (121205 mg/kg to 123650 mg/kg) to precipitate S as Burkeite. This increased value is 2% higher than the calculated charge balance value, which is well within the analytical uncertainty.
5. The predicted undissolved solids were slightly higher than measured. The actual hydration of the species after a total solids determination is not known so the correct choice of solid species in OLI cannot be made. Generally, the OLI predicted species contain more water of hydration than are probably present in the solids determination product.

3.7 Sludge Washing

The purpose of washing is to remove soluble species from sludge slurry, primarily Na. However, with the SRNL washing of the decanted Tank 51/Tank 4 blend, the primary purpose was to determine if currently insoluble sulfur would dissolve, and to determine the extent of sulfur removal during washing. Sulfur removal, along with the other primarily soluble species, Fe, and Al is presented as a mass balance in Table 3-3 below. The Al values shown in the table appear to be biased low based on those predicted values from the OLI model. They may in fact be biased low as a result of the aqua regia digestion and the observed presence of some undissolved white solids that in the past has been an Al species.

Table 3-3. Mass Balance for Washing the Decanted-Tank 51/Tank 4 Sludge Blend

	Al (mg)	Soluble Al (mg)	Fe (mg)	Na (mg)	S (mg)	NO_2^- (mg)	NO_3^- (mg)
Replicate 1							
Initial Amount [1]	553	362	307	3901	182	1233	1658
Initial Decant Removed	-120	-120	ND	-1414	-73	-447	-636
Decant A removed	-150	-150	ND	-1763	-88	-562	-838
Decant B Removed	-46	-46	ND	-544	-29	-170	-241
Decant C Removed	-6	-6	ND	-75	-4	-24	-34
Calculated remaining	231	40	307	105	-12	30	-91
Analyzed remaining [2]	53 [4]	1.3	318	141	7	NM	NM
Percent remaining [3]	10	0.4	100	4	4	NA	NA
Replicate 2							
Initial Amount [1]	580	380	322	4092	191	1293	1739
Initial Decant Removed	-143	-143	ND	-1679	-85	-550	-779
Decant A removed	-144	-144	ND	-1664	-82	-529	-787
Decant B Removed	-47	-47	ND	-556	-29	-172	-244
Decant C Removed	-5	-5	ND	-61	-4	-20	-27
Calculated remaining	241	41	322	133	-9	22	-98
Analyzed remaining [2]	56 [4]	1.2	339	150	8	NM	NM
Percent remaining [3]	10	0.3	100	4	4	NA	NA

[1] Based on a calculated composition of Tank 51/Tank 40 (see Table 2-13).
[2] Results based on ICP-AES analysis of slurry remaining after washing.
[3] Based on the initial amount and analyzed remaining.
[4] The aluminum result from the slurry digestion may be biased low. An aqua regia digestion was used which may not dissolve some Al species.
NA ≡ Not Applicable
ND ≡ Not Detected
NM ≡ Not Measured

Presented in Table 3-4 is the cumulative percent of the soluble species and Al removed following each decant. As can be seen, following Decant B, greater than 100 percent of the S and nitrate is calculated to have been removed. This could be due to analytical uncertainties of the supernate analyses and uncertainties in the calculation of the initial amounts. Note that initial amounts were calculated by mass balance with inputs being the Tank 4 analyses and the Tank 51 analyses followed by adjustment for the decant prior to mixing with Tank 4. It should also be noted that the Tank 51/Tank 4 combination was made by volume and converted to mass using measured densities, another source of uncertainty. In the calculation of insoluble S, the wt% insoluble solids, a key input, was assumed, not measured Unfortunately, due to these uncertainties and assumptions, a definitive quantification of insoluble S is not possible. Based on the digestion results of the washed slurry, 96% of the S was removed (see Table 3-5). This is equivalent to the percent of Na removed. Based on the washing data, greater than 100% of the S was removed, indicating that the initial S concentration *could* have been higher than calculated; however, the calculated S remaining is on the order of the measurement uncertainty.

Table 3-4. Cumulative Component Percent Removed Following Each Decant

	Al	Na	S	NO_2^-	NO_3^-
Replicate 1					
Initial Decant	21	36	41	36	39
Decant A	48	80	89	82	90
Decant B	57	94	106	96	105
Decant C	58	96	108	98	107
Replicate 2					
Initial Decant	24	40	45	43	45
Decant A	49	80	88	83	91
Decant B	57	94	104	97	105
Decant C	58	95	106	98	107

Based on the OLI modeling about 12%, or 780 mg/kg, of the S was initially insoluble. If 2% of the initially soluble S (5450 mg/kg) remained (~110 mg/kg) and 4% of the total S remained (~250 mg/kg), then about 140 mg/kg of the initially insoluble S remained. This is about 18% of the initially insoluble S, so based on the final washed sludge analysis for S, 78% of the initially insoluble S may have dissolved. If the washing data for S is used, the amount dissolved is 100%. That 78-100% of the initially insoluble S dissolved supports the hypothesis that most of the initially insoluble S was Burkeite. From the amounts of Ba in the samples, and assuming $BaSO_4$ is totally insoluble, $BaSO_4$ accounts for about 2.2% of the initially insoluble S.

Analyzing this another way by comparing the analysis of Decant C, which represents the soluble Na and S in the final decanted slurry, and that of the final digestion, which represents the total solids. If it is assumed that the final slurry is 20% insoluble solids, the Decant C results can be placed on a slurry basis (1-weight fraction of insoluble solids = weight fraction of supernate). An estimate of the amount of insoluble Na and S can then be made by comparing the soluble amounts to the total amounts. The results of this exercise are given in Table 3-5. As can be seen, 90% of the detected Na and S are insoluble after the washing is performed, representing 3-4% of the Na and S in the unwashed sludge. This insoluble Na and S would likely not be dissolved with normal washing. Converting the soluble Na and S values from Table 2-14 to a slurry basis and subtracting these from the final washed slurry values in Table 2-15, multiplying the result by the remaining slurry mass from Table 2-12, and finally dividing the result into the initial mass balance value shown in Table 3-3, one calculates the percent insoluble relative to initial shown below.. This exercise shows that Na and S behave similarly, and the majority of S (on the order of 95% in these tests) is either soluble or does dissolve during washing.

Table 3-5. Calculations of Insoluble Na and S in Washed Slurry with Comparison to Initial Amounts.

Species	Replicate 1 (mg/kg slurry)	Replicate 2 (mg/kg slurry)
Na (soluble)	2520	2040
Na (total)	20700	20600
S (soluble)	146	122
S (total)	1100	948
	(Percent)	**(Percent)**
Na (insoluble relative to initial)	3	3
S (insoluble relative to initial)	3	4

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4.0 CONCLUSIONS

The following conclusions were drawn from the Tank 4 analytical results reported here:

- Any projected blend of Tank 4 and the current Tank 51 contents will produce a SB6 composition that is lower in Ca and U than the current SB5 composition being processed by DWPF.

- Unwashed Tank 4 has a relatively large initial S concentration of 3.68 wt% on a total solids basis, and approximately 10% of the total S is present as an insoluble or undissolved form.

- There is 19% more S than can be accounted for by IC sulfate measurement. This additional soluble S is detected by ICP-AES analysis of the supernate.

- Total supernate and slurry sulfur by ICP-AES should be monitored during washing in addition to supernate sulfate in order to avoid under estimating the amount of sulfur species removed or remaining in the supernate.

- OLI simulation calculations show that the presence of undissolved Burkeite in the Tank 4 sample is reasonable, assuming a small difference in the Na concentration that is well within the analytical uncertainties of the reported value.

The following conclusions were drawn from the blend studies of Tank 4 and decanted Tank 51-E1:

- The addition of Tank 4 slurry to a decanted Tank 51-E1 sample significantly improved the degree and time for settling.

- The addition of Tank 4 slurry to a decanted Tank 51-E1 sample significantly improved the plastic viscosity and yield stress.

- The SRNL washing test, where nearly all of the wash solution was decanted from the solids, indicates that approximately 96% or more of the total S was removed from the blend in these tests, and the removal of the sulfur tracks closely with that of Na. Insoluble (undissolved) S remaining in the washed sludge was calculated from an estimate of the final slurry liquid fraction, the S result in the slurry digestion, and the S in the final decant (which was very close to the method detection limit). Based on this calculated result, about 4% of the initial total S remained after these washes; this amount is equivalent to about 18% of the initially undissolved S.

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