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Sludge Batch 7 Preparation: Tank 4 and 12 Characterization

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EXECUTIVE SUMMARY

Samples of PUREX sludge from Tank 4 and HM sludge from Tank 12 were characterized in preparation for Sludge Batch 7 (SB7) formulation in Tank 51. SRNL analyses on Tank 4 and Tank 12 were requested in separate Technical Assistance Requests (TAR).[i, ii] The Tank 4 samples were pulled on January 19, 2010 following slurry operations by F-Tank Farm. The Tank 12 samples were pulled on February 9, 2010 following slurry operations by H-Tank Farm.

At the Savannah River National Laboratory (SRNL), two 200 mL dip samples of Tank 4 and two 200 mL dip samples of Tank 12 were received in the SRNL Shielded Cells. Each tank's samples were composited into clean 500 mL polyethylene storage bottles and weighed. The composited Tank 4 sample was 428.27 g and the composited Tank 12 sample was 502.15 g.

As expected there are distinct compositional differences between Tank 4 and Tank 12 sludges. The Tank 12 slurry is much higher in Al, Hg, Mn, and Th, and much lower in Fe, Ni, S, and U than the Tank 4 slurry.

The Tank 4 sludge definitely makes the more significant contribution of S to any sludge batch blend. This S, like that observed during SB6 washing, is best monitored by looking at the total S measured by digesting the sample and analyzing by inductively coupled plasma – atomic emission spectroscopy (ICP-AES). Alternatively, one can measure the soluble S by ICP-AES and adjust the value upward by approximately 15% to have a pretty good estimate of the total S in the slurry. Soluble sulfate measurements by ion chromatography (IC) will be biased considerably lower than the actual total S, the difference being due to the non-sulfate soluble S and the undissolved S.

Tank 12 sludge is enriched in U-235, and hence samples transferred into SRNL from the Tank Farm will need to be placed on the reportable special nuclear material inventory and tracked for total U per SRNL procedure requirements.

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i. Colleran, H. Q. *Tank 4 Slurry Samples in Support of Sludge Batch 7 Preparation*, 2010-LWOTF-002, Rev. 0, Savannah River Site, Aiken, SC 29808 (2010).

ii. Martin, K. B. *Tank 12 Slurry Samples in Support of Sludge Batch 7 (SB7)*, 2010-LWOTF-001, Rev. 0, Savannah River Site, Aiken, SC 29808 (2010).

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AD	Analytical Development
CVAA	Cold Vapor Atomic Absorption Spectroscopy
DOE	Department of Energy
DWPF	Defense Waste Processing Facility
g	gram
HM	H-Area Modified PUREX
ICP-AES	Inductively Couple Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
L	liter
PUREX	Plutonium Uranium Reduction Extraction
SB6	Sludge Batch 6
SB7	Sludge Batch 7
SNRL	Savannah River National Laboratory
SRS	Savannah River Site
TAR	Technical Assistance Request
Wt% TS	Weight Percent of Total Solids

1.0 Introduction

Samples of PUREX sludge from Tank 4 and HM sludge from Tank 12 were characterized in preparation for Sludge Batch 7 (SB7) formulation in Tank 51. SRNL analyses on Tank 4 and Tank 12 were requested in separate Technical Assistance Requests (TAR).[1,2] The Tank 4 samples were pulled on January 19, 2010 following slurry operations by F-Tank Farm. The Tank 12 samples were pulled on February 9, 2010 following slurry operations by H-Tank Farm.

2.0 Experimental Procedure

At the Savannah River National Laboratory (SRNL), two 200 mL dip samples of Tank 4 and two 200 mL dip samples of Tank 12 were received in the SRNL Shielded Cells. Each tank's samples were composited into clean 500 mL polyethylene storage bottles and weighed. The composited Tank 4 sample was 428.27 g and the composited Tank 12 sample was 502.15 g.

Eight separate aliquots of the slurry were digested, four with HNO_3/HCl (aqua regia[3]) in sealed Teflon® vessels and four in Na_2O_2 (alkali or peroxide fusion[4]) 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, and alkaline earth metals, such as Ca, that may be contaminants in the Na_2O_2 are not determined from this preparation. Three Analytical Reference Glass – 1[5] (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 and 12 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 and ion chromatography (IC).

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[6]. 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.

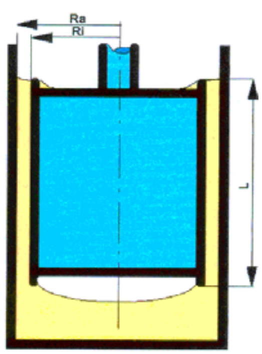
The bob typically used for measuring tank sludge is the MV I rotor. The shape, dimensions, and geometric constants for the MV I rotor are provided in Table 2-1.

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 with the control being a calculated viscosity within ± 10% of the reported NIST viscosity at a given temperature. 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.

$$\tau = \tau_o + \mu_\infty \dot{\gamma} \tag{1}$$

Table 2-1. 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

3.0 Results and Discussion

Table 3-1 presents the measured Tank 4 and Tank 12 densities and weight percent solids data.[7] The Tank 4 results compare quite well with those reported on the previous Tank 4 slurry sample.[8] Particularly close was the calculated insoluble solids content of the two samples, 3.62 wt% for the sample taken last summer during SB6 preparations, and 3.64 wt% for this sample of Tank 4.

Table 3-1. Densities and Weight Percent Solids for the Tank 4 and Tank 12 SB7 Preparation Samples

Property	Tank 4 Result (%RSD)	Tank 12 Result (%RSD)
Slurry Density (g/mL)	1.34 (0.1) †	1.27 (0.9) ‡
Supernate Density (g/mL)	1.32 (0.3) †	1.23 (0.9) ‡
Wt% Total Solids (Slurry Basis)	34.5 (0.3)	28.8 (0.2)
Wt% Dissolved Solids[a] (Supernate Basis)	32.0 (0.1)	24.9 (0.3)
Wt% Soluble Solids[b] (Slurry Basis)	30.9 (0.1) [c]	23.6 (0.2) [c]
Wt% Insoluble Solids (Slurry Basis)	3.64 (2.1) [c]	5.23 (1.2) [c]

† Temperature at time of density measurements was 10°C.
‡ Temperature at time of density measurements was 16 °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

Rheological measurements were made on the Tank 12 material, but not on the Tank 4 material. Previous measurements on Tank 4 [8] indicated essentially no yield stress and a plastic viscosity of 6.7 cP, and the current sample also appeared to be very thin. Figure 3-1 and Figure 3-2 provide the shear stress versus shear rate flow curves for the Tank 12 sample. The resulting plastic viscosities in cP and yield stresses in Pa are summarized in Table 3-2.

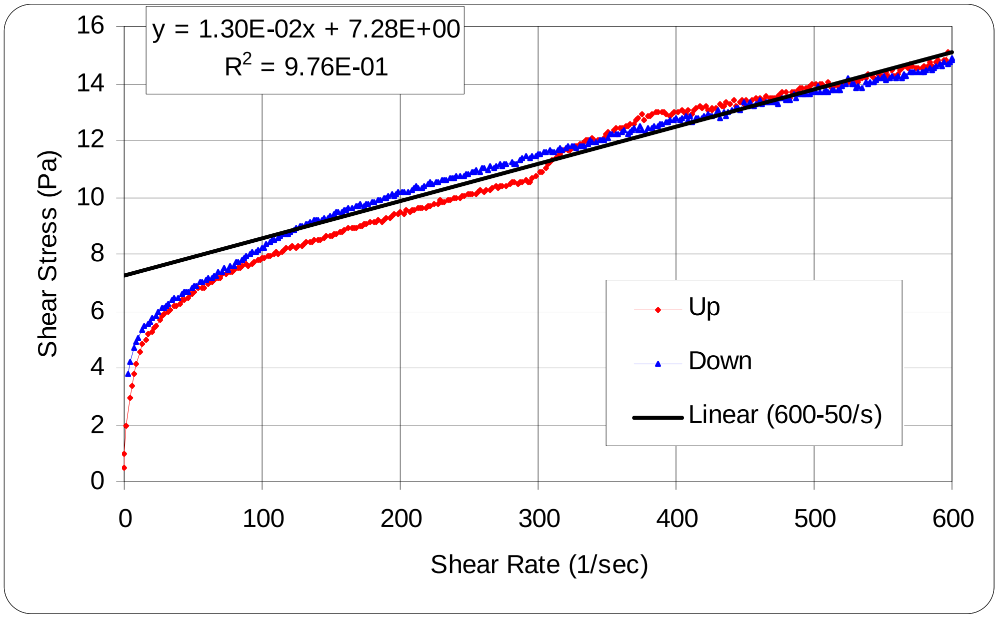


Figure 3-1. Tank 12 Shear Stress vs. Shear Rate Replicate 1

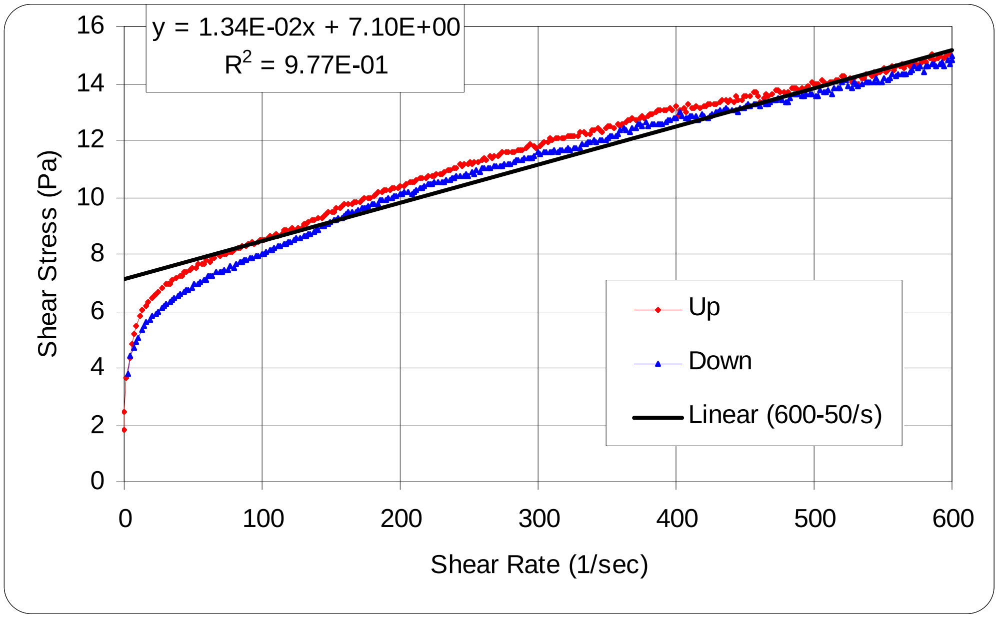


Figure 3-2. Tank 12 Shear Stress vs. Shear Rate Replicate 2

Table 3-2. Rheology Summary for Tank 12.

Replicate	Consistency (cP)	Yield Stress (Pa)
Run 1	13.0	7.3
Run 2	13.4	7.1
Average	13.2	7.2

Table 3-3 provides the elemental composition for both Tank 4 and Tank 12 slurry on a weight percent dried total solids (Wt% TS) basis. Elemental compositions were determined by digestion of the samples and analyses by ICP-AES, ICP-MS, and CVAA. A less than value is reported for the lowest quantification limit measured for the analyzed set of replicates. For elements determined by ICP-MS, the following isotopes were used for each determination: ***Cd*** in Tank 4, all isotopes were below their quantification limit, and varied across two orders of magnitude, hence the less than value reported is that from the ICP-AES; for Tank 12, the average for masses **111**, **112**, and **114** was used since the distribution appeared to be natural. Mass **139** was used for ***La***. ***Ce*** was the sum of masses **140** and **142** since it was not a natural distribution. For ***Gd***, the sum of masses **155**, **156**, **157**, **158**, and **160** was used since it was not a natural distribution. For ***Pb***, the average for masses **206**, **207**, and **208** was used since the distribution appeared to be natural. ***Th*** was determined from mass **232** since mass 230 was below the quantification limit. ***U*** was determined from the sum of masses **233**, **234**, **235**, **236**, and **238** since it was not a natural distribution.

Table 3-3. Concentrations of Elements in Total Dried Solids for the Tank 4 and Tank 12 SB7 Preparation Samples. Results are Averages of Dissolution and Analysis of Three to Eight Aliquots of the Respective Slurry

Element	Tank 4 Wt% Total Solids	%RSD	Prep Method	Tank 12 Wt% Total Solids	%RSD	Prep Method	Instrument
Al	2.23	1.7	PF	7.83	9.3	PF	ICP-AES
B	0.0105	3.1	AR	0.0221	5.9	AR/PF	ICP-AES
Ba	0.0207	2.7	AR/PF	0.0123	6.9	AR/PF	ICP-AES
Be	<0.00054	-	AR	<0.00057	-	AR	ICP-AES
Ca	0.117	1.1	AR	0.0580	1.7	AR	ICP-AES
Cd	<0.0022*	-	AR	0.00105	19	AR	ICP-MS
Ce	0.0277	0.8	AR	0.0189	1.6	AR	ICP-MS
Co	0.00286	2.3	AR	<0.0019	-	AR	ICP-AES
Cr	0.0936	2.7	AR/PF	0.0347	5.6	AR/PF	ICP-AES
Cu	0.00770	1.4	AR	0.0110†	0.0	AR	ICP-AES
Fe	3.46	1.7	AR/PF	1.37	8.9	AR/PF	ICP-AES
Gd	0.00131	1.4	AR	0.000776	2.5	AR	ICP-MS
Hg	0.0374	3.8	AR	0.760	7.5	AR	CVAA
K	0.165	1.3	AR	0.280	2.8	AR	ICP-AES
La	0.0147	2.1	AR	0.00941	3.9	AR	ICP-MS
Li	0.00252	4.2	AR	<0.0062	-	AR	ICP-AES
Mg	0.0121	4.8	AR/PF	0.0223	13	AR/PF	ICP-AES
Mn	0.165	2.9	AR/PF	0.906	5.3	AR/PF	ICP-AES
Mo	0.0143	2.6	AR	0.0115	4.0	AR	ICP-AES
Na	34.3	1.1	AR	31.1	2.0	AR	ICP-AES
Ni	0.900	2.5	AR/PF	0.116	6.9	AR/PF	ICP-AES
P	0.0825	3.5	AR	0.0634	1.9	AR	ICP-AES
Pb	0.00408	9.0	AR	0.00129	12	AR	ICP-MS
S	3.67	2.9	AR/PF	0.440	6.6	AR	ICP-AES
Sb	<0.017	-	AR	<0.011	-	AR	ICP-AES
Si	0.155	6.8	PF	0.244	14	PF	ICP-AES
Sn	<0.028	-	AR	<0.011	-	AR	ICP-AES
Sr	0.00766	4.6	AR/PF	0.00557	5.7	AR/PF	ICP-AES
Ti	<0.0039	-	AR	0.00227	12	AR/PF	ICP-AES
Th	<0.00068	-	AR	0.589	2.3	AR	ICP-MS
U	0.995	2.1	AR	0.256	1.7	AR	ICP-MS
V	<0.0039	-	AR	<0.0042	-	AR	ICP-AES
Zn	<0.0060	-	AR	0.00619	1.8	AR	ICP-AES
Zr	0.0467	1.5	AR	0.0271	2.5	AR	ICP-AES

* The Tank 4 Cd value is reported from ICP-AES because all ICP-MS isotopic values were also below their quantification limit.
† Reported result is the average of two replicates.
ICP-MS ≡ inductively coupled plasma – mass spectrometry, ICP-AES ≡ inductively coupled plasma – atomic emission spectroscopy, CVAA ≡ cold vapor atomic absorption spectroscopy, AR ≡ aqua regia digestion, PF ≡ peroxide fusion digestion

Surveying the results given in Table 3-3, the Tank 12 slurry is much higher in Al, Hg, Mn, and Th, and much lower in Fe, Ni, S, and U than the Tank 4 slurry. Not unexpected for a HM sludge (Tank 12) versus a PUREX sludge (Tank 4). Typically, a value for S above the quantification limit from the peroxide fusion digestion is not obtained, but for this set of Tank 4 digestions, a value was obtained. The result was consistent with that obtained from the aqua regia digestion. Both preparations were averaged and reported for the Tank 4 slurry.

The fission product noble metal and silver concentrations for both Tank 4 and Tank 12 sludge are given in Table 3-4. The values were calculated from the 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 3-4. Concentrations of Noble Metals and Silver in Total Dried Solids for the Tank 4 and Tank 12 SB7 Preparation Samples. Results are Averages of Aqua Regia Digestions and ICP-MS Analyses of Four Slurry Aliquots.

Element	Tank 4 Wt% Total Solids	%RSD	Tank 12 Wt% Total Solids	%RSD
Ru	0.0243	0.6	0.0119	1.2
Rh	0.00549	2.4	0.00257	5.2
Pd	0.00171	1.4	0.000598	4.6
Ag	0.0150	2.0	0.00279	2.2

The soluble elemental, i.e. those in the supernate, values above the quantification limit are provided in Table 3-5. While Fe was detected in three replicates in the Tank 12 sample, there was considerable scatter in the results. Iron was not detected at all in the Tank 4 supernate. The most significant difference is the difference in the S values between the two tanks. Tank 4, as observed for the total S value (Table 3-3), has more than an order of magnitude more soluble S as compared to Tank 12.

Table 3-5. Concentrations of Soluble Elements for the Tank 4 and Tank 12 SB7 Preparation Samples. Results are Averages of Four Supernate Aliquots

Element	Tank 4 Molarity	%RSD	Tank 12 Molarity	%RSD
Al	0.345	0.1	0.268	0.4
B	0.00252	2.0	0.00573	1.6
Ca	0.000218	6.4	0.000135	2.5
Cr	0.00796	1.0	0.00209	0.5
Fe	ND	-	0.0000704†	54
K	0.0194	2.6	0.0274	0.9
Mg	ND	-	0.0000227†	3.0
Mo	0.000722	1.4	0.000382	1.7
Na	6.89	0.5	5.06	0.2
P	0.00719	2.1	0.00491	1.5
S	0.529	0.5	0.0456	2.6

ND = not detected
† Reported result is based on three replicates.

Table 3-6 provides the supernate anion concentrations for both Tank 4 and 12 samples determined by IC. Comparing the total soluble S (Table 3-5) with the soluble S as sulfate (Table 3-6), 4.5% of the Tank 4 sulfur is present as non-sulfate species, much lower than that seen during SB6 Qualification sample washing in SRNL where the average was 17.5%. The non-sulfate species in Tank 12 accounts for about 18% of the total soluble S. The Tank 4 material has more than an order of magnitude more soluble S than does the material in Tank 12.

Converting the soluble S, given in Table 3-6, to a wt% total solids basis gives a value of 3.61 wt% S for Tank 4 and 0.392 wt% S for Tank 12. Comparing these to the total S determination in Table 3-5 indicates that <2% of the Tank 4 total S is undissolved, while 11% of the Tank12 total S is undissolved. The difference between the soluble S and sulfate species also varied considerably between the two tank samples. For Tank 4, the difference is 4.5% while for Tank 12 the difference is 18%.

Table 3-6. Concentrations of Anions on a Supernate Basis for the Tank 4 and Tank 12 SB7 Preparation Samples. Results are Averages of Four Supernate Aliquots

Anion	Tank 4 Molarity	%RSD	Tank 12 Molarity	%RSD
Br^-	<0.03	-	<0.02	-
Cl^-	<0.006	-	<0.05	-
F^-	<0.01	-	<0.1	-
HCO_2^-	<0.005	-	<0.04	-
NO_3^-	1.13	1.6	1.11	4.8
NO_2^-	0.972	0.8	1.05	0.5
$C_2O_4^{2-}$	<0.02	-	<0.02	-
PO_4^{3-}	<0.02	-	<0.02	-
SO_4^{2-}	0.505	1.2	0.0374	1.3

During SB6 Qualification sample washing, about 15% of the total S as an undissolved species was observed (see Figure 3-3). Note that "undissolved" does not imply that this S cannot dissolve during washing operations; it just indicates that it is not soluble under the current sludge conditions. It can also be seen in Figure 3-3 that there is a small, but consistent difference between the soluble S from ICP-AES and the soluble sulfate by IC, expressed in the figure on a molar basis, for the SB6 Qualification sample during washing. This difference averaged 17.5 % (Std. Dev. 2.6 %) across the eight washes, but the as-received material did not show any difference between soluble S and sulfate. Hence the difference in soluble S and sulfate seen during sludge batch washing appears to be due to the contribution of S from the HM sludge rather than the PUREX sludge.

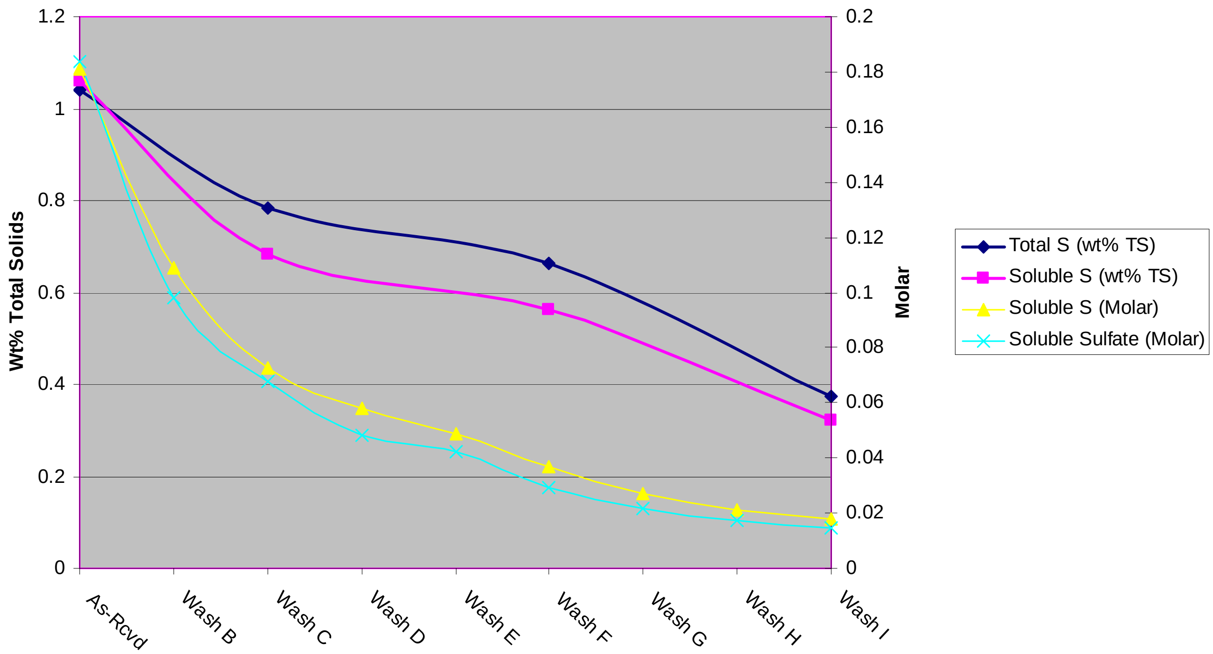


Figure 3-3. Sulfur and Sulfate Measured During the SRNL SB6 Qualification Sample Washing

Table 3-7 provides the isotopic U distribution in terms of wt% of total solids. Based upon this data, the Tank 4 slurry has a U enrichment of 0.39%, while the Tank 12 slurry has a U enrichment of 0.99%.

Table 3-7. Uranium Isotopes in Tank 4 and Tank 12 SB7 Preparation Samples. Results are Averages of Four Slurry Aliquots by ICP-MS

Isotope	Tank 4 Wt% Total Solids	%RSD	Tank 12 Wt% Total Solids	%RSD
U-233	<1.3E-04	-	2.18E-04	19
U-234	<6.5E-05	-	1.56E-04	14
U-235	3.83E-03	1.0	2.52E-03	2.4
U-236	1.98E-04 †	4.7	2.59E-04	7.5
U-238	9.91E-01	4.2	2.53E-01	3.5

† Reported result is based on three replicates.

4.0 Conclusions

As expected there are distinct compositional differences between Tank 4 and Tank 12 sludges. The Tank 12 slurry is much higher in Al, Hg, Mn, and Th, and much lower in Fe, Ni, S, and U than the Tank 4 slurry.

The Tank 4 sludge definitely makes the more significant contribution of S to any sludge batch blend. This S, like that observed during SB6 washing, is best monitored by looking at the total S measured by digesting the sample and analyzing by ICP-AES. Alternatively, the soluble S can be measured by ICP-AES and the value adjusted upward by approximately 15% to have a pretty good estimate of the total S in the slurry. Soluble sulfate measurements by IC will be biased considerably lower than the actual total S, the difference being due to the non-sulfate soluble S and the undissolved S.

Tank 12 sludge is enriched in U-235, and hence samples transferred into SRNL from the Tank Farm will need to be placed on the reportable special nuclear material inventory and tracked for total U per SRNL procedure requirements.

5.0 References

1. Colleran, H. Q. *Tank 4 Slurry Samples in Support of Sludge Batch 7 Preparation*, 2010-LWOTF-002, Rev. 0, Savannah River Site, Aiken, SC 29808 (2010).

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Distribution:

A. B. Barnes, 999-W
D. A. Crowley, 773-43A
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
A. M. Murray, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
D. K. Peeler, 999-W
M. E. Stone, 999-W
N. E. Bibler, 773-A
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S. H. Reboul, 773-A
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J. M. Gillam, 766-H
B. A. Hamm, 766-H
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D. D. Larsen, 766-H
D. J. McCabe, 773-42A
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J. W. Ray, 704-S
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