

Key Words:
DWPF
CPC, sludge

Retention:
Permanent

**DWPF SB6 INITIAL CPC FLOWSHEET TESTING
SB6-1 TO SB6-6 4L TESTS OF SB6-A AND SB6-B SIMULANTS**

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JULY 2009

Savannah River National Laboratory
Savannah River Nuclear Solutions
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Contract Number DE-AC09-08SR22470**



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LIST OF ACRONYMS

Aiken County Technology Laboratory	ACTL
Analytical Development	AD
Chemical Process Cell	CPC
Continuous Stirred Tank Reactor	CSTR
Defense Waste Processing Facility	DWPF
Formic Acid Vent Condenser	FAVC
Mercury Water Wash Tank	MWWT
Process Science Analytical Laboratory	PSAL
Reduction/Oxidation Potential	REDOX
Sludge Batch 5	SB5
Sludge Batch 6	SB6
Sludge Receipt and Adjustment Tank	SRAT
Slurry Mix Evaporator Condensate Tank	SMECT
Slurry Mix Evaporator	SME
Task Technical and Quality Assurance Plan	TT&QAP
Technical Task Request	TTR

1.0 EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) will transition from Sludge Batch 5 (SB5) processing to Sludge Batch 6 (SB6) processing late in fiscal year 2010. Tests were conducted using non-radioactive simulants of the expected SB6 composition to determine the impact of varying the acid stoichiometry during the Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) processes. The work was conducted to meet the Technical Task Request (TTR) HLW/DWPF/TTR-2008-0043¹, Rev.0 and followed the guidelines of a Task Technical and Quality Assurance Plan² (TT&QAP).

The flowsheet studies are performed to evaluate the potential chemical processing issues, hydrogen generation rates, and process slurry rheological properties as a function of acid stoichiometry. These studies were conducted with the estimated SB6 composition at the time of the study. This composition assumed a blend of 101,085 kg of Tank 4 insoluble solids and 179,000 kg of Tank 12 insoluble solids. For Tank 12, the assumption was that aluminum dissolution would be performed in Tank 51 to dissolve 75% of the aluminum.³

Six DWPF process simulations were completed in 4-L laboratory-scale equipment using two projections of the SB6 blend simulant composition⁴ (Tank 40 composition after Tank 51 transfer on a 40" Tank 40 heel is complete). The more washed simulant (SB6-A washed to nominally 1M Na) had a set of four SRAT and SME simulations at varying acid stoichiometry levels (90%, 100%, 120% and 150%). Two additional SRAT simulations were made using SB6-B blend simulant (nominally 1.2 M Na) at 100% and 120% of acid stoichiometry. Acid predictions used the Koopman Acid Prediction Calculation, which was approximately 3% higher than the Hsu equation.⁵

Two SB6 processing issues were noted during testing. First, the highest hydrogen generation rate exceeded the DWPF SME hydrogen processing limit of 0.223 lb/hr in Run SB6-4, the highest acid stoichiometry (150%) experiment. Also, in the lower acid runs (90% and 100%), the SRAT product mercury concentration exceeded the 0.45 wt % Hg in the total solids DWPF SRAT limit after 12 hours of total boiling time.

Processing of SB6B was very similar to SB6-A, but acid requirements were higher due to the higher concentrations of hydroxide, nitrite, and carbonate.

The yield stress of the SRAT and SME products produced during the testing was very low, and some products were below the DWPF process limits. It should be noted that simulants have been indicated lower yield stress than actual waste during past runs and it is not known how the SB6 simulants compare to the real waste. Yield stress can be increased by targeting high solids content, therefore no processing issues are expected from the low yield stresses noted during the testing.

A black film was formed on the agitator shaft and impellers during SB6-4. This run had the highest acid stoichiometry tested and exceeded the hydrogen limits during SME processing. The film was resistant to rinsing, was not removed by soaking in nitric acid and required

mechanical cleaning to remove. The material deposited on the shaft contained mercury and likely contained noble metals, but speciation was not performed.

The following information was requested as part of the TTR.

1. Hydrogen and nitrous oxide generation rates as a function of acid stoichiometry

Hydrogen generation was significantly impacted by the changes in acid stoichiometry from 90% to 150% (1.96 to 2.73 moles acid per liter of SB6-A sludge or 1.28 to 1.79 moles acid per liter of SB6-B sludge). For the SB6-A sludge, the hydrogen generation rate exceeded the process limit during the SME cycle at the highest acid stoichiometry (150%). The nitrous oxide generation peak was relatively insensitive to acid stoichiometry and was relatively low due to the low starting nitrite concentration.

2. Acid quantities and processing times required for mercury removal

Mercury was added to the sludge simulant at the start of the SRAT cycle as mercuric oxide at 1.5 wt% (total solids basis) based on the expected composition of the SB6 blend. Boiling flux was maintained at a scaled rate of 5,000 lb/hr for a total of 12 hours, so a total of 60,000 lb of steam flow in DWPF would be needed to remove this same 120 lb of mercury. Acid quantities from 120% to 150% resulted in satisfactory mercury removal (product mercury below the 0.45 wt % SRAT limit) with 12 hours of boiling time. However, the lower acid stoichiometry runs (90% and 100% acid stoichiometry) with both the SB6-A and SB6-B simulants resulted in unsatisfactory mercury removal with 12 hours of boiling. If DWPF experiences problems stripping mercury, increasing the acid stoichiometry or boiling time is likely to improve mercury removal but may increase hydrogen generation. Longer boiling times will be used in future SB6 testing to ensure the mercury concentration is below the SRAT limit.

3. Acid quantities and processing times required for nitrite destruction

Acid quantities from 100% to 150% resulted in satisfactory nitrite destruction with 12 hours of boiling. In all but the 90% run, the amount of nitrite present in the SRAT product was less than the 1,000 mg/kg limit. The low starting nitrite concentration helped to reduce the nitrite by the end of the SRAT cycle. Both runs at 100% stoichiometry met the nitrite limit, but contained some residual nitrite.

2.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) will transition from Sludge Batch 5 (SB5) processing to Sludge Batch 6 (SB6) processing in late fiscal year 2010. Tests were conducted using non-radioactive simulants of the expected SB6 composition to determine the impact of varying the acid stoichiometry during the Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) processes. The work was conducted to meet the Technical Task Request (TTR) HLW/DWPF/TTR-2008-0043⁶, Rev.0 and followed the guidelines of a Task Technical and Quality Assurance Plan (TT&QAP).

The flowsheet studies are performed to evaluate the potential chemical processing issues, hydrogen generation rates, and process slurry rheological properties as a function of acid stoichiometry. These studies were conducted with the estimated SB6 composition at the time of the study. This composition assumed a blend of 101,085 kg of Tank 4 insoluble solids and 179,000 kg of Tank 12 insoluble solids. The current plans are to subject Tank 12 sludge to aluminum dissolution. Liquid Waste Operations assumed that 75% of the aluminum would be dissolved during this process. After dissolution and blending of Tank 4 sludge slurry, plans included washing the contents of Tank 51 to ~1M Na. After the completion of washing, the plan assumes that 40% on Tank 40 slurry would remain for blending with the qualified SB6 material.

There are several parameters that are noteworthy concerning SB6 sludge:

- This is the second batch DWPF will be processing that contains sludge that has had a significant fraction of aluminum removed through aluminum dissolution.
- The sludge is high in mercury, but the projected concentration is lower than SB5.
- The sludge is high in noble metals, but the projected concentrations are lower than SB5
- The sludge is high in U and Pu – components that are not added in sludge simulants.

Six DWPF process simulations were completed in 4-L laboratory-scale equipment using two projections of the SB6 blend simulant composition (Tank 40 simulant after Tank 51 transfer is complete). The more washed simulant (SB6-A) had a set of four SRAT and SME simulations at varying acid stoichiometry levels (90%, 100%, 120% and 150%) using the Koopman Acid Prediction Calculation. Two additional SRAT simulations were made using SB6-B blend simulant at 100% and 120% of acid stoichiometry. SME cycles were noted performed for the SB6B simulants to allow the SRAT products to be used for melt rate testing.

3.0 DISCUSSION

Four SRAT/SME runs (SB6-1,2,3, and 4) were completed during this study using acid stoichiometries of 90%, 100%, 120%, and 150% with the Tank 40 blend simulant (SB6-A). Two SRAT runs (SB6-5, 6) were completed with the Tank 40 blend simulant (SB6-B) based on Variation 8, with one less wash/decant. These runs were completed and samples analyzed using the practices and procedures typical for Chemical Process Cell (CPC) simulations at the Aiken County Technology Laboratory (ACTL), as described below.

3.1.1 Simulant Preparation

Two simulant batches were prepared, one simulating the best estimate of the SB6 Tank 40 composition (SB6-A baseline sludge simulant) and the other simulating one of the processing options with one less wash/decant (SB6-B sludge simulant, one less wash). The SB6-A baseline sludge simulant used targets specified by Jeff Gillam, and David Larsen. Since the insoluble solids in both simulants were very similar, the same insoluble solids basis was used to prepare both simulants. Compositions of the simulants are shown in Table 1⁷.

Table 1 SB6-A and SB6-B Final Slurry Targets

Component	SB6-A	SB6-B
Total Solids, wt %	17.31	19.61
Insoluble Solids, wt %	12.0	12.98
Al, calcined wt %	12.9	15.28
Fe, calcined wt %	16.2	18.6
Na, calcined wt %	18.6	13.9
Mn, calcined wt %	6.16	6.86
Ni, calcined wt %	3.52	3.92
Nitrite, mg/kg	14,200	14,000
Nitrate, mg/kg	9,800	8,830
Sulfate, mg/kg	1,540	1,000
Soluble TIC, mg/kg	525	490

The preparation of a simulant for Sludge Batch 6 involved six steps: precipitation of manganese (IV) oxide, caustic precipitation of a metal nitrate solution, addition of sodium carbonate, washing of the precipitated solids, addition of minor insoluble species, and addition of soluble species. The precipitation of metal nitrates to form insoluble oxides and hydroxides was conducted in a Continuous Stirred Tank Reactor (CSTR) and involved generation of a metal nitrate solution followed by precipitation of the metal nitrates through the addition of sodium hydroxide. Following the addition of sodium carbonate, the material was washed then soluble/insoluble species were added. Procedure L29 ITS-00124⁸, “SRS HLW Sludge Simulant Preparation” was utilized to perform the tests.

The simulants were prepared using facilities at both ACTL and in 735-11A. The MnO₂ precipitation, the precipitation in the CSTR and the precipitation of the insoluble carbonate species were each completed in one day. The washing and concentration of the precipitate took approximately three weeks, while the final insoluble and soluble species were added in

one day. The final slurry was sampled and analyzed at ACTL, the Process Science Analytical Laboratory (PSAL), and by Analytical Development (AD). The results of these analyses are summarized in Table 2. As can be seen, the results agreed well with the planned targets summarized in Table 1. The SB6 simulants were very thin rheologically, especially because of the low insoluble solids targets

Table 2 Simulant Composition for SB6 Flowsheet Testing

Analyses	SB6-A	SB6-B	Analyses	SB6-A	SB6-B
Elemental	Wt% calcined solids		Solids Data	Wt %	
Al	15.2	14.8	Total Solids	17.73	19.93
Ba	0.216	0.216	Insoluble Solids	11.10	13.37
Ca	2.09	2.17	Calcined Solids	12.95	14.72
Cr	0.251	0.253	Soluble Solids	6.63	6.56
Cu	0.084	0.095	Anions	mg/kg	
Fe	18.3	18.3	Chloride	169	191
K	0.103	0.097	Nitrite	14,000	13,600
Mg	1.50	1.56	Nitrate	8,710	9,040
Mn	6.74	6.98	Formate	<100	<100
Na	14.4	15.2	Sulfate	1,190	1,150
Ni	3.85	4.02	Oxalate	<100	<100
P	<0.100	<0.100	Phosphate	<100	<100
Pb	0.013	0.027	Total Carbonate	11,900	13,000
S	0.309	0.308	Other Results		
Si	0.502	0.598	Base Equivalents (molar)	0.620	0.732
Ti	0.020	0.021	Slurry Density (g/ml)	1.14	1.165
Zn	0.155	0.155	pH	12.7	12.7
Zr	0.359	0.362	Soluble Total Inorganic C	2390	

Noble metals, mercury, and rinse water were added to the sludge simulant prior to performing the SRAT cycle. The noble metal concentrations were based on 100% of the estimated amount in the sludge batch. The concentrations of each trim chemical added are shown in Table 3. Noble Metal Concentrations were based on lanthanum concentration predicted by Ned Bibler using David Larsen's lanthanum estimate with typical fission yield.

Table 3. Trim Chemical Additions, wt % on Total Solids Basis

TRIM CHEMICAL	SB5	SB6
Trimmed Sludge Target Ag metal content	0.0137	0.0002
Trimmed Sludge Target wt% Hg dry basis	2.38	1.50
Trimmed Sludge Target Pd metal content	0.0036	0.0158
Trimmed Sludge Target Rh metal content	0.0227	0.0202
Trimmed Sludge Target Ru metal content	0.0980	0.0760

3.1.2 Experimental Apparatus

The testing was performed at the ACTL using the four-liter kettle setup. The SRAT rigs were assembled following the guidelines of SRNL-PSE-2006-00074⁹. The intent of the equipment is to functionally replicate the DWPF processing vessels. The 4-liter glass kettle is used to replicate both the SRAT and the SME, and it is connected to the SRAT Condenser, the Mercury Water Wash Tank (MWWT), and the Formic Acid Vent Condenser (FAVC). The Slurry Mix Evaporator Condensate Tank (SMECT) is represented by a sampling bottle that is used to remove condensate through the MWWT. For the purposes of this paper, the condensers and wash tank are referred to as the offgas components. A sketch of the experimental setup is given as Figure 1.

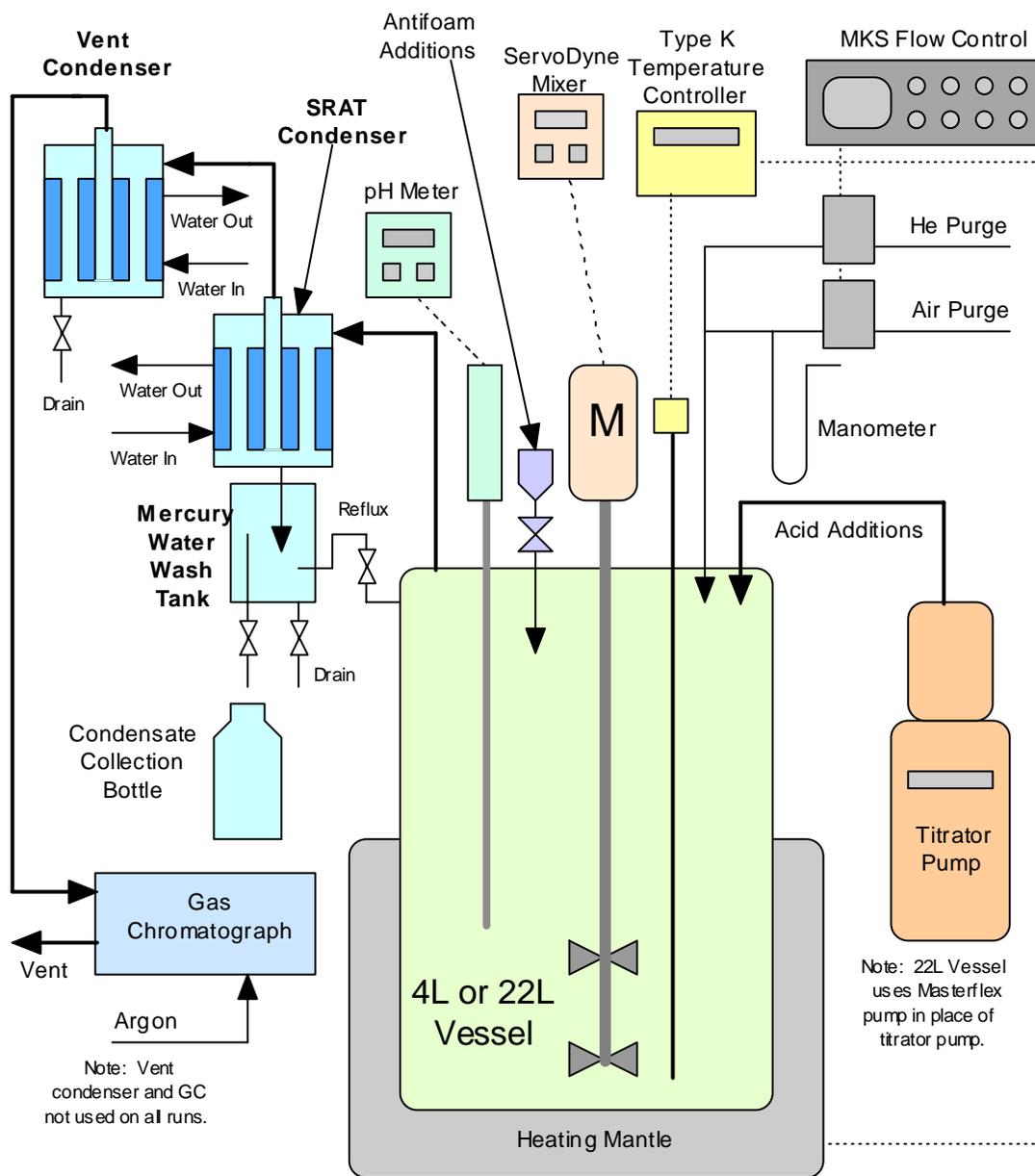


Figure 1. Schematic of SRAT Equipment Set-Up

The flowsheet runs were performed using the guidance of Procedure ITS-0094¹⁰ (“Laboratory Scale Chemical Process Cell Simulations”) of Manual L29. Offgas hydrogen, oxygen, nitrogen, nitrous oxide, and carbon dioxide concentrations were measured during the experiments using in-line instrumentation. Helium was introduced at a concentration of 0.5% of the total air purge as an inert tracer gas so that total amounts of generated gas and peak generation rates could be calculated. During the runs, the kettle was monitored to observe reactions that were occurring to include foaming, air entrainment, rheology changes,

loss of heat transfer capabilities, and offgas carryover. Observations were recorded on data sheets and pasted into a laboratory notebook¹¹.

Concentrated nitric acid (50-wt%) and formic acid (90-wt%) were used to acidify the sludge and perform neutralization and reduction reactions during processing. The amounts of acid to add for each run were determined using the proposed Koopman DWPF acid addition equation. The split of the acid was determined using the REDOX equation currently being used in DWPF processing¹². The REDOX target ($\text{Fe}^{2+}/\Sigma\text{Fe}$) was 0.2. To account for the reactions and anion destructions that occur during processing, assumptions about nitrite destruction, nitrite to nitrate conversion, and formate destruction were made for each run.

To prevent foaming during SRAT processing, 200 ppm IIT 747 antifoam was added before acid addition, 100 ppm was added after nitric acid addition was complete and 500 ppm was added at the completion of formic acid addition. SRAT processing included 12-hours at boiling (dewater time plus reflux time). The SME processing did not include the addition of canister dewaterers. The frit addition was split into two equal portions. The frit was added with water and formic acid at DWPF prototypical conditions. Concentration was performed after each frit addition and then the vessel was allowed to cool to approximately 90 degrees. An addition of ½ of the frit water was made to further cool the vessel, then the frit was added followed by the remaining water.. A final concentration was performed at the end of the run to meet the target total solids of 45 wt%. The SRAT condenser was maintained at 25° C during the run, while the vent condenser was maintained at 4° C.

3.2 SRAT CYCLE RESULTS

Four SRAT/SME runs (SB6-1, 2, 3, and 4) were completed during this study using acid stoichiometries of 90%, 100%, 120%, and 150% with the Tank 40 blend simulant (SB6-A) as shown in Table 4.. Two SRAT runs (SB6-5, 6) were completed with the Tank 40 blend simulant (SB6-B) using acid stoichiometries of 100% and 120% based on Liquid Waste's Variation 8, with one less wash/decant, as shown in Table 5. A unique run number was assigned to each run. All runs targeted a predicted glass REDOX of 0.2 by adjusting the ratio of formic to nitric acid during the SRAT cycle and using the current REDOX equation.

To prevent foaming during SRAT processing, 200 ppm IIT 747 antifoam was added before acid addition, 100 ppm was added after nitric acid addition was complete and 500 ppm was added at the completion of formic acid addition. SRAT processing included 12-hours* at boiling (dewater time plus reflux time). The boiling time calculation represents the minimum expected time that adequate mercury stripping could be expected and was used to allow differences in processing characteristics to be noted.

* The minimum boiling time to strip mercury is calculated by using the acid calc spreadsheet, assuming 500 lb steam is required to strip 1 lb elemental mercury with a 5000 lb/hr steam flowrate for a 6000 gallon sludge batch. This is based on shielded cells mercury stripping data.

Minimum boil time, min = (sludge solids mass * Hg wt%/100-SRAT product solids mass *.45/100)*500 g steam/g Hg/4.24 g steam/min = 505.3 g * 1.5 g Hg/100 g – 585.65 g *0.45 g Hg/100)*500 g Hg/500 g steam * 4.24 g steam /minute = 604 minutes or 10 hours and four minutes.

The SME processing did not include the addition of canister dewaterers. The frit addition was split into two equal portions. The frit was added with water and formic acid at DWPF prototypical conditions. Concentration was performed after each frit addition and then heat was removed to allow for the next frit addition. A final concentration was performed at the end of the run to meet the target total solids. The SRAT condenser was maintained at 25° C during the run, while the vent condenser was maintained at 4° C.

3.3 SRAT CYCLE RESULTS

Four SRAT/SME runs (SB6-1, 2, 3, and 4) were completed during this study using acid stoichiometries of 90%, 100%, 120%, and 150% with the Tank 40 blend simulant (SB6-A) as shown in Table 4. Two SRAT runs (SB6-5, 6) were completed with the Tank 40 blend simulant (SB6-B) using acid stoichiometries of 100% and 120% based on Liquid Waste's Variation 8, with one less wash/decant, as shown in Table 5. A unique run number was assigned to each run. All runs targeted a predicted glass REDOX ($Fe^{2+}/\Sigma Fe$) of 0.2 by adjusting the ratio of formic to nitric acid during the SRAT cycle and using the current REDOX equation.

Table 4 SB6-A Baseline Sludge Simulant SRAT/SME Tests

Run Number	Acid Stoichiometry	REDOX Target	Process Frit	Waste Loading
SB6-1	100%	0.2	418	38
SB6-2	90%	0.2	418	38
SB6-3	120%	0.2	418	38
SB6-4	150%	0.2	418	38

Table 5 SB6-B Sludge Simulant (One Less Wash) SRAT Tests

Run Number	Acid Stoichiometry	REDOX Target
SB6-5	100%	0.2
SB6-6	120%	0.2

All six experiments included a SRAT cycle, designed to simulate the chemical processing in the DWPF SRAT. The SRAT cycles were completed using conservative design basis inputs such as acid addition flowrates, air purges, steam flowrates, although these may be different than the typical flowrates used during DWPF SRAT processing.

3.3.1 Acid Addition Calculation

An acid calculation was completed prior to each experiment to estimate a number of scaled parameters necessary to complete each experiment at the conditions specified with the inputs such as kettle power (designed to simulate steam flow), acid addition flowrate, offgas purge, acid volume, etc. Results from the acid calculation and other run data are summarized in Appendix C.

3.3.1.1 Calculation Inputs

The SRAT cycle acid calculation utilizes the amount of nitrite, mercury, manganese, carbonate, and base equivalents to calculate the stoichiometric amount of acid to be added. Nitric acid and formic acid amounts are calculated¹³ based on the applied stoichiometric factor and the ratio needed to achieve the predicted glass REDOX target of $0.2 \text{ Fe}^{+2}/\Sigma\text{Fe}$. The equation for prediction of glass REDOX utilizes estimates of the amount of formate, oxalate, nitrate, nitrite, manganese, and total solids in the SME product. The estimation of the final concentration for the anions requires assumptions to be made concerning how these species will react during the SRAT and SME cycles. Formate and oxalate are destroyed by reactions with oxidizing species and by catalytic reactions with noble metals. Nitrite is typically consumed during acid additions, but can react to form different species including nitrate.

Three different acid addition predictions were used. The Hsu equation, an equation with inputs for total base, slurry carbonate, nitrite, manganese and mercury, has been used for estimating the acid requirement in DWPF since startup. Two new acid equations, which more accurately predict the acid requirement in DWPF, have been developed. The Koopman equation adds inputs for supernate (not slurry) carbonate, calcium and magnesium to better predict the acid requirement. The cation equation uses cations (manganese, sodium, potassium, mercury, cesium, strontium, calcium, nickel, and magnesium) to predict the acid demand with credits for anions (nitrite, nitrate, sulfate, chloride, formate and phosphate). Both of these new equations were developed for minimum acid (just enough acid to destroy nitrite with very little hydrogen generation) and nominal acid (enough acid to destroy nitrite, reduce mercury, and without making too much hydrogen). The minimum Koopman equation's prediction of acid requirement was used throughout the testing and the other results are summarized in Table 6. The acid calculation inputs and assumptions are shown in Table 8 and Table 9 for runs SB6-1 to SB6-6. The same assumptions and inputs were used for all runs, with the exception of the acid stoichiometry. Conversion of minimum Koopman stoichiometric factors into the equivalent Hsu equation stoichiometry is shown in

Table 7. Note that the conversion of SB6A and SB6B shiochiometry results in the same factors for these sludges, but this is not expected to be the same for all sludges.

Table 6 Acid Calculation Results at 100% Stoichiometry

Equation	SB6-A	SB6-B
Hsu Equation, M	1.56	1.77
Minimum Koopman, M	1.61	1.83
Nominal Koopman, M	1.96	2.24
Minimum Cation, M	1.29	1.62
Nominal Cation, M	1.56	1.95

Table 7. Conversion of Minimum Koopman Stoichiometry into Hsu Stoichiometry

Minimum Koopman Stoichiometric Factor	Hsu Equivalent Stoichiometry	Nominal Koopman Stoichiometry
90	93	74
100	103	82
120	124	99
150	155	123

Table 8 SRAT Cycle Processing Parameters and Assumptions

Description	Units	SB6-1,2,3,4	SB6-5,6
		SB6-A Baseline	SB6-B One Less Wash
Sludge			
Conversion of Nitrite to Nitrate in SRAT Cycle	gmol NO ₃ ⁻ /100 gmol NO ₂ ⁻	20.00	20.00
Destruction of Nitrite in SRAT and SME cycle	% of starting nitrite	100.00	100.00
Destruction of Formic acid charged in SRAT	%	20.00	20.00
Destruction of oxalate charged	%	100.00	100.00
Percent Acid in Excess Stoichiometric Ratio	%	100.00	100.00
SRAT Product Target Solids	%	25.00	25.00
Nitric Acid Molarity	Molar	10.534	10.534
Formic Acid Molarity	Molar	23.600	23.600
Scaled Nitric Acid addition Rate	gallons per minute	2.0	2.0
Scaled Formic Acid addition Rate	gallons per minute	2.0	2.0
REDOX Target	Fe ⁺² / ΣFe	0.200	0.200
Trimmed Sludge Target Ag metal content	total wt% dry basis	0.0002	0.0002
Trimmed Sludge Target wt% Hg dry basis	total wt% dry basis	1.5000	1.5000
Trimmed Sludge Target Pd metal content	total wt% dry basis	0.0158	0.0158
Trimmed Sludge Target Rh metal content	total wt% dry basis	0.0202	0.0202
Trimmed Sludge Target Ru metal content	total wt% dry basis	0.0760	0.0760
Water to dilute fresh sludge and/or rinse trim chemicals	g	50	50
Mass of SRAT cycle samples	g	250	250
Wt% Active Agent In Antifoam Solution	%	10	10
Basis Antifoam Addition for SRAT (generally 100 mg antifoam/kg slurry)	mg/kg slurry	100	100
Number of basis antifoam additions added during SRAT cycle		8	8

Table 9 SME Processing Parameters and Assumptions

Description	Units	SB6-1,2,3,4
Sludge		SB6-A Blend
Frit type		418
Destruction of Formic acid in SME	%	10.00
Destruction of Nitrate in SME	%	10.00
Assumed SME density	kg / L	1.450
Basis Antifoam Addition for SME cycle	mg/kg slurry	100
Number of basis antifoam additions added during SME cycle		5
Sludge Oxide Contribution in SME (Waste Loading)	%	38
Frit Slurry Formic Acid Ratio	g 90 wt% FA/100 g Frit	1.50
Target SME Solids total Wt%	wt%	45.0
Number of frit additions in SME Cycle		2

3.3.1.2 Acid Calculation Results

The acid calculation determines the values for a large number of processing parameters as well as the amount of formic and nitric acid to be used. Selected values are shown in Table 10 and Table 11. The stoichiometric acid addition for the sludge simulant was calculated to be 1.61 moles per liter for SB6-A and 1.83 moles per liter for SB6-B. The minimum stoichiometric acid requirement is based on a new acid addition equation developed by David Koopman. As acid stoichiometry increased, the ratio of formic acid to the total amount of acid decreased. This decrease is due to the presence of nitrate and nitrite in the initial sludge simulant lowering the amount of nitrate or oxidizers needed to balance the formic acid at lower acid stoichiometries. The frit addition increased slightly due to the process samples being more dilute in terms of the original feed as acid stoichiometry increased.

Table 10 Selected Process Values for Testing with SB6-A Baseline Sludge Simulant

Acid Stoichiometry	Total Acid Required (mol/L)	Formic Acid Ratio (% of Total Acid)	Frit Addition Amount (grams)
90%	1.45	88.4	537.7
100%	1.61	87.1	539.7
120%	1.93	85.2	543.3
150%	2.42	83.2	548.1

Table 11 Selected Process Values for Testing with SB6-B Sludge Simulant (One Less Wash)

Acid Stoichiometry	Total Acid Required (mol/L)	Formic Acid Ratio (% of TOTAL ACID)	Calculated Frit Addition Amount (grams)
100%	1.83	87.5	558.5
120%	2.20	85.5	549.1

3.3.2 Processing Observations

Overall processing during the testing went smoothly with no interruptions or upsets occurring during process runs. The sludge became less viscous during acid additions and no problems were noted with mixing during the runs. Agitator speeds of 250 RPM[†] were sufficient to mix the sludge simulants.

3.3.2.1 Foaming

No additional antifoam was required during any of the nine experiments. No foaming problems were noted during SRAT or SME processing.

3.3.2.2 pH Profiles

The pH profiles of the four SB6-A runs in general matched profiles noted during previous CPC simulations¹⁴. As shown in Figure 2, the pH was lower for runs with higher acid additions. Formic acid decomposition during high acid runs can result in lower pH at higher acid stoichiometries, but the decomposition noted during the flowsheet testing was not high enough to raise the pH of the higher acid runs above the lower acid runs in the SRAT cycle. All three runs with acid stoichiometries above 100% had a minimum pH near 4.0 at the end of acid addition. The pH profiles for runs with SB6B were very similar.

[†] The mixing geometry of the lab-scale apparatus is not prototypic of the DWPF SRAT/SME vessels and mixing was adjusted as required during testing to ensure that the process chemistry was captured. Agitator speed is reported only to give an indication of changes in rheological properties during the testing.

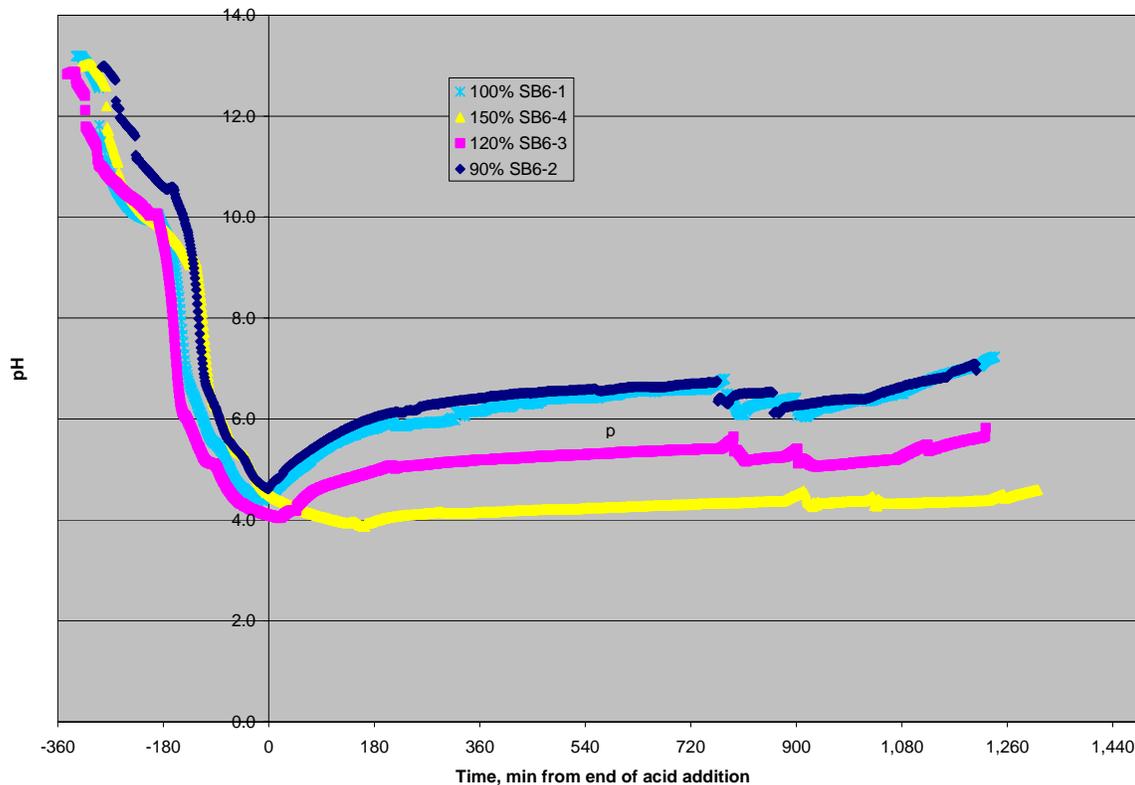


Figure 2. SB6 Flowsheet Testing pH Profiles

3.3.3 SRAT Cycle Sample Results

Samples were pulled at the conclusion of the SRAT cycle. The total solids, mercury, anions, and soluble elemental species were analyzed for all samples. Samples were taken of the SRAT dewater and the MWWT contents at the completion of the SRAT cycle. All sample results are tabulated in Appendix B.

3.3.3.1 Nitrite, Nitrate, Formate

Nitrite destruction met the process requirement of <math><1000\text{ mg/kg}</math> at the end of the SRAT cycle for all runs except the 90% stoichiometry run. For runs at 120% stoichiometry (100% nominal Koopman) or higher, there was no detectable nitrite at the end of the SRAT cycle. Note that the total time at boiling was 12 hours for each of these experiments. Anion results are summarized in Table 12 and Table 13.

Table 12 SRAT Product Anion Concentration from Tests with SB6-A Baseline Sludge Simulant, mg/kg

ACID STOICHIOMETRY	Sample #09-	F	Cl	NO ₂	NO ₃	SO ₄	PO ₄	HCO ₂
90% SB6-2	SB6-2-2711	<100	360	1960	24,600	<100	<100	52,600
100% SB6-1	SB6-1-2677	<100	393	200	27,000	<100	<100	54,600
120% SB6-3	SB6-3-2719	<100	349	<100	31,900	801	<100	57,600
150% SB6-4	SB6-4-2694	<100	335	<100	37,700	256	<100	67,000

Table 13 SRAT Product Anion Concentration from Tests with SB6-B Sludge Simulant (One Less Wash), mg/kg

ACID STOICHIOMETRY	Sample #09-	F	Cl	NO ₂	NO ₃	SO ₄	PO ₄	HCO ₂
100%	SB6-5-2748	<100	362	518	24,200	<100	<100	52,400
120%	SB6-6-2765	<100	331	<100	29,300	<100	<100	56,900

In a “typical run”, approximately one-third of the nitrite is converted to nitrate and the other two-thirds are converted to NO_x and N₂O. In all of these runs (Table 14 and Table 15), some additional nitrate was present in the SRAT product due to the destruction of nitrite.

Formate is destroyed by reduction of Mn, Hg and catalytic destruction of nitrite ion to primarily produce NO, N₂O, NO₂, and CO₂. Formic acid is destroyed catalytically to produce primarily CO₂, and hydrogen. An overall trend of higher formate loss with higher acid stoichiometry is indicated, which matches previous results and the amount of formate loss is consistent with previous testing.

Table 14 SRAT Anion Conversions from Tests with SB6-A Baseline Sludge Simulant

ACID STOICHIOMETRY	SRAT CYCLE		
	Formate Destruction	Nitrite to Nitrate Conversion	Nitrite Destruction
SB6-2 90%	16.7%	10.5%	88.8%
SB6-1 100%	18.3%	11.9%	98.8%
SB6-3 120%	22.3%	18.7%	100%
SB6-4 150%	19.8%	26.1%	100%

Table 15 SRAT Product Anion Conversions from Tests with SB6-B Sludge Simulant (One Less Wash)

ACID STOICHIOMETRY	SRAT CYCLE		
	Formate Destruction	Nitrite to Nitrate Conversion	Nitrite Destruction
SB6-5 100%	22.4%	5.4%	96.5%
SB6-6 120%	24.4%	12.6%	100%

3.3.3.2 Mercury

The SRAT product samples were analyzed for mercury content to evaluate the stripping of mercury during the SRAT cycle. The SRAT product must be below 0.45 wt% (solids basis) mercury to meet process specifications. Sludge batches 1A, 2 and 3 met this requirement without mercury removal, but SB6 is estimated to contain approximately 1.5 wt% mercury in the incoming blended feed solids. The mercury concentration in the six hour sample and in the SRAT product sample (12-hour of boiling) is summarized in Table 15 and Table 16.

Table 16 SRAT Product Mercury Results from Tests with SB6-A Baseline Sludge Simulant

Acid Stoichiometry	SRAT 6-hour Mercury, Wt % Total Solids Basis	SRAT Product Mercury, Wt % Total Solids Basis
90% (SB6-2)	0.971	0.855
100% (SB6-1)	0.729	0.525
120% (SB6-3)	1.387	0.094
150% (SB6-4)	0.136	0.064

Table 17 SRAT Product Mercury Results from Tests with SB6-B Sludge Simulant (One Less Wash)

Acid Stoichiometry	SRAT 6-hour Mercury, Wt % Total Solids Basis	SRAT Product Mercury, Wt % Total Solids Basis
100%	0.734	0.714
120%	0.347	0.518

The four runs with the lowest acid additions exceeded the DWPF mercury limit, although the 120% run with SB6B had a process sample that met the limit. Since it is not known which result is more valid, it was assumed that the higher result from the process sample was representative. If DWPF has problems achieving the mercury limit, a higher acid stoichiometry or longer stripping time may improve mercury removal. However, higher acid stoichiometry may also lead to higher hydrogen generation. Note also that the simulant testing was completed without a heel.

3.3.4 SRAT Cycle Offgas Composition Results

A typical offgas concentration profile is shown in Figure 3, while charts from all runs are shown in Appendix A. Helium and nitrogen show reduced concentrations during periods with large quantities of offgas generation due to dilution, while oxygen showed reduced concentrations during these periods due to dilution and from consumption. In general, hydrogen generation began after nitrous oxide emissions had ceased and carbon dioxide emission was noted in conjunction with the hydrogen. The patterns of offgas emissions noted during the runs were typical of offgas generation during the SRAT cycle.

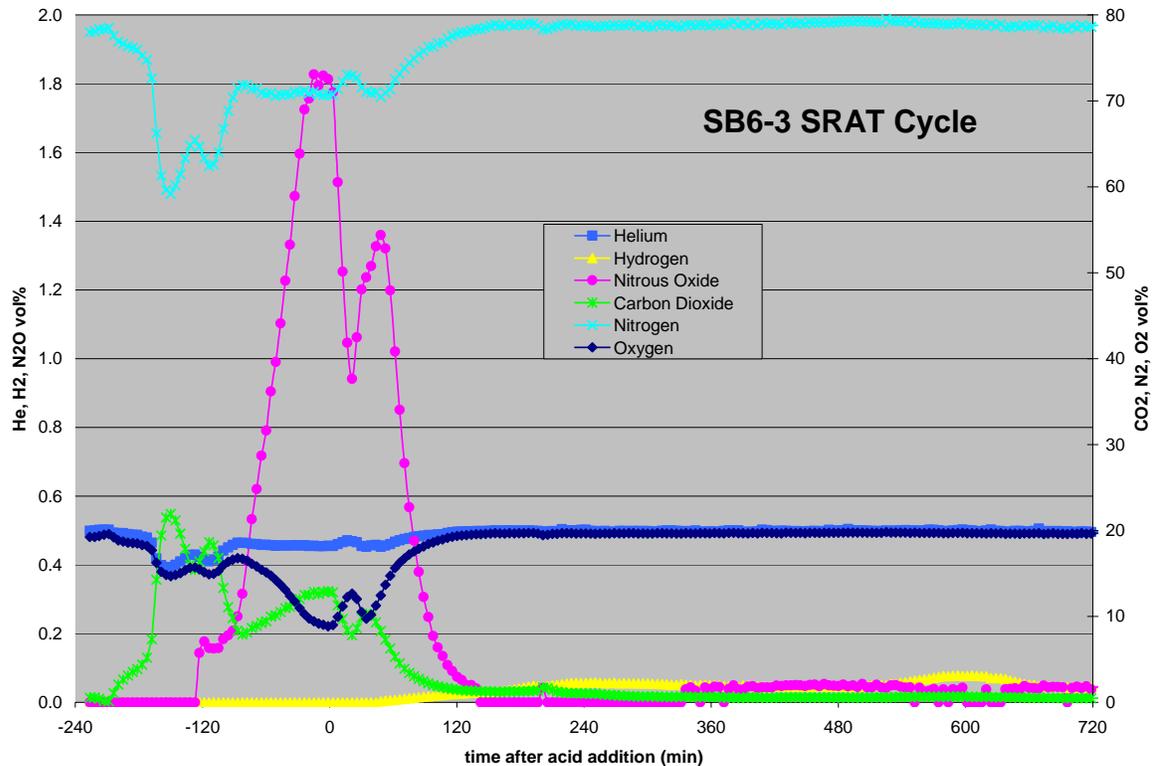


Figure 3. Typical SRAT Offgas Profile 120% Acid Stoichiometry, SB6-A Baseline Sludge Simulant

3.3.4.1 SRAT Hydrogen Evolution

The peak hydrogen concentration for each SRAT run is shown in Figure 4. In general, the peak hydrogen generation rate increased with increased acid addition. None of the rates exceeded the DWPF SRAT processing limits of 0.65 lb/hr, as shown in Table 18, which shows the peak hydrogen generation after scaling to the DWPF process.

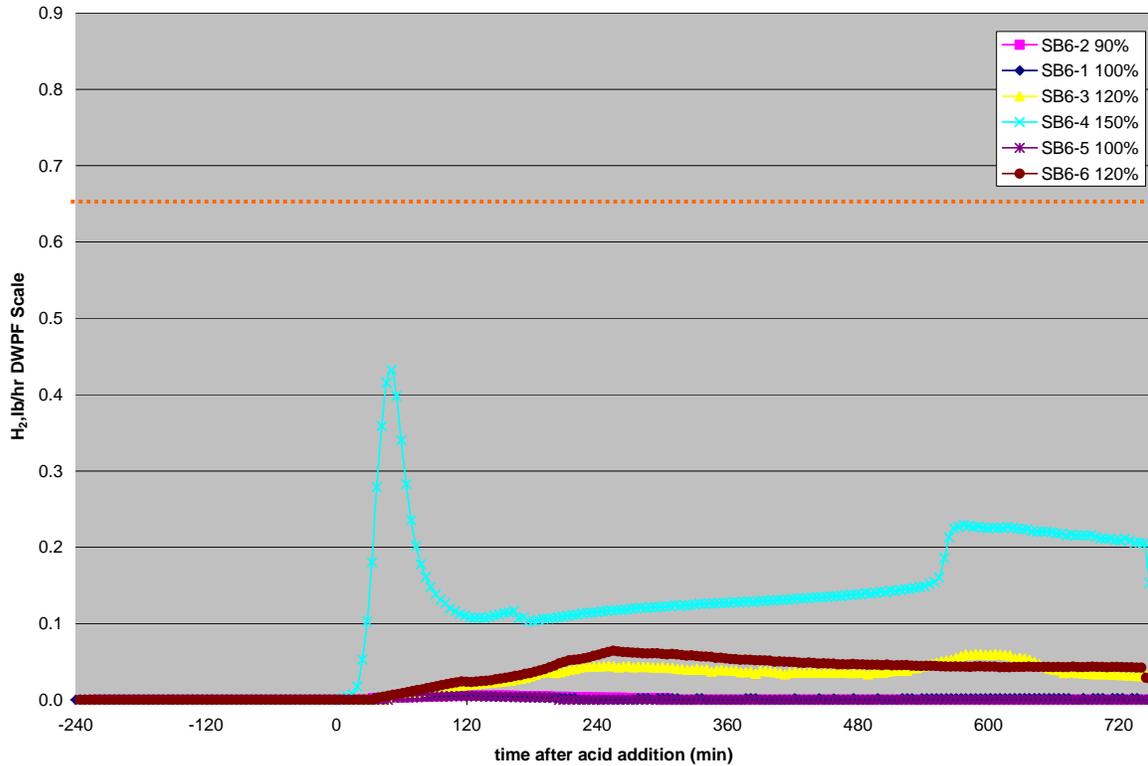


Figure 4. SRAT Cycle Hydrogen Peaks

Table 18 SRAT Cycle Hydrogen Peak Generation Rate

		Acid Stoichiometry			
SRAT Hydrogen Peak	Units	90%	100%	120%	150%
SB6-A Simulant	lb/hr	0.006	0.006	0.060	0.432
SB6-B Simulant	lb/hr		0.0050	0.064	

3.3.4.2 Other Species

The nitrous oxide peak concentrations slightly increased as acid addition was increased, while the carbon dioxide peak was very similar for all runs. The peak generation of these species is less dependent on acid concentration than hydrogen since more acid is added than needed to destroy carbonate and nitrite, the compounds that are responsible for the highest emissions. The peak generation rates are shown in Table 19 and Table 20 after scaling to the DWPF process scale.

Table 19 SRAT Cycle Nitrous Oxide and Carbon Dioxide Peak Generation Rates from Tests with SB6-A Baseline Sludge Simulant

		Acid Stoichiometry			
		90%	120%	120%	150%
SRAT Nitrous Oxide Peak	lb/hr	20.9	29.1	33.8	35.8
SRAT Carbon Dioxide Peak	lb/hr	547	539	469	470

Table 20 SRAT Cycle Nitrous Oxide and Carbon Dioxide Peak Generation Rates from Tests with SB6-B Sludge Simulant (One Less Wash)

		Acid Stoichiometry	
		100%	120%
SRAT Nitrous Oxide Peak	lb/hr	22.1	31.5
SRAT Carbon Dioxide Peak	lb/hr	545	493

3.3.5 SRAT Product Rheological Properties

The rheological properties of SRAT products were measured for the four runs produced with the simulant (SB6-A). The rheological properties were outside the processing limits for yield stress and consistency for SRAT products (yield stress 1.5 to 5 Pa and Consistency 5 to 12 cP)[‡] except for the 90 and 100% acid runs which were within the operation limits for yield stress and consistency. The yield stress and consistency of the SRAT products are shown in Table 21. It should be noted that differences between the rheological properties of the simulants and the actual waste have been noted during past qualification testing. Past simulants have typically had lower yield stress than actual waste.

Table 21 SRAT Product Rheological Properties with SB6-A Baseline Sludge Simulant

Run	Acid %	Yield Stress, Pa	Consistency, cP	Insoluble Solids, wt %	Total Solids, wt %
SB6-2	90	2.73	9.40	15.7	26.3
SB6-1	100	3.24	8.22	14.7	26.1
SB6-3	120	0.36	4.47	12.9	25.3
SB6-4	150	0.05	3.44	14.6	25.8

3.4 SME CYCLE RESULTS

The four SME cycles were performed immediately following the SRAT cycle and utilized the estimated amount of frit based on the initial sludge additions and the expected amount of SRAT samples. The SME cycles for Runs SB6-1, 2, 3 and 4 did not include the addition of water simulating decon water additions but all included two frit slurry additions. As stated earlier, the SME cycle targeted a final solids concentration of 45 wt % total solids based on

[‡] “Technical Data Summary for the Defense Waste Processing Facility: Sludge Plant”, DPSTD-80-38-2

earlier testing with SB4 and 5 that resulted in extremely viscous slurries at the end of the SME cycle¹⁵.

3.4.1 Processing Observations

Only hydrogen generation was noted as a potential processing issue during the SME cycle. The hydrogen generation in the highest acid run exceeded the DWPF hydrogen limit at during the final dewater at the completion of the SME cycle. Mixing was not an issue during processing. Mixer speed was maintained at 250 RPM throughout each run.

As shown in Figure 5, the pH profile of each SME cycle followed a similar profile with a dip in pH as the frit is added due to the formic acid content of the frit slurry followed by a gradual rise in pH as the slurry mix is evaporated.

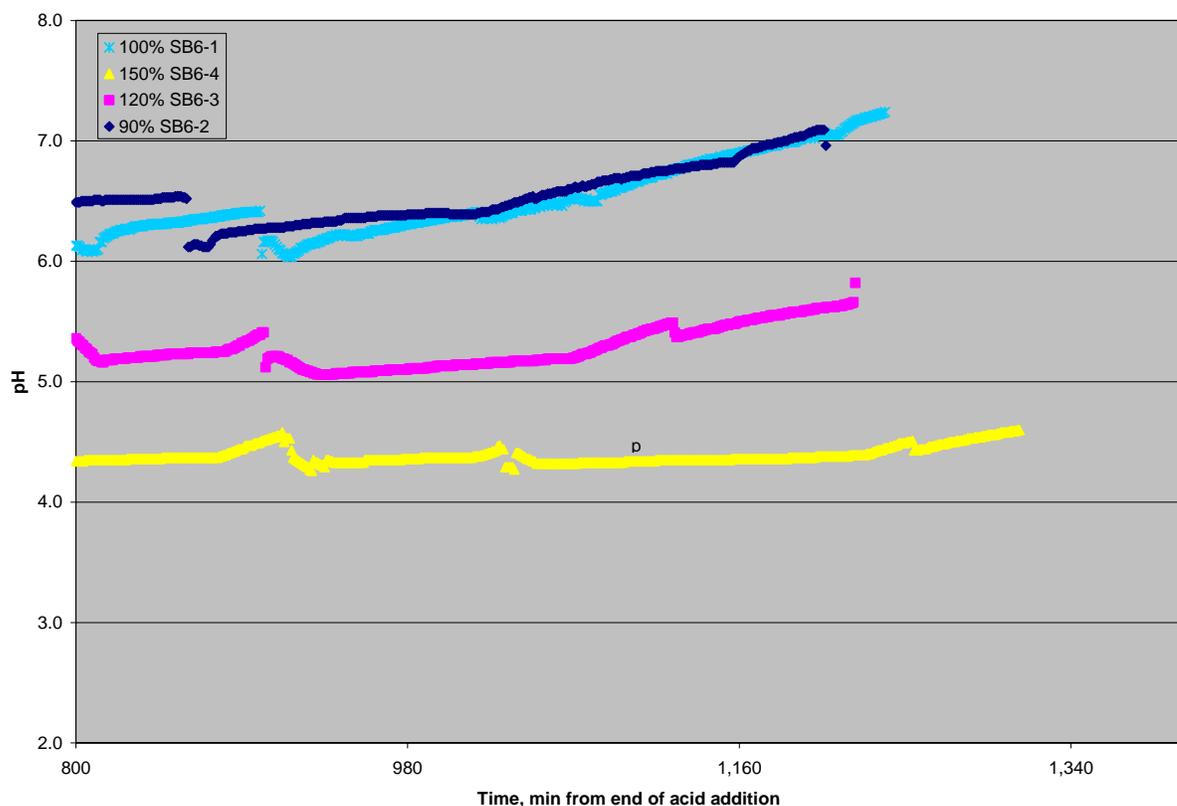


Figure 5. SME pH Profile from Tests with SB6-A Baseline Sludge Simulant

3.4.2 SME Cycle Sample Results

Samples were pulled at the conclusion of the SME cycle and analyzed for total solids, anions, soluble elemental species, TOC, and mercury. Samples were taken of the composite SME dewater and the FAVC contents at the completion of the SME cycle.

The solids contents of the SME products are shown in Table 22 along with the calculated waste loading and pH. The solids contents generally were higher than targeted, but the waste loading targets were lower than the 38% target. Waste loadings were calculated from the PSAL analyzed lithium content of the SME product (the frit 418 was 7.42% Li).

Table 22 SME Product Results from Tests with SB6-A Baseline Sludge Simulant

Acid %	pH	Total Solids wt%	Lithium Oxide Content (wt % Calcined solids)	Waste Loading [§] Wt %
SB6-2 90%	7.38	47.15	4.71	36.5%
SB6-1 100%	7.67	46.8	4.74	36.1%
SB6-3 120%	5.85	46.9	4.82	35.0%
SB6-4 150%	4.97	47.4	4.94	33.5%

Loss of formate varied during the SME cycles, as shown in Table 23. The range of values noted during the testing is similar to results from previous runs.

Table 23 SME Product Anion Conversions from Tests with SB6-A Baseline Sludge Simulant

ACID STOICHIOMETRY	SME Cycle	
	Formate Destruction	Nitrate Destruction
SB6-2 90%	6.1%	3.4%
SB6-1 100%	6.0%	4.1%
SB6-3 120%	2.8%	4.3%
SB6-4 150%	10.3%	6.8%

Total Organic Carbon (TOC) results are shown in Appendix B, but appear to be biased high based on the formate results. The TOC values listed exceed the TOC limits for the current sludge batch, but the values obtained based on the formate results meet TOC limits. The formate analysis is typically more accurate than the TOC analysis.

3.4.3 SME Cycle Offgas Composition Results

The amount of offgas generated during the runs generally increased as acid stoichiometry increased, as indicated by the helium concentration in the offgas since helium is added at a constant 0.5 wt% of the incoming air purge. A typical offgas concentration profile is shown in Figure 6. The patterns of offgas emissions noted during the runs were typical of offgas generation during the SME cycle with hydrogen and carbon dioxide emissions occurring during dewatering after each frit addition. The dip in concentrations near the run time of 900 minutes is the result of cooling the vessel and adding the second frit addition.

[§] % Waste Loading = (1-Lithium in SME product/7.42)*100%

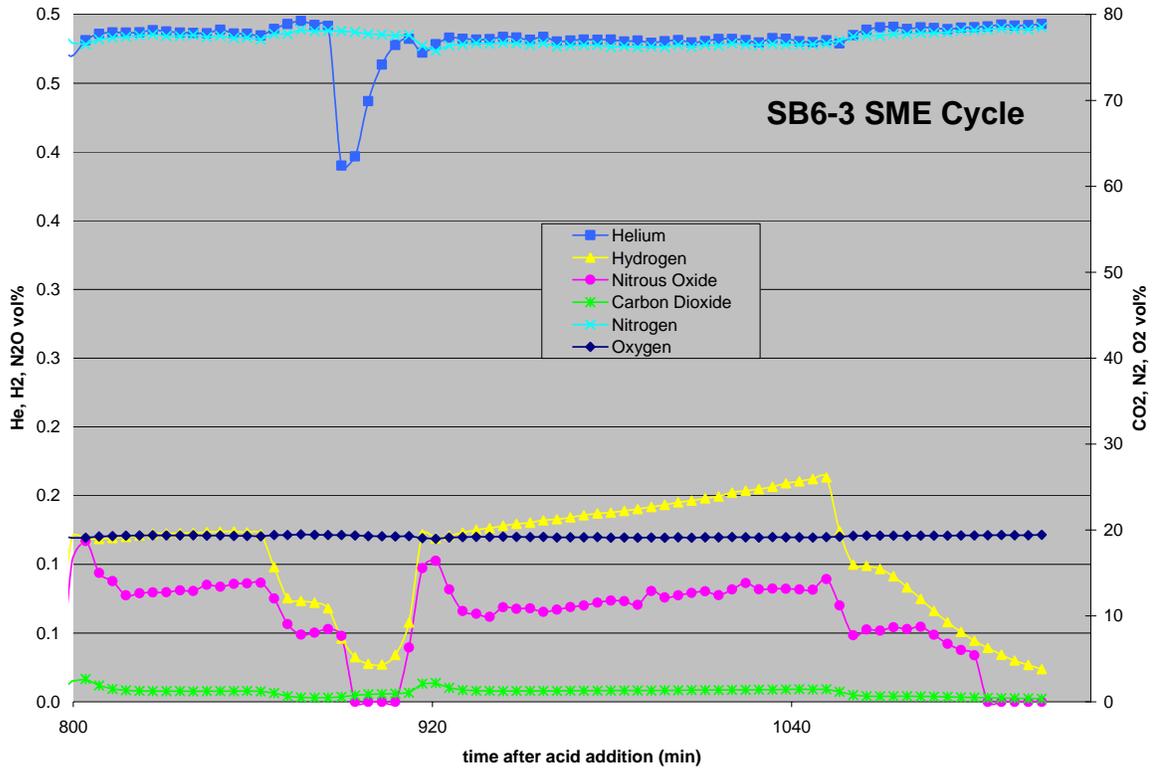


Figure 6. Typical SME Offgas Profile 120% Acid Stoichiometry, SB6-A Baseline Sludge Simulant

3.4.3.1 Hydrogen Evolution

The peak hydrogen generation rates were generally noted as sharp spikes in the data immediately following the start of dewater, as shown in Figure 6 above. Hydrogen reached concentrations higher than noted in the SRAT cycle due to the decreased purge during the SME cycle. Peak hydrogen concentrations reached close to 0.5 volume %, as shown in Figure 7 and Figure 8, and were a function of acid stoichiometry. Peak generation rates scaled to the DWPF process are shown in Table 24 and were all below the SME process limit of 0.223 lb/hr, except for the 150% stoichiometry.

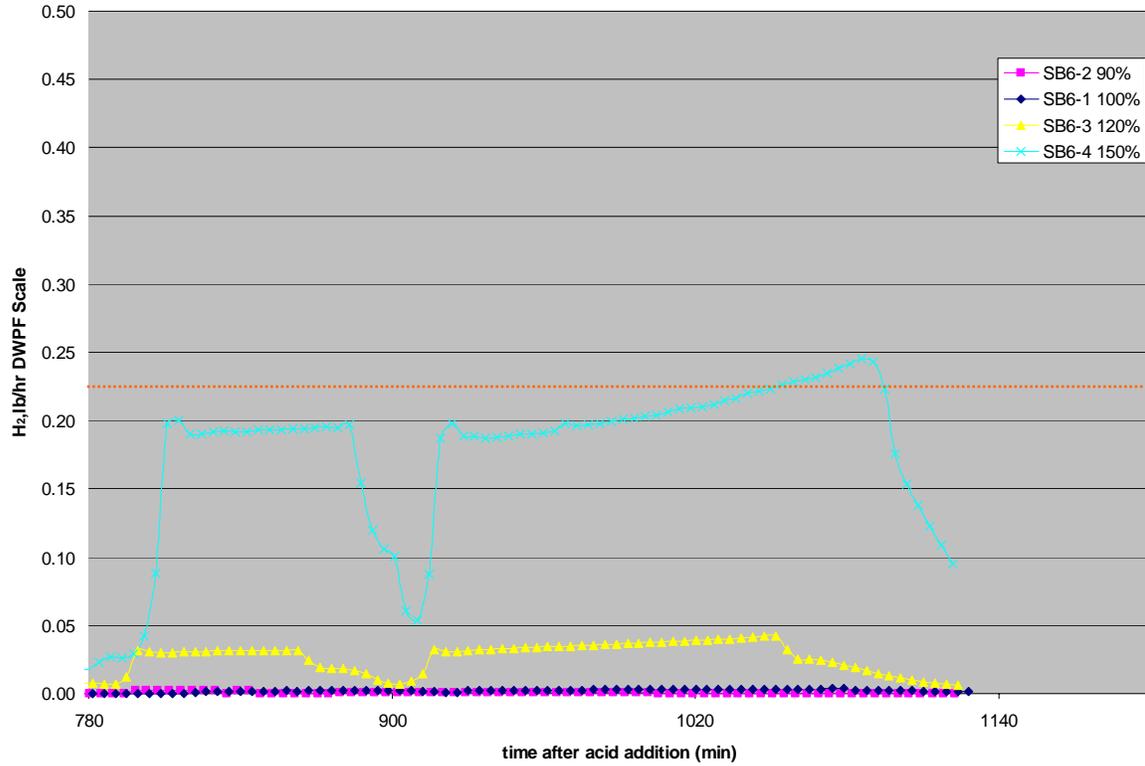


Figure 7. SME Hydrogen Generation

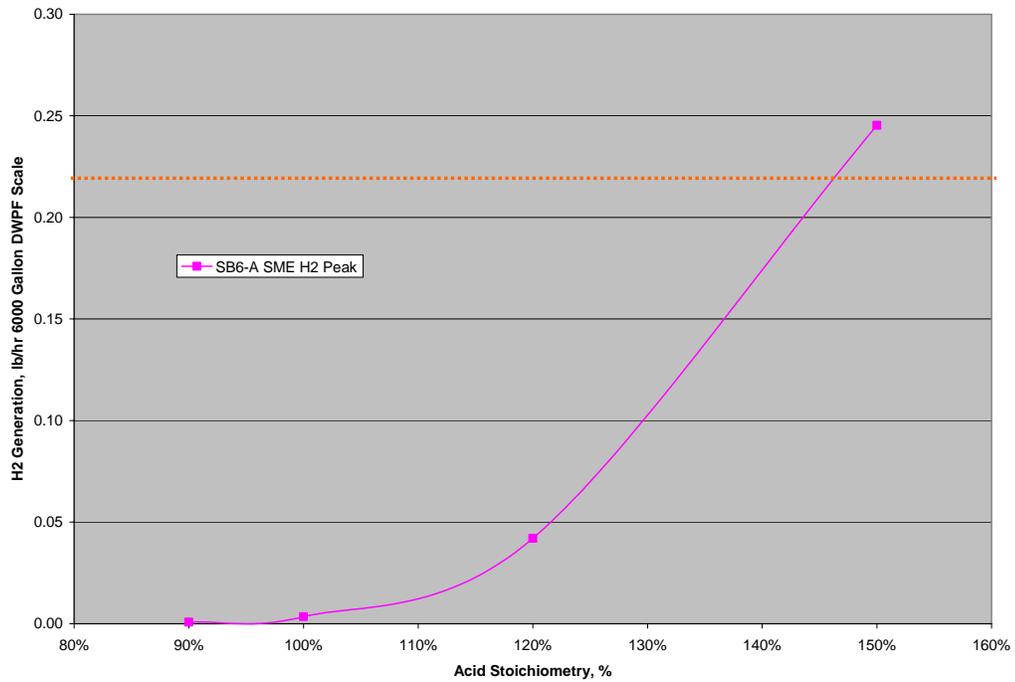


Figure 8. Peak Hydrogen Generation during SME Cycle, SB6-A Baseline Sludge Simulant

Table 24 SME Cycle Hydrogen Peak Generation Rate, SB6-A Baseline Sludge Simulant

SME Hydrogen Peak		Acid Stoichiometry			
		90%	100%	120%	150%
SB6-A Simulant	lb/hr	0.001	0.004	0.042	0.245

3.4.3.2 Other Species

Carbon dioxide, as shown in Table 25, was generally the only other gas of any significance emitted during the SME cycle (the lower acid runs contained a small amount of nitrous oxide emissions from the nitrite remaining after the SRAT cycle).

Table 25 SME Cycle Nitrous Oxide and Carbon Dioxide Peak Generation Rates from Tests with SB6-A Baseline Sludge Simulant

		Acid Stoichiometry			
		90%	100%	120%	150%
SRAT Nitrous Oxide Peak	lb/hr	0.95	0.44	0.66	0.00
SRAT Carbon Dioxide Peak	lb/hr	19.1	21.3	14.9	17.9

3.4.4 SME Product Rheological Properties

The rheological properties of each SME product were measured. Higher acid stoichiometry lowered the yield stress and consistency of the SME products. All runs were within the process limits for yield stress (2.5 to 15 Pa) and consistency (10 to 40 cP)** as shown in Table 26.

Table 26 SME Product Rheological Properties from Tests with SB6-A Baseline Sludge Simulant

Run	Acid %	Yield Stress, Pa	Consistency, cP	Total Solids, wt %
SB6-2	90%	8.56	32.34	46.8
SB6-1	100%	4.95	25.56	47.1
SB6-3	120%	3.58	30.04	46.4
SB6-4	150%	4.50	16.75	47.4

3.4.5 Other Notable Observations

Two notable observations by the technicians were unusual and are noted below.

1. The agitator blades and shaft from SB6-4 had black deposits covering all surfaces (Figure 9). For comparison, the agitator from run SB6-1, the lowest acid run, was virtually without deposits of solids on the surfaces. The film was resistant to rinsing, was not removed by soaking in nitric acid and required mechanical cleaning to remove. The material deposited on the shaft contained mercury and likely contained noble metals, but speciation was not performed.

** "Technical Data Summary for the Defense Waste Processing Facility: Sludge Plant", DPSTD-80-38-2

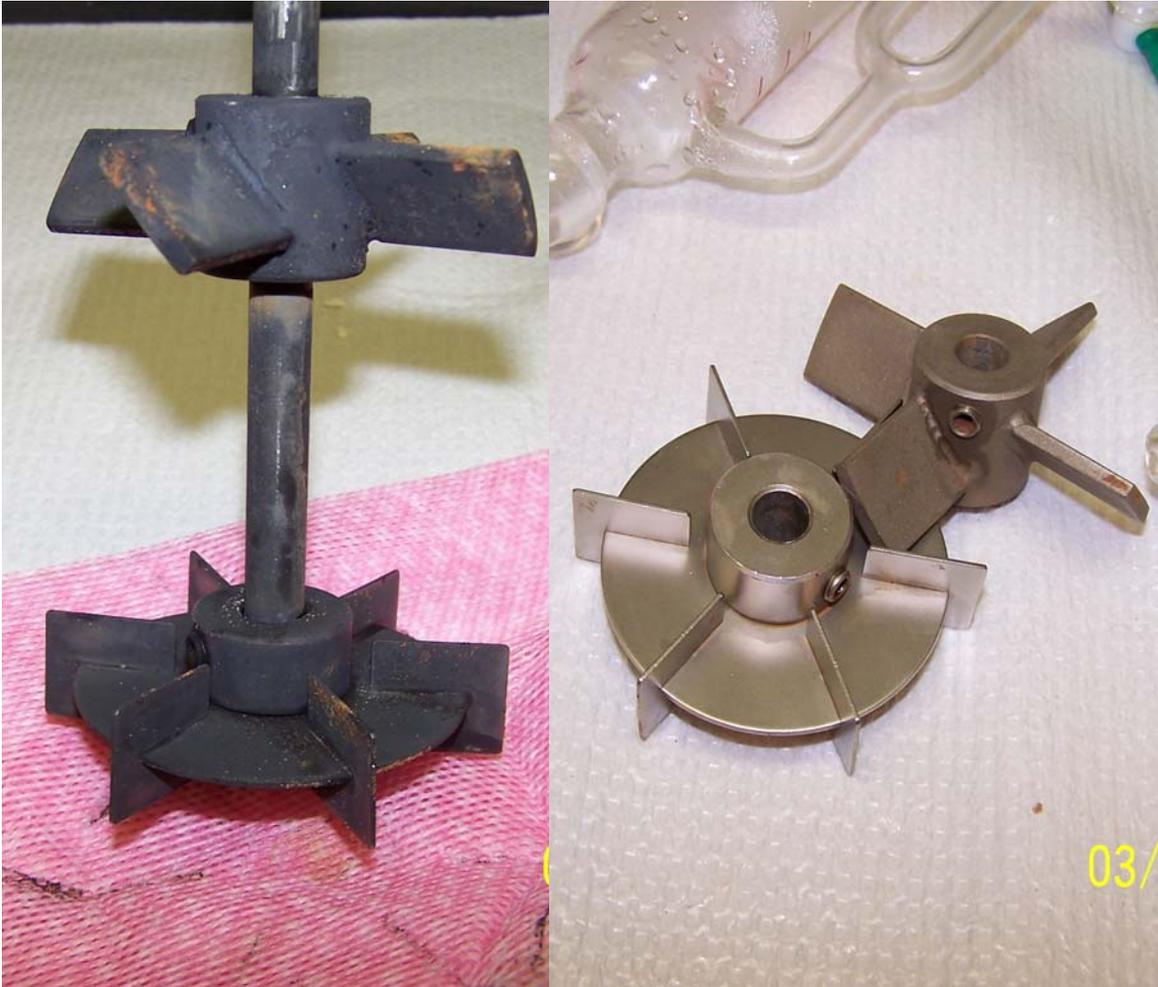


Figure 9. Photos of SB6-4 Agitator Blades and Shaft (left) and SB6-1 Agitator Blades (right)

2. The SME products were saved for melt rate testing. Before combining the four SME products, the technicians noted dark solids in the three lowest acid runs.

4.0 CONCLUSIONS

Six DWPF process simulations were completed in 4-L laboratory-scale equipment using two projections of the SB6 blend simulant composition (Tank 40 composition after Tank 51 transfer on a 40" Tank 40 heel is complete). The more washed simulant (SB6-A washed to nominally 1M Na) had a set of four SRAT and SME simulations at varying acid stoichiometry levels (90%, 100%, 120% and 150%). Two additional SRAT simulations were made using SB6-B blend simulant (nominally 1.2 M Na) at 100% and 120% of acid stoichiometry. Acid predictions used the Koopman Acid Prediction Calculation.¹⁶

Two SB6 processing issues were noted during initial flowsheet testing. First, a high hydrogen generation rate was measured during the SB6-A experiment with the highest acid stoichiometry. Second, in the lower acid runs for both SB6-A and SB6-B sludge, the mercury concentration exceeded the 0.45 wt % Hg in SRAT product solids limit.

Three items were specifically requested in the TTR and are discussed below.

1. Hydrogen and nitrous oxide generation rates as a function of acid stoichiometry

Hydrogen generation was significantly impacted by the changes in acid stoichiometry from 90% to 150% (1.45 to 2.42 moles acid per liter of SB6-A sludge or 1.83 to 2.20 moles acid per liter of SB6-B sludge). For the SB6-A sludge, the hydrogen generation rate was within DWPF limits in the SRAT cycle, but exceeded the process limit during the SME cycle at the highest acid stoichiometry (150%). Both of the SB6-B experiments were within the process limits throughout the SRAT even though the stoichiometric acid requirement was higher. No SME cycles were performed with the SB6-B feed. The nitrous oxide generation peak was relatively insensitive to acid stoichiometry and was relatively low due to the low starting nitrite concentration. Hydrogen generation and nitrous oxide generation scaled to DWPF are shown in Table 27 and Table 28.

Table 27. Offgas Peak Summary – SB6-A Baseline Sludge Simulant

		Acid Stoichiometry			
		90%	100%	120%	150%
SRAT Hydrogen Peak	lb/hr	0.0056	0.0058	0.0602	0.4322
SME Hydrogen Peak	lb/hr	0.001	0.004	0.042	0.245
SRAT Nitrous Oxide Peak	lb/hr	20.9	29.1	33.8	35.8
SME Nitrous Oxide Peak	lb/hr	0.95	0.44	0.66	0.00
SRAT Carbon Dioxide Peak	lb/hr	547	539	469	470
SME Carbon Dioxide Peak	lb/hr	19.1	21.3	14.9	17.9

Table 28. Offgas Peak Summary – SB6-B Sludge Simulant (One Less Wash)

		Acid Stoichiometry	
		100%	120%
SRAT Hydrogen Peak	lb/hr	0.0050	0.0642
SRAT Nitrous Oxide Peak	lb/hr	22.1	31.5
SRAT Carbon Dioxide Peak	lb/hr	545	493

2. Acid quantities and processing times required for mercury removal

Mercury was added to the sludge simulant at the start of the SRAT cycle as mercuric oxide at 1.5 wt% (total solids basis) based on the expected composition of the SB6 blend. In the higher acid runs, 12 hours of boiling was sufficient time to strip mercury below the 0.45 wt % SRAT limit. Boiling flux was maintained at a scaled rate of 5,000 lb/hr so a total of 60,000 lb of steam flow in DWPF would be needed to remove 120 lb of mercury. Acid quantities from 120% to 150% resulted in satisfactory mercury removal with 12 hours of boiling time. The two lowest acid stoichiometry runs with the SB6-A simulant did not meet the process limit after 12 hours of boiling and neither run with the SB6-B simulant met the 0.45wt% limit. A general trend of more efficient stripping was noted at higher acid stoichiometries. If DWPF experiences problems stripping mercury, increasing the boiling time or increasing the acid stoichiometry is likely to improve mercury removal but may also increase hydrogen generation. Longer boiling times will be used in future SB6 testing to ensure the mercury concentration is below the SRAT limit.

3. Acid quantities and processing times required for nitrite destruction

Acid quantities from 100% to 150% resulted in satisfactory nitrite destruction with 12 hours of boiling. In all runs, except the 90% run, the amount of nitrite present in the SRAT product was less than the 1,000 mg/kg target. The low starting nitrite concentration helped to reduce the nitrite by the end of the SRAT cycle.

5.0 REFERENCES

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- ¹³ Lambert, D. P., *Acid Calculation Spreadsheet for DWPF Simulations, Revision 1 (Dated 8/14/06)*, SRNL-PSE-2006-00173, Savannah River Site, Aiken, SC 29808 (2006).
- ¹⁴ Lambert, D.P., M. E. Stone, B. R. Pickenheim, D. R. Best, D. C. Koopman, *Sludge Batch 5 Simulant Flowsheet Studies*, SRNS-STI-2008-00024, Revision 0, Savannah River Site, Aiken, SC 29808 (2008).
- ¹⁵ Stone, M. E., *FY06 Feed Preparation for Melt Rate Testing*, WSRC-STI-2006-0007, Savannah River Site, Aiken, SC 29808 (2006).

- ¹⁶ Koopman, D.C., A.I. Fernandez, B.R. Pickenheim, *Preliminary Evaluations of Two Proposed Stoichiometric Acid Equations, Revision 0*, SRNL-L3100-2009-00146, Savannah River Site, Aiken, SC 29808 (2009).

APPENDIX A. Offgas Data

The following graphs were generated using offgas composition information using Agilent micro gas chromatographs. The data was reprocessed after each run by John Pareizs to recalculate the concentration of any missed peaks or other instrument anomalies.

The following graphs are included in this appendix:

- Figure A1 SB6-1 Offgas Profile (SRAT and SME Cycle)
- Figure A2 SB6-2 Offgas Profile (SRAT and SME Cycle)
- Figure A3 SB6-3 Offgas Profile (SRAT and SME Cycle)
- Figure A4 SB6-4 Offgas Profile (SRAT and SME Cycle)
- Figure A5 SB6-5 Offgas Profile (SRAT Cycle Only)
- Figure A6 SB6-6 Offgas Profile (SRAT Cycle Only)

Figure A1—SB6-1 SRAT and SME Cycle GC Data

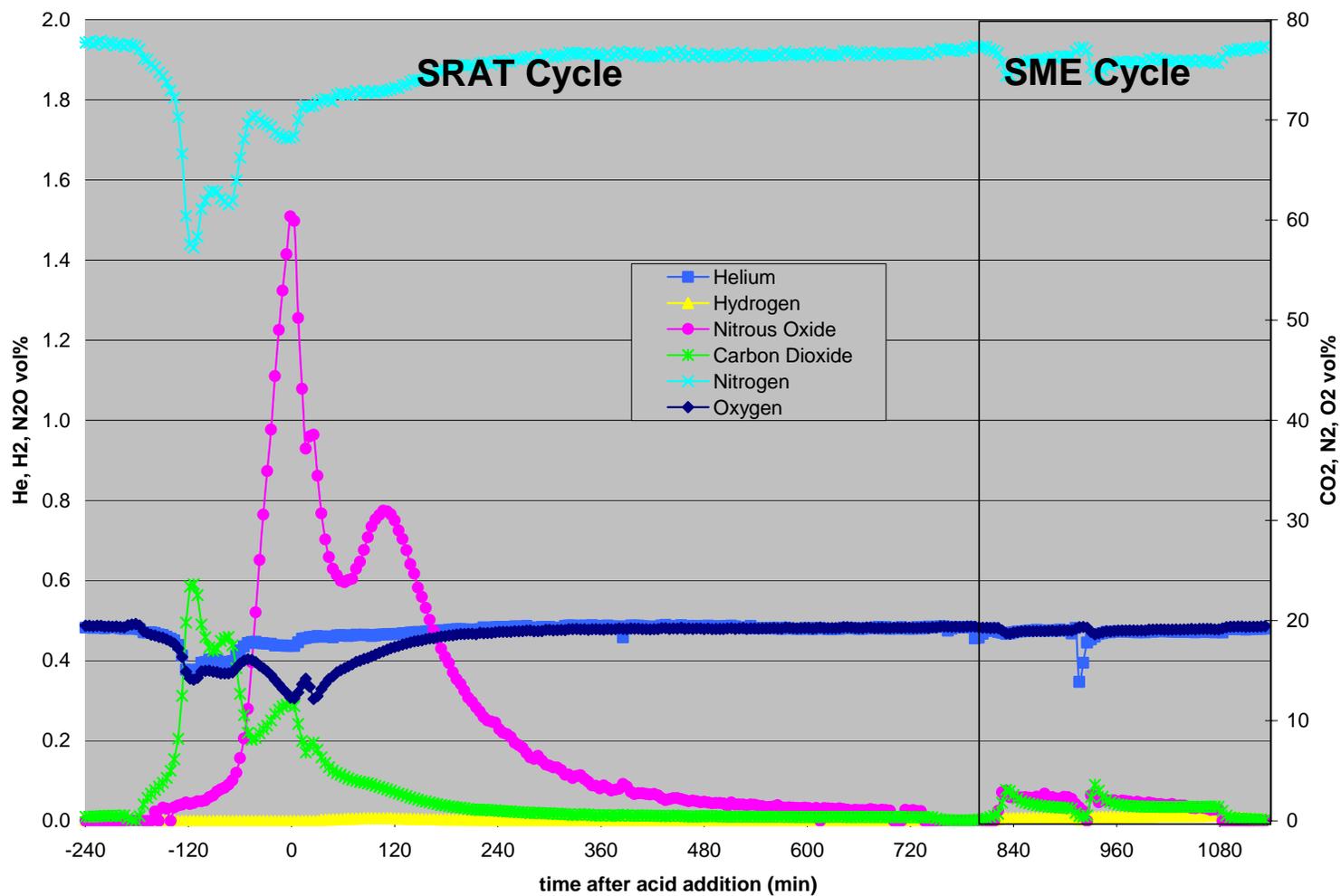


Figure A2—SB6-2 SRAT and SME Cycle GC Data

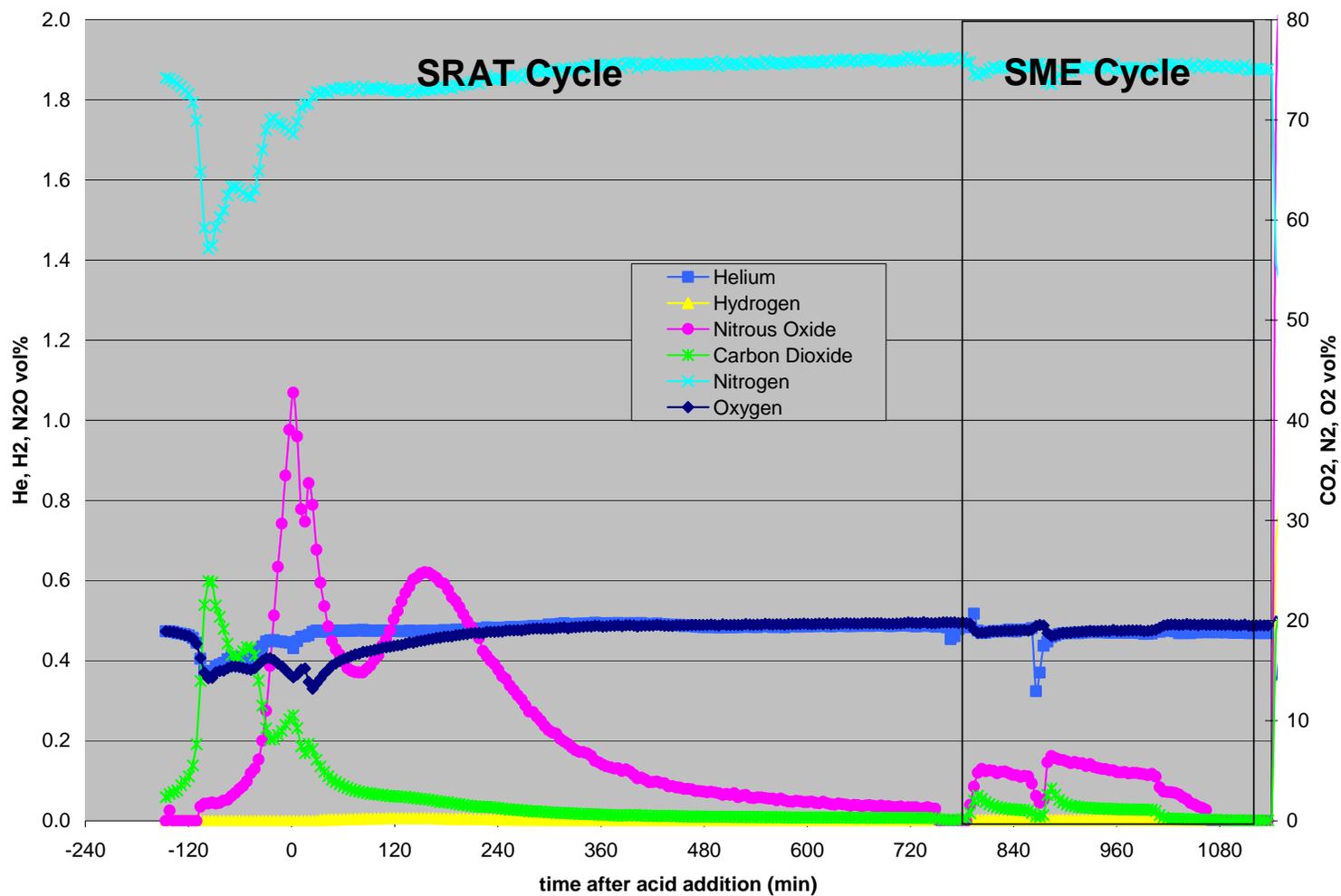


Figure A3—SB6-3 SRAT and SME Cycle GC Data

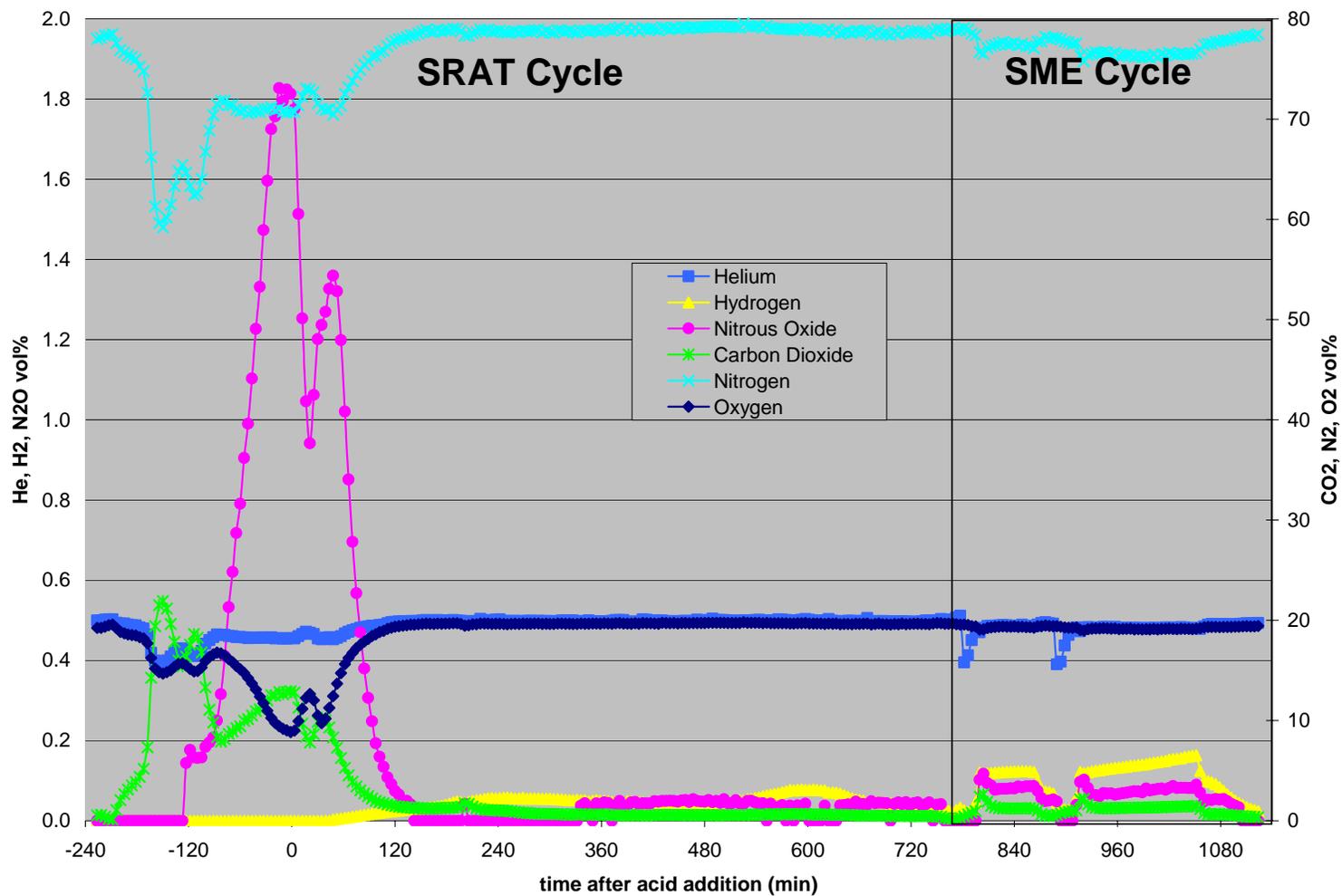


Figure A4—SB6-4 SRAT and SME Cycle GC Data

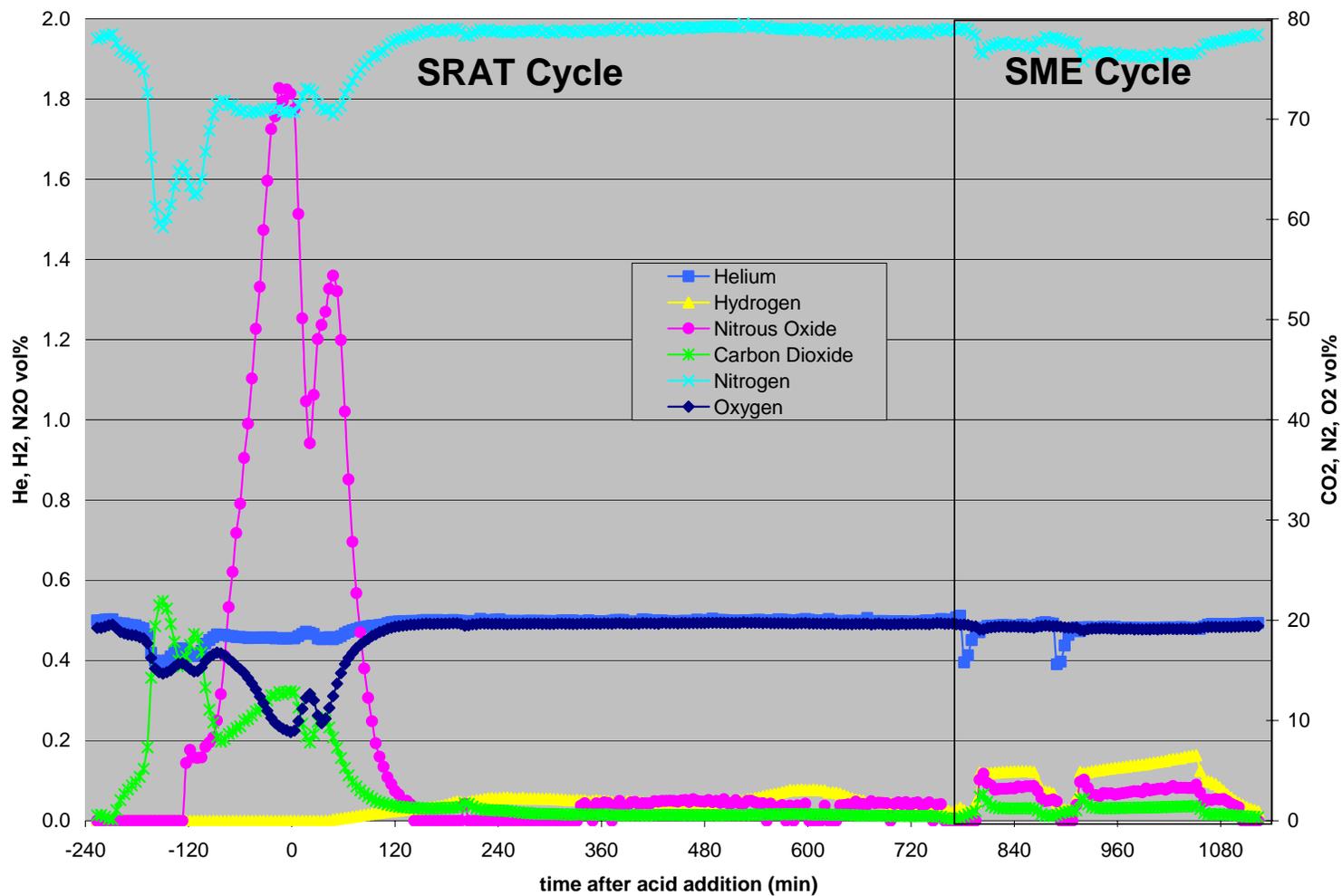


Figure A5—SB6-5 SRAT Cycle GC Data (No SME Cycle)

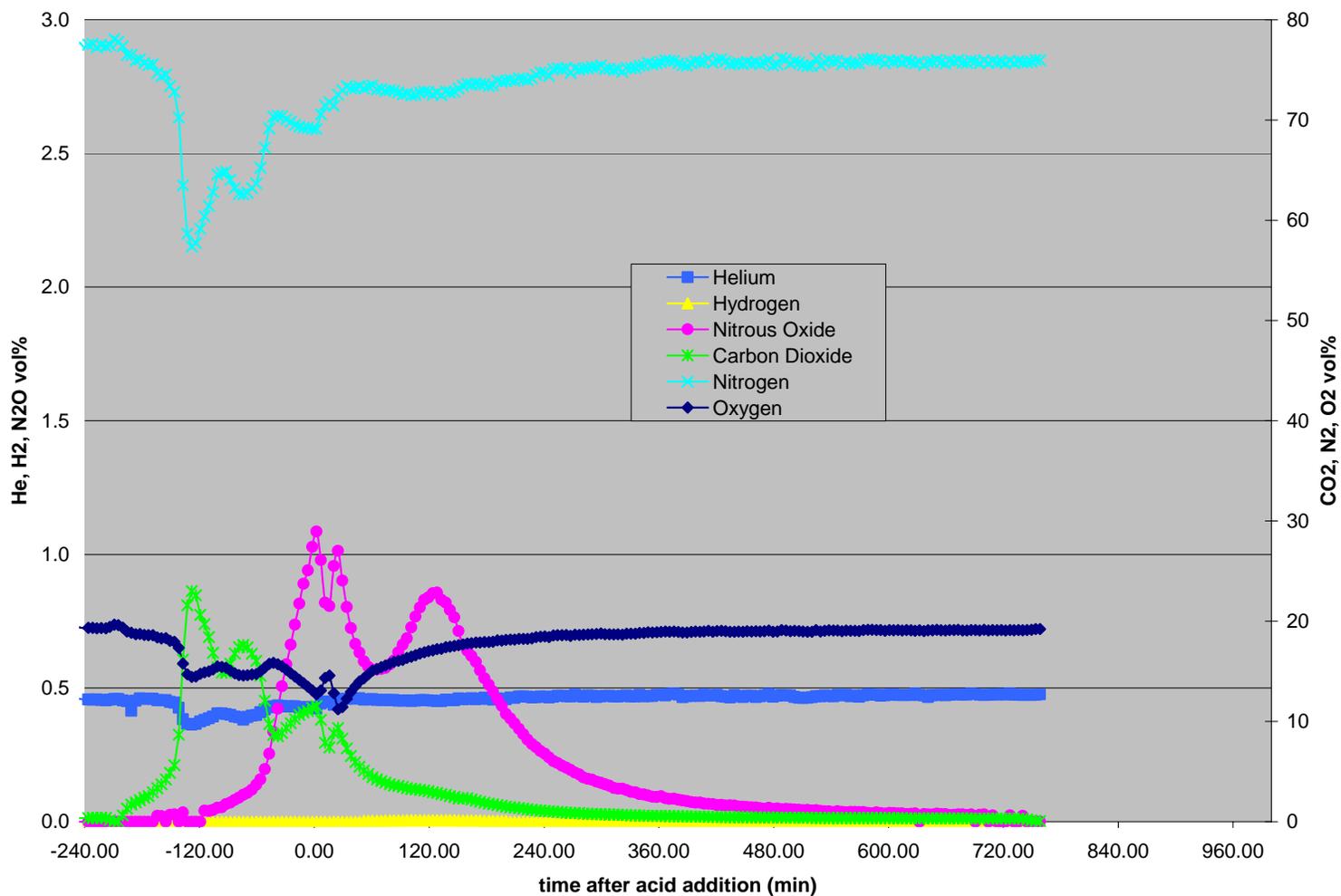
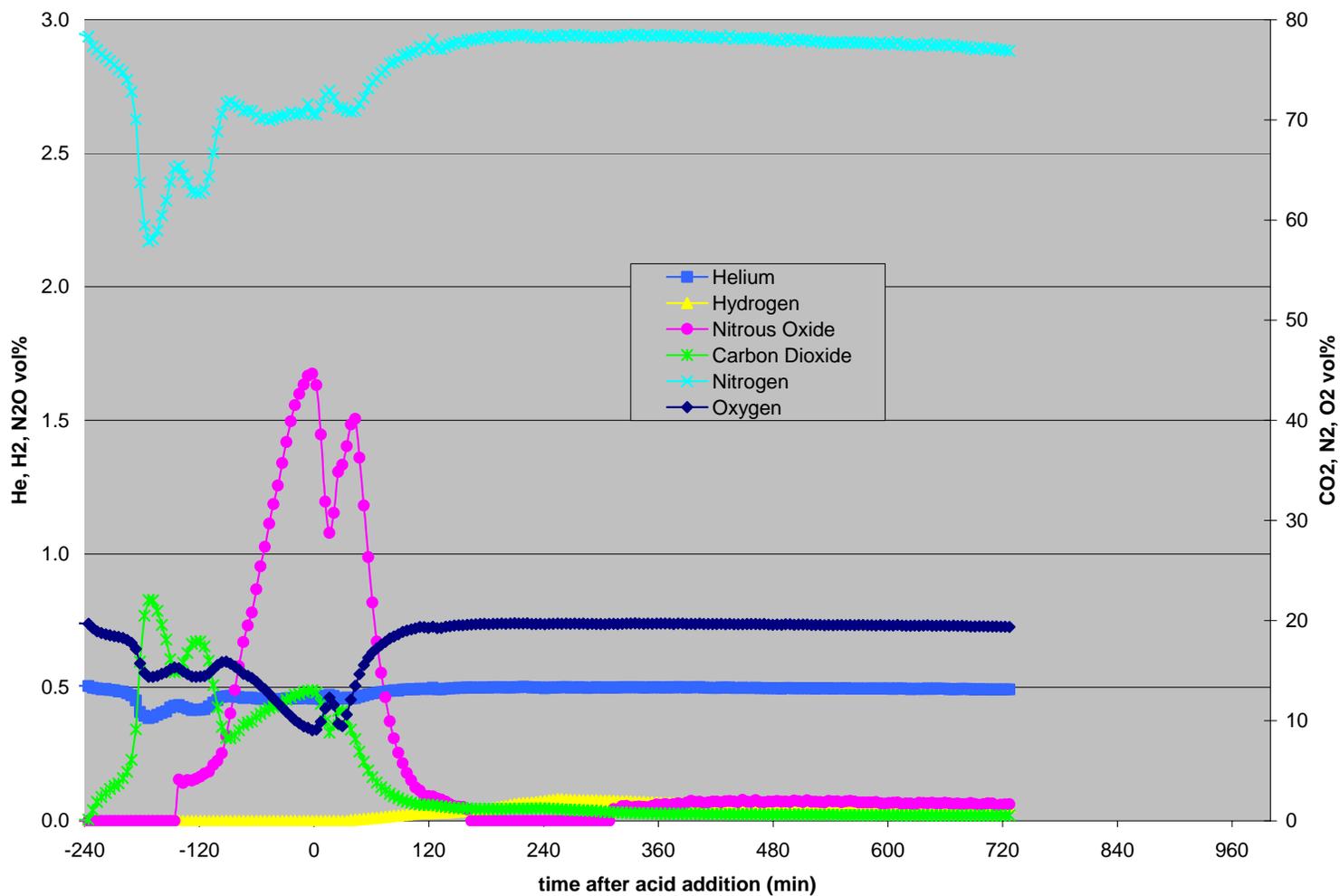


Figure A6—SB6-6 SRAT Cycle GC Data (No SME Cycle)



APPENDIX B. ANALYTICAL DATA

The Process Science Analytical Laboratory and Analytical Development personnel analyzed samples as requested. The sample results presented in this appendix:

Table B1	SB6-A and SB6-B Sludge Analytical Data (SRAT Receipt Sample)
Table B2	SB6-1 to SB6-6 SRAT Product Analytical Data
Table B3	SB6-1 to SB6-6 SME Product Analytical Data
Table B4	SB6-1 to SB6-6 SRAT Dewater Analytical Results
Table B5	SB6-1 to SB6-6 SRAT Intermediate Slurry Sample Analytical Results
Table B6	SB6-1 to SB6-6 SRAT Intermediate Centrifuged Sample Analytical Results
Table B7	AD Mercury Analytical Results, wt % total solids
Table B8	AD Ammonium and Carbon Analytical Results on SRAT and SME product

NOTE: The following protocol was used to number the samples from the SRAT runs: 09-SB6-x-xxxx-A/B where:

09	=	year (not included in many tables since this number is the same for all samples in study)
SB6-x	=	Run number
xxxx	=	Unique sample number
A/B	=	Analytical replicate

Table B1
SB6-A and SB6-B Sludge Slurry Analysis

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 3/25/09

Sample ID: 09-SB6-A-2665, 09-SB6-B-2665 (5) and 09-SB6-B-2665 (6) Lab ID: 09-0139 09-0194 and 09-0195

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	<u>Al</u>	<u>Ba</u>	<u>Ca</u>	<u>Ce</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>K</u>	<u>La</u>	<u>Mg</u>	<u>Mn</u>	<u>Na</u>
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	15.3	0.214	2.08	0.180	0.248	0.086	18.4	0.104	0.093	1.52	6.70	14.4
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	15.2	0.218	2.10	0.180	0.253	0.082	18.2	0.101	0.093	1.49	6.77	14.5
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	14.9	0.215	2.20	0.188	0.251	0.093	18.3	0.098	0.093	1.56	7.01	15.4
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	14.9	0.215	2.20	0.189	0.252	0.096	18.3	0.097	0.092	1.56	7.01	15.2
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	14.6	0.217	2.15	0.191	0.256	0.100	18.2	0.096	0.093	1.55	6.90	15.2
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	14.7	0.215	2.15	0.188	0.254	0.089	18.4	0.096	0.092	1.58	6.99	15.1
	<i>oxide wt% - calcined 1100C</i>	Lab ID	<u>Al2O3</u>	<u>BaO</u>	<u>CaO</u>	<u>CeO2</u>	<u>Cr2O3</u>	<u>CuO</u>	<u>Fe2O3</u>	<u>K2O</u>	<u>La2O3</u>	<u>MgO</u>	<u>MnO2</u>	<u>Na2O</u>
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	28.8	0.240	2.91	0.221	0.362	0.108	26.4	0.125	0.109	2.52	10.6	19.4
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	28.7	0.244	2.94	0.221	0.369	0.103	26.0	0.121	0.109	2.47	10.7	19.6
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	28.2	0.241	3.08	0.232	0.366	0.116	26.2	0.118	0.109	2.59	11.1	20.8
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	28.1	0.241	3.07	0.232	0.368	0.120	26.2	0.117	0.108	2.59	11.1	20.6
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	27.6	0.243	3.01	0.235	0.374	0.125	26.0	0.115	0.108	2.58	10.9	20.5
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	27.7	0.241	3.01	0.231	0.371	0.112	26.3	0.116	0.108	2.62	11.1	20.4

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	<u>Ni</u>	<u>P</u>	<u>Pb</u>	<u>S</u>	<u>Si</u>	<u>Ti</u>	<u>Zn</u>	<u>Zr</u>	
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	3.82	<0.100	0.012	0.311	0.504	0.020	0.153	0.358	
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	3.87	<0.100	0.013	0.306	0.500	0.020	0.156	0.360	
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	4.06	<0.100	0.021	0.304	0.588	0.023	0.156	0.361	
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	4.01	<0.100	0.021	0.304	0.622	0.020	0.154	0.361	
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	3.96	<0.100	0.033	0.312	0.594	0.020	0.155	0.367	
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	4.04	<0.100	0.032	0.314	0.588	0.020	0.156	0.361	
	<i>oxide wt% - calcined 1100C</i>	Lab ID	<u>NiO</u>	<u>P2O5</u>	<u>PbO</u>	<u>SO4</u>	<u>SiO2</u>	<u>TiO2</u>	<u>ZnO</u>	<u>ZrO2</u>	<u>Totals</u>
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	4.85	0.000	0.013	0.933	1.08	0.000	0.190	0.483	99.3
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	4.91	0.000	0.014	0.918	1.07	0.000	0.193	0.486	99.1
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	5.16	0.00	0.023	0.913	1.26	0.038	0.193	0.488	101
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	5.10	0.00	0.022	0.911	1.33	0.034	0.191	0.487	101
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	5.03	0.00	0.036	0.935	1.27	0.033	0.192	0.495	99.7
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	5.13	0.00	0.034	0.941	1.26	0.033	0.193	0.487	100

Units: mg/Kg

Sample Description	Sample ID	Lab ID	F	Cl	NO ₂	NO ₃	SO ₄	HCO ₂	C ₂ O ₄	PO ₄
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	<100	168	14000	8650	1180	<100	<100	<100
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	<100	170	14000	8760	1190	<100	<100	<100
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	<100	191	13700	9130	1150	<100	<100	<100
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	<100	190	13500	9080	1140	<100	<100	<100
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	<100	191	13600	8940	1150	<100	<100	<100
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	<100	191	13500	9010	1150	<100	<100	<100

Weight % Solids Calculations

Sample Description	Sample	Lab ID	Total Solids	Insoluble Solids	Calcined Solids	Soluble Solids		Density, g/mL	pH
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	17.7%	11.2%	13.0%	6.50%		1.14	12.7
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	17.7%	11.0%	12.9%	6.76%			
SB6B Simulant	09-SB6-B-2665 (5) (A)	09-0194	20.0%	13.5%	14.7%	6.48%		1.07	12.7
SB6B Simulant	09-SB6-B-2665 (5) (B)	09-0194	20.0%	13.4%	14.7%	6.60%			
SB6B Simulant	09-SB6-B-2665 (6) (A)	09-0195	19.9%	13.3%	14.7%	6.56%		1.11	12.7
SB6B Simulant	09-SB6-B-2665 (6) (B)	09-0195	19.9%	13.3%	14.7%	6.62%			

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 3/13/09

Sample ID: 09-SB6-A-2665

Lab ID: 09-0139

Comments: Auto Titration

Units: mmol/gram

Sample Description	Sample ID	Lab ID	pH - 7.0 Result (mmol/gram)	pH - 5.5 Result (mmol/gram)
	0.1 mmol/gram NaOH Standard		0.1009	0.1009
	0.1 mmol/gram NaOH Standard		0.1010	0.1010
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	0.5434	0.6470
SB6B Simulant	09-SB6-A-2665 (B)	09-0139	0.5437	0.6464
	0.1 mmol/gram NaOH Standard		0.1005	0.1005

Total Base Titration Result, M

0.6350

Sample Description	<i>supernate (mg/L)</i>		<u>Al</u>	<u>Ba</u>	<u>Ca</u>	<u>Ce</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>K</u>	<u>La</u>	<u>Mg</u>	<u>Mn</u>	<u>Na</u>
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	2690	<0.100	5.87	<0.100	13.5	<0.100	<0.100	235	<0.100	<0.100	<0.100	23000
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	2700	<0.100	5.86	<0.100	13.6	<0.100	<0.100	233	<0.100	<0.100	<0.100	22900

Sample Description	<i>supernate (mg/L)</i>		<u>Ni</u>	<u>P</u>	<u>Pb</u>	<u>S</u>	<u>Si</u>	<u>Ti</u>	<u>Zn</u>	<u>Zr</u>
SB6A Simulant	09-SB6-A-2665 (A)	09-0139	<0.100	<1.00	<0.100	408	<0.100	<0.100	<0.100	<0.100
SB6A Simulant	09-SB6-A-2665 (B)	09-0139	<0.100	<1.00	<0.100	407	<0.100	<0.100	<0.100	<0.100

AD Sample Results (SB6-B TIC values calculated from SB6a results)

Sample Id	User SampleID	LIMS Method	Total Carbon	Inorganic Carbon	Organic Carbon	1 Sigma % Unc	Units	Rv
Slurry								
300258058	09_SB6_A_2667A	TIC/TOC (B154)	133	118	15	10	mg C/mL	1
300258059	09_SB6_A_2667B	TIC/TOC (B154)	150	121	29	10	mg C/mL	
300258060	09_SB6_A_2667C	TIC/TOC (B154)	173	125	48	10	mg C/mL	1
Supernate			1290	1270	20			
300258061	09_SB6_A_2668A	TIC/TOC (B154)	1280	1270	10	10	mg C/mL	1
300258062	09_SB6_A_2668B	TIC/TOC (B154)	1290	1270	20	10	mg C/mL	1
300258063	09_SB6_A_2668C	TIC/TOC (B154)	1300	1280	20	10	mg C/mL	1

Sample Id	User SampleID	Sample Mass, g	Total Mass, g	Corrected TIC	Carbonate
300258058	09_SB6_A_2667A	2.002	40.084	2360	11,800
300258059	09_SB6_A_2667B	2.097	40.14	2320	11,600
300258060	09_SB6_A_2667C	2.018	40.031	2480	12,400

Table B2
PSAL SRAT Product Analytical Results

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 4/2/09, 4/6/09

Sample ID: SB6-1-2677, SB6-4-2694, SB6-2-2711, SB6-3-2729, SB6-5-2748 and SB6-6-2765 (SRAT Products)

Lab ID: 09-0165, 09-0168, 09-0217 and 09-0218

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	<u>Al</u>	<u>Ba</u>	<u>Ca</u>	<u>Ce</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>K</u>	<u>La</u>	<u>Mg</u>	<u>Mn</u>
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	14.5	0.211	2.03	0.189	0.262	0.091	17.2	0.122	0.092	1.44	6.49
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	14.4	0.213	1.98	0.188	0.259	0.097	17.1	0.119	0.092	1.42	6.45
SB6-2 SRAT Product (90%)	SB6-2-2711 (A)	09-0167	14.5	0.203	2.00	0.186	0.259	0.097	17.2	0.088	0.091	1.44	6.51
SB6-2 SRAT Product (90%)	SB6-2-2711 (B)	09-0167	14.5	0.203	2.07	0.186	0.259	0.087	17.1	0.090	0.091	1.43	6.47
SB6-3 SRAT Product (120%)	SB6-3-2729 (A)	09-0168	14.5	0.204	2.02	0.187	0.258	0.043	17.2	0.090	0.091	1.41	6.23
SB6-3 SRAT Product (120%)	SB6-3-2729 (B)	09-0168	14.6	0.205	2.07	0.187	0.259	0.033	17.2	0.093	0.092	1.42	6.29
SB6-4 SRAT Product (150%)	SB6-4-2694 (A)	09-0166	14.7	0.223	1.96	0.188	0.263	0.030	17.4	0.134	0.092	1.39	6.50
SB6-4 SRAT Product (150%)	SB6-4-2694 (B)	09-0166	14.9	0.226	1.94	0.190	0.266	0.033	17.6	0.134	0.093	1.39	6.49
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	14.5	0.211	2.08	0.194	0.261	0.096	17.6	0.095	0.095	1.49	6.68
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	14.3	0.208	2.06	0.193	0.260	0.089	17.4	0.095	0.094	1.48	6.65
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	14.3	0.213	2.05	0.193	0.262	0.066	17.4	0.084	0.094	1.46	6.56
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	14.4	0.209	2.07	0.192	0.257	0.057	17.3	0.087	0.094	1.46	6.48

Sample Description	<i>oxide wt% - calcined 1100C</i>		<u>Al₂O₃</u>	<u>BaO</u>	<u>CaO</u>	<u>CeO₂</u>	<u>Cr₂O₃</u>	<u>CuO</u>	<u>Fe₂O₃</u>	<u>K₂O</u>	<u>La₂O₃</u>	<u>MgO</u>	<u>MnO₂</u>
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	27.5	0.236	2.84	0.232	0.383	0.113	24.6	0.147	0.108	2.40	10.3
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	27.1	0.238	2.78	0.231	0.378	0.121	24.5	0.143	0.107	2.36	10.2
SB6-2 SRAT Product (90%)	SB6-4-2694 (A)	09-0166	27.8	0.250	2.74	0.231	0.383	0.038	24.9	0.161	0.108	2.31	10.3
SB6-2 SRAT Product (90%)	SB6-4-2694 (B)	09-0166	28.1	0.253	2.72	0.233	0.388	0.042	25.1	0.160	0.108	2.31	10.2
SB6-3 SRAT Product (120%)	SB6-2-2711 (A)	09-0167	27.3	0.227	2.80	0.229	0.378	0.122	24.6	0.106	0.106	2.39	10.3
SB6-3 SRAT Product (120%)	SB6-2-2711 (B)	09-0167	27.4	0.227	2.90	0.229	0.378	0.109	24.5	0.108	0.107	2.38	10.2
SB6-4 SRAT Product (150%)	SB6-3-2729 (A)	09-0168	27.4	0.228	2.83	0.230	0.377	0.053	24.5	0.108	0.107	2.34	9.85
SB6-4 SRAT Product (150%)	SB6-3-2729 (B)	09-0168	27.7	0.229	2.89	0.230	0.379	0.041	24.6	0.112	0.107	2.36	9.94
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	27.3	0.236	2.91	0.238	0.381	0.120	25.1	0.114	0.111	2.47	10.6
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	27.1	0.233	2.88	0.237	0.379	0.112	24.9	0.114	0.110	2.45	10.5
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	27.1	0.239	2.86	0.237	0.382	0.083	24.9	0.101	0.110	2.43	10.4
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	27.2	0.234	2.89	0.236	0.376	0.071	24.8	0.105	0.110	2.42	10.2

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	<u>Ni</u>	<u>P</u>	<u>Pb</u>	<u>Pd</u>	<u>Rh</u>	<u>S</u>	<u>Si</u>	<u>Ti</u>	<u>Zn</u>	<u>Zr</u>	
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	3.76	<0.010	0.017	<0.010	<0.100	0.332	0.599	0.020	0.148	0.358	
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	3.74	<0.010	0.021	<0.010	<0.100	0.337	0.595	0.019	0.148	0.357	
SB6-2 SRAT Product (90%)	SB6-2-2711 (A)	09-0167	3.76	<0.010	0.014	<0.010	<0.100	0.325	0.615	0.019	0.147	0.359	
SB6-2 SRAT Product (90%)	SB6-2-2711 (B)	09-0167	3.74	<0.010	0.011	<0.010	<0.100	0.339	0.605	0.019	0.159	0.359	
SB6-3 SRAT Product (120%)	SB6-3-2729 (A)	09-0168	3.75	<0.010	0.016	<0.010	<0.100	0.328	0.583	0.018	0.148	0.353	
SB6-3 SRAT Product (120%)	SB6-3-2729 (B)	09-0168	3.77	<0.010	0.016	<0.010	<0.100	0.346	0.584	0.019	0.154	0.363	
SB6-4 SRAT Product (150%)	SB6-4-2694 (A)	09-0166	3.78	<0.010	0.046	<0.010	<0.100	0.334	0.593	0.019	0.155	0.370	
SB6-4 SRAT Product (150%)	SB6-4-2694 (B)	09-0166	3.78	<0.010	0.047	<0.010	<0.100	0.336	0.593	0.020	0.151	0.371	
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	3.83	<0.100	0.022	<0.010	<0.100	0.314	0.615	0.019	0.152	0.374	
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	3.80	<0.100	0.021	<0.010	<0.100	0.322	0.622	0.019	0.152	0.371	
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	3.79	<0.100	0.021	<0.010	<0.100	0.314	0.625	0.020	0.151	0.371	
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	3.78	<0.100	0.020	<0.010	<0.100	0.313	0.619	0.019	0.149	0.369	
Sample Description	<i>oxide wt% - calcined 1100C</i>		<u>NiO</u>	<u>P2O5</u>	<u>PbO</u>	<u>PdO</u>	<u>RhO2</u>	<u>SO4</u>	<u>SiO2</u>	<u>TiO2</u>	<u>ZnO</u>	<u>ZrO2</u>	Totals
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	4.78		0.018			0.996	1.28	0.033	0.184	0.483	98.5
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	4.75		0.023			1.01	1.27	0.031	0.184	0.482	97.8
SB6-2 SRAT Product (90%)	SB6-2-2711 (A)	09-0167	4.78		0.015			0.976	1.32	0.031	0.182	0.484	98.3
SB6-2 SRAT Product (90%)	SB6-2-2711 (B)	09-0167	4.75		0.012			1.02	1.29	0.031	0.197	0.485	97.9
SB6-3 SRAT Product (120%)	SB6-3-2729 (A)	09-0168	4.76		0.018			0.985	1.25	0.031	0.184	0.477	97.8
SB6-3 SRAT Product (120%)	SB6-3-2729 (B)	09-0168	4.79		0.017			1.04	1.25	0.032	0.191	0.491	98.3
SB6-4 SRAT Product (150%)	SB6-4-2694 (A)	09-0166	4.81		0.049			1.00	1.27	0.032	0.192	0.500	98.1
SB6-4 SRAT Product (150%)	SB6-4-2694 (B)	09-0166	4.80		0.050			1.01	1.27	0.033	0.188	0.501	98.9
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	4.86		0.024			0.942	1.32	0.032	0.189	0.504	98.4
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	4.82		0.023			0.966	1.33	0.032	0.188	0.501	97.6
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	4.82		0.023			0.943	1.34	0.033	0.187	0.501	97.5
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	4.80		0.022			0.938	1.32	0.032	0.185	0.498	97.9

Units: mg/Kg

Sample Description	Sample ID	Lab ID	F	Cl	NO2	NO3	SO4	HCO2	C2O4	PO4
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	<100	395	201	27100	<100	54500	<100	<100
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	<100	392	200	27000	<100	54800	<100	<100
SB6-2 SRAT Product (90%)	SB6-2-2711 (A)	09-0167	<100	361	1940	24600	<100	52900	<100	<100
SB6-2 SRAT Product (90%)	SB6-2-2711 (B)	09-0167	<100	359	1980	24700	<100	52200	<100	<100
SB6-3 SRAT Product (120%)	SB6-3-2729 (A)	09-0168	<100	350	<100	31800	841	57500	<100	<100
SB6-3 SRAT Product (120%)	SB6-3-2729 (B)	09-0168	<100	348	<100	32000	761	57700	<100	<100
SB6-4 SRAT Product (150%)	SB6-4-2694 (A)	09-0166	<100	334	<100	37700	237	67100	<100	<100
SB6-4 SRAT Product (150%)	SB6-4-2694 (B)	09-0166	<100	336	<100	37700	275	67000	<100	<100
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	<100	364	521	24300	<100	52700	<100	<100
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	<100	359	514	24200	<100	52100	<100	<100
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	<100	331	<100	29300	<100	56800	<100	<100
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	<100	330	<100	29400	<100	57000	<100	<100

Weight % Solids Calculations, Density, pH

Sample Description	Sample	Lab ID	Total Solids	Insoluble Solids	Wt % Calcined	Soluble Solids	Density	pH
SB6-1 SRAT Product (100%)	SB6-1-2677 (A)	09-0165	26.1%	14.6%	16.1%	11.5%	1.14	7.55
SB6-1 SRAT Product (100%)	SB6-1-2677 (B)	09-0165	26.1%	14.7%	16.2%	11.5%		
SB6-2 SRAT Product (90%)	SB6-2-2711 (A)	09-0167	26.3%	15.6%	16.6%	10.8%	1.18	7.57
SB6-2 SRAT Product (90%)	SB6-2-2711 (B)	09-0167	26.3%	15.9%	16.5%	10.4%		
SB6-3 SRAT Product (120%)	SB6-3-2729 (A)	09-0168	25.3%	13.0%	15.2%	12.3%	1.19	5.79
SB6-3 SRAT Product (120%)	SB6-3-2729 (B)	09-0168	25.3%	12.9%	15.2%	12.4%		
SB6-4 SRAT Product (150%)	SB6-4-2694 (A)	09-0166	25.9%	14.7%	14.4%	11.3%	1.18	4.64
SB6-4 SRAT Product (150%)	SB6-4-2694 (B)	09-0166	25.8%	14.4%	14.3%	11.3%		
SB6-5 SRAT Product (100%)	SB6-5-2748 (A)	09-0217	25.9%	15.1%	16.2%	10.9%	1.19	6.20
SB6-5 SRAT Product (100%)	SB6-5-2748 (B)	09-0217	25.9%	15.1%	16.2%	10.9%		
SB6-6 SRAT Product (120%)	SB6-6-2765 (A)	09-0218	25.2%	13.4%	15.2%	11.9%	1.18	7.45
SB6-6 SRAT Product (120%)	SB6-6-2765 (B)	09-0218	25.2%	13.3%	15.2%	11.9%		

Table B3
PSAL SME Product Analytical Results

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 4/6/09

Sample ID: SB6-1-2684, SB6-4-2701, SB6-2-2719, SB6-3-2737 (SME Products)

Lab ID: 09-0190 and 09-0193

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	Al	B	Ba	Ca	Ce	Cr	Cu	Fe	K	La	Li	M
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	5.74	1.60	0.085	0.739	0.071	0.105	0.044	6.55	0.095	0.032	2.19	0.
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	5.74	1.52	0.084	0.747	0.071	0.105	0.047	6.56	0.096	0.032	2.22	0.
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	5.65	1.45	0.081	0.749	0.069	0.103	0.047	6.40	0.078	0.031	2.21	0.
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	5.69	1.42	0.081	0.754	0.069	0.104	0.042	6.43	0.076	0.032	2.17	0.
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	5.57	1.44	0.081	0.718	0.068	0.102	0.031	6.29	0.078	0.031	2.24	0.
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	5.60	1.49	0.082	0.729	0.069	0.102	0.030	6.32	0.079	0.031	2.24	0.
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	5.32	1.51	0.078	0.643	0.065	0.098	0.016	6.11	0.097	0.029	2.28	0.
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	5.29	1.49	0.078	0.645	0.065	0.102	0.026	6.04	0.096	0.029	2.31	0.

Sample Description	<i>oxide wt% - calcined 1100C</i>		Al ₂ O ₃	B ₂ O ₃	BaO	CaO	CeO ₂	Cr ₂ O ₃	CuO	Fe ₂ O ₃	K ₂ O	La ₂ O ₃	Li ₂ O	M
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	10.9	5.15	0.095	1.03	0.088	0.153	0.055	9.36	0.114	0.038	4.71	0.
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	10.8	4.89	0.094	1.05	0.087	0.153	0.058	9.37	0.116	0.038	4.77	0.
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	10.7	4.67	0.090	1.05	0.085	0.150	0.059	9.15	0.093	0.037	4.75	0.
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	10.8	4.57	0.091	1.06	0.085	0.152	0.052	9.20	0.091	0.037	4.67	0.
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	10.5	4.64	0.091	1.01	0.084	0.149	0.038	8.99	0.094	0.036	4.82	0.
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	10.6	4.80	0.091	1.02	0.084	0.150	0.038	9.03	0.095	0.036	4.82	0.
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	10.0	4.86	0.087	0.90	0.080	0.144	0.020	8.74	0.116	0.034	4.90	0.
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	10.0	4.80	0.087	0.90	0.079	0.148	0.032	8.64	0.115	0.034	4.97	0.

elemental wt%-calcined 1100C

Sample Description	Sample ID	Lab ID	Na	Ni	P	Pb	Pd	Rh	S	Si	Ti	Zn	Zr
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	9.47	1.40	<0.010	0.055	<0.010	<0.100	0.104	23.0	0.046	0.055	0.224
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	9.47	1.40	<0.010	0.053	<0.010	<0.100	0.107	23.0	0.047	0.059	0.224
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	9.61	1.37	<0.010	0.052	<0.010	<0.100	0.088	23.2	0.045	0.053	0.221
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	9.56	1.39	<0.010	0.052	<0.010	<0.100	0.087	23.2	0.046	0.055	0.223
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	9.35	1.38	<0.010	0.053	<0.010	<0.100	0.090	23.3	0.047	0.054	0.220
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	9.67	1.35	<0.010	0.053	<0.010	<0.100	0.094	23.6	0.046	0.053	0.221
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	9.11	1.32	<0.010	0.047	<0.010	<0.100	0.082	24.1	0.047	0.051	0.217
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	9.09	1.39	<0.010	0.049	<0.010	<0.100	0.078	23.8	0.047	0.051	0.214

Sample Description	<i>oxide wt% - calcined 1100C</i>		Na2O	NiO	P2O5	PbO	PdO	RhO2	SO4	SiO2	TiO2	ZnO	ZrO2
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	12.8	1.77	LTD	0.059	LTD	LTD	0.313	49.2	0.078	0.068	0.302
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	12.8	1.78	LTD	0.057	LTD	LTD	0.320	49.1	0.078	0.073	0.302
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	13.0	1.74	LTD	0.057	LTD	LTD	0.265	49.6	0.076	0.066	0.298
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	12.9	1.76	LTD	0.056	LTD	LTD	0.261	49.8	0.077	0.068	0.300
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	12.6	1.76	LTD	0.057	LTD	LTD	0.270	49.9	0.078	0.066	0.297
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	13.1	1.71	LTD	0.057	LTD	LTD	0.281	50.4	0.077	0.066	0.298
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	12.3	1.68	LTD	0.051	LTD	LTD	0.247	51.5	0.078	0.063	0.293
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	12.3	1.77	LTD	0.053	LTD	LTD	0.235	51.0	0.078	0.064	0.289

Units: mg/Kg

Sample Description	Sample ID	Lab ID	F	Cl	NO2	NO3	HCO2	C2O4	PO4
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	<100	365	<100	23700	49700	<100	<100
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	<100	365	<100	23600	49700	<100	<100
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	<100	333	995	21600	47700	<100	<100
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	<100	314	967	21400	47300	<100	<100
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	<100	328	<100	28400	55800	<100	<100
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	<100	278	<100	28300	54300	<100	<100
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	<100	363	<100	33800	59700	<100	<100
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	<100	342	<100	34200	61800	<100	<100

Weight % Solids Calculations

			Insoluble	Wt %	Soluble				
Sample Description	Sample	Lab ID	Total Solids	Solids	Calcined	Solids		Density	pH
SB6-1 SME Product (100%)	SB6-1-2684 (A)	09-0190	46.8%	36.8%	38.0%	10.0%		1.35	7.67
SB6-1 SME Product (100%)	SB6-1-2684 (B)	09-0190	46.8%	36.9%	38.0%	9.94%			
SB6-2 SME Product (90%)	SB6-2-2719 (A)	09-0192	47.1%	37.9%	38.5%	9.18%		1.37	7.38
SB6-2 SME Product (90%)	SB6-2-2719 (B)	09-0192	47.2%	38.0%	38.6%	9.19%			
SB6-3 SME Product (120%)	SB6-3-2737 (A)	09-0193	46.5%	36.1%	37.0%	10.4%		1.36	5.85
SB6-3 SME Product (120%)	SB6-3-2737 (B)	09-0193	46.3%	36.0%	36.8%	10.4%			
SB6-4 SME Product (150%)	SB6-4-2701 (A)	09-0191	47.3%	37.7%	36.9%	9.59%		1.35	4.97
SB6-4 SME Product (150%)	SB6-4-2701 (B)	09-0191	47.5%	37.8%	37.0%	9.65%			

Table B4
PSAL SRAT Dewater Analytical Results

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 3/26/09, 3/31/2009, 4/13/09

Sample ID: Post SRAT Dewater: SB6-1-2681, SB6-2-2716, SB6-3-2734, SB6-4-2698, SB6-5-2752, SB6-6-2770, Post SME Dewater: SB6-1-2683, SB6-4-2700, SB6-2-2718, SB6-3-2736, MWWT Samples: SB6-1-2680, SB6-2-2715, SB6-3-2733, SB6-4-2697, SB6-5-2750, SB6-6-2768, Post SRAT FAVC Sample: SB6-1-2680, SB6-2-2715, SB6-3-2733, SB6-4-2697, SB6-5-2751, SB6-6-2769, Post SME FAVC: SB6-1-2682, SB6-2-2717, SB6-3-2735, SB6-4-2699

Lab ID: 09-0169 - 09-0188, 09-0219 through 09-0224

Units: mg/L									
<u>Sample ID</u>	<u>Lab ID</u>	<u>F</u>	<u>Cl</u>	<u>NO2</u>	<u>NO3</u>	<u>SO4</u>	<u>HCO2</u>	<u>C2O4</u>	<u>PO4</u>
SRAT Dewater Composite									
SB6-1-2681 (A)	09-0181	<100	<100	275	5390	<100	509	<100	<100
SB6-1-2681 (B)	09-0181	<100	<100	301	5480	<100	523	<100	<100
SB6-2-2716 (A)	09-0185	<100	<100	590	4260	<100	395	<100	<100
SB6-2-2716 (B)	09-0185	<100	<100	576	4090	<100	403	<100	<100
SB6-3-2734 (A)	09-0187	<100	<100	150	5940	<100	1040	<100	<100
SB6-3-2734 (B)	09-0187	<100	<100	159	5940	<100	1050	<100	<100
SB6-4-2698 (A)	09-0183	<100	<100	<100	1660	<100	4620	<100	<100
SB6-4-2698 (B)	09-0183	<100	<100	<100	1680	<100	4680	<100	<100
SB6-5-2752 (A)	09-0223	<100	<100	608	7300	<100	614	<1000	<100
SB6-5-2752 (B)	09-0223	<100	<100	615	7380	<100	652	<1000	<100
SB6-6-2770 (A)	09-0224	<100	<100	289	10300	<100	2200	<1000	<100
SB6-6-2770 (B)	09-0224	<100	<100	289	10200	<100	2190	<1000	<100
SME Dewater Composite									
SB6-1-2683 (A)	09-0182	<100	<100	<100	152	<100	<100	<100	<100
SB6-1-2683 (B)	09-0182	<100	<100	<100	154	<100	<100	<100	<100
SB6-4-2700 (A)	09-0184	<100	<100	<100	<100	<100	3080	<100	<100
SB6-4-2700 (B)	09-0184	<100	<100	<100	<100	<100	3000	<100	<100
SB6-2-2718 (A)	09-0186	<100	<100	<100	634	<100	<100	<100	<100
SB6-2-2718 (B)	09-0186	<100	<100	<100	625	<100	<100	<100	<100
SB6-3-2736 (A)	09-0188	<100	<100	<100	<100	<100	506	<100	<100
SB6-3-2736 (B)	09-0188	<100	<100	<100	<100	<100	499	<100	<100

MWWT Samples									
Units: mg/L									
<u>Sample ID</u>	<u>Lab ID</u>	<u>F</u>	<u>Cl</u>	<u>NO2</u>	<u>NO3</u>	<u>SO4</u>	<u>HCO2</u>	<u>C2O4</u>	<u>PO4</u>
SB6-1-2679 (A)	09-0171	<100	<100	<100	232	<100	<100	<100	
SB6-1-2679 (B)	09-0171	<100	<100	<100	219	<100	<100	<100	
SB6-2-2714 (A)	09-0177	<100	<100	<100	461	<100	<100	NM	<100
SB6-2-2714 (B)	09-0177	<100	<100	<100	373	<100	<100	NM	<100
SB6-4-2696 (A)	09-0174	<100	<100	<100	<100	<100	3000	NM	<100
SB6-4-2696 (B)	09-0174	<100	<100	<100	<100	<100	3080	NM	<100
SB6-3-2732 (A)	09-0180	<100	<100	<100	<100	<100	509	NM	<100
SB6-3-2732 (B)	09-0180	<100	<100	<100	<100	<100	467	NM	<100
SB6-5-2750 (A)	09-0219	<100	<100	<100	232	<100	<100	<1000	<100
SB6-5-2750 (B)	09-0219	<100	<100	<100	243	<100	<100	<1000	<100
SB6-6-2768 (A)	09-0221	<100	<100	<100	181	<100	298	<1000	<100
SB6-6-2768 (B)	09-0221	<100	<100	<100	180	<100	251	<1000	<100
Post SRAT FAVC Sample									
SB6-1-2680 (A)	09-0170	<100	<100	<100	686000	<100	308	NM	<100
SB6-1-2680 (B)	09-0170	<100	<100	<100	685000	<100	269	NM	<100
SB6-2-2715 (A)	09-0176	<100	<100	<100	657000	<100	230	NM	<100
SB6-2-2715 (B)	09-0176	<100	<100	<100	669000	<100	229	NM	<100
SB6-3-2733 (A)	09-0179	<100	<100	<100	633000	<100	240	NM	<100
SB6-3-2733 (B)	09-0179	<100	<100	<100	629000	<100	220	NM	<100
SB6-4-2697 (A)	09-0173	<100	<100	<100	542000	<100	772	NM	<100
SB6-4-2697 (B)	09-0173	<100	<100	<100	528000	<100	779	NM	<100
SB6-5-2751 (A)	09-0220	<100	<100	<100	536000	<100	157	<1000	<100
SB6-5-2751 (B)	09-0220	<100	<100	<100	536000	<100	181	<1000	<100
SB6-6-2769 (A)	09-0222	<100	<100	<100	486000	<100	272	<1000	<100
SB6-6-2769 (B)	09-0222	<100	<100	<100	488000	<100	242	<1000	<100

Units: g/mL		
<u>Sample ID</u>	<u>Lab ID</u>	<u>Density</u>
SRAT Dewater Composite		
SB6-1-2681	09-0181	1.02
SB6-2-2716	09-0185	1.02
SB6-3-2734	09-0187	1.01
SB6-4-2698	09-0183	1.01
SB6-5-2752	09-0223	1.00
SB6-6-2770	09-0224	1.00
SME Dewater Composite		
SB6-1-2683	09-0182	1.01
SB6-2-2718	09-0186	1.01
SB6-3-2736	09-0188	1.01
SB6-4-2700	09-0184	1.01
MWWT Samples		
SB6-1-2679	09-0171	1.09
SB6-2-2714	09-0177	1.01
SB6-3-2732	09-0180	1.01
SB6-4-2696	09-0174	1.01
SB6-5-2750	09-0219	0.998
SB6-6-2768	09-0221	1.20
Post SRAT FAVC Sample		
SB6-1-2680	09-0170	1.24
SB6-2-2715	09-0176	1.23
SB6-3-2733	09-0179	1.22
SB6-4-2697	09-0173	1.19
SB6-5-2751	09-0220	0.998
SB6-6-2769	09-0222	1.19

Table B5
PSAL SRAT Intermediate Slurry Sample Analytical Results

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 4/6/09, 4/14/09

Sample ID: SB6-1-2672, SB6-1-2676, SB6-4-2689, SB6-4-2693, SB6-2-2706, SB6-2-2710, SB6-3-2724, SB6-3-2728 (slurry), SB6-5-2743, SB6-5-2747, SB6-6-2760, and SB6-6-2765

Lab ID: 09-0157 - 09-0164, 09-0225 - 09-0228

Units: mg/Kg

Sample ID	Lab ID	F	Cl	NO2	NO3	SO4	HCO2	C2O4	PO4
Post Formic Slurry Sample (corrected for caustic dilution)									
SB6-1-2672 (A)	09-0157	<108	<108	6,590	19,742	<108	43,800	<108	<108
SB6-1-2672 (B)	09-0157	<108	<108	6,690	19,958	<108	43,584	<108	<108
SB6-2-2706 (A)	09-0161	<108	252.3	8,853	17,793	117	41,517	<108	<108
SB6-2-2706 (B)	09-0161	<108	251.3	8,875	17,577	112	41,517	<108	<108
SB6-3-2724 (A)	09-0163	<107	138.2	4,522	24,538	189	50,362	<107	<107
SB6-3-2724 (B)	09-0163	<107	133.9	4,522	24,967	193	51,219	<107	<107
SB6-4-2689 (A)	09-0159	<108	152.4	<108	32,759	227	69,086	<108	<108
SB6-4-2689 (B)	09-0159	<108	150.3	<108	33,516	280	72,870	<108	<108
30 min prior to SRAT Complete									
SB6-1-2676 (A)	09-0158	<100	379	258	26000	<100	50100	<100	<100
SB6-1-2676 (B)	09-0158	<100	387	259	25500	<100	49600	<100	<100
SB6-2-2710 (A)	09-0162	<100	337	1980	23500	<100	48200	<100	<100
SB6-2-2710 (B)	09-0162	<100	337	1960	23600	<100	49100	<100	<100
SB6-3-2728 (A)	09-0164	<100	314	<100	30200	<100	55500	<100	<100
SB6-3-2728 (B)	09-0164	<100	302	<100	30400	<100	55700	<100	<100
SB6-4-2693 (A)	09-0160	<100	347	<100	41500	440	73700	<100	<100
SB6-4-2693 (B)	09-0160	<100	312	<100	37000	466	70200	<100	<100
SB6-5-2747 (A)	09-0226	<100	362	594	24500	<100	53300	<100	<100
SB6-5-2747 (B)	09-0226	<100	357	583	24700	<100	53500	<100	<100
SB6-6-2765 (A)	09-0228	<100	332	<100	29600	<100	57200	<100	<100
SB6-6-2765 (B)	09-0228	<100	333	<100	31900	<100	57900	<100	<100

Table B6
PSAL SRAT Intermediate Centrifuged Sample Analytical Results

Process Science Analytical Laboratory

Customer: Dan Lambert

Date: 4/2/09, 4/8/09

Sample ID: Mid Formic: SB6-1-2670, SB6-2-2704, SB6-3-2722, SB6-4-2687, SB6-5-2741, SB6-6-2758 (centrifuged supernate)

Sample ID: Post Formic: SB6-1-2671, SB6-2-2705, SB6-3-2723, SB6-4-2688, SB6-5-2742, SB6-6-2759 (centrifuged supernate)

Sample ID: Post Dewater: SB6-1-2673, SB6-2-2707, SB6-3-2725, SB6-4-2690, SB6-5-2744, SB6-6-2761 (centrifuged supernate)

Lab ID: 09-0229-0234

elemental mg/L

<u>Sample ID</u>	<u>Lab ID</u>	<u>Al</u>	<u>Ba</u>	<u>Ca</u>	<u>Cd</u>	<u>Ce</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>K</u>	<u>Mn</u>	<u>Na</u>	<u>Ni</u>
Mid Formic													
SB6-1-2670	09-0145	1.92	0.593	1710	<0.100	<10.0	<0.100	0.152	1.44	293	1150	24900	56.9
SB6-2-2704	09-0151	0.483	0.413	1300	<0.100	<10.0	<0.100	0.133	<0.100	446	1060	25300	51.9
SB6-3-2722	09-0154	1.62	0.335	629	<0.100	<10.0	<0.100	<0.100	1.16	270	503	25200	16.5
SB6-4-2687	09-0148	1.11	0.743	795	<0.100	<10.0	<0.100	<0.100	<0.100	275	1150	25100	48.1
SB6-5-2741	09-0229	0.589	3.41	2020	<0.100	<10.0	<0.100	0.557	<0.100	293	2120	27800	141.0
SB6-6-2758	09-0232	0.605	0.491	675	<0.100	<10.0	<0.100	<0.100	<0.100	277	967	27300	12.2
Post Formic													
SB6-1-2671	09-0146	40.0	2.01	2670	<0.100	<10.0	2.71	13.8	1.94	344	6060	24700	1460
SB6-2-2705	09-0152	49.9	3.28	2640	<0.100	<10.0	2.54	10.0	1.26	656	4910	25200	1250
SB6-3-2723	09-0155	260	2.34	2630	<0.100	<10.0	14.8	28.4	12.09	289	7560	24200	2360
SB6-4-2688	09-0149	861	1.93	2590	<0.100	<10.0	39.2	59.8	63.0	304	9060	23500	3450
SB6-5-2742	09-0230	41.9	6.11	3300	<0.100	<10.0	3.24	15.0	1.62	302	6820	9150	1430
SB6-6-2759	09-0233	149	4.87	3110	<0.100	<10.0	11.5	27.9	7.76	301	8990	26900	2270
Post Dewater													
SB6-1-2673	09-0147	0.548	4.51	3490	<0.100	<10.0	<0.100	0.225	<0.100	598	5350	35900	23.3
SB6-2-2707	09-0153	0.581	4.63	3550	<0.100	<10.0	<0.100	<0.100	<0.100	485	3270	37500	11.1
SB6-3-2725	09-0156	1.30	5.36	3330	<0.100	<10.0	0.301	5.08	<0.100	430	11000	33200	440
SB6-4-2690	09-0150	276	4.66	3340	<0.100	<10.0	24.7	77.0	2160	428	7290	30800	2460
SB6-5-2744	09-0231	1.150	4.31	3490	<0.100	<10.0	<0.100	0.191	0.365	419	6200	34300	41.3
SB6-6-2761	09-0234	3.91	7.51	3520	<0.100	<10.0	0.531	8.72	0.372	383	10700	33100	843

elemental mg/L

<u>Sample ID</u>	<u>Lab ID</u>	<u>P</u>	<u>Pb</u>	<u>Pd</u>	<u>Rh</u>	<u>Ru</u>	<u>Si</u>	<u>Ti</u>	<u>Zn</u>	<u>Zr</u>
Mid Formic										
SB6-1-2670	09-0145	<10.0	<0.100	<1.00	8.88	3.01	21.6	<0.100	<0.100	<1.00
SB6-2-2704	09-0151	<10.0	<0.100	<1.00	9.61	2.32	23.6	<0.100	<0.100	<1.00
SB6-3-2722	09-0154	<10.0	<0.100	<1.00	6.36	<1.00	31.0	<0.100	<0.100	<1.00
SB6-4-2687	09-0148	<10.0	<0.100	<1.00	9.50	1.90	48.1	<0.100	<0.100	<1.00
SB6-5-2741	09-0229	<10.0	<0.100	<1.00	6.94	NM	20.1	<0.100	<0.100	<1.00
SB6-6-2758	09-0232	<10.0	<0.100	<1.00	6.70	NM	17.0	<0.100	<0.100	<1.00
Post Formic										
SB6-1-2671	09-0146	<10.0	1.91	<1.00	12.0	32.0	22.6	<0.100	12.1	<1.00
SB6-2-2705	09-0152	<10.0	0.607	<1.00	11.7	22.4	27.8	<0.100	9.10	<1.00
SB6-3-2723	09-0155	<10.0	5.25	<1.00	18.5	33.8	27.1	<0.100	24.9	<1.00
SB6-4-2688	09-0149	<10.0	0.432	<1.00	0.632	<1.00	62.1	<0.100	4.95	<1.00
SB6-5-2742	09-0230	<10.0	1.93	<1.00	8.65	NM	22.0	<0.100	12.0	<1.00
SB6-6-2759	09-0233	<10.0	5.47	<1.00	15.7	NM	22.9	<0.100	22.6	<1.00
Post Dewater										
SB6-1-2673	09-0147	<10.0	<0.100	<1.00	2.80	<1.00	32.4	<0.100	<0.100	<1.00
SB6-2-2707	09-0153	<10.0	<0.100	<1.00	6.39	<1.00	22.5	<0.100	<0.100	<1.00
SB6-3-2725	09-0156	<10.0	0.432	<1.00	0.632	<1.00	62.1	<0.100	4.95	<1.00
SB6-4-2690	09-0150	<10.0	28.4	<1.00	3.35	38.5	111	<0.100	38.4	<1.00
SB6-5-2744	09-0231	<10.0	<0.100	<1.00	7.07	NM	32.9	<0.100	<0.100	<1.00
SB6-6-2761	09-0234	<10.0	1.50	<1.00	1.06	NM	30.0	<0.100	8.06	<1.00

NM=Not Measured

Density, g/mL

<u>Sample ID</u>	<u>Lab ID</u>	<u>Density, g/mL</u>
Mid Formic		
SB6-1-2670	09-0145	1.05
SB6-2-2704	09-0151	1.05
SB6-3-2722	09-0154	1.05
SB6-4-2687	09-0148	1.05
SB6-5-2741	09-0229	1.06
SB6-6-2758	09-0232	1.05
Post Formic		
SB6-1-2671	09-0146	1.07
SB6-2-2705	09-0152	1.06
SB6-3-2723	09-0155	1.07
SB6-4-2688	09-0149	1.08
SB6-5-2742	09-0230	1.07
SB6-6-2759	09-0233	1.08
Post Dewater		
SB6-1-2673	09-0147	1.08
SB6-2-2707	09-0153	1.08
SB6-3-2725	09-0156	1.09
SB6-4-2690	09-0150	1.09
SB6-5-2744	09-0231	1.08
SB6-6-2761	09-0234	1.09

Table B7
AD Mercury Analytical Results, wt % total solids

Sample Id	User SampleID	Sample Type	LIMS Method	Component	Sample Mass	Result	1 SIGMA % UNC	Units	Rv	Corrected Result
300258542	09_SB6_1_2674	6 hr	CVAA HG (B143)	Hg	2.52	61.2	20	mg/L	1	0.971
300258543	09_SB6_1_2675	Product	CVAA HG (B143)	Hg	1.671	35.7	20	mg/L	1	0.855
300258546	09_SB6_2_2708	6 hr	CVAA HG (B143)	Hg	1.5693	28.6	20	mg/L	1	0.729
300258547	09_SB6_2_2709	Product	CVAA HG (B143)	Hg	2.24	29.4	20	mg/L	1	0.525
300258548	09_SB6_3_2726	6 hr	CVAA HG (B143)	Hg	2.0539	71.2	20	mg/L	1	1.387
300258549	09_SB6_3_2727	Product	CVAA HG (B143)	Hg	1.0827	2.55	20	mg/L	1	0.094
300258544	09_SB6_4_2691	6 hr	CVAA HG (B143)	Hg	1.23	4.18	20	mg/L	1	0.136
300258545	09_SB6_4_2692	Product	CVAA HG (B143)	Hg	1.18	1.89	20	mg/L	1	0.064
300258550	09_SB6_5_2745	6 hr	CVAA HG (B143)	Hg	1.2103	22.2	20	mg/L	1	0.734
300258551	09_SB6_5_2746	Product	CVAA HG (B143)	Hg	1.11	19.8	20	mg/L	1	0.714
300258552	09_SB6_6_2763	6 hr	CVAA HG (B143)	Hg	1.0393	9.02	20	mg/L	1	0.347
300258553	09_SB6_6_2764	Product	CVAA HG (B143)	Hg	1.0342	13.4	20	mg/L	1	0.518

Table B8
AD Ammonium and Carbon Analytical Results on SRAT and SME product

Product?	Sample Id	User SampleID	LIMS Method	Component	Result	1 SIGMA % UNC	Units
SRAT Product	300258554	09 SB6 1 2678	TIC/TOC (B154)	Inorganic Carbon	24.2	10	ug C/mL
SRAT Product	300258558	09 SB6 2 2713	TIC/TOC (B154)	Inorganic Carbon	29.6	10	ug C/mL
SRAT Product	300258560	09 SB6 3 2731	TIC/TOC (B154)	Inorganic Carbon	8.68	10	ug C/mL
SRAT Product	300258556	09 SB6 4 2695	TIC/TOC (B154)	Inorganic Carbon	35.8	10	ug C/mL
SRAT Product	300258562	09 SB6 5 2749	TIC/TOC (B154)	Inorganic Carbon	20.4	10	ug C/mL
SRAT Product	300258554	09 SB6 1 2678	TIC/TOC (B154)	Organic Carbon	16,600	10	ug C/mL
SRAT Product	300258558	09 SB6 2 2713	TIC/TOC (B154)	Organic Carbon	15,100	10	ug C/mL
SRAT Product	300258560	09 SB6 3 2731	TIC/TOC (B154)	Organic Carbon	17,200	10	ug C/mL
SRAT Product	300258556	09 SB6 4 2695	TIC/TOC (B154)	Organic Carbon	15,000	10	ug C/mL
SRAT Product	300258562	09 SB6 5 2749	TIC/TOC (B154)	Organic Carbon	15,300	10	ug C/mL
SRAT Product	300258554	09 SB6 1 2678	TIC/TOC (B154)	Total Carbon	16,600	10	ug C/mL
SRAT Product	300258558	09 SB6 2 2713	TIC/TOC (B154)	Total Carbon	15,100	10	ug C/mL
SRAT Product	300258560	09 SB6 3 2731	TIC/TOC (B154)	Total Carbon	17,200	10	ug C/mL
SRAT Product	300258556	09 SB6 4 2695	TIC/TOC (B154)	Total Carbon	15,000	10	ug C/mL
SRAT Product	300258562	09 SB6 5 2749	TIC/TOC (B154)	Total Carbon	15,300	10	ug C/mL
SME Product	300258564	09 SB6 6 2767	TIC/TOC (B154)	Inorganic Carbon	16.4	10	ug C/mL
SME Product	300258564	09 SB6 6 2767	TIC/TOC (B154)	Organic Carbon	17,500	10	ug C/mL
SME Product	300258564	09 SB6 6 2767	TIC/TOC (B154)	Total Carbon	17,500	10	ug C/mL
SME Product	300258555	09 SB6 1 2685	TIC/TOC (B154)	Inorganic Carbon	21.2	10	ug C/mL
SME Product	300258559	09 SB6 2 2720	TIC/TOC (B154)	Inorganic Carbon	25.7	10	ug C/mL
SME Product	300258561	09 SB6 3 2738	TIC/TOC (B154)	Inorganic Carbon	20.9	10	ug C/mL
SME Product	300258557	09 SB6 4 2702	TIC/TOC (B154)	Inorganic Carbon	78.4	10	ug C/mL
SME Product	300258555	09 SB6 1 2685	TIC/TOC (B154)	Organic Carbon	20,600	10	ug C/mL
SME Product	300258559	09 SB6 2 2720	TIC/TOC (B154)	Organic Carbon	19,700	10	ug C/mL
SME Product	300258561	09 SB6 3 2738	TIC/TOC (B154)	Organic Carbon	19,000	10	ug C/mL
SME Product	300258557	09 SB6 4 2702	TIC/TOC (B154)	Organic Carbon	16,800	10	ug C/mL
SME Product	300258555	09 SB6 1 2685	TIC/TOC (B154)	Total Carbon	20,600	10	ug C/mL
SME Product	300258559	09 SB6 2 2720	TIC/TOC (B154)	Total Carbon	19,700	10	ug C/mL
SME Product	300258561	09 SB6 3 2738	TIC/TOC (B154)	Total Carbon	19,000	10	ug C/mL
SME Product	300258557	09 SB6 4 2702	TIC/TOC (B154)	Total Carbon	16,900	10	ug C/mL
SRAT Product	300258554	09 SB6 1 2678	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SRAT Product	300258558	09 SB6 2 2713	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL

Product?	Sample Id	User SampleID	LIMS Method	Component	Result	1 SIGMA % UNC	Units
SRAT Product	300258560	09_SB6_3_2731	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SRAT Product	300258556	09_SB6_4_2695	IC Cations (B134)	Ammonium Ion	<25 ^{††}	10	ug/mL
SRAT Product	300258562	09_SB6_5_2749	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SME Product	300258555	09_SB6_1_2685	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SME Product	300258559	09_SB6_2_2720	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SME Product	300258561	09_SB6_3_2738	IC Cations (B134)	Ammonium Ion	<25	10	ug/mL
SME Product	300258557	09_SB6_4_2702	IC Cations (B134)	Ammonium Ion	27	10	ug/mL

^{††} Trace ammonium peak present below quant. limits

APPENDIX C. RUN DATA

Data was collected throughout the run with automated equipment to record offgas composition, temperatures, helium and air purge flowrates, acid flowrates, acid cumulative flow, pH, ORP, agitator speed and torque. This data is stored on a server. In addition, the weight of any solution added or removed is recorded to allow mass balances and other checks to be completed for each run. This is completed as an ancillary spreadsheet in the acid calculation spreadsheet. The checks and balances data for each run is reported below.

Table C1	SB6-1 Checks and Balances
Table C2	SB6-2 Checks and Balances
Table C3	SB6-3 Checks and Balances
Table C4	SB6-4 Checks and Balances
Table C5	SB6-5 Checks and Balances
Table C6	SB6-6 Checks and Balances

Table C1 SB6-1 Checks and Balances

SRAT Mass Balance SB6-1	Planned, g	Actual, g	Delta, g
Sludge Simulant (grams)	2,850.00	2,850.00	0.00
AgNO ₃ (grams)	0.0016	0.0016	0.00
HgO (grams)	8.3416	8.3419	0.00
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5329	0.5332	0.00
Coal/Carbon source (grams)	0.0000	0.0000	0.00
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.0703	2.0701	0.00
RuCl ₃ (grams)	0.9378	0.9381	0.00
Water to dilute/rinse trim chemicals (grams)	50.00	50.00	0.00
Sodium Oxalate (grams)	0.0000	0.0000	0.00
Total Slurry (grams)	2,911.88	2,911.88	0.00
Sample Trimmed Sludge (grams)	0.00	0.00	0.00
Slurry Mass after sample (grams)	2,911.88	2,911.88	0.00
SRAT Antifoam (and water) (grams)	46.48	46.46	-0.02
Nitric Acid solution (grams)	64.76	64.60	-0.16
Formic Acid solution (grams)	179.03	178.30	-0.73
Total Dewater (grams)	777.97	777.53	8.42
MWWT Dewater mass (grams)		0.16	
SRAT FAVC Dewater mass (grams)		8.70	
SRAT Sample #1 (grams)		15.44	
SRAT Sample #2 (grams)		14.57	
SRAT Sample #3 (grams)		12.69	
SRAT Sample #4 (grams)		15.30	
SRAT Sample #5 (grams)	250.00	2.52	-0.17
SRAT Sample #6 (grams)		1.67	
SRAT Sample #7 (grams)		13.94	
SRAT Product Sample #1 (grams)		110.18	
SRAT Product Sample #2 (grams)		63.52	
SRAT Product after sampling (grams)		2,102.98	
SRAT Product Mass after sampling (grams)	2,092.16	2,102.98	10.82
Expected Mass Loss (CO ₂ , NO _x , etc., g)	70.90	62.04	
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta
SRAT Product Analysis:			
SRAT Product Total Solids, wt %	25.00	26.10	1.1
SRAT Calcined Solids, wt %	15.81	16.15	0.3
SRAT Mn, wt. % calcined element	6.74	6.47	
SRAT Formate, mg/kg	52,773	54,600	1,827
SRAT Nitrite, mg/kg	0	200	200
SRAT Nitrate, mg/kg	29,057	27,000	-2,057
SRAT Formate Added as acid (best basis, grams)	157.936	157.292	-1
Nitrate Added as acid (best basis, grams)	32.213	32.133	0
Nitrite in Feed (grams)	39.90	39.90	0
Nitrate in Feed & trim chemicals (grams)	25.09	25.09	0
Nitrite in SRAT product (grams)	0.00	0.47	0
Nitrate in SRAT product (grams)	68.06	63.53	

Formate in SRAT product (grams)	126.35	128.46	
SRAT Formate Destruction (grams)	31.587	28.828	-2.8
SRAT Formate Destruction (%) planned/actual	20.0	18.3	-1.7
SRAT Nitrite Destruction (grams)	39.9	39.4	-0.5
SRAT Nitrite Destruction (%)	100.0	98.8	-1.2
Nitrite to Nitrate Conversion (grams)	10.76	6.30	-4.5
Nitrate from nitrite in SRAT product, mol	0.173	0.102	-0.07
Moles of nitrite reacted	0.867	0.857	-0.010
% nitrite conversion to nitrate (SRAT product based)	20.0	11.9	-8.1
Stop at SRAT Product Redox Check:	Planned	Actual	
Predicted SME product mass from forwarded SRAT mass	2361.6	2451.1	
Predicted SME Product Formate, gmol/kg SME slurry	1.054	1.041	
Predicted SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Nitrate, gmol/kg SME slurry	0.330	0.374	
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.004	
Predicted SME Product Mn, gmol/kg SME slurry	0.170	0.163	
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.20	0.211	
SME Mass Balance SB6-1	Planned, g	Actual, g	Delta, g
SRAT Product Mass to SME cycle (grams)	2,092.16	2,102.98	10.82
Calcined solids mass to SME cycle (grams)	330.78	339.63	8.85
Canister Water Addition (grams)	0.00	0.00	0.00
Canister Water Removal (grams)	0.00	0.00	0.00
SME Antifoam Solution Addition (grams)	4.18	4.18	0.00
Frit #1 (grams)	269.85	269.90	0.05
Frit #2 (grams)	269.85	269.90	0.05
Frit Water #1 (grams)	265.80	265.80	0.00
Frit Water #2 (grams)	265.80	265.80	0.00
Formic Acid #1 (grams)	4.05	4.05	0.00
Formic Acid #2 (grams)	4.05	4.05	0.00
SME Dewater #1 (grams)	269.85	263.10	-6.75
SME Dewater #2 (grams)	269.85	265.77	-4.08
Final SME Dewater (grams)	257.75	269.02	11.27
SME FAVC (grams)	0.00	0.76	0.76
Total Dewater (grams)	797.45	798.65	1.20
SME Product Sample #1 (grams)		148.70	
SME Product Sample #2 (grams)		151.54	
SME Product after sampling (grams)		2,007.50	
Potential Total SME Product (no samples removed, g)	2,378.30	2,307.74	-70.56
Expected Mass Loss (CO ₂ , NO _x , etc., g)	0.00	80.27	
Projected SME Product Waste Loading, %	38.00	38.62	
Predicted SME Product Mass (Acid Calc's, g)	2,378.30		
Anion Conversion Balance (SME Cycle)	Planned, g	Actual, g	Delta

SME Product Analysis:			
SME Product Total Solids, wt %	45.00	46.8	2
SME Calcined Solids, wt %	36.60	38	1
SME Manganese, calcined wt %	2.55	2.44	
SME Formate, mg/kg	45,407	49,700	4,293
SME Oxalate, mg/kg	0	0	
SME Nitrate, mg/kg	23,005	23,600	595
SME Nitrite, mg/kg	0	0	
SME Feed formate (grams)	112.86	114.82	
SME Feed nitrate (grams)	60.79	56.78	
SME Formate Added as acid (best basis, grams)	7.13	7.13	
Nitrate in SME product (grams)	54.71	54.46	
Formate in SME product (grams)	107.99	114.69	
SME Formate Destruction (grams)	12.00	7.26	
SME Formate Destruction (%) planned/actual	10.0	6.0	
SME Nitrate Destruction (grams)	6.08	2.32	
SME Nitrate Destruction (%) planned/actual	10.0	4.1	
SME Product Formate, gmol/kg SME slurry	1.009	1.104	
SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
SME Product Coal, gmol/kg SME slurry	0.000	0.000	
SME Product Nitrate, gmol/kg SME slurry	0.371	0.381	
SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
SME Product Manganese, gmol/kg SME slurry	0.170	0.169	
Predicted Fe⁺²/Fe total in glass	0.200	0.230	

Table C2 SB6-2 Checks and Balances

SRAT Mass Balance SB6-2	Planned, g	Actual, g	Delta, g
Sludge Simulant (grams)	2,850.00	2,850.00	0.00
AgNO ₃ (grams)	0.0016	0.0015	0.00
HgO (grams)	8.3416	8.3432	0.00
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5329	0.5340	0.00
Coal/Carbon source (grams)	0.0000	0.0000	0.00
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.0703	2.0713	0.00
RuCl ₃ (grams)	0.9378	0.9321	-0.01
Water to dilute/rinse trim chemicals (grams)	50.00	50.00	0.00
Sodium Oxalate (grams)	0.0000	0.0000	0.00
Total Slurry (grams)	2,911.88	2,911.88	0.00
Sample Trimmed Sludge (grams)	0.00	0.00	0.00
Slurry Mass after sample (grams)	2,911.88	2,911.88	0.00
SRAT Antifoam (and water) (grams)	46.48	46.46	-0.02
Nitric Acid solution (grams)	52.47	52.40	-0.07
Formic Acid solution (grams)	163.51	163.00	-0.51
Total Dewater (grams)	822.59	826.30	24.90
MWWT Dewater mass (grams)		7.48	
SRAT FAVC Dewater mass (grams)		13.71	
SRAT Sample #1 (grams)		15.73	
SRAT Sample #2 (grams)		13.78	
SRAT Sample #3 (grams)		12.99	
SRAT Sample #4 (grams)		14.40	
SRAT Sample #5 (grams)	250.00	1.57	2.41
SRAT Sample #6 (grams)		2.24	
SRAT Sample #7 (grams)		13.99	
SRAT Product Sample #1 (grams)		102.12	
SRAT Product Sample #2 (grams)		75.59	
SRAT Product after sampling (grams)		2,026.93	
SRAT Product Mass after sampling (grams)	2,022.53	2,026.93	4.40
Expected Mass Loss (CO ₂ , NO _x , etc., g)	68.10	46.91	
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta
SRAT Product Analysis:			
SRAT Product Total Solids, wt %	25.00	26.30	1.3
SRAT Calcined Solids, wt %	16.30	15.8	-0.5
SRAT Mn, wt. % calcined element	6.74	6.49	
SRAT Formate, mg/kg	49,674	52,550	2,876
SRAT Nitrite, mg/kg	0	1,960	1,960
SRAT Nitrate, mg/kg	27,259	24,650	-2,609
SRAT Formate Added as acid (best basis, grams)	144.241	143.795	0
Nitrate Added as acid (best basis, grams)	26.102	26.065	0
Nitrite in Feed (grams)	39.90	39.90	0
Nitrate in Feed & trim chemicals (grams)	25.09	25.09	0
Nitrite in SRAT product (grams)	0.00	4.47	4
Nitrate in SRAT product (grams)	61.95	56.19	

SME Product Total Solids, wt %	45.00	47.15	2
SME Calcined Solids, wt %	37.14	38.55	1
SME Manganese, calcined wt %	2.55	2.39	
SME Formate, mg/kg	42,318	47,500	5,182
SME Oxalate, mg/kg	0	0	
SME Nitrate, mg/kg	21,248	21,500	252
SME Nitrite, mg/kg	0	0	
SME Feed formate (grams)	102.70	106.52	
SME Feed nitrate (grams)	55.13	49.96	
SME Formate Added as acid (best basis, grams)	7.10	7.10	
Nitrate in SME product (grams)	49.62	48.29	
Formate in SME product (grams)	98.82	106.69	
SME Formate Destruction (grams)	10.98	6.93	
SME Formate Destruction (%) planned/actual	10.0	6.1	
SME Nitrate Destruction (grams)	5.51	1.67	
SME Nitrate Destruction (%) planned/actual	10.0	3.4	
SME Product Formate, gmol/kg SME slurry	0.940	1.055	
SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
SME Product Coal, gmol/kg SME slurry	0.000	0.000	
SME Product Nitrate, gmol/kg SME slurry	0.343	0.347	
SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
SME Product Manganese, gmol/kg SME slurry	0.172	0.168	
Predicted Fe⁺²/Fe total in glass	0.200	0.244	

Table C3 SB6-3 Checks and Balances

SRAT Mass Balance SB6-3	Planned, g	Actual, g	Delta, g
Sludge Simulant (grams)	2,850.00	2,850.00	0.00
AgNO ₃ (grams)	0.0016	0.0025	0.00
HgO (grams)	8.3416	8.3412	0.00
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5329	0.5393	0.01
Coal/Carbon source (grams)	0.0000	0.0000	0.00
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.0703	2.0719	0.00
RuCl ₃ (grams)	0.9378	0.9381	0.00
Water to dilute/rinse trim chemicals (grams)	50.00	50.00	0.00
Sodium Oxalate (grams)	0.0000	0.0000	0.00
Total Slurry (grams)	2,911.88	2,911.89	0.01
Sample Trimmed Sludge (grams)	0.00	0.00	0.00
Slurry Mass after sample (grams)	2,911.88	2,911.89	0.01
SRAT Antifoam (and water) (grams)	46.48	46.47	-0.01
Nitric Acid solution (grams)	89.39	89.20	-0.19
Formic Acid solution (grams)	210.05	209.40	-0.65
Total Dewater (grams)	688.72	688.40	14.50
MWWT Dewater mass (grams)		0.63	
SRAT FAVC Dewater mass (grams)		14.19	
SRAT Sample #1 (grams)		15.72	
SRAT Sample #2 (grams)		15.98	
SRAT Sample #3 (grams)		14.09	
SRAT Sample #4 (grams)		15.81	
SRAT Sample #5 (grams)	250.00	2.05	11.25
SRAT Sample #6 (grams)		1.08	
SRAT Sample #7 (grams)		14.27	
SRAT Product Sample #1 (grams)		104.84	
SRAT Product Sample #2 (grams)		77.40	
SRAT Product after sampling (grams)		2,230.82	
SRAT Product Mass after sampling (grams)	2,231.48	2,230.82	-0.66
Expected Mass Loss (CO ₂ , NO _x , etc., g)	76.50	75.95	
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta
SRAT Product Analysis:			
SRAT Product Total Solids, wt %	25.00	25.30	0.3
SRAT Calcined Solids, wt %	14.92	15.2	0.3
SRAT Mn, wt. % calcined element	6.74	6.26	
SRAT Formate, mg/kg	58,441	57,600	-841
SRAT Nitrite, mg/kg	0	0	0
SRAT Nitrate, mg/kg	32,364	31,900	-464
SRAT Formate Added as acid (best basis, grams)	185.303	184.727	-1
Nitrate Added as acid (best basis, grams)	44.466	44.370	0
Nitrite in Feed (grams)	39.90	39.90	0
Nitrate in Feed & trim chemicals (grams)	25.09	25.09	0
Nitrite in SRAT product (grams)	0.00	0.00	0
Nitrate in SRAT product (grams)	80.31	79.50	

Formate in SRAT product (grams)	148.24	143.54	
SRAT Formate Destruction (grams)	37.061	41.185	4.1
SRAT Formate Destruction (%) planned/actual	20.0	22.3	2.3
SRAT Nitrite Destruction (grams)	39.9	39.9	0.0
SRAT Nitrite Destruction (%)	100.0	100.0	0.0
Nitrite to Nitrate Conversion (grams)	10.76	10.04	-0.7
Nitrate from nitrite in SRAT product, mol	0.173	0.162	-0.01
Moles of nitrite reacted	0.867	0.867	0.000
% nitrite conversion to nitrate (SRAT product based)	20.0	18.7	-1.3
Stop at SRAT Product Redox Check:	Planned	Actual	
Predicted SME product mass from forwarded SRAT mass	2447.1	2483.6	
Predicted SME Product Formate, gmol/kg SME slurry	1.202	1.149	
Predicted SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Nitrate, gmol/kg SME slurry	0.378	0.462	
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Mn, gmol/kg SME slurry	0.165	0.156	
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.200	0.173	
SME Mass Balance SB6-3	Planned, g	Actual, g	Delta, g
SRAT Product Mass to SME cycle (grams)	2,231.48	2,230.82	-0.66
Calcined solids mass to SME cycle (grams)	333.00	339.08	6.08
Canister Water Addition (grams)	0.00	0.00	0.00
Canister Water Removal (grams)	0.00	0.00	0.00
SME Antifoam Solution Addition (grams)	4.46	4.46	0.00
Frit #1 (grams)	271.66	271.70	0.04
Frit #2 (grams)	271.66	271.70	0.04
Frit Water #1 (grams)	267.59	267.60	0.01
Frit Water #2 (grams)	267.59	267.60	0.01
Formic Acid #1 (grams)	4.07	4.07	0.00
Formic Acid #2 (grams)	4.07	4.07	0.00
SME Dewater #1 (grams)	271.66	273.06	1.40
SME Dewater #2 (grams)	271.66	279.20	7.54
Final SME Dewater (grams)	315.38	306.46	-8.92
SME FAVC (grams)	0.00	1.86	1.86
Total Dewater (grams)	858.70	860.58	1.88
SME Product Sample #1 (grams)		145.88	
SME Product Sample #2 (grams)		140.62	
SME Product after sampling (grams)		2,109.20	
Potential Total SME Product (no samples removed, g)	2,463.88	2,395.70	-68.18
Expected Mass Loss (CO ₂ , NO _x , etc., g)	0.00	65.74	
Projected SME Product Waste Loading, %	38.00	38.42	
Predicted SME Product Mass (Acid Calc's, g)	2,463.88		
Anion Conversion Balance (SME Cycle)	Planned, g	Actual, g	Delta

SME Product Analysis:			
SME Product Total Solids, wt %	45.00	46.4	1
SME Calcined Solids, wt %	35.57	36.9	1
SME Manganese, calcined wt %	2.55	2.37	
SME Formate, mg/kg	51,315	55,050	3,735
SME Oxalate, mg/kg	0	0	
SME Nitrate, mg/kg	26,380	28,350	1,970
SME Nitrite, mg/kg	0	0	
SME Feed formate (grams)	133.31	128.50	
SME Feed nitrate (grams)	72.22	71.16	
SME Formate Added as acid (best basis, grams)	7.18	7.17	
Nitrate in SME product (grams)	65.00	67.92	
Formate in SME product (grams)	126.43	131.88	
SME Formate Destruction (grams)	14.05	3.78	
SME Formate Destruction (%) planned/actual	10.0	2.8	
SME Nitrate Destruction (grams)	7.22	3.24	
SME Nitrate Destruction (%) planned/actual	10.0	4.6	
SME Product Formate, gmol/kg SME slurry	1.140	1.223	
SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
SME Product Coal, gmol/kg SME slurry	0.000	0.000	
SME Product Nitrate, gmol/kg SME slurry	0.425	0.457	
SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
SME Product Manganese, gmol/kg SME slurry	0.165	0.159	
Predicted Fe⁺²/Fe total in glass	0.200	0.205	

Table C4 SB6-4 Checks and Balances

SRAT Mass Balance SB6-4	Planned, g	Actual, g	Delta, g
Sludge Simulant (grams)	2,850.00	2,850.00	0.00
AgNO ₃ (grams)	0.0016	0.0018	0.00
HgO (grams)	8.3416	8.3415	0.00
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5329	0.5333	0.00
Coal/Carbon source (grams)	0.0000	0.0000	0.00
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.0703	2.0702	0.00
RuCl ₃ (grams)	0.9378	0.9381	0.00
Water to dilute/rinse trim chemicals (grams)	50.00	50.00	0.00
Sodium Oxalate (grams)	0.0000	0.0000	0.00
Total Slurry (grams)	2,911.88	2,911.88	0.00
Sample Trimmed Sludge (grams)	0.00	0.00	0.00
Slurry Mass after sample (grams)	2,911.88	2,911.88	0.00
ARP Simulant	0.00	0.00	0.00
Nitric Acid solution (grams)	126.27	126.00	-0.27
Formic Acid solution (grams)	256.61	256.00	-0.61
Total Dewater (grams)	554.85	555.80	9.57
MWWT Dewater mass (grams)		-4.67	
SRAT FAVC Dewater mass (grams)		13.30	
SRAT Sample #1 (grams)		15.30	
SRAT Sample #2 (grams)		14.57	
SRAT Sample #3 (grams)		14.00	
SRAT Sample #4 (grams)		16.38	
SRAT Sample #5 (grams)	250.00	1.23	0.01
SRAT Sample #6 (grams)		1.18	
SRAT Sample #7 (grams)		13.29	
SRAT Product Sample #1 (grams)		98.84	
SRAT Product Sample #2 (grams)		75.22	
SRAT Product after sampling (grams)		2,449.65	
SRAT Product Mass after sampling (grams)	2,440.39	2,449.65	9.26
Expected Mass Loss (CO ₂ , NO _x , etc., g)	84.89	89.56	
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta
SRAT Product Analysis:			
SRAT Product Total Solids, wt %	25.00	25.80	0.8
SRAT Calcined Solids, wt %	13.76	14.3	0.5
SRAT Mn, wt. % calcined element	6.74	6.49	
SRAT Formate, mg/kg	65,852	67,050	1,198
SRAT Nitrite, mg/kg	0	0	0
SRAT Nitrate, mg/kg	36,670	37,700	1,030
SRAT Formate Added as acid (best basis, grams)	226.379	225.837	-1
Nitrate Added as acid (best basis, grams)	62.811	62.675	0
Nitrite in Feed (grams)	39.90	39.90	0
Nitrate in Feed & trim chemicals (grams)	25.09	25.09	0
Nitrite in SRAT product (grams)	0.00	0.00	0
Nitrate in SRAT product (grams)	98.66	101.78	

Formate in SRAT product (grams)	181.10	181.01	
SRAT Formate Destruction (grams)	45.276	44.825	-0.5
SRAT Formate Destruction (%) planned/actual	20.0	19.8	-0.2
SRAT Nitrite Destruction (grams)	39.9	39.9	0.0
SRAT Nitrite Destruction (%)	100.0	100.0	0.0
Nitrite to Nitrate Conversion (grams)	10.76	14.01	3.3
Nitrate from nitrite in SRAT product, mol	0.173	0.226	0.05
Moles of nitrite reacted	0.867	0.867	0.000
% nitrite conversion to nitrate (SRAT product based)	20.0	26.1	6.1
Stop at SRAT Product Redox Check:	Planned	Actual	
Predicted SME product mass from forwarded SRAT mass	2573.7	2674.6	
Predicted SME Product Formate, gmol/kg SME slurry	1.409	1.364	
Predicted SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Nitrate, gmol/kg SME slurry	0.446	0.557	
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Mn, gmol/kg SME slurry	0.158	0.155	
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.200	0.167	
SME Mass Balance SB6-4	Planned, g	Actual, g	Delta, g
SRAT Product Mass to SME cycle (grams)	2,440.39	2,449.65	9.26
Calcined solids mass to SME cycle (grams)	335.90	350.30	14.40
Canister Water Addition (grams)	0.00	0.00	0.00
Canister Water Removal (grams)	0.00	0.00	0.00
SME Antifoam Solution Addition (grams)	4.88	4.88	0.00
Frit #1 (grams)	274.02	274.00	-0.02
Frit #2 (grams)	274.02	274.00	-0.02
Frit Water #1 (grams)	269.91	269.90	-0.01
Frit Water #2 (grams)	269.91	269.90	-0.01
Formic Acid #1 (grams)	4.11	4.11	0.00
Formic Acid #2 (grams)	4.11	4.11	0.00
SME Dewater #1 (grams)	274.02	281.97	7.95
SME Dewater #2 (grams)	274.02	274.01	-0.01
Final SME Dewater (grams)	402.68	394.78	-7.90
SME FAVC (grams)	0.00	1.57	1.57
Total Dewater (grams)	950.73	952.33	1.60
SME Product Sample #1 (grams)		151.13	
SME Product Sample #2 (grams)		153.16	
SME Product after sampling (grams)		2,227.90	
Potential Total SME Product (no samples removed, g)	2,590.64	2,532.19	-58.45
Expected Mass Loss (CO ₂ , NO _x , etc., g)	0.00	66.03	
Projected SME Product Waste Loading, %	38.00	39.00	
Predicted SME Product Mass (Acid Calc's, g)	2,590.64		
Anion Conversion Balance (SME Cycle)	Planned, g	Actual, g	Delta

SME Product Analysis:			
SME Product Total Solids, wt %	45.00	47.4	2
SME Calcined Solids, wt %	34.12	36.9	3
SME Manganese, calcined wt %	2.55	2.29	
SME Formate, mg/kg	59,584	60,750	1,166
SME Oxalate, mg/kg	0	0	
SME Nitrate, mg/kg	31,089	34,000	2,911
SME Nitrite, mg/kg	0	0	
SME Feed formate (grams)	164.27	164.25	
SME Feed nitrate (grams)	89.49	92.35	
SME Formate Added as acid (best basis, grams)	7.24	7.24	
Nitrate in SME product (grams)	80.54	86.09	
Formate in SME product (grams)	154.36	153.83	
SME Formate Destruction (grams)	17.15	17.66	
SME Formate Destruction (%) planned/actual	10.0	10.3	
SME Nitrate Destruction (grams)	8.95	6.26	
SME Nitrate Destruction (%) planned/actual	10.0	6.8	
SME Product Formate, gmol/kg SME slurry	1.324	1.350	
SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
SME Product Coal, gmol/kg SME slurry	0.000	0.000	
SME Product Nitrate, gmol/kg SME slurry	0.501	0.548	
SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
SME Product Manganese, gmol/kg SME slurry	0.158	0.154	
Predicted Fe⁺²/Fe total in glass	0.200	0.169	

Table C5 SB6-5 Checks and Balances

SRAT Mass Balance SB6-5	Planned, g	Actual, g
Sludge Simulant (grams)	2,589.40	2,589.40
AgNO ₃ (grams)	0.0017	0.0019
HgO (grams)	8.5210	8.5226
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5444	0.5474
Coal/Carbon source (grams)	0.0000	0.0000
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.1148	2.1206
RuCl ₃ (grams)	0.9579	0.4546
Water to dilute/rinse trim chemicals (grams)	50.00	50.00
Sodium Oxalate (grams)	0.0000	0.0000
Total Slurry (grams)	2,651.54	2,651.05
Sample Trimmed Sludge (grams)	0.00	0.00
Slurry Mass after sample (grams)	2,651.54	2,651.05
SRAT Antifoam (and water) (grams)	42.23	42.25
Nitric Acid solution (grams)	63.09	62.20
Formic Acid solution (grams)	181.32	180.30
Total Dewater (grams)	462.89	462.60
MWWT Dewater mass (grams)		-0.36
SRAT FAVC Dewater mass (grams)		16.95
SRAT Sample #1 (grams)	250.00	14.77
SRAT Sample #2 (grams)		15.03
SRAT Sample #3 (grams)		13.31
SRAT Sample #4 (grams)		15.23
SRAT Sample #5 (grams)		1.21
SRAT Sample #6 (grams)		1.11
SRAT Sample #7 (grams)		15.60
SRAT Product Sample #1 (grams)		263.19
SRAT Product Sample #2 (grams)		75.85
SRAT Product after sampling (grams)		
SRAT Product Mass after sampling (grams)	2,145.00	1,939.45
Expected Mass Loss (CO ₂ , NO _x , etc., g)	69.42	117.46
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g
SRAT Product Analysis:		
SRAT Product Total Solids, wt %	25.00	25.90
SRAT Calcined Solids, wt %	15.96	16.2
SRAT Mn, wt. % calcined element	6.98	6.66
SRAT Formate, mg/kg	52,268	52,400
SRAT Nitrite, mg/kg	0	518
SRAT Nitrate, mg/kg	26,953	24,250
SRAT Formate Added as acid (best basis, grams)	159.953	159.056
Nitrate Added as acid (best basis, grams)	31.384	30.940
Nitrite in Feed (grams)	35.15	35.15
Nitrate in Feed & trim chemicals (grams)	23.69	23.69
Nitrite in SRAT product (grams)	0.00	1.22
Nitrate in SRAT product (grams)	64.55	57.10

Formate in SRAT product (grams)	127.96	123.39
SRAT Formate Destruction (grams)	31.991	35.667
SRAT Formate Destruction (%) planned/actual	20.0	22.4
SRAT Nitrite Destruction (grams)	35.2	33.9
SRAT Nitrite Destruction (%)	100.0	96.5
Nitrite to Nitrate Conversion (grams)	9.47	2.47
Nitrate from nitrite in SRAT product, mol	0.153	0.040
Moles of nitrite reacted	0.764	0.738
% nitrite conversion to nitrate (SRAT product based)	20.0	5.4
Stop at SRAT Product Redox Check:	Planned	Actual
Predicted SME product mass from forwarded SRAT mass	2432.8	2255.4
Predicted SME Product Formate, gmol/kg SME slurry	1.106	1.001
Predicted SME Product Oxalate, gmol/kg SME slurry	0.000	0.000
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000
Predicted SME Product Nitrate, gmol/kg SME slurry	0.381	0.336
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.010
Predicted SME Product Mn, gmol/kg SME slurry	0.177	0.169
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.200	0.223

Table C6 SB6-6 Checks and Balances

SRAT Mass Balance SB6-6	Planned, g	Actual, g	Delta, g
Sludge Simulant (grams)	2,535.30	2,535.30	0.00
AgNO ₃ (grams)	0.0016	0.0018	0.00
HgO (grams)	8.3430	8.3488	0.01
Pd(NO ₃) ₂ *H ₂ O (grams solution)	0.5330	0.5310	0.00
Coal/Carbon source (grams)	0.0000	0.0000	0.00
Rh(NO ₃) ₃ *2H ₂ O (grams solution)	2.0706	2.0932	0.02
RuCl ₃ (grams)	0.9379	0.9378	0.00
Water to dilute/rinse trim chemicals (grams)	50.00	50.00	0.00
Sodium Oxalate (grams)	0.0000	0.0000	0.00
Total Slurry (grams)	2,597.19	2,597.21	0.03
Sample Trimmed Sludge (grams)	0.00	0.00	0.00
Slurry Mass after sample (grams)	2,597.19	2,597.21	0.03
SRAT Antifoam (and water) (grams)	41.35	41.35	0.00
Nitric Acid solution (grams)	86.05	85.30	-0.75
Formic Acid solution (grams)	208.15	207.70	-0.45
Total Dewater (grams)	366.20	365.70	15.30
MWWT Dewater mass (grams)		0.20	
SRAT FAVC Dewater mass (grams)		15.60	
SRAT Sample #1 (grams)		14.69	
SRAT Sample #2 (grams)		14.54	
SRAT Sample #3 (grams)		12.62	
SRAT Sample #4 (grams)		16.17	
SRAT Sample #5 (grams)	250.00	1.04	178.86
SRAT Sample #6 (grams)		1.03	
SRAT Sample #7 (grams)		14.32	
SRAT Product Sample #1 (grams)		275.68	
SRAT Product Sample #2 (grams)		78.77	
SRAT Product after sampling (grams)		2,006.72	
SRAT Product Mass after sampling (grams)	2,232.40	2,006.72	-225.68
Expected Mass Loss (CO ₂ , NO _x , etc., g)	73.49	128.80	
Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta
SRAT Product Analysis:			
SRAT Product Total Solids, wt %	25.00	25.20	0.2
SRAT Calcined Solids, wt %	15.08	15.2	0.1
SRAT Mn, wt. % calcined element	6.98	6.52	
SRAT Formate, mg/kg	57,891	56,900	-991
SRAT Nitrite, mg/kg	0	0	0
SRAT Nitrate, mg/kg	30,325	29,350	-975
SRAT Formate Added as acid (best basis, grams)	183.628	183.228	0
Nitrate Added as acid (best basis, grams)	42.802	42.430	0
Nitrite in Feed (grams)	34.42	34.42	0
Nitrate in Feed & trim chemicals (grams)	23.20	23.20	0
Nitrite in SRAT product (grams)	0.00	0.00	0
Nitrate in SRAT product (grams)	75.28	71.48	

Formate in SRAT product (grams)	146.90	138.58	
SRAT Formate Destruction (grams)	36.726	44.643	7.9
SRAT Formate Destruction (%) planned/actual	20.0	24.4	4.4
SRAT Nitrite Destruction (grams)	34.4	34.4	0.0
SRAT Nitrite Destruction (%)	100.0	100.0	0.0
Nitrite to Nitrate Conversion (grams)	9.28	5.86	-3.4
Nitrate from nitrite in SRAT product, mol	0.150	0.094	-0.06
Moles of nitrite reacted	0.748	0.748	0.000
% nitrite conversion to nitrate (SRAT product based)	20.0	12.6	-7.4
Stop at SRAT Product Redox Check:	Planned	Actual	
Predicted SME product mass from forwarded SRAT mass	2460.4	2229.7	
Predicted SME Product Formate, gmol/kg SME slurry	1.250	1.138	
Predicted SME Product Oxalate, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Nitrate, gmol/kg SME slurry	0.441	0.426	
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.000	
Predicted SME Product Mn, gmol/kg SME slurry	0.172	0.162	
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.200	0.200	

Distribution List

S. L. Marra	773-A	M. T. Keefer	766-H
A. B. Barnes	999-W	J. E. Occhipinti	704-S
D. A. Crowley	999-W	D. C. Sherburne	704-S
C. C. Herman	999-W	R. N. Hinds	704-S
N. E. Bibler	773-A	R. T. McNew	704-27S
C. M. Jantzen	773-A	J. W. Ray	704-S
B. J. Giddings	786-5A	J. F. Iaukea	704-30S
J. P. Vaughan	773-41A	H. H. Elder	704-24S
S. R. Loflin	773-41A	H. B. Shah	766-H
J. M. Pareizs	773-A	J. M. Gillam	766-H
C. J. Bannochie	773-42A	D. D. Larsen	766-H
D. K. Peeler	999-W	B. A. Hamm	766-H
M. E. Stone	999-W	D. R. Click	773-A
D. C. Koopman	773-42A	B. N. Attaway	773-A
B. R. Pickenheim	999-W	S. L. Beard	773-A
F. M. Pennebaker	773-42A	L. M. Chandler	773-A
S. D. Fink	773-A	M. J. Barnes	773-A
C. W. Gardner	773-A	L. H. Connelly	773-A
R. H. Young	773-A	C. M. Gregory	773-A
D. P. Lambert	773-A	L. W. Brown	773-A
T. L. Fellingner	704-26S	J. M. Bricker	704-27S