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DISCLAIMER

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CHARACTERIZATION OF A COMPOSITE OF SAMPLES HTF-E-03-162, 163, AND 164 FROM TANK 51H (U)

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1.0 INTRODUCTION

Three dip samples of suspended sludge slurry were obtained from Tank 51H in December, 2003 after addition of corrosion control chemicals to the tank. The samples were sent to the Savannah River Technology Center (SRTC) for analysis. The analyses requested included those required for Extended Sludge Processing (ESP) washing controls and corrosion controls.[1] The washing controls require the measurement of the sodium concentration, density, and weight percent solids of the sample. The corrosion control analyses require determination of the nitrate, nitrite, and free hydroxide concentrations in the supernate.

2.0 SAMPLE RECEIVING, COMPOSITING, AND ANALYSIS

The three dip samples of suspended sludge from Tank 51H were labeled HTF-E-03-162, HTF-E-03-163, and HTF-E-03-164. The samples numbers were entered in the SRTC Shielded Cells logbook as required by sample tracking procedures. The samples were composited into a single vessel. After the solids had settled in the composite sample, a portion of the supernate was decanted and used to rinse any remaining solids from the dip sample vials into the compositing vessel. The total volume of the composite sample was ~250 mL.

A portion of the composite slurry sample was filtered through a 0.45 μ nylon disposable filter to obtain the clear supernate phase. The density of the filtered supernate and the well mixed slurry sample were measured in the Shielded Cells using calibrated vials with a nominal volume of ~8.25 mL. The density measurements were conducted in triplicate on each phase of the sample.

The weight percent total solids in the slurry sample were measured in the Shielded Cells using a conventional drying oven at 100 °C. The sample was dried until repeated weights indicated no further loss of water. The weight percent dissolved solids in a sample of the filtered supernate were measured in the same manner. All weight percent solids measurements were made in triplicate. The weight percent insoluble solids and weight percent soluble solids in the slurry sample were calculated using the equations shown below.

$$Wis = (Wts - Wds)/(1-Wds) \quad \text{and} \quad Wss = Wts - Wis$$

where:

Wis = weight fraction of insoluble solids in the slurry
Wss = weight fraction of soluble solids in the slurry
Wts = weight fraction of total solids in the slurry
Wds = weight fraction of dissolved solids in the filtered supernate

Thus:

Wt% dissolved solids =	(wt dissolved solids/wt of supernate) x 100
Wt% total solids =	(wt total solids/wt of total slurry) x 100
Wt% insoluble solids =	(wt insoluble solids/wt of total slurry) x 100
Wt% soluble solids =	(wt of dissolved solids/wt of total slurry) x 100

Aliquots of the filtered supernate were diluted with 0.5 M HNO3 and with de-ionized distilled water and sent to the Analytical Development Section (ADS) for analysis. A blank containing de-ionized water pipetted into the diluent was also submitted for each set of samples. Triplicate dilutions with each diluent were sent for analysis. The corrosion control species, nitrate and nitrite, were determined using Ion Chromatography (IC). The free hydroxide was determined using an automated titration method. Inductively Coupled Plasma-Emission Spectroscopy (ICP-ES) was used to determine the sodium concentration in the filtered supernate.

3.0 RESULTS AND DISCUSSION

Table 3-1 shows the results of the analysis of the composite sample from Tank 51H. The IC analysis for anion concentrations and free hydroxide were conducted on the water diluted sample. The results from acid diluted sample were used for the ICP-ES metal concentrations since an acid matrix generally provides better results for the method.

The low percent relative standard deviations for all analytical results indicate good analytical precision for the three replicates. The blanks submitted with the sample show no contamination of the samples from reagents used in the sample preparations. The anion/cation balance for the results show reasonable agreement with a difference of 14%. The addition of carbonate to the anion sum would reduce the difference in the anion/cation balance further, however, the carbonate concentration in the sample was not determined. The sulfate concentration measured in the supernate by IC and by ICP-ES (as sulfur) show a difference of less than 12% also. The results of the density and weight percent solids measurements appear consistent with the sodium concentration measured in the supernate.

For comparison, Table 3-2 shows the average results of the current sample versus the last dip sample obtained from Tank 51H in July 2003.[2] Since the previous sample was obtained, the tank was decanted and corrosion inhibitor's added so a direct comparison for all species cannot be made. For example, the sodium, nitrate, nitrite, and hydroxide concentrations would be expected to change as a result of the addition of corrosion inhibitor's. All of the weight percent solids results would also change as a result of the decanting and inhibitor additions to the tank.

Table 3-1. Composition of the Composite Sample (HTF-E-03-162, 163, and 164) from Tank 51H

Analyte	1st Replicate (M)	2nd Replicate (M)	3rd Replicate (M)	Average (M)	%RSD	Blank (M)
NO_3^- (IC)	3.18E-01	3.00E-01	3.13E-01	3.10E-01	3.0%	<1.6E-03
NO_2^- (IC)	5.25E-01	4.96E-01	5.19E-01	5.13E-01	3.0%	<2.2E-03
PO_4^{3-} (IC)	<1.1E-03	<1.1E-02	<1.1E-03	<4.3E-03	-	<1.1E-03
SO_4^{2-} (IC)	2.44E-02	2.26E-02	2.36E-02	2.35E-02	3.9%	<5.3E-04
$C_2O_4^{2-}$ (IC)	3.28E-02	2.96E-02	3.15E-02	3.13E-02	5.1%	<1.1E-03
Cl^- (IC)	1.15E-03	<5.7E-03	1.15E-03	1.15E-03*	0.0%	<5.7E-04
F^- (IC)	1.72E-02	1.49E-02	1.66E-02	1.62E-02	7.4%	<1.1E-03
OH^-_{free} (T)	2.62E-01	2.80E-01	2.69E-01	2.70E-01	3.3%	<1.2E-06
Na (ICP-ES)	1.47E+00	1.48E+00	1.52E+00	1.49E+00	1.8%	<1.0E-05
K (ICP-ES)	4.65E-03	4.48E-03	4.06E-03	4.39E-03	6.9%	<2.5E-04
Al (ICP-ES)	4.38E-02	4.35E-02	4.43E-02	4.39E-02	0.9%	<5.0E-05
P (ICP-ES)	1.72E-04	2.04E-04	<1.5E-04	1.88E-04*	12%	<1.5E-04
S (ICP-ES)	2.65E-02	2.61E-02	2.63E-02	2.63E-02	0.8%	<4.4E-05
$Density_{supernate}$ (g/mL)	1.08	1.10	1.07	1.08	1.2%	-
$Density_{slurry}$ (g/mL)	1.21	1.21	1.22	1.21	0.1%	-
Wt% Total Solids	26.0%	25.9%	25.9%	25.9%	0.3%	-
Wt% Dissolved Solids	6.59%	6.64%	6.38%	6.54%	2.1%	-
Wt% Insoluble Solids	20.8%	20.7%	20.8%	20.8%	0.4%	-
Wt% Soluble Solids	5.22%	5.27%	5.05%	5.18%	2.2%	-

* Average of two replicates
IC – Ion Chromatography method
T – Titration method
ICP-ES – Inductively-Coupled Plasma Emission Spectroscopy method

Table 3-2. Comparison of the Composition of the Current Composite Sample and the Previous Sample (HTF-E-03-76) from Tank 51H

Analyte	Current Sample Average (M)	Previous Sample Average (M)
NO_3^- (IC)	3.10E-01	1.95E-01
NO_2^- (IC)	5.13E-01	3.25E-01
PO_4^{3-} (IC)	<4.3E-03	3.0E-03
SO_4^{2-} (IC)	2.35E-02	2.01E-02
$C_2O_4^{2-}$ (IC)	3.13E-02	2.71E-02
Cl^- (IC)	1.15E-03*	1.1E-03
F^- (IC)	1.62E-02	1.6E-02
OH^-_{free} (T)	2.70E-01	2.61E-01
Na (ICP-ES)	1.49E+00	1.23E+00
K (ICP-ES)	4.39E-03	5.5E-03
Al (ICP-ES)	4.39E-02	-
P (ICP-ES)	1.88E-04*	-
S (ICP-ES)	2.63E-02	-
$Density_{supernate}$ (g/mL)	1.08	1.06
$Density_{slurry}$ (g/mL)	1.21	1.12
Wt% Total Solids	25.9%	15.7%
Wt% Dissolved Solids	6.54%	7.82%
Wt% Insoluble Solids	20.8%	8.53%
Wt% Soluble Solids	5.18%	7.15%

* Average of two replicates
IC – Ion Chromatography method
T – Titration method
ICP-ES – Inductively-Coupled Plasma Emission Spectroscopy method

4.0 REFERENCES

1. Davis, P., *Technical Task Request: Sludge Batch 3 Confirmation Sample Analysis*, HLE-TTR-2004-015, Rev. 0, September 15, 2003.

2. Swingle, R. F., *Results of Tank 51H Sludge Batch 3 Qualification Sample WAC and Corrosion Control Analyses (HTF-E-03-76)*, SRT-LWP-2003-00062, July 22, 2003.