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Revision 0

CHARACTERIZATION OF THE MARCH 2004 TANK 40 (SLUDGE BATCH 3) DIP SAMPLES

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May 2004

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EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) has begun processing Sludge Batch 3 (SB3). Sludge Batch 3 consists of the heel in Tank 40 (Sludge Batch 2), the contents of Tank 51, and a Np stream from H Canyon. Two dip samples were pulled from Tank 40 in March 2004 after the initial Tank 51 to 40 transfer and the first transfer of Np material from H Canyon. These samples were combined into one sample and characterized by the Savannah River Technology Center (SRTC). The purpose of this characterization is to provide DWPF with a current Tank 40 (SB3) composition for comparison to Sludge Receipt and Adjustment Tank (SRAT) receipt analyses as they transition to the new sludge batch. The key analytical results for this sample are given below.

Slurry Density	1.185 g/mL of slurry	Coal (mg/kg)	71 – 240 mg/kg of slurry
Supernate Density	1.048 g/mL of supernate	Base Equivalents	0.43 Eq/L slurry of slurry
Total Solids	20.0 wt% of slurry	Al	6.04 wt% of total solids
Soluble Solids	5.17 wt% of slurry	Ca	1.66 wt% of total solids
Insoluble Solids	14.8 wt% of slurry	Fe	19.4 wt% of total solids
Fluoride	235 mg/kg of slurry	Mg	1.67 wt% of total solids
Formate	<1,000 mg/kg of slurry	Mn	3.95 wt% of total solids
Chloride	<200 mg/kg of slurry	Na	12.1 wt% of total solids
Nitrite	15,500 mg/kg of slurry	Ni	1.09 wt% of total solids
Nitrate	10,500 mg/kg of slurry	S	0.288 wt% of total solids
Phosphate	<1,000 mg/kg of slurry	U	6.99 wt% of total solids
Sulfate	1,690 mg/kg of slurry	Consistency	5.6 cp
Oxalate	1,030 mg/kg of slurry	Yield Stress	33 dynes/cm^2

The conclusions from this analysis are:

- Coal content of the Tank 40 sample was similar to that predicted using analysis of the Tank 51 qualification sample.

- Most, if not all, the sulfur was soluble and in the form of sulfate. Ion Chromatography (IC) analysis of the water dilution of the slurry is adequate for sulfate determination in the SB3 sample.

- Most, if not all, the oxalate was soluble. IC analysis of the water dilution of the slurry is adequate and the acid strike method is not necessary for oxalate determination in SB3.

- The yield stress and the consistency for the March 2004 SB3 sample is within the DWPF Operating Region.

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LIST OF ACRONYMS

DWPF	Defense Waste Processing Facility
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma Emissions Spectroscopy
NIST	National Institute of Standards and Technology
SB3	Sludge Batch 3
SRAT	Slurry Receipt Adjustment Tank
SRTC	Savannah River Technology Center
TGA	Thermogravimetric Analysis

1.0 INTRODUCTION AND BACKGROUND

The Defense Waste Processing Facility (DWPF) has begun processing Sludge Batch 3 (SB3). Sludge Batch 3 consists of the heel in Tank 40 (Sludge Batch 2), the contents of Tank 51, and a Np stream from H Canyon.

Two dip samples were pulled from Tank 40 after the initial Tank 51 to 40 transfer and the first transfer of Np material from H Canyon[1]. These samples were combined into one sample and characterized by SRTC. The purpose of this characterization is to provide DWPF with a current Tank 40 (SB3) composition for comparison to SRAT receipt analyses as they transition to the new sludge batch.

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[1] Initial plans were to transfer all of Tank 51 and all of a Np stream from H Canyon to Tank 40 prior to Sludge Batch 3 processing. Due to operational problems, only about 70% of the Tank 51 to 40 transfer and 55% of the H Canyon to Tank 40 transfers were completed. Final transfers are planned for late spring of this year.

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2.0 CHEMICAL CHARACTERIZATION

2.1 Results

The two dip samples from Tank 40 were received by SRTC in March 2004 and combined into one sample of approximately 350 mL. Chemical and physical analyses, including rheology, were performed using aliquots of slurry subsampled from this combined sample. To obtain supernate, a portion of slurry was filtered using a 0.45 μm Nalgene® filter.

Slurry and supernate densities were determined by weighing slurry and supernate in vessels of known volume. Weight percent total solids were determined by drying slurry samples. Weight percent dissolved solids (solids in the supernate) were measured by drying supernate samples. The measured weight percent total and dissolved solids were then used to calculate the weight percent soluble and insoluble solids in the slurry:

$$W_{is} = \frac{W_{ts} - W_{ds}}{100 - W_{ds}} \cdot 100 \text{ and } W_{ss} = \frac{100 - W_{ts}}{100 - W_{ds}} \cdot W_{ds}$$

where,
W_{ts} = weight percent solids in the slurry (weight percent total solids)
W_{ds} = weight percent solids in the supernate (weight percent dissolved solids)
W_{is} = weight percent insoluble solids in the slurry
W_{ss} = weigh percent soluble solids in the slurry.

Weight percent calcined solids were determined by heating slurry samples to 1000°C in alumina crucibles. Density and weight percent solids results of the slurry are presented in Table 2-1.

Table 2-1. Density and Weight Percent Solids of the Tank 40 Dip Sample

	Result With 95% Confidence Interval
Slurry Density (g/mL) [a]	1.185 ± 0.018
Supernate Density (g/mL) [a]	1.048 ± 0.002
Wt% Total Solids [b] (Wt% solids in slurry)	20.0 ± 1.9
Wt% Dissolved Solids [c] (Wt% solids in supernate)	6.07 ± 0.03
Wt% Soluble Solids [d] (Wt% soluble solids in slurry)	5.17 ± 0.18
Wt% Insoluble Solids [d] (Wt% insoluble solids in slurry)	14.8 ± 2.0
Wt% Calcined Solids [e] (Wt% calcined solids in slurry)	17.4 ± 0.2

[a] Based on triplicate measurements
[b] Based on quadruplicate samples weighed three times each.
[c] Based on triplicate measurements
[d] Calculated from weight percent total solids and weight percent dissolved solids)
[e] Based on five measurements.

Supernate was characterized by Ion Chromatography (IC) for anions and Inductively Coupled Plasma Emissions Spectroscopy (ICP-ES) for sodium, the primary cation in the supernate. IC provides quantification of the primary anions in the supernate except hydroxide. ICP-ES provides the elemental composition of the supernate. These results are given in Table 2-2. Presented are all the anions, excluding bromide, that are routinely reported by IC and the major cation - sodium.

Table 2-2. Supernate Composition

	Concentration with 95% Confidence Interval (M)
Fluoride [a]	0.0109 ± 0.0006
Formate [a]	0.0020 ± 0.0001
Chloride [a]	<0.0009
Nitrite [a]	0.420 ± 0.011
Nitrate [a]	0.191 ± 0.010
Phosphate [a]	<0.0006
Sulfate [a]	0.0218 ± 0.0007
Oxalate [a]	0.0160 ± 0.0005
Sodium [a]	1.00 ± 0.02

[a] Based on four replicates.

Table 2-3 lists the anion composition and base equivalents (equivalents of acid needed to reach pH 7) of the Tank 40 slurry. The anions were determined by IC analysis of slurry diluted by a factor of 100 (nominally 1 g of slurry diluted to 100 mL) with water. Solids in the slurry were allowed to settle to minimize solids uptake during sampling prior to submitting for IC analysis. The oxalate by acid strike was determined by IC analysis of slurry diluted with acid. The purpose of the acid strike is to dissolve any insoluble oxalate so it can be measured.

Table 2-3. Anions and Base Equivalents of the Tank 40 Slurry

	Concentration in Slurry with 95% Confidence Interval (mg/kg)
Fluoride (mg/kg) [a]	235 ± 84
Formate (mg/kg) [a]	<1,000
Chloride (mg/kg) [a]	<200
Nitrite (mg/kg) [a]	15,500 ± 900
Nitrate (mg/kg) [a]	10,500 ± 600
Phosphate (mg/kg) [a]	<1,000
Sulfate (mg/kg) [a]	1,690 ± 240
Oxalate (mg/kg) [a]	1,030 ± 110
Oxalate (Acid Strike) (mg/kg) [a]	920 ± 220
Base Equivalents (Eq/L slurry) [b]	0.43

[a] Based on four replicates.
[b] Based on two replicates. Because there were only two replicates, a confidence interval was not calculated.

Coal content was determined by thermogravimetric analysis (TGA) of slurry solids with a particle size greater than 38 μm. Maximum coal content was determined based on the total weight loss of these solids during TGA analysis. The minimum coal content was based on the weight loss of these solids attributed to fixed carbon. Experimental results are presented in Table 2-4. Coal content of the Tank 40 sample with a comparison to the Tank 51 qualification sample is presented in Table 2-5.

Table 2-4. Experimental Results of the Thermogravimetric Analysis of Sieved Solids from the March 2004 Tank 40 Sample

Weight of Slurry Sample Sieved (g)	Weight of Solids Collected on Sieve (g)[a]	Total Weight % Loss Upon TGA Analysis	Weight % Loss Due to Fixed Carbon Loss Upon TGA Analysis
31.154	0.005	40.07	12.18
30.089	0.036	16.19	0.88
29.989	0.034	34.99	12.24

[a] Solids collected on 400 mesh (38 μm) sieve screen.

Table 2-5. Coal Content of the Tank 40 Sludge Batch 3 and Tank 51 Qualification Samples

	Tank 40 Sludge Batch 3 Sample	Tank 51 Qualification Sample
Maximum Coal (mg/kg) [a]	240 [c]	320
Minimum Coal (mg/kg) [b]	71 [c]	54

[a] Calculated according to the following:

$$\frac{\text{wt of solids collected on sieve}}{\text{wt of slurry sample}} \times \frac{\text{\% total wt loss}}{\text{ave \% wt loss of coal std}} \times 1{,}000{,}000$$, with the average % weight loss of the coal standard equal to 89.9.

[b] Calculated according to the following:

$$\frac{\text{wt of solids collected on sieve}}{\text{wt of slurry sample}} \times \frac{\text{\% total wt loss fr fixed C}}{\text{ave \% wt loss of coal std fr fixed C}} \times 1{,}000{,}000$$, with the average % weight loss of the coal standard from fixed carbon equal to 79.6. .

[c] A 95% confidence interval was not calculated for these analyses. Analyses were completed in triplicate with % relative standard deviations of 40% for the maximum coal determination and 70% for the minimum.

The measured Tank 40 coal concentration is similar to the predicted concentration (200 ppm) based upon the values obtained from the Tank 51 qualification sample multiplied by the ratio of sludge batch 2 to 3 blend now feeding the DWPF[1,2,3]. It is important to note, that if all 7000 lbs of coal from the K Area sand filters was backwashed into Tank 7, the approximate coal concentration in Tank 40 would be 1000 ppm based upon the mass of slurry presently in Tank 40. It is estimated that only a small portion of the coal was sent to Tank 7 [4].

Table 2-6 gives the elemental composition of the dried slurry solids. Composition was determined by an aqua regia dissolution of dried solids. Included in the table are elements present at greater than 0.5 wt% of the solids plus sulfur. Although silicon may be present at greater than 0.5%, it is not reported; aqua regia is not a suitable dissolution for silicon determination.

Table 2-6. Elemental Composition of Dried Solids

	Wt% of Total Solids with 95% Confidence Interval
Al	6.04 ± 0.23
Ca	1.66 ± 0.08
Fe	19.4 ± 1.7
Mg	1.67 ± 0.05
Mn	3.95 ± 0.11
Na	12.1 ± 0.8
Ni	1.09 ± 0.04
S	0.288 ± 0.015
U	6.99 ± 0.32

[a] Based on four aqua regia dissolutions of dried solids.

A comparison between supernate anion results (from Table 2-2) and the results from the water dilution of the slurry (from Table 2-3) is presented in Table 2-7. The supernate results were converted to a slurry basis using the supernate density, weight percent dissolved solids in the supernate, and weight percent total solids in the slurry:

$$C_i = \frac{M_i \cdot MW_i}{D_{supn}} \cdot \frac{(100 - W_{ts})}{(100 - W_{ds})} \cdot 1000$$

or, using the calculated soluble solids,

$$C_i = \frac{M_i \cdot MW_i}{D_{supn}} \cdot \frac{W_{ss}}{W_{ds}} \cdot 1000$$

where,

C_i is the concentration of i in the slurry (mg/kg)
M_i is the molarity of i in the supernate (mol/L)
MW_i is the molecular weight of i (g/mol)
W_{ts} is the weight percent total solids in the slurry
W_{ds} is the weight percent dissolved solids in the supernate
W_{ss} is the weight percent soluble solids in the slurry
D_{supn} is the density of the supernate (kg/L)
1000 is the conversion from g to mg.

Table 2-7. Comparison between Supernate Analysis and Water Dilution of Slurry

	Concentration Calculated from Supernate Analysis with 95% Confidence Interval (mg/kg)	Concentration from Water Dilution of Slurry with 95% Confidence Interval (mg/kg)
Fluoride	168 ± 9	235 ± 84
Formate	<80	<1,000
Chloride	<30	<200
Nitrite	15,700 ± 500	15,500 ± 900
Nitrate	9,620 ± 550	10,500 ± 600
Phosphate	<50	<1,000
Sulfate	1,700 ± 70	1,690 ± 240
Oxalate	1,140 ± 50	1,030 ± 110

As shown in Table 2-7, there is good agreement between the concentrations calculated from the supernate results and the concentrations from the slurry dilution within the 95% confidence limits. It should also be noted that the precision for the supernate results is generally better than the results from the slurry dilution. The conclusion from this comparison is that a supernate analysis and a slurry dilution give comparable results for these anions in the SB3 sample.

2.2 Comparison of Methods for Sulfate and Oxalate Determination

A comparison of the various methods for determining sulfate content in the slurry is given in Figure 2-1. Sulfate, on a slurry basis, was measured/calculated four ways:

- IC analysis of supernate – this method measures only soluble sulfate.
- IC analysis of water diluted slurry – this method measures soluble sulfate plus any insoluble sulfate dissolved in the dilution.
- ICP-ES analysis of supernate – this method measures soluble S; the result is converted to sulfate for comparison to IC results.
- ICP-ES analysis of dried solids – this method measures all the S in the slurry; the result is then converted to sulfate for comparison to IC results.

As can be seen from Figure 2-1, all of the methods yield a similar sulfate concentration in the slurry. This implies that, for this sample, all the sulfur is in the form of sulfate, and all the sulfate is soluble. These results are similar to earlier analyses of Tank 40 and Tank 51.[5] In these analyses it was shown that sulfur was soluble and in the form of sulfate for the Tank 40 and 51 samples.

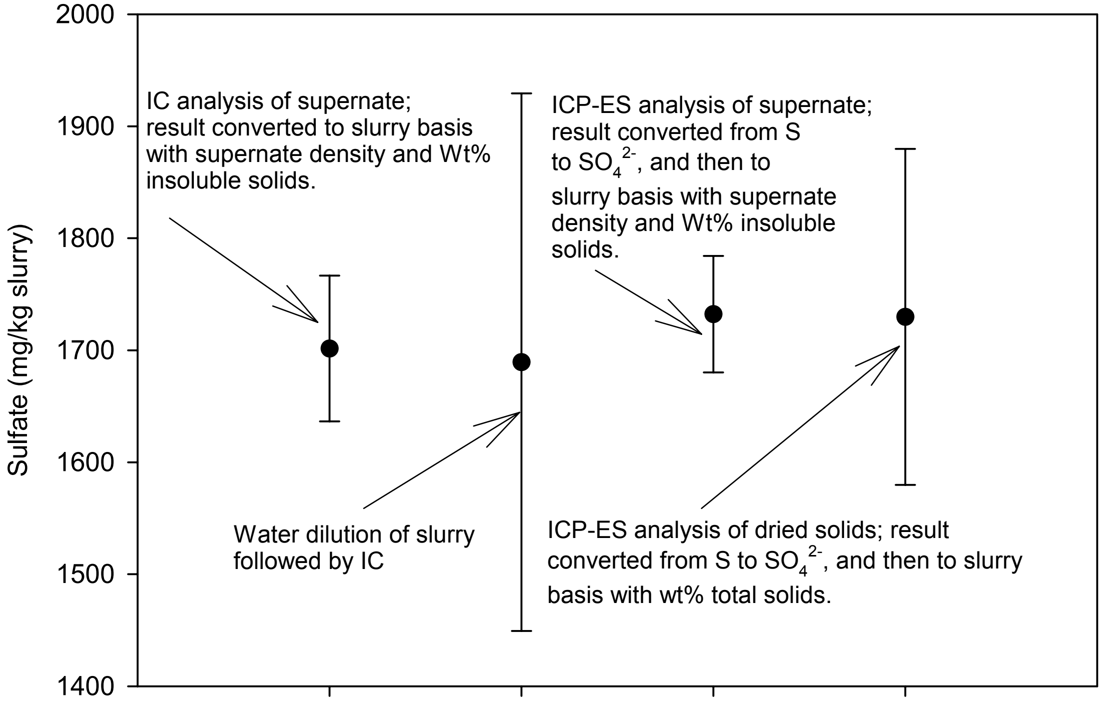

Figure 2-1. Graphical Comparison of the Average of Four Sulfate Determinations With 95% Confidence Intervals

A similar comparison of methods was completed for oxalate (see Figure 2-2). Three methods were compared:

- IC analysis of supernate – this method measures only the soluble oxalate.
- IC analysis of water diluted slurry – this method measures soluble oxalate plus any insoluble oxalate dissolved in the water dilution.
- IC analysis of acid strike of slurry – this method measures the soluble oxalate plus any additional oxalate dissolved by the acid in the slurry.

An examination of the results of the three methods shows that there is little, if any, insoluble oxalate in this sample. The acid strike method, designed to dissolve insoluble oxalate, actually gave a lower result than the other two methods. Note, however, that with the 95% confidence intervals taken into consideration, one cannot conclude that the acid strike result is significantly different than the other two methods. One can conclude that the acid strike method is less precise and is certainly not better than the other two methods for this SB3 sample. Therefore, it appears that the acid strike method is not necessary for oxalate quantification since no insoluble oxalate was identified.

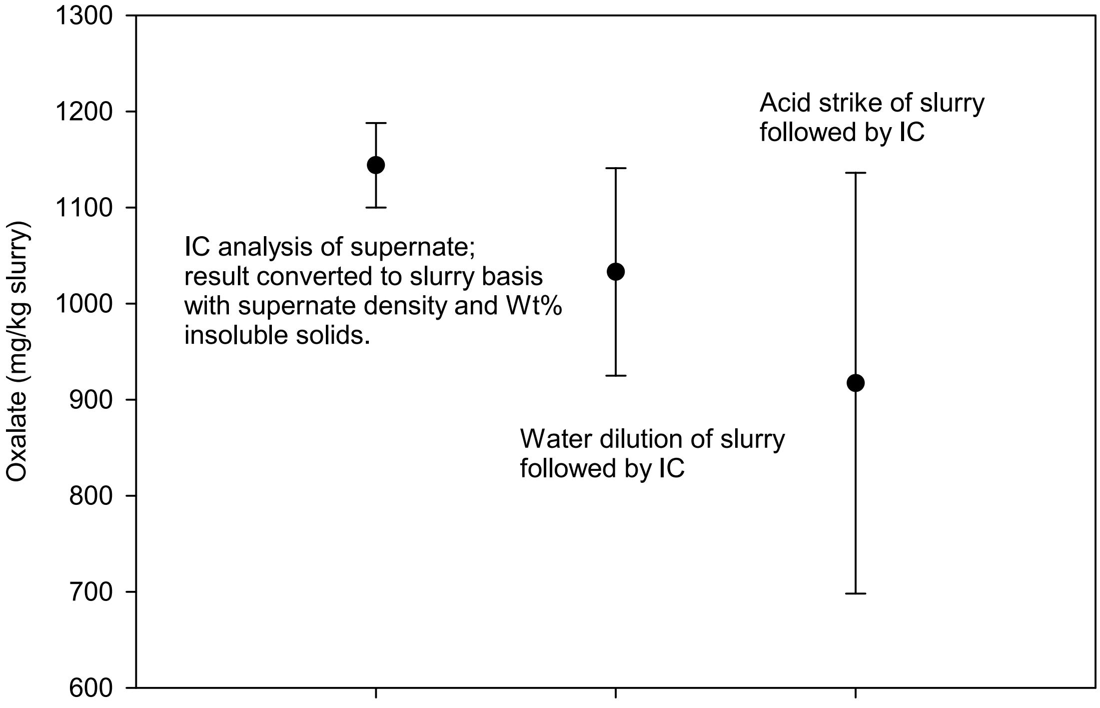

Figure 2-2. Graphical Comparison of the Average of Three Oxalate Determinations With 95% Confidence Intervals

2.3 Comparison of SB3 Sample Results to Tank Farm Predictions and DWPF SRAT Receipt Results

Table 2-8 shows a comparison between the SB3 sample supernate results and Tank Farm predictions. Except for sodium, results compare well, indicating that actual Tank Farm processing (transfers, etc.) occurred as planned. The sodium discrepancy is likely due to precipitation of sodium compounds. For the SB3 sample, if one converts the soluble sodium to a total solids basis, only 77% of the sodium is soluble.

Table 2-8. Comparison of Sludge Batch 3 Supernate Results to Tank Farm Predictions

	SB3 Supernate Results [a]	Tank Farm Prediction [b]
Nitrite (M)	0.42	0.44
Nitrate (M)	0.19	0.20
Sulfate (M)	0.022	0.023
Oxalate (M)	0.016	0.017
Sodium (M)	1.00	1.13

[a] See Table 2-2.
[b] Values taken from a spreadsheet from J. M. Gillam of Tank Farm Engineering.

Table 2-9 shows a comparison between the SB3 sample and DWPF SRAT Receipt analyses for batch 275. In comparing total solids, calcined solids, oxalate, sulfate, iron, sodium, and uranium, it appears that DWPF is nearly transitioned to the new sludge batch. Nitrite, nitrate, and base equivalents cannot be compared because these results are affected by the SRAT heel, which is higher in formate and nitrate, and lover in nitrite than incoming sludge.

Table 2-9. Comparison of SB3 Sample Results to DWPF SRAT 275 Receipt Analyses

	SB3 Sample [a]	SRAT Receipt for Batch 275 [b]
Total Solids (wt%)	20.0	20.4
Calcined Solids (wt%)	17.4	16.8
Density (g/mL)	1.19	1.11
Nitrite (mg/kg)	15,500	10,500
Nitrate (mg/kg)	10,500	11,300
Oxalate (mg/kg)	1,030 [a]	1,050 [a]
Sulfate (mg/kg)	1,690	1,520
Base Eq. (Eq/L) pH=7	0.43	0.155
Fe (wt% of solids)	19.4	17.4
Na (wt% of solids)	12.1	11.4
U (wt% of solids)	7.0	6.7

[a] Values from Table 2-1, Table 2-3, and Table 2-6. The oxalate value is from the water dilution of the slurry.
[b] These results were provided by R. N. Mahannah of DWPF Laboratories.

3.0 RHEOLOGY

3.1 Description of the Apparatus Used for the Rheological Measurements

All of the rheological measurements for the sludge slurry sample were obtained using the Haake RV30/M5 system located in Cell 2 of the Shielded Cells Facility. The Haake RV30/M5 system is a controlled shear rate rheometer that is operated remotely in the Shielded Cells environment. A water bath/circulator supplies water to maintain the temperature of the water jacket used to keep the sample at a specified temperature. The M5 measuring head can be equipped with different rotors, with rotor group having a specified measuring cup. The selection of the rotor/cup combination depends on the sample to be analyzed. The specifications for the instrument can be found in a previous publication[6]. A National Institute of Standards and Technology (NIST) traceable Newtonian oil standard (~14 cp @ 25°C) was used to verify the functionality/operability of the RV30/M5 system prior to the start and at the completion of a set of samples. All measurements for the Newtonian oil standard were within ±10% of the standards viscosity. The MVI rotor and MV cup was used in all of the measurements obtained. Specifications for the MVI rotor and cup have been published previously[6].

The same programming times and shear rate ranges were used for the oil standard and sludge slurry sample. Table 3-1 contains the programming times and shear rate ranges for the sludge slurry samples and the oil standard. Raw data for the flow curves are located in notebook WSRC-NB-2000-00056.

Table 3-1. Programming Times and Shear Rate Ranges Selected for the Sludge Slurry and Standard Oil Samples

	Sludge Slurry		Standard Oil	
	Shear Rate Range (s^{-1})	Time (minutes)	Shear Rate Range (s^{-1})	Time (minutes)
Up Curve	0 – 800	5	0 – 800	5
Hold	800	1	800	1
Down Curve	800 – 0	5	800 – 0	5

3.2 March 2004 SB3 Sludge Slurry Observations and Rheology Measurements

After the dip samples had been combined and mixed, a sample (~125mL) was removed for rheology measurements. The sludge slurry sample was mixed and poured into the measuring cup. While pouring the samples into the measuring cup, a visual observation of the sample was noted. The sludge slurry sample appeared to be dark brown and very fluid. No clumps were observed while pouring the sample into the measuring cup. Once the sample was placed into the measuring cup, the in-cell camera was used to look inside the measuring cup. Several air bubbles appeared to be readily popping at the surface of the sample. The majority of the air bubbles were small (less than ~1/16 inches in diameter). The measuring cup was loaded into the instrument and the measurement was successfully completed. All subsequent measurements were performed in the same fashion and were also successfully completed.

The raw data from the rheometer (up flow curve only) for the sludge slurry is plotted in Figure 3-1. The plotted data, specifically the shear rate, is that of a Newtonian fluid and has not been corrected. The complete data set in Figure 3-1 was curve fitted using the Bingham Plastic model and was plotted against the DWPF operating region along. The DWPF operating region was created by using the Bingham Plastic parameters (consistency and yield stress) provided in reference DPSTD-80-38-2[7] and the Bingham Plastic equation ($\tau_{upper}\left(Pa\right)=0.012\cdot\dot{\gamma}+10$) and lower ($\tau_{lower}\left(Pa\right)=0.004\cdot\dot{\gamma}+2.5$). The Bingham Plastic model is defined as:

$$\text{Tau} = \text{Tau}_o + \eta\text{D or } \{\ \tau_{BP}\left(\frac{dynes}{cm^2}\right)=\frac{\eta}{100}\cdot\dot{\gamma}+\tau_o \text{ or } \tau_{BP}\left(Pa\right)=\eta\cdot\dot{\gamma}+\tau_o\ \}$$

Where: Tau (τ) = Shear stress {Dynes/cm^2 or Pa}
Tau$_o$ (τ_o) = Shear stress at D = 0 s^{-1} {Dynes/cm^2 or Pa} or Yield Stress
η = Consistency {centipoise = cP or Pa-sec}
D ($\dot{\gamma}$) = shear rate {s^{-1}}

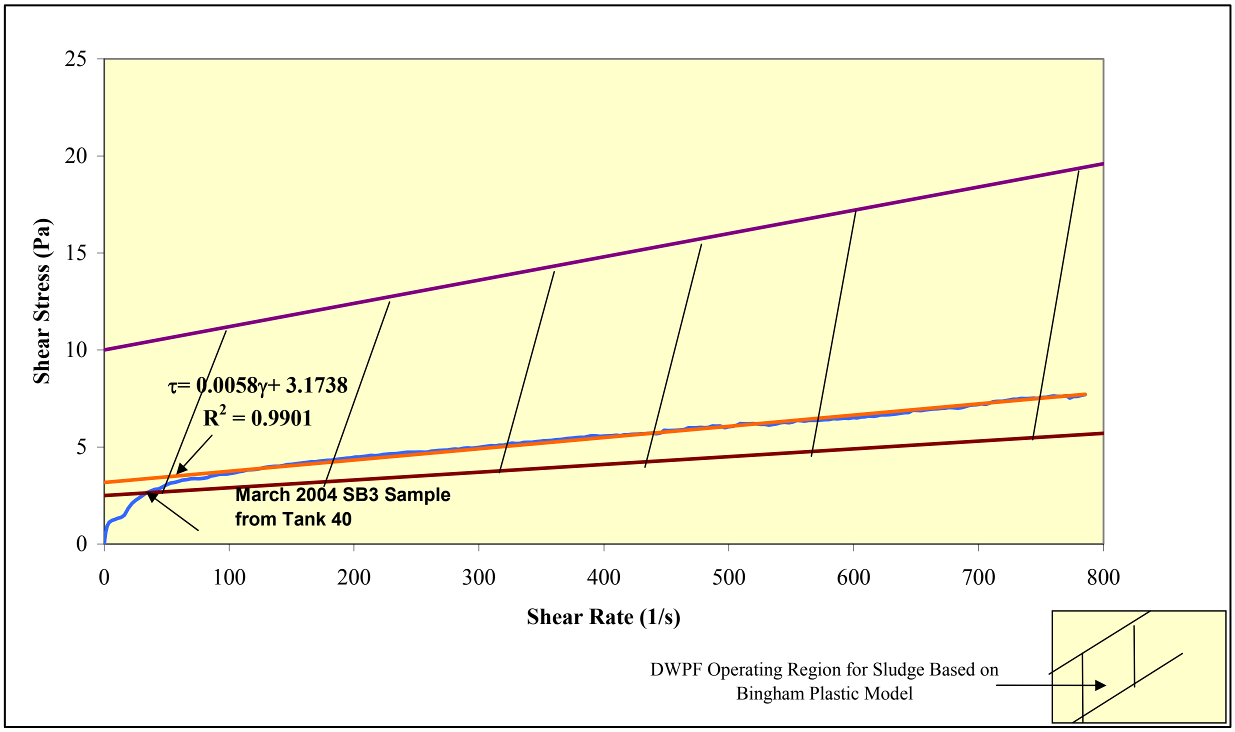

Figure 3-1. Uncorrected Flow Curve for the March 2004 SB3 Sludge Slurry Sample Compared to the DWPF Operating Region

As seen in Figure 3-1, the sample flow curve is within the DWPF operating region for the sludge slurry. The sample was fitted using the Bingham Plastic model from a shear rate range of 40 to 785 sec^{-1}. Table 3-2 contains the results of the sludge slurry samples, the SRTC SB3 qualification sample[8], and the DWPF operating region. The table includes weight percent solids, insoluble solids, yield stress, consistency, and pH. If a value is not available, “N/A” is used in the table.

Table 3-2. Summary of the Results Obtained from the SB3 Sludge Slurry Samples Compared to the SRTC SB3 Qualification Sample and the DWPF Operating Region

Sample ID	Total Solids (wt.%)	Insoluble Solids (wt.%)	Yield Stress (dynes/cm^2)	Consistency (cp)	pH
2004 SB3 Dip Samples	20.0	14.8	33	5.6	12.6
SRTC SB3 Qualification Sample	27.2	17.1	40.6	7.37	13.4
DWPF Operating Region	13 – 19	N/A	25 – 100	4 – 12	N/A

As can be seen in Table 3-2, the 2004 SB3 sample is within all of the DWPF operation parameters listed except for the total weight percent solids. The total weight percent solids value is higher due to the Canyon additions (Pu/Gd, Am/Cm, and Np) to Tank 40. DWPF Engineering evaluated these additions and has accepted the higher wt% total solids[9].

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4.0 CONCLUSIONS

- Coal content of the Tank 40 sample was similar to that predicted using analysis of the Tank 51 qualification sample.

- Most, if not all, the sulfur was soluble and in the form of sulfate. IC analysis of the water dilution of the slurry is adequate for sulfate determination in the SB3 sample.

- Most, if not all, the oxalate was soluble. IC analysis of the water dilution of the slurry is adequate and the acid strike method is not necessary for oxalate determination in SB3.

- The yield stress and the consistency for the March 2004 SB3 sample is within the DWPF Operating Region. The consistency was 5.6 cp and yield stress was 33 dynes/cm^2.

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