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Revision 0

SLUDGE BATCH 4 SIMULANT FLOWSHEET STUDIES: PHASE II RESULTS

M. E. Stone
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Process Science and Engineering Section
Savannah River National Laboratory
Aiken, SC 29808

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REVIEWS AND APPROVALS

AUTHORS:

M. E. Stone, Process Engineering Technology Date

D. R. Best, Process Engineering Technology Date

TECHNICAL REVIEWER:

D. P. Lambert, Process Chemistry Science Date

APPROVERS:

C. C. Herman, Manager, Process Engineering Technology Date

R. E. Edwards, Manager, Process Science and Engineering Date

J. E. Occhipinti, Manager, Date
Process Cognizant Engineering - Waste Solidification Engineering

EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) will transition from Sludge Batch 3 (SB3) processing to Sludge Batch 4 (SB4) processing in early fiscal year 2007. Tests were conducted using non-radioactive simulants of the expected SB4 composition to determine the impact of varying the acid stoichiometry during the Sludge Receipt and Adjustment Tank (SRAT) process. The work was conducted to meet the Technical Task Request (TTR) HLW/DWPF/TTR-2004-0031 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. Initial SB4 flowsheet studies were conducted to guide decisions during the sludge batch preparation process. These studies were conducted with the estimated SB4 composition at the time of the study. The composition has changed slightly since these studies were completed due to changes in the sludges blended to prepare SB4 and the estimated SB3 heel mass. The SB4 simulant used in this testing was based on Case 15C Blend 1.

No significant processing issues with processing SB4 were noted during the run.

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

Hydrogen generation was significantly impacted by the changes in acid stoichiometry from 130% to 170% (1.39 to 1.82 mole acid per liter of sludge), but all generation rates were within process limits.

- 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 1.0 wt% (solids basis) based on the expected composition of the SB4 blend. Acid quantities from 130% to 170% resulted in satisfactory mercury removal with 12 hours of reflux boiling. Mercury accumulation was noted on the agitator shaft and impellers during the 130% acid run. This material would not be transferred to the SME cycle, therefore it does not represent failure to remove mercury. However, accumulation of deposits on processing equipment is not desirable and discussions concerning whether or not the accumulation on the impellers would continue or the mercury would slough off during subsequent processing would be highly speculative.

- Acid quantities and processing times required for nitrite destruction

Acid quantities from 130% to 170% resulted in satisfactory nitrite destruction with 12 hours of reflux boiling. 130% probably represents the lower end of the window since there was still a small amount of nitrite present but was less than 1000 mg/kg.

- Impact of SB4 composition (in particular, manganese, nickel, mercury, and aluminum) on DWPF processing (i.e. acid addition strategy, foaming, hydrogen generation, REDOX control, rheology, etc.)

Acid quantities from 130% to 170% resulted in satisfactory process performance with no significant issues noted. Foaming was noted during formic acid addition, but lab-scale operations did not utilize an antifoam addition between the nitric and formic acid additions. Addition of

antifoam equal to the amount added at DWPF between the acid additions was sufficient to control foaming. Increased solubility of Mn and Ni were noted as acid stoichiometry increased.

Except for the 150% run, all SRAT products were outside the process limits for yield stress with the lowest acid (130%) being above the process limit and the 160% and 170% runs being below the process limit. The process limits for SME product yield stress were met for the 150% acid run at 47% solids, but the 130% acid run was above process limits and the 160% and 170% runs were slightly below process limits. The 150% acid run exceeded the upper limit for SME product yield stress when concentrated to 50 wt% solids. It should be noted that the trend seen in rheological properties of the simulants are expected to be similar for the DWPF process slurries, but the absolute values for the simulants are not expected to be prototypical in yield stress or consistency. Adjustment in the solids concentration targets and/or acid stoichiometry should be made if processing problems due to viscous process slurries are noted in DWPF.

The pH of the condensate generated was typically acidic, but the 130% acid run resulted in condensate that was basic before the end of the SRAT cycle and throughout the SME cycle with a pH of approximately 9. All condensates from all other runs had a pH of less than 4.

Measured REDOX values for all runs were significantly below the predicted values. REDOX values increased slightly as acid stoichiometry was increased. The issues with REDOX using SB4 simulants have been evaluated in a separate study and will be documented in a separate report.

The following preliminary recommendations apply for DWPF SB4 processing:

- An acid stoichiometry of 150% is recommended for initial SB4 processing with a corresponding acid window of 130% to 170%. The SB4 simulant used during the testing had a stoichiometric acid requirement of 1.07 mol/L, giving an acid addition of 1.60 mol/L at 150% acid.

- The manganese term in the electron equivalence REDOX model should be changed from a coefficient of "2" to a coefficient of "5" for SB4 processing. This recommendation and basis will be documented in a separate report.

- No changes to the antifoam addition strategy, acid addition rate, reflux time, or SME solids targets are recommended.

The recommendation for acid addition during the Shielded Cells processing studies will be finalized once the acid inputs are determined. Final recommendations to DWPF on SB4 processing will be made after the Shielded Cells testing and will be based on the results of this study and the Shielded Cells test.

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ACTL	Aiken County Technologies Laboratory
AD	Analytical Development
ASP	Analytical Study Plan
CPC	Chemical Process Cell
CS	Calcine Solids
DWPF	Defense Waste Processing Facility
FAVC	Formic Acid Vent Condenser
GC	Gas Chromatograph
HLW	High Level Waste
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
IS	Insoluble Solids
MWWT	Mercury Water Wash Tank
PSAL	Process Science Analytical Laboratory
PSE	Process Science and Engineering Section
QA	Quality Assurance
REDOX	REDuction / OXidation potential
SB3	Sludge Batch 3
SB4	Sludge Batch 4
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	Sludge Receipt and Adjustment Tank
SS	Soluble Solids
TIC	Total Inorganic Carbon
TS	Total Solids
TT&QAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request

1.0 Introduction and Background

The Defense Waste Processing Facility (DWPF) will transition from Sludge Batch 3 (SB3) processing to Sludge Batch 4 (SB4) processing in early fiscal year 2007. Tests were conducted using non-radioactive simulants of the expected SB4 composition to determine the impact of varying the acid stoichiometry during the Sludge Receipt and Adjustment Tank (SRAT) process. The work was conducted to meet the Technical Task Request (TTR) HLW/DWPF/TTR-2004-0031[1] and followed the guidelines of a Task Technical and Quality Assurance Plan (TT&QAP)[2].

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. Initial SB4 flowsheet studies were conducted to guide decisions during the sludge batch preparation process[3,4]. These studies were conducted with the estimated SB4 composition at the time of the study. The composition has changed slightly since these studies were completed due to changes in the sludges blended to prepare SB4 and the estimated SB3 heel mass.

The following TTR requirements were addressed in this testing:

- Hydrogen and nitrous oxide generation rates as a function of acid stoichiometry
- Acid quantities and processing times required for mercury removal
- Acid quantities and processing times required for nitrite destruction
- Impact of SB4 composition (in particular, oxalate, manganese, nickel, mercury, and aluminum) on DWPF processing (i.e. acid addition strategy, foaming, hydrogen generation, REDOX control, rheology, etc.)

2.0 Approach

Four SRAT/SME runs were completed during this study using acid stoichiometries of 130%, 150%, 160% and 170%. 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.

2.1 Simulant Preparation

The simulant was based on the composition estimate for the 15C Blend 1 option[5] in January 2006. Simulant was prepared by blending the Spintek simulant[6] with precursors-based simulant[3]. Composition of the simulant is shown in Table 1. The simulant matches the expected SB4 composition to within 15% for major species and was deemed to be acceptable for testing based on engineering judgment. The composition estimates for SB4 were revised in June 2006[a], but the revised compositions did not vary from the earlier estimates sufficiently to warrant a new recipe for the sludge simulant.

Table 1. Simulant Composition for SB4 Flowsheet Testing

Elemental	Wt% calcined solids	June 2006 Estimates	Solids Data	Wt %
Al	14.31	12.60	Total	17.36
Ba	0.16	0.11	Insoluble	11.92
Ca	1.85	1.68	Soluble	5.45
Cr	0.14	0.14	Calcined	12.41
Cu	0.06	0.05	**Anions**	**mg/Kg**
Fe	17.85	18.34	Chloride	141
K	0.25	0.27	Nitrite	14495
Mg	1.37	1.50	Nitrate	9331
Mn	4.05	4.19	Formate	<100
Na	15.15	17.76	Sulfate	1537
Ni	1.39	1.22	Oxalate	622
P	0.03	n/a	Phosphate	<100
Pb	<0.020	0.08	Carbonate	1,157
S	0.38	n/a	**Other Results**	
Si	1.75	1.86	Base Equivalents (molar)	0.443
Ti	0.03	0.02	Density (g/ml)	1.138
Zn	0.17	0.08	pH	12.75
Zr	0.21	0.17	**Rheological Properties**	
			Yield Stress (Pa)	8.82
			Consistency (cP)	7.3

Noble metals, mercury, and rinse water were added to the sludge simulant prior to performing the SRAT cycle. Samples were not taken after the additions as the amount of these additions is small compared to the sludge. The noble metal concentrations were based on 125% of the estimated

[a] E-mail message from H. B. Shah to C. C. Herman dated 6/22/06.

amount in the sludge batch[7] after transfer onto the heel of SB3. The concentrations of each trim chemical added are shown in Table 2.

Table 2. Trim Chemical Additions

Trim Chemical	Wt% in Total Solids
Hg	1.0
Ag	0.0112
Pd	0.0015
Rh	0.0108
Ru	0.0493

2.2 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-2006-00074[8]. 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.

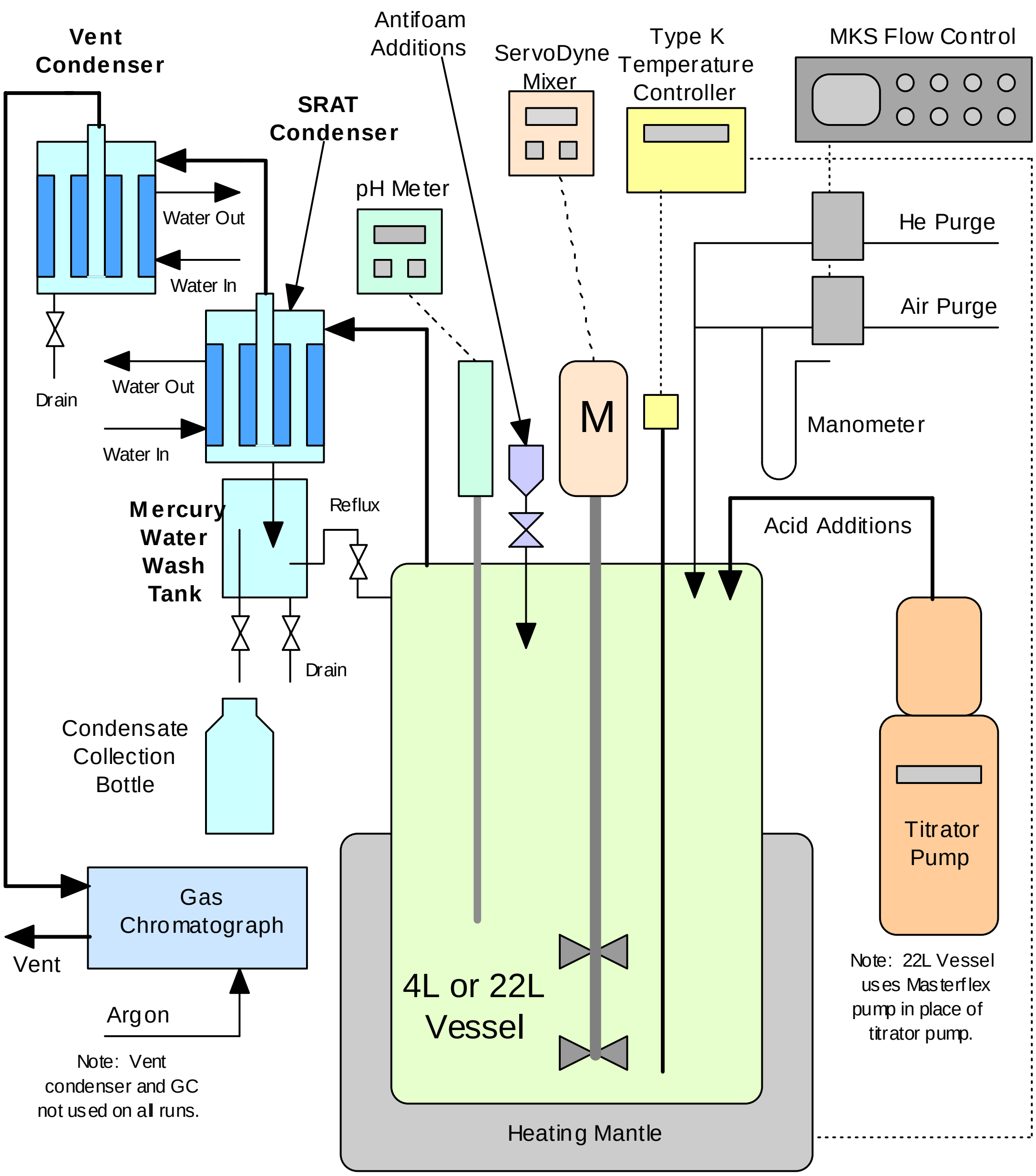

Figure 1. Schematic of SRAT Equipment Set-Up

SRAT and SME processing parameters are summarized in Appendix A. The flowsheet runs were performed using the guidance of Procedure ITS-0094 ("Laboratory Scale Chemical Process Cell Simulations") of Manual L29[9]. 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 in laboratory notebook WSRC-NB-2006-00087[10] and are discussed in Section 3.0.

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 existing DWPF acid addition equation[11]. The split of the acid was determined using the redox equation currently being used in DWPF processing[12] with a slight modification for the manganese term. The redox target ($Fe^{2+}/\Sigma Fe$) 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. The values used for each run are provided in Section 3.0.

To prevent foaming during processing, 200 ppm IIT 747 antifoam was added during heat-up at 40°C and 500 ppm was added at the completion of acid addition. The addition strategy was conservative relative to the current DWPF addition strategy to increase sensitivity to foaming issues, and no recommendations on changes to the antifoam addition strategy will be made based on this testing. SRAT processing included the dewater time in boiling plus an additional 12 hours of reflux to simulate DWPF processing conditions. SME processing did not include the addition of canister dewaters. 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.

2.3 Analytical Methods

Analyses for this task used guidance of Analytical Study Plan (ASP) SRNL-GPD-2005-00001[13]. Sample request forms were used for samples to be analyzed, and analyses followed the guidelines and means of sample control stated in the ASP for the task. A unique lab identification number was assigned to each sample for tracking purposes. Analyses were performed using approved analytical and Quality Assurance (QA) procedures.

The sludge simulant was analyzed as part of the sludge fabrication process; therefore, those results were used to support this testing and no discussion of the methods will be presented here. Samples were taken throughout the run and of the SRAT and SME products to evaluate the process chemistry. In-process supernate and slurry samples were taken in both of the runs to help support programs focused on understanding the SRAT chemical reactions. The samples were analyzed at the Process Science Analytical Laboratory (PSAL) and Analytical Development (AD). The PSAL performed analyses on the in-process and product samples to determine the chemical composition, total and dissolved solids, density, and pH. The chemical composition was determined in duplicate by calcining the samples at 1100°C and then dissolving the product using Na_2O_2/NaOH fusion and a lithium metaborate fusion. The preparations were then analyzed using Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) to measure the cations present.

The in-process samples were centrifuged immediately after pulling the samples from the SRAT vessel in an attempt to stop the reactions occurring with the other solids. The centrifuged supernates were analyzed by ICP-AES. For the SRAT products, the products were filtered using a 0.45 μm filter and the separated supernates were analyzed using ICP-AES to determine the soluble cations present. Sludge samples for anion analyses were prepped using weighted dilutions and were analyzed using Ion Chromatography (IC). The in-process supernates were also analyzed on the IC to determine the soluble anions. The total and dissolved solids were measured on two aliquots and the insoluble and soluble solids fractions were calculated from the results. Density and pH measurements of the samples were also performed on the in-process samples. Rheological properties of the SRAT and SME products (yield stress and plastic viscosity) were measured and evaluated as a function of the test conditions.

Gases were monitored during the runs using a high-speed Agilent model 3000 micro Gas Chromatograph (GC) to provide insight into the reactions occurring during processing and to determine whether a flammable mixture was formed. As mentioned above, helium was used as a purge gas tracer. Two calibration standards were used to calibrate the GCs before each run to attempt to bound the quantities of the expected gases. The concentrations of these calibration standards were 0.5 mol% helium, 0 and 1 mol% hydrogen, 0 and 21% oxygen, 55 and 66.5 mol% nitrogen, 2.5 mol% nitrous oxide, 0.5 mol% carbon monoxide, 20 mol% carbon dioxide, and 0 and 10 mol% nitric oxide. Calibration checks were performed before and after each run.

The GC is self-contained and is designed specifically for fast and accurate analysis. The GCs have five main components. The first is the carrier gas (argon for this testing) to transport the sample through the MolSieve 5A PLOT (Channel A) and PLOT Q (Channel B) columns. The second is the injector, which introduces a measured amount of sample into the inlet of the analytical columns where it is separated. Injection time is 50 milliseconds for the Channel A gases (helium, hydrogen, nitrogen, oxygen, nitric oxide and carbon monoxide) and 100 milliseconds for the Channel B gases (carbon dioxide and nitrous oxide). The third component is the column, which is capillary tubing coated or packed with a chemical substance known as the stationary phase that preferentially attracts the sample components. As a result, components separate as they pass through the column based on their solubility. Since solubility is affected by temperature, column temperature is controlled during the run. The Channel A column is set at 60ºC, while the Channel B column is set at 70ºC. The fourth component is a micro-machine thermo conductivity detector. The solid state detector monitors the carrier and senses a change in its composition when a component in the sample elutes from the column. The fifth component is the data system, Cerity. Its main purpose is to generate both qualitative and quantitative data. It provides a visual recording of the detector output and an area count of the detector response. The detector response is used to identify the sample composition and measure the amount of each component by comparing the area counts of the sample to the analysis of known calibration standards. A sample was taken approximately once every 3 minutes.

The Total Inorganic Carbon (TIC), mercury, and Semi-Volatile Organic Analysis (SVOA) analysis were performed by Analytical Development. TIC was analyzed with an OI 1010 High Temperature Total Carbon Analyzer. Mercury was analyzed using Atomic Adsoption Spectroscopy following an aqua regia preparation. SVOA analysis was performed using a Gas Chromatograph-Mass Spectrometer.

3.0 Results

Four SRAT/SME cycles were conducted during this study, as shown in Table 3. A unique run number was assigned to each run[14,15,16,17]. All runs targeted a predicted glass REDOX of 0.2 by adjusting the ratio of formic to nitric acid during the SRAT cycle and assumed a coefficient of five for the manganese term in the REDOX prediction. Frit 503 was utilized during the SME cycle and a waste loading of 35% was targeted.

Table 3. SRAT/SME Tests

RUN NUMBER	ACID STOICHIOMETRY	REDOX TARGET	PROCESS FRIT	WASTE LOADING
SB4-61	130%	0.2	503	35
SB4-62	150%	0.2	503	35
SB4-63	160%	0.2	503	35
SB4-64	170%	0.2	503	35

3.1 SRAT Cycle Results

3.1.1 Acid Addition Calculation

3.1.1.1 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 calculated[11] based on the applied stoichiometric factor and the ratio needed to achieve the predicted glass redox target of 0.2 $Fe^{+2}/\Sigma Fe$. 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 require 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. The acid calculation inputs and assumptions are shown in Table 4, Table 5, and Table 6 for SB4-61. The same assumptions and inputs were used for all four runs, with the exception of the acid stoichiometry.

Table 4. Sludge Trimming Parameters and Assumptions

Description	Units	SB4-61
Fresh Sludge Mass without trim chemicals	g slurry	2,845.0
Fresh Sludge Weight % Total Solids	wt%	17.36
Fresh Sludge Weight % Calcined Solids	wt%	12.41
Fresh Sludge Weight % Insoluble Solids	wt%	11.92
Fresh Sludge Density	kg / L slurry	1.138
Fresh Sludge Nitrite	mg/kg slurry	14,495
Fresh Sludge Nitrate	mg/kg slurry	9,331
Fresh Sludge Oxalate	mg/kg slurry	622
Fresh Sludge Formate	mg/kg slurry	0
Fresh Sludge Manganese (% of Calcined Solids)	wt % calcined basis	4.046
Fresh Sludge Slurry TIC (treated as Carbonate)	mg/kg slurry	1,157
Fresh Sludge Hydroxide (Base Equivalents) pH = 7	Equiv Moles Base/L slurry	0.443
Fresh Sludge Mercury (% of Total Solids in untrimmed sludge)	wt% dry basis	0.00
Fresh Sludge Supernate manganese	mg/L supernate	0
Fresh Sludge Supernate density	kg / L supernate	1.04

Table 5. SRAT Cycle Processing Parameters and Assumptions

Description	Units	SB4-61
Conversion of Nitrite to Nitrate in SRAT Cycle	gmol NO_3^-/100 gmol NO_2^-	15.00
Destruction of Nitrite in SRAT and SME cycle	% of starting nitrite	100.00
Destruction of Formic acid charged in SRAT	%	25.00
Destruction of oxalate charged	%	50.00
Percent Acid in Excess Stoichiometric Ratio	%	130.00
SRAT Product Target Solids	%	25.00
Nitric Acid Molarity	Molar	10.573
Formic Acid Molarity	Molar	23.604
DWPF Nitric Acid addition Rate	gallons per minute	2.0
DWPF Formic Acid addition Rate	gallons per minute	2.0
REDOX Target	Fe^{+2} / ΣFe	0.200
REDOX Equation (7 for Mn^{+7}, otherwise assumes Mn^{+4})		7
Trimmed Sludge Target Ag metal content	total wt% dry basis	0.011200
Trimmed Sludge Target wt% Hg dry basis	total wt% dry basis	1.0000
Trimmed Sludge Target Pd metal content	total wt% dry basis	0.0015
Trimmed Sludge Target Rh metal content	total wt% dry basis	0.0108
Trimmed Sludge Target Ru metal content	total wt% dry basis	0.0493
Trimmed Sludge Target oxalate after trim (wt % not mg/kg)	total wt% dry basis	0.3530
Water to dilute fresh sludge and/or rinse trim chemicals	g	50.00
Sample Mass of Trimmed sludge (SRAT Receipt sample, if any)	g	0.00
Mass of SRAT cycle samples	g	250.00
Wt% Active Agent In Antifoam Solution	%	10
Basis Antifoam Addition for SRAT (generally 100 mg antifoam/kg slurry)	mg/kg slurry	100
Number of basis antifoam additions added during SRAT cycle		7

Table 6. SME Processing Parameters and Assumptions

Description	Units	SB4-61
Frit type		503
Destruction of Formic acid in SME	%	8.00
Destruction of Nitrate in SME	%	5.00
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		7
Sludge Oxide Contribution in SME (Waste Loading)	%	35.00
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

3.1.1.2 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 7 with all values tabulated in Appendix A and selected values graphically shown in Appendix B. The stoichiometry acid addition for the sludge simulant was calculated to be 1.07 moles per liter. 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 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 7. Selected Process Values

ACID STOICHIOMETRY	TOTAL ACID REQUIRED (MOL/L)	FORMIC ACID RATIO (% OF TOTAL ACID)	FRIT ADDITION AMOUNT (GRAMS)
130%	1.388	94.77	583.8
150%	1.601	92.47	586.6
160%	1.708	91.55	587.8
170%	1.815	90.71	589.0

3.1.2 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 350 RPM[b] were needed to mix the sludge simulants. The agitator speed was reduced to 250 RPM during acid addition to reduce the size of the central vortex. This speed was maintained for the remainder of the SRAT cycle for all runs except for SB4-61 (130% acid). The agitator speed was increased to 320 RPM after dewater to maintain a vortex. During this run, mercury beads were noted on the agitator shaft and impellers after the SME cycle similar to (but less than) the accumulation seen during the SB4 qualification testing with simulants. Mercury accumulation was not noted during the other three runs.

3.1.2.1 Foaming

Additional antifoam (100 ppm) was required during the formic acid addition for two of the runs (SB4-61 and SB4-63). The antifoam protocol used during DWPF operation has an additional 100 ppm antifoam addition between the nitric and formic additions that was not performed during simulant testing. The extra antifoam addition (one 100 ppm addition) made to each run resulted in the total antifoam addition matching the amount of antifoam that would be added at DWPF.

3.1.2.2 pH Profiles

The pH profiles of the four runs in general matched profiles noted during previous CPC simulations[4]. As shown in Figure 2, the pH of the runs 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. All three runs with acid stoichiometries above 130% had a minimum pH near 4.0 at the end of acid addition.

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

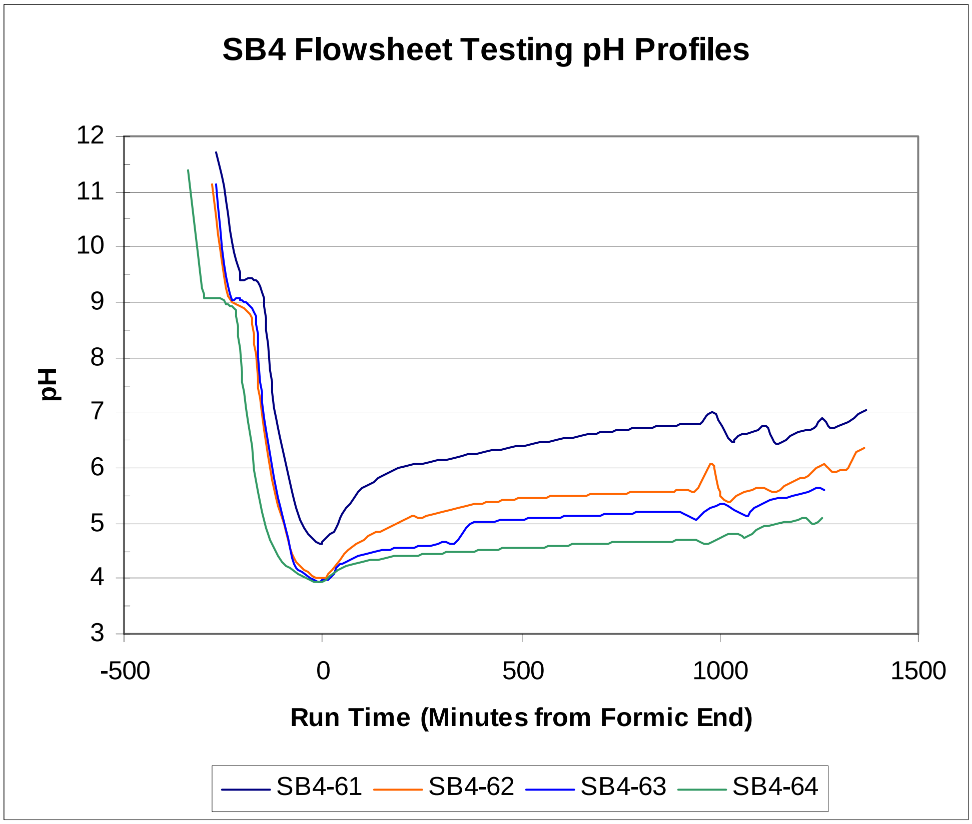

Figure 2. SB4 Flowsheet Testing pH Profiles

3.1.3 SRAT Cycle Sample Results

Samples were pulled during the SRAT cycle after acid addition was completed, at the onset of hydrogen generation[c], at the peak of hydrogen generation, and at the conclusion of the SRAT cycle. The total solids, anions, and soluble elemental species were analyzed for all samples. Mercury analysis was performed on all samples except after acid addition. 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 A while graphical presentations are shown in Appendix B.

3.1.3.1 Nitrite

Nitrite destruction met the process requirement of <1000 mg/kg at the end of the SRAT cycle for all runs and was 100% complete for all runs except SB4-61, as shown in Figure 3. Higher acid stoichiometries led to more rapid destruction of nitrite while nitrite destruction at 130% acid was not 100% complete (i.e., still above the detection limit) at the end of the SME cycle.

[c] SB4-61 (130%) did not have samples for the hydrogen onset and hydrogen peak as no hydrogen generation was noted after the completion of acid addition.

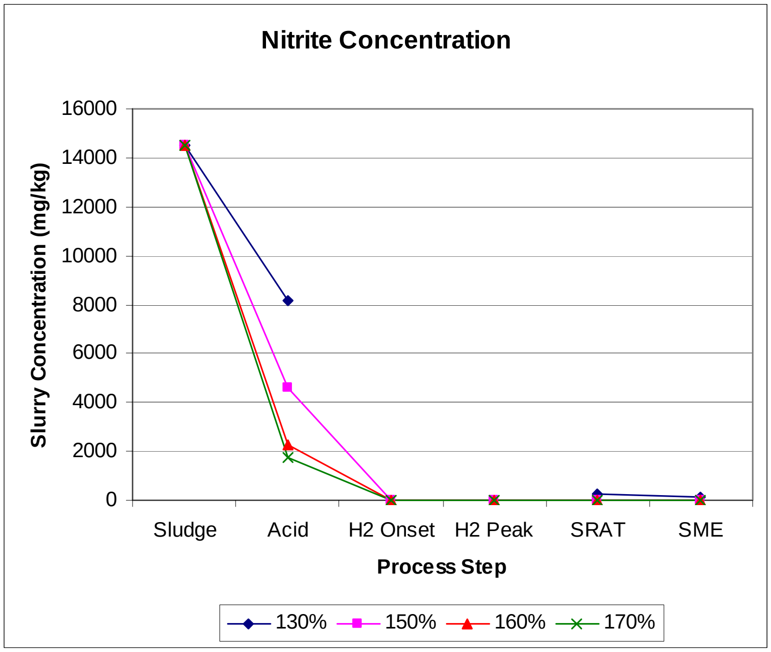

Figure 3. Nitrite Concentration Profile

Conversion of nitrite to nitrate indicated a trend of higher conversions of nitrite to nitrate as the acid stoichiometry was increased, as shown in Figure 4.

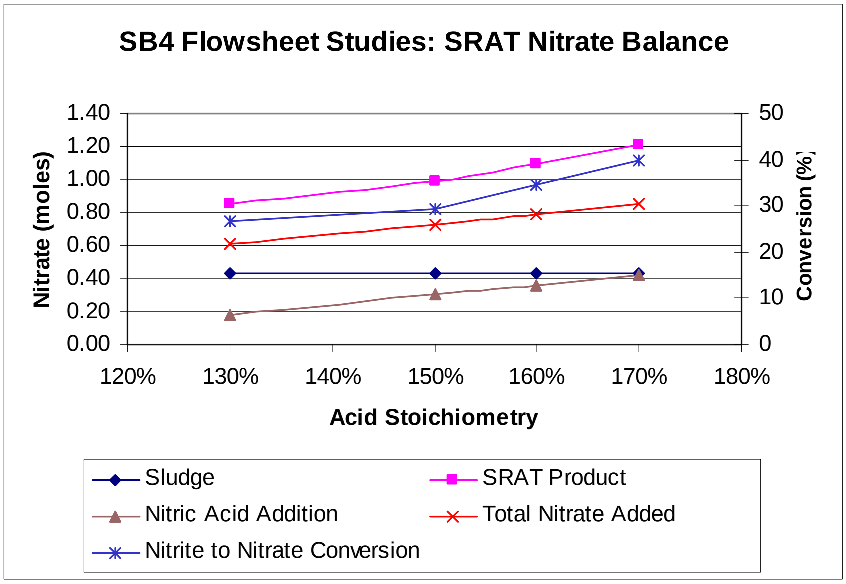

Figure 4. SRAT Nitrate Balance

3.1.3.2 Formate

Formate destruction during the runs is shown in Figure 5. 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[4].

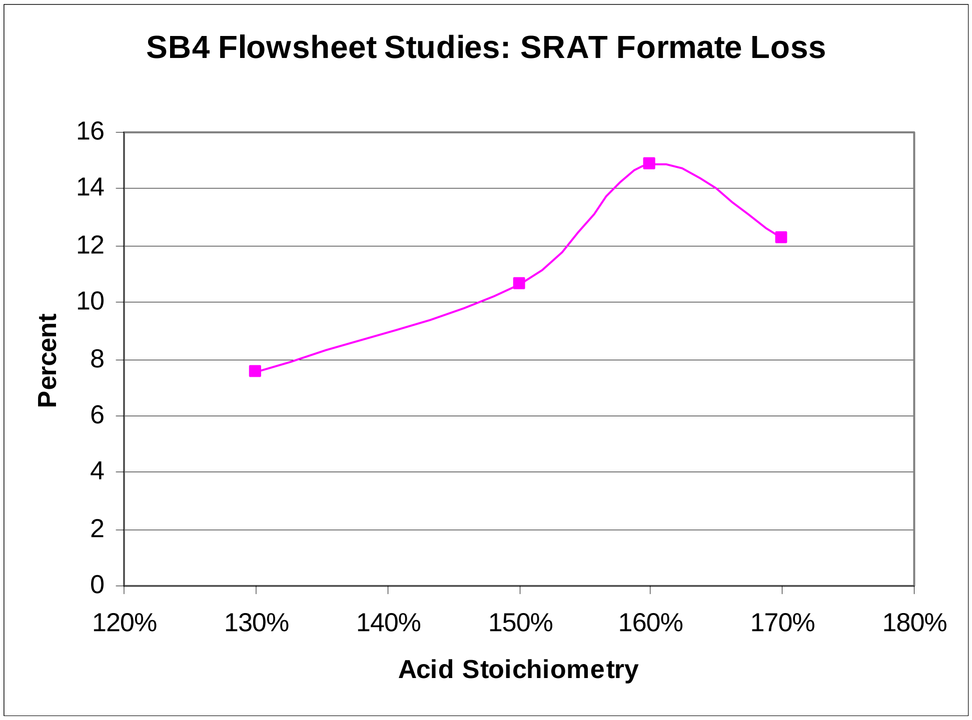

Figure 5. SRAT Cycle Formate Destruction

3.1.3.3 Oxalate

Initial oxalate concentration in the simulant was approximately 600 ppm. No oxalate was noted in any of the SRAT product samples, as shown in Figure 6. The amount of oxalate noted in the 130% acid SME product is just above the minimum sensitivity of the sample analysis.

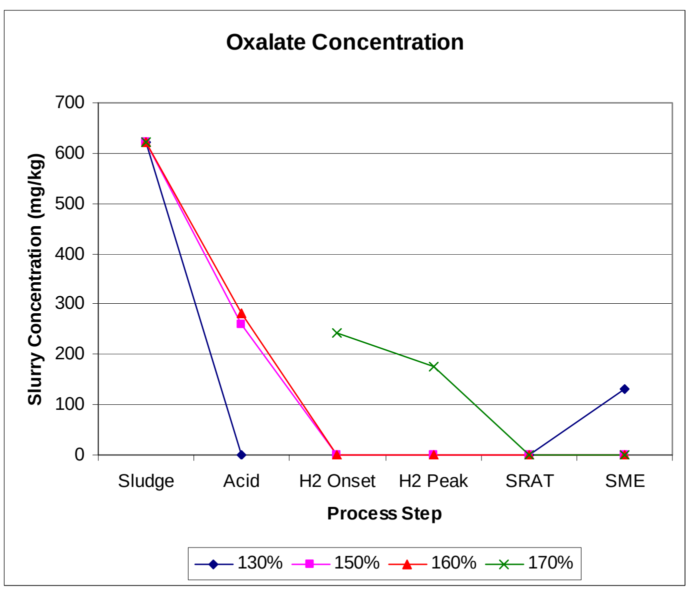

Figure 6. Oxalate Concentration Profile

3.1.3.4 Mercury

The SRAT product and process 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. Previous sludge batches met this requirement without mercury removal, but SB4 is estimated to contain approximately 1.0 wt% mercury in the incoming blended feed. As shown in Table 8, the mercury was reduced to acceptable levels by the end of the SRAT cycle for all runs. The samples taken during the SRAT cycle had considerable variability in the Hg results and some results indicated more mercury than was added to the sludge, as shown in Figure 7. The scatter in these process sample results is likely the result of the small sample size and the difficulty in taking a representative sample when elemental mercury is present.

The mercury data indicates that the experimental setup may have an impact on the results. Two setups were used during the testing: Rig 1 and Rig 2. The two rigs were made with identical setups, but the use of custom glass fabrication for the vessel, lid, and offgas components cause slight variations between the rigs. The 130% and 160% were performed in the Rig 1 while the 150% and 170% runs were performed in Rig 2. The results for the runs performed in the same rig are very similar; however, the amount of scatter and sampling error typical for mercury analysis would make conclusions from only four runs highly speculative.

Table 8. SRAT and SME Product Mercury Results

ACID STOICHIOMETRY	SRAT PRODUCT	SME PRODUCT
%	wt% (solids basis)	wt% (solids basis)
130	0.256	0.088
150	0.340	0.189
160	0.245	0.097
170	0.385	0.138

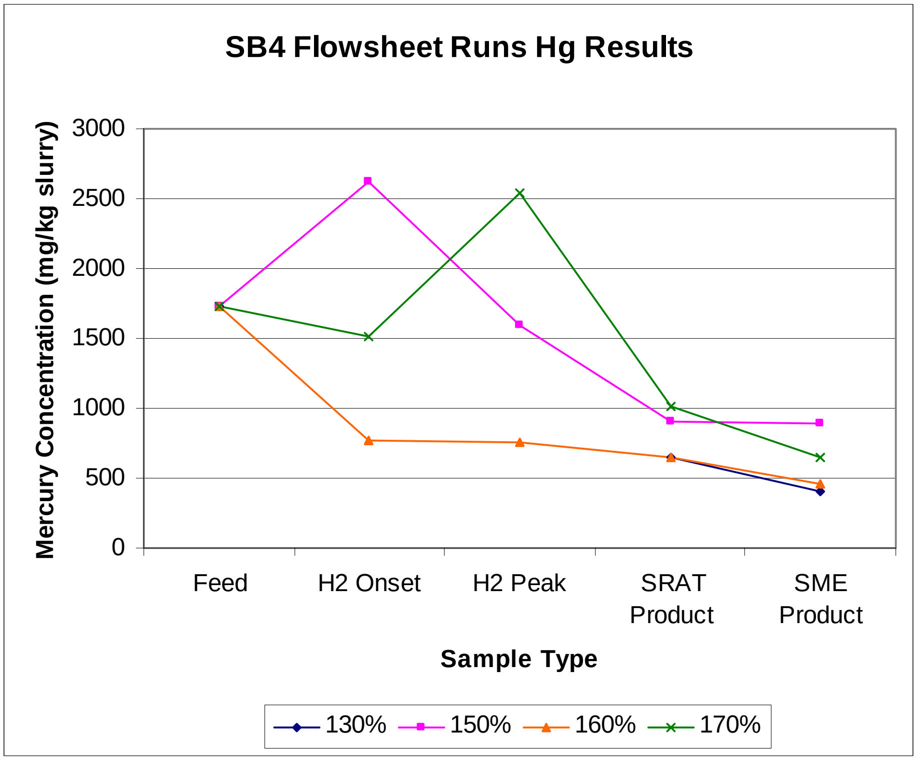

Figure 7. Mercury Results

3.1.3.5 Soluble Species

The amount of soluble metal species present in the SRAT samples is an indirect measurement of the effectiveness of the acid addition and provides data to allow the process chemistry to be evaluated. The percentage soluble for each species was calculated by dividing the amount of the species in the supernate by the amount of the species in the total slurry. The overall trend for most species (phosphorus and palladium being exceptions) is higher solubility as acid stoichiometry is increased. Most species indicated a maximum in soluble concentration after acid addition with decreased solubility as the pH rose during the dewater and reflux steps. Calcium, magnesium, potassium, sodium, and sulfur were mostly soluble in the SRAT products. Several species (Ba, Ni, Pb, and Zn) exhibited peaks in solubility at the peak of hydrogen generation. Silicon solubility was likely strongly influenced by antifoam additions and subsequent breakdown. Iron solubility was significantly higher than noted in previous CPC testing. Solubility charts for each species as a function of process step are shown in Appendix B.

Manganese is insoluble when in the Mn^{+4} state, therefore soluble manganese indicates the presence of Mn^{+2} (higher valences are also soluble but are not expected to be present during the SRAT cycle). The solubility of Mn^{+2} is pH dependent, therefore insoluble manganese does not necessarily represent Mn^{+4}. As shown in Table 9, the higher acid stoichiometries resulted in all the manganese being soluble. Nickel solubility was also shown to be strongly dependent on the pH.

Table 9. Manganese and Nickel Solubility in SRAT Product

ACID STOICHIOMETRY	pH	MANGANESE	NICKEL
%		% soluble	% soluble
130	8.17	22.6	0.0
150	6.83	81.0	1.1
160	5.27	105.2	28.7
170	4.78	108.3	45.3

3.1.3.6 Condensates

The sample results for all condensate samples are tabulated in Appendix A along with corresponding charts in Appendix B. The major species noted in the condensate were nitrate, formate, and silicon. The nitrate and formate were most likely present as acid since no major cation species were detected. General trends indicate increased concentrations of species in the condensate as acid stoichiometry is increased. The higher amounts of formate and nitrate are expected as higher acid additions lead to higher levels of these species in the vessel. The silicon in the condensate has been shown to be the result of antifoam degradation[18]. Higher acid stoichiometry lowers the pH of the SRAT slurry and increases the rate of antifoam degradation.

Condensate pH was generally lower as acid stoichiometry was increased, as shown in Figure 8. The condensate pH of the 130% run was basic at the end of the SRAT cycle as indicated by the pH of the MWWT results. The MWWT was drained at the end of the SRAT cycle and (generally) represents the last condensate generated during that cycle. The nitrate results for the SRAT dewater during SB4-62 (150% acid) are most likely in error, but the sample was not re-analyzed.

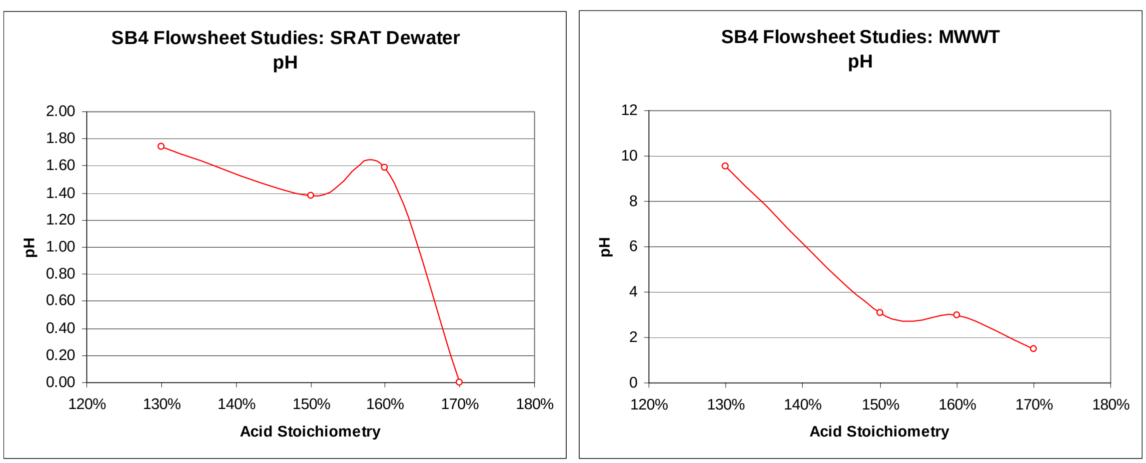

Figure 8. SRAT Dewater and MWWT pH

SVOA analysis was performed on an aggregate sample of the condensate from each run. The aggregate sample was a weighted mixture of all condensates from the SRAT and SME cycles. Past tests had indicated the presence of methyl-mercuric chlorides, but all samples from this testing were below detection limits for these species.

3.1.4 SRAT Cycle Offgas Composition Results

A typical offgas concentration profile is shown in Figure 9 while charts from all runs are shown in Appendix C. Note that Figure 9 also shows the SME cycle, which started at a run time of approximately 1000 minutes. 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.

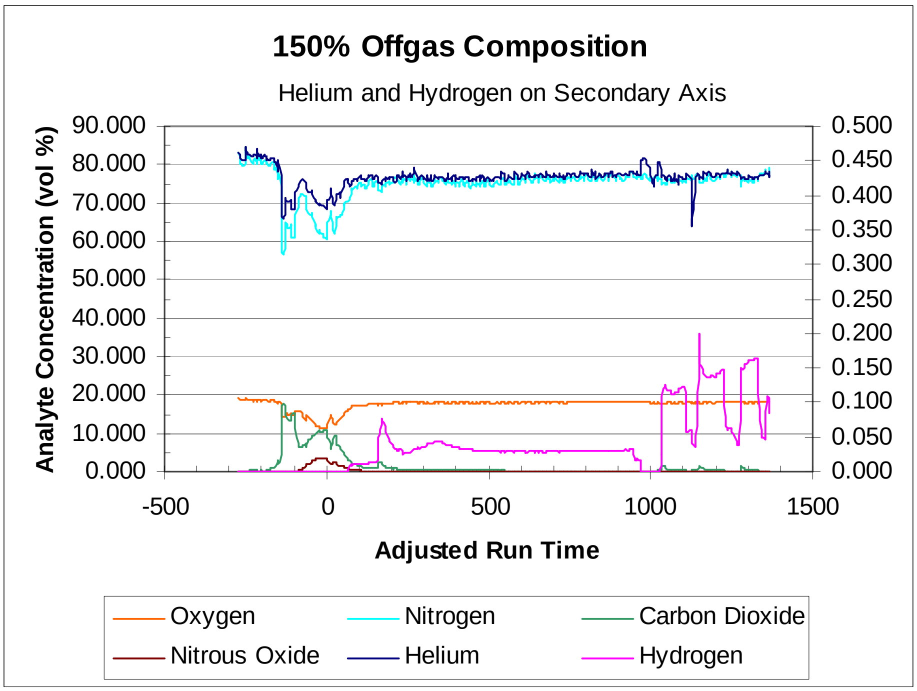

Figure 9. Offgas Data from SB4-62 (150% Acid Stoichiometry)

3.1.4.1 Hydrogen Evolution

The peak hydrogen generation for each run is shown in Figure 10, along with the peak carbon dioxide and nitrous oxide rates. In general, the peak hydrogen generation rate increased with increased acid addition. None of the rates approached the SRAT processing limits of 0.65 lb/hr, as shown in Table 10 which shows the peak hydrogen generation after scaling to the DWPF process. A review of the run data for SB4-64 (170% acid) and SB4-63 (160% acid) was conducted. No process upsets or issues with the acid addition and/or noble metals addition was noted which would explain the lower hydrogen emission from run SB4-64 than SB4-63.

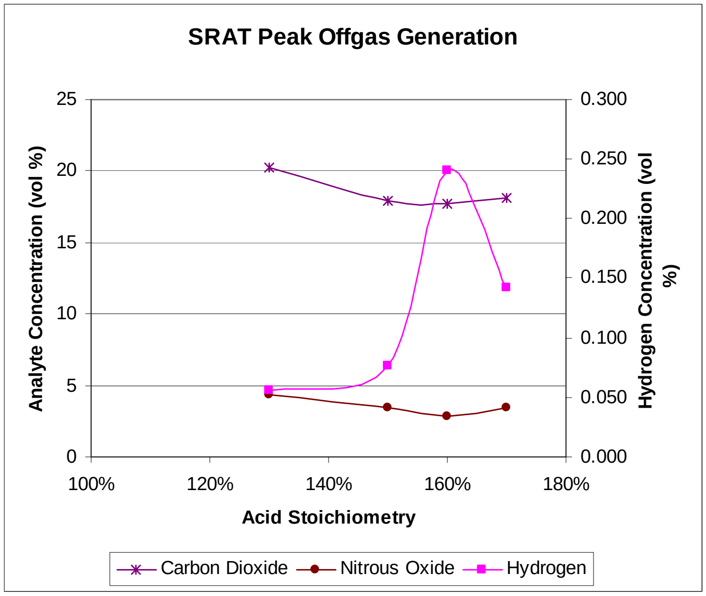

Figure 10. SRAT Cycle Peak Offgas Concentrations

Table 10. SRAT Cycle Hydrogen Peak Generation Rate

		Acid Stoichiometry			
		130%	**150%**	**160%**	**170%**
SRAT Hydrogen Peak	lb/hr	0.045	0.070	0.167	0.117

The hydrogen evolution as a function of time is shown in Figure 11. Note that the SME cycle is also shown for comparison. Increased acid stoichiometry decreased the time between the start of boiling and the onset of hydrogen emission.

Hydrogen emission was only noted for the 130% acid run during acid addition. Hydrogen emission is generally not noted during this time period and may have been the result of a stagnant area in the vessel. The SRAT product initially thickens as acid is added, then thins as the pH drops below 6. A stagnant area could result in higher acid concentrations in the portion of the vessel that is being mixed or localized areas that are superheated in the portion that is stagnant.

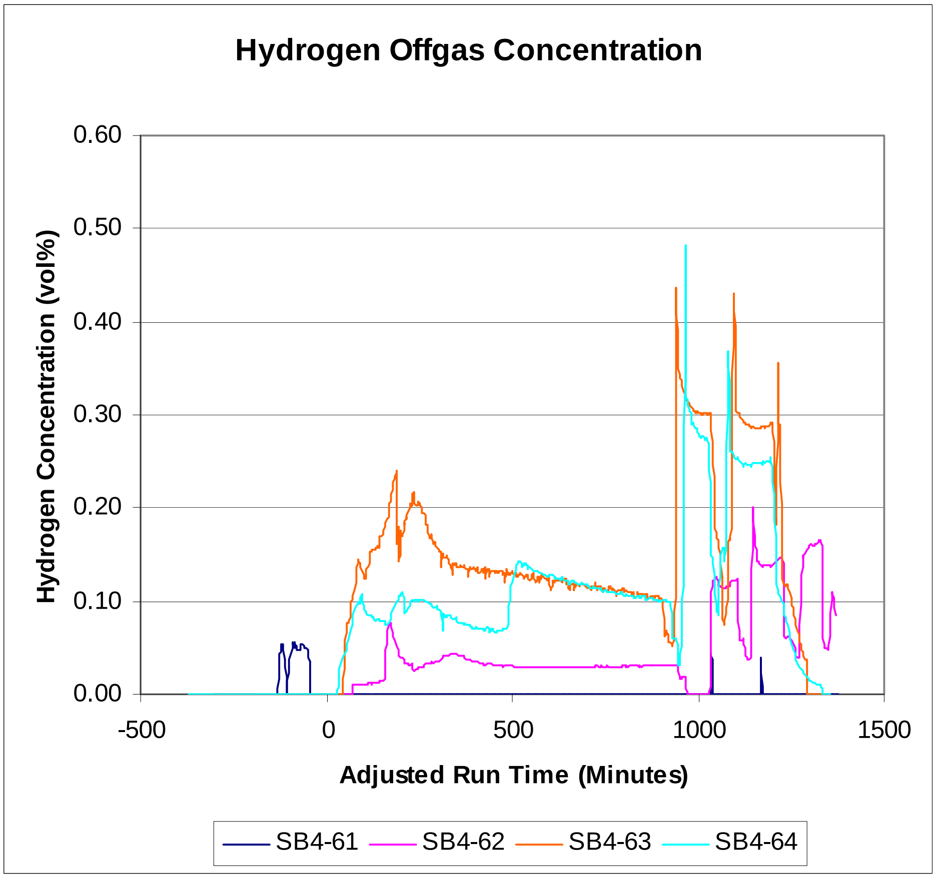

Figure 11. SRAT Cycle Hydrogen Evolution

3.1.4.2 Other Species

As shown above in Figure 10, carbon dioxide and nitrous oxide peak concentrations may have slightly decreased as acid addition was increased. The peak generation of these species is less dependent on acid concentration than hydrogen since the compounds that are responsible for the highest emissions are already present in the sludge as carbonate and nitrite. The peak generation rates are shown in Table 11 after scaling to the DWPF process scale.

Table 11. SRAT Cycle Nitrous Oxide and Carbon Dioxide Peak Generation Rates

		Acid Stoichiometry			
		130%	**150%**	**160%**	**170%**
SRAT Nitrous Oxide Peak	lb/hr	27.0	27.3	17.0	25.3
SRAT Carbon Dioxide Peak	lb/hr	134.9	149.5	116.3	143.9

3.1.5 SRAT Product Rheological Properties

The rheological properties of SRAT products 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)[d] except

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

for the 150% acid run. The yield stress of the 130% run was higher than the upper limit for yield stress while the 160% and 170% runs were below the minimum yield stress. The yield stress and consistency of the SRAT products are shown in Table 12. The flow curve shape varied considerably, but the overall trend can be seen in the data that runs with higher acid additions were less viscous. All SRAT cycles targeted 25 wt% solids and the measured results indicate very little variability in the total solids between runs, so the difference in rheological behavior results from the different acid stoichiometries and corresponding soluble species. The flow curves generated during the testing are shown in Appendix D.

Table 12. Rheological Properties of SRAT Products

	Up Curve		Down Curve	
	Yield Stress	Consistency	Yield Stress	Consistency
	Pa	cP	Pa	cP
SB4-61-1	10.07	10.82	10.80	7.18
SB4-61-2	11.90	5.95	9.62	7.16
130% Ave	**10.99**	**8.38**	**10.21**	**7.17**
SB4-62-1	3.15	8.93	1.85	10.94
SB4-62-2	3.42	8.61	1.91	11.04
150% Ave	**3.29**	**8.77**	**1.88**	**10.99**
SB4-63-1	1.03	8.05	0.81	8.09
SB4-63-2	1.01	7.96	0.83	8.00
160% Ave	**1.02**	**8.01**	**0.82**	**8.04**
SB4-64-1	0.69	7.47	0.52	7.42
SB4-64-2	0.73	7.48	0.55	7.49
170% Ave	**0.71**	**7.48**	**0.54**	**7.45**

The shape of the flow curves is expected to be that of a Bingham Plastic, but the more viscous SRAT products (130 and 150% runs) had flow curves that contained humps. This type of flow curve has been noted during previous testing and has been evaluated previously[19]. The yield stress and consistency of the down curve is considered to be more representative when flow curves are shaped in the manner of the curves for the 130% and 150% acid run.

As shown in Figure 12, the impact of increased acid addition diminished as acid was increased over 160%. The diminished impact may be the result of the behavior of manganese and other soluble species. At 160%, acid manganese was 100% soluble. Increasing the acid stoichiometry to 170% did dissolve some of the nickel, but the impact of nickel solubility would be less then the impact of manganese due to the smaller amount of nickel in the slurry.

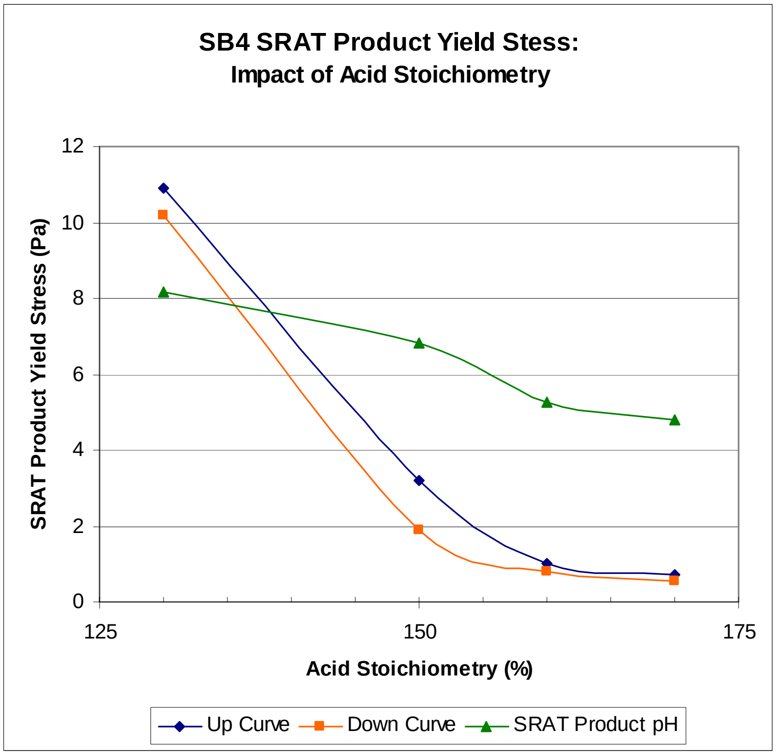

Figure 12. Impact of Acid Stoichiometry on SRAT Product Yield Stress

3.2 SME Cycle Results

The SME cycle was 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. As stated earlier, the SME cycle targeted a final solids concentration of 45 wt % total solids based on earlier testing using the same simulants that resulted in extremely viscous slurries at the end of the SME cycle[20].

3.2.1 Processing Observations

No processing issues were noted during the SME cycle. Mixer speed was maintained at 250 RPM throughout each run except for the 130% run. Agitator speed was increased to 320 RPM during this run to maintain mixing, which is similar to the conditions at the start of the SRAT cycle.

As shown in Figure 2 above, 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.

3.2.2 SME Cycle Sample Results

Samples were pulled at the conclusion of the SME cycle and analyzed for total solids, anions, soluble elemental species, mercury, and REDOX. Samples were taken of the SME dewater and the FAVC contents at the completion of the SME cycle. All sample results are tabulated in Appendix A while graphical presentations are shown in Appendix B.

3.2.2.1 SME Product Results

The solids content of the SME products are shown in Table 13 along with the calculated waste loading and pH. The solids content generally were ~2 wt% higher than targeted, but the waste loading targets were generally very close to the 35% target as calculated from the lithium content of the SME product. Mercury continued to be steam stripped from the process slurry and final levels of mercury were reduced slightly compared to the SRAT products.

Table 13. SME Product Results

RUN ID	PH	TOTAL SOLIDS	LITHIUM OXIDE CONTENT	WASTE LOADING
		wt%	wt % Calcined solids	wt %
130%	7.43	46.65	5.17	35.4
150%	6.98	47.57	5.22	34.7
160%	5.72	46.83	5.15	35.6
170%	5.35	47.13	5.06	36.7

Loss of formate was noted during the SME cycle, as shown in Figure 13 and Table 14. The values noted during the testing are similar to results from previous runs[4] and are relatively uniform from run to run. The amount of nitrate loss was small for all runs, with the highest acid stoichiometry indicating a loss of 8.5%. The negative values for the lower acid stoichiometries likely result from the expected analytical error and cumulative errors in the mass balance as various samples are pulled.

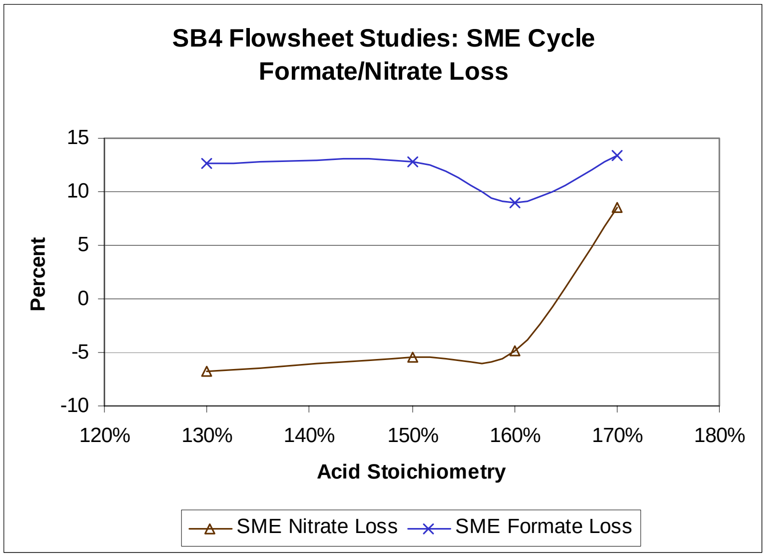

Figure 13. SME Cycle Formate / Nitrate Loss

Table 14. SME Cycle Formate / Nitrate Loss

		130%	150%	160%	170%
Nitrate Lost	%	-6.83	-5.41	-4.92	8.58
Formate Lost	%	12.65	12.77	8.98	13.42

3.2.2.2 Condensates

The condensate from SME dewater followed the same trends as the SRAT cycle condensate. pH was generally higher than the SRAT dewater and the SRAT MWWT. The pH was higher during runs with the lowest acid addition, as shown in Figure 14. As with the SRAT condensate, the major species present were formate and silicon, but nitrate was not noted. The dominant species in the FAVC was nitrate with silicon as the major cation.

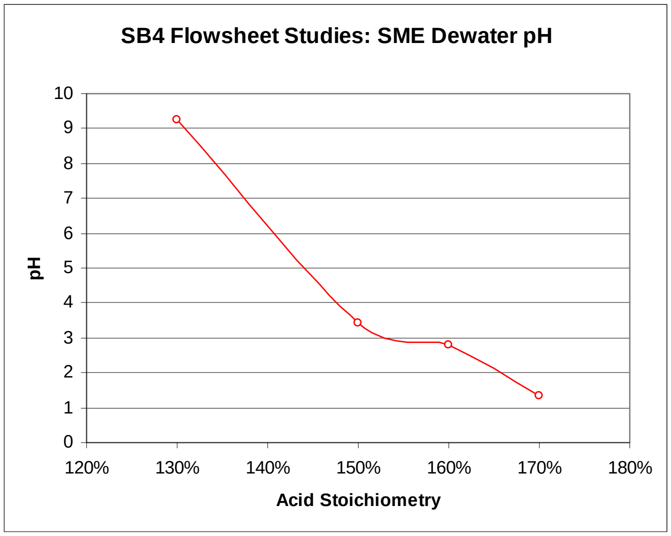

Figure 14. SME Dewater pH

3.2.3 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 15 (the SME cycle starts at ~ 1000 minutes) while charts from all runs are shown in Appendix C. 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.

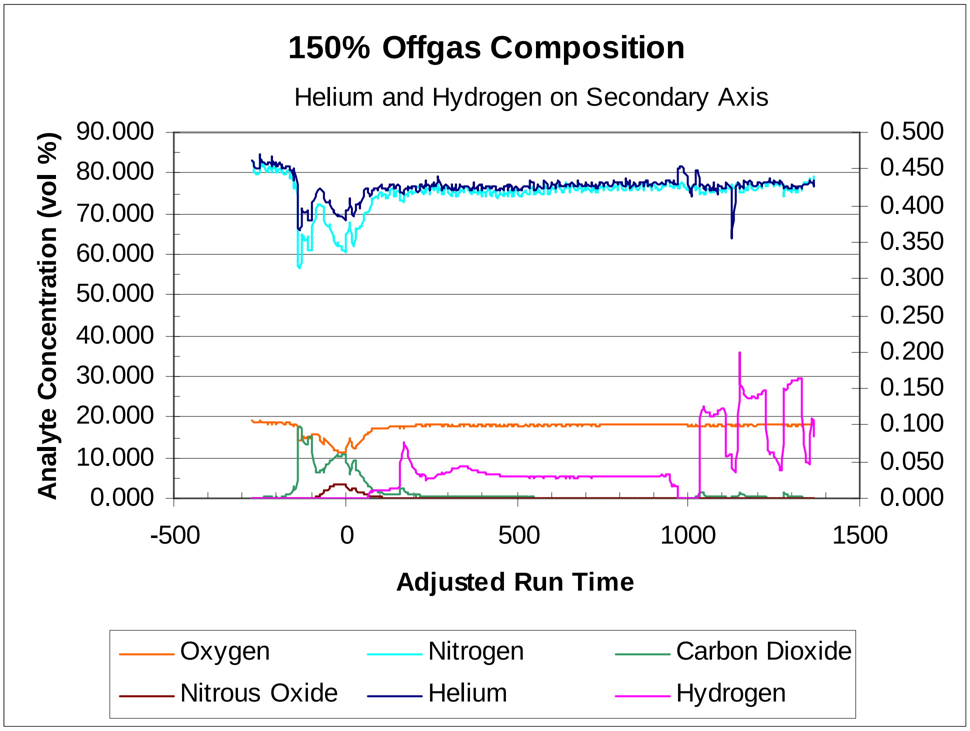

Figure 15. Typical Offgas Profile

3.2.3.1 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 11 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 16 and were a function of acid stoichiometry. Peak generation rates scaled to the DWPF process are shown in Table 15 and were all below the SME process limit of 0.223 lb/hr.

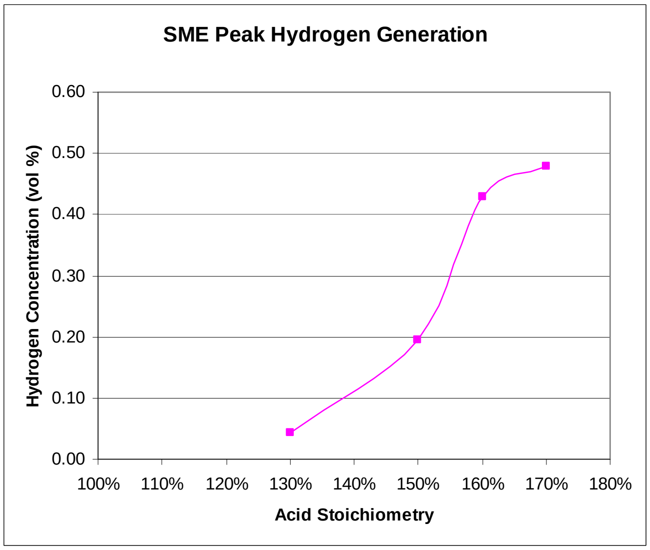

Figure 16. Peak Hydrogen Generation during SME Cycle

Table 15. SME Cycle Hydrogen Peak Generation Rates

		Acid Stoichiometry			
		130%	**150%**	**160%**	**170%**
SME Hydrogen Peak	lb/hr	0.009	0.050	0.089	0.121

3.2.3.2 Other Species

Carbon dioxide was generally the only other gas of any significance emitted during the SME cycle (the 130% acid run contained a small amount of nitrous oxide emissions from the nitrite remaining after the SRAT cycle). Peak carbon dioxide generation is shown in Figure 17.

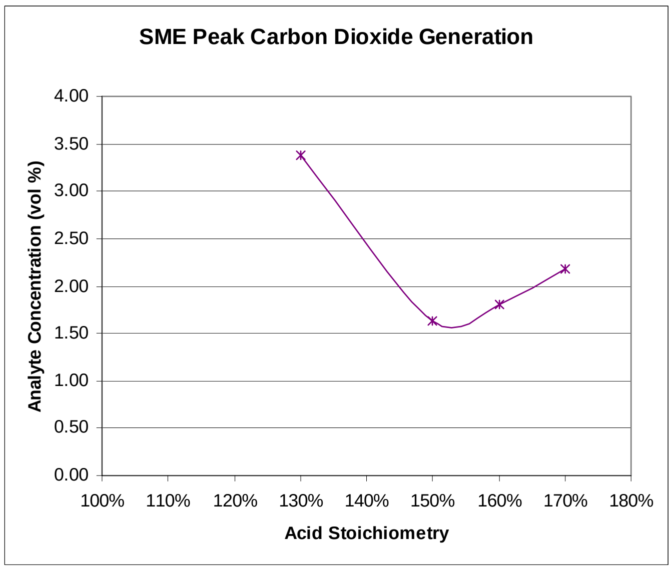

Figure 17. SME Cycle Carbon Dioxide Peak Generation Rates

Table 16. SME Cycle Carbon Dioxide and Nitrous Oxide Peak Generation Rates

		Acid Stoichiometry			
		130%	**150%**	**160%**	**170%**
SME Nitrous Oxide Peak	lb/hr	0.141	0.00	0.00	0.00
SME Carbon Dioxide Peak	lb/hr	5.32	3.34	3.01	4.41

3.2.4 SME Product Rheological Properties

The rheological properties of each SME product were measured as well as the rheological properties of the 150% acid run at different solids concentrations. Four 250 ml samples of the SME product were pulled and centrifuged at 1000 RPM for 10 minutes in an IEC Centra GP8 centrifuge. After centrifuging, supernate was decanted or added to reach the solids concentration target. The highest targeted solids concentration (52%) could not be reached as the supernate layer was not sufficient to remove the targeted amount. Solids content of each sample was measured to verify the total solids in each sample were correct.

Higher acid stoichiometry lowered the yield stress and consistency of the SME products, as shown in Figure 18. The 130% acid run exceeded the upper process limit for yield stress (15 Pa)[e]. All runs met the process limits for consistency (10 to 40 cP) and the lower limit for yield stress (2.5 Pa), as shown in Table 17. The 150% acid run yield stress and consistency increased sharply as solids content was increased, as shown in Figure 19. This result is consistent with previous results. Previous testing with sludge simulants has indicated that centrifuging and resuspending the solids can impact the yield stress[21]. The baseline sample during the solids study was repeated to evaluate the impact of the centrifuging process. The baseline sample yield stress was increased slightly by centrifuging, as shown in Table 18.

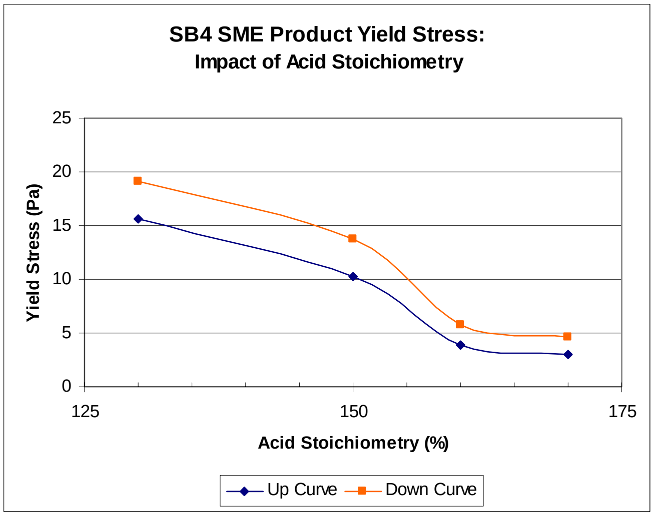

Figure 18. SME Product Yield Stress

Table 17. SME Product Rheological Properties

	Up Curve		Down Curve	
	Yield Stress	Consistency	Yield Stress	Consistency
	Pa	cP	Pa	cP
SB4-61-1	15.88	21.79	19.34	20.62
SB4-61-2	15.50	23.06	18.99	21.97
130% Ave	**15.69**	**22.43**	**19.16**	**21.29**
SB4-62-1	10.41	24.18	14.09	20.31
SB4-62-2	10.09	25.22	13.40	23.00
150% Ave	**10.25**	**24.70**	**13.74**	**21.66**
SB4-63-1	3.92	19.67	5.63	17.88
SB4-63-2	3.87	20.13	5.89	16.67
160% Ave	**3.89**	**19.90**	**5.76**	**17.28**
SB4-64-1	3.04	17.80	4.55	18.17
SB4-64-2	3.07	7.93	4.58	18.22
170% Ave	**3.06**	**12.87**	**4.57**	**18.19**

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

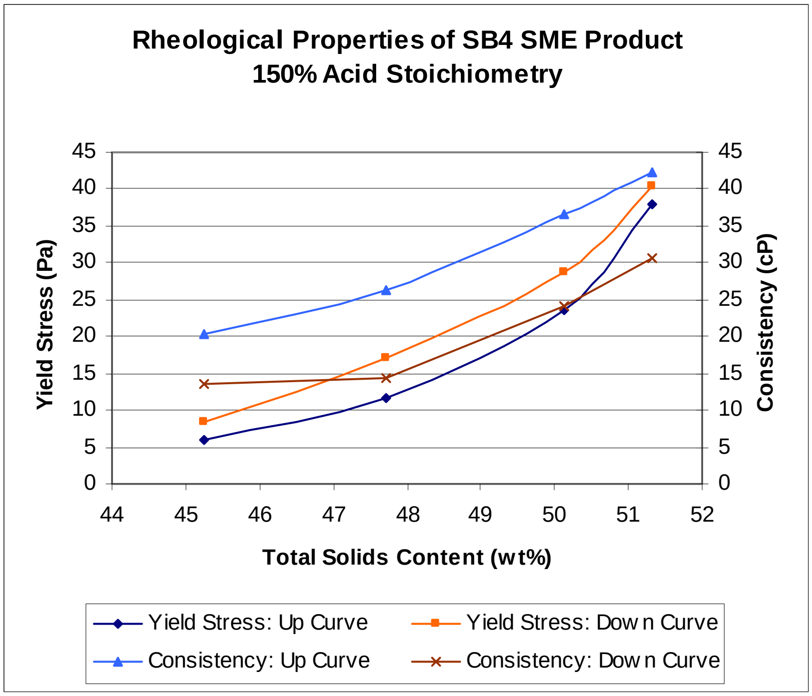

Figure 19. 150% Acid Rheological Properties versus Solids Content

Table 18. SME Product Rheology versus Solids Content

Total	Up		Down	
Solids	**Yield Stress**	**Consistency**	**Yield Stress**	**Consistency**
wt%	**Pa**	**cP**	**Pa**	**cP**
45.26	6.01	20.27	8.44	13.59
47.72 (prior to centrifuge)	10.25	24.70	13.74	21.66
47.72	11.66	26.34	17.07	14.48
50.14	23.57	36.52	28.79	24.07
51.33	37.88	42.21	40.35	30.58

3.2.5 REDOX Results

Previous testing with SB4 simulants has indicated that the Reduction-oxidization (REDOX) potential of the glass[f] has been more oxidizing than predicted[22]. Evaluation of the discrepancies between the predicted and measured REDOX values has focused on the role of manganese in the melter REDOX. The current equation for prediction of glass REDOX assumes that manganese must be converted from the Mn^{+4} state to Mn^{+2} in the cold cap, yielding a coefficient of “2” for manganese based on the number of electrons transferred. Assuming a coefficient of “5” in the prediction equation yields results closer to the measured values and has been used for this testing. A coefficient of “5” would result if the manganese was in the Mn^{+7} state in the melter feed or converted to Mn^{+7} in the cold cap.

[f] REDOX is measured by determining the ratio of Fe^{+2} to total Fe in the glass. A REDOX target of 0.2 $Fe^{+2}/\Sigma FE$ was utilized for this testing assuming Mn^{+7} in the melter feed.

The predicted REDOX values shown in Table 19 are based on the sample results of the SME product. Measured values are the average of duplicate samples generated by the guidelines of L29 ITS-0052 "Vitrification of Melter Slurries for Glass Redox (Fe2+/~Fe) & Chemical Composition Measurement"[23].

Table 19. SME Product REDOX

Calculation Inputs	SB4-61	SB4-62	SB4-63	SB4-64
Nitrite (mg/kg)	101.5	0	0	0
Nitrate (mg/kg)	20200	22550	24950	23000
Formate (mg/kg)	51110	55500	57450	58600
Oxalate (mg/kg)	130	0	0	0
Mn (wt% calcine)	1.46	1.47	1.48	1.52
Wt% Solids	46.65	47.57	46.83	47.13
Calcine Solids	38.81	39.11	38.31	38.16
Nitrite (mol/kg)	0.0022	0.0000	0.0000	0.0000
Nitrate (mol/kg)	0.3258	0.3637	0.4024	0.3710
Formate (mol/kg)	1.1358	1.2333	1.2767	1.3022
Oxalate (mol/kg)	0.0000	0.0000	0.0000	0.0000
Mn (mol/kg)	0.1031	0.1049	0.1032	0.1056
Predicted Redox ($Fe^{+2}/\Sigma Fe$)				
Mn^{+7}	0.216	0.217	0.199	0.235
Mn^{+4}	0.273	0.273	0.256	0.292
Measured Redox				
($Fe^{+2}/\Sigma Fe$)	0.056	0.075	0.084	0.088

As shown in Figure 20, the measured REDOX increased with increased acid stoichiometry, but all results are significantly less than the predicted values. The predictions are based on SME product sample results, therefore the predicted values are not biased due to assumptions made during the acid calculations.

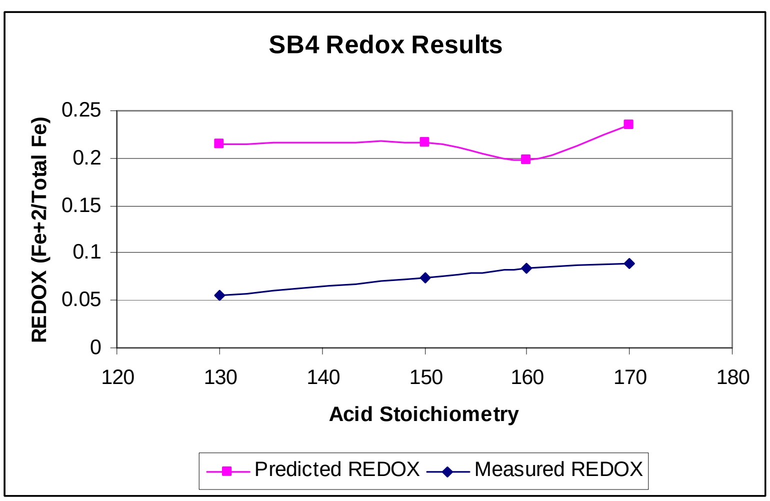

Figure 20. SB4 Flowsheet Testing REDOX Results

4.0 Conclusions

No significant processing issues with processing SB4 were noted during the run.

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

Hydrogen generation was significantly impacted by the changes in acid stoichiometry from 130% to 170% (1.39 to 1.82 mole acid per liter of sludge), but all generation rates were within process limits. Hydrogen generation and nitrous oxide generation scaled to DWPF are shown in Table 20.

Table 20. Offgas Peak Summary

		Acid Stoichiometry			
		130%	**150%**	**160%**	**170%**
SRAT Hydrogen Peak	lb/hr	0.045	0.070	0.167	0.117
SME Hydrogen Peak	lb/hr	0.009	0.050	0.089	0.121
SRAT Nitrous Oxide Peak	lb/hr	27.0	27.3	17.0	25.3
SME Nitrous Oxide Peak	lb/hr	0.141	0.00	0.00	0.00
SRAT Carbon Dioxide Peak	lb/hr	134.9	149.5	116.3	143.9
SME Carbon Dioxide Peak	lb/hr	5.32	3.34	3.01	4.41

- 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 1.0 wt% (solids basis) based on the expected composition of the SB4 blend. Acid quantities from 130% to 170% resulted in satisfactory mercury removal with 12 hours of reflux boiling. Mercury accumulation was noted on the agitator shaft and impellers during the 130% acid run. This material would not be transferred to the SME cycle, therefore it does not represent failure to remove mercury. However, accumulation of deposits on processing equipment is not desirable and discussions concerning whether or not the accumulation on the impellers would continue or the mercury would slough off during subsequent processing would be highly speculative.

- Acid quantities and processing times required for nitrite destruction

Acid quantities from 130% to 170% resulted in satisfactory nitrite destruction with 12 hours of reflux boiling. 130% probably represents the lower end of the window since there was still a small amount of nitrite present but was less than 1000 mg/kg.

- Impact of SB4 composition (in particular, manganese, nickel, mercury, and aluminum) on DWPF processing (i.e. acid addition strategy, foaming, hydrogen generation, REDOX control, rheology, etc.)

Acid quantities from 130% to 170% resulted in satisfactory process performance with no significant issues noted. Foaming was noted during formic acid addition, but lab-scale operations did not utilize an antifoam addition between the nitric and formic acid additions. Addition of antifoam equal to the amount added at DWPF between the acid additions was sufficient to control foaming. Increased solubility of Mn and Ni were noted as acid stoichiometry increased.

Except for the 150% run, all SRAT products were outside the process limits for yield stress with the lowest acid (130%) being above the process limit and the 160% and 170% runs being below the process limit. The process limits for SME product yield stress were met for the 150% acid run at 47% solids, but the 130% acid run was above process limits and the 160% and 170% runs were slightly below process limits. The 150% acid run exceeded the upper limit for SME product yield stress when concentrated to 50 wt% solids. It should be noted that the trend seen in rheological properties of the simulants are expected to be similar for the DWPF process slurries, but the absolute values for the simulants are not expected to be prototypical in yield stress or consistency. Adjustment in the solids concentration targets and/or acid stoichiometry should be made if processing problems due to viscous process slurries are noted in DWPF.

The pH of the condensate generated was typically acidic, but the 130% acid run resulted in condensate that was basic before the end of the SRAT cycle and throughout the SME cycle with a pH of approximately 9. All condensates from all other runs had a pH of less than 4.

Measured REDOX values for all runs were significantly below the predicted values. REDOX values increased slightly as acid stoichiometry was increased. The issues with REDOX using SB4 simulants have been evaluated in a separate study and will be documented in a separate report.

5.0 Recommendations

Based on this series of runs, an acid stoichiometry of 150% is recommended for initial SB4 processing with an acid window of 130% to 170%. The SB4 simulant used during the testing had a stoichiometric acid requirement of 1.07 mol/L, giving an acid addition of 1.60 mol/L at 150% acid. The actual DWPF recommendation will be finalized once processing studies are completed with the blended sample.

The manganese term in the electron equivalence REDOX model should be changed from a coefficient of “2” to a coefficient of “5” for SB4 processing. Additional studies are needed to determine the cause of the lower than predicted REDOX values in this program.

No changes to the DWPF antifoam addition strategy, acid addition rate, reflux time, or SME solids targets are recommended.

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Appendix A. Sample and Run Results: Tabulated Presentations

Table A- 1. SRAT Process Sample Results

Process Samples: After Acid				
	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%
Supernate elementals (mg/L)				
Al	140.5	405.5	268.5	429
B	8.42	7.725	6.015	4.225
Ba	0.939	1.46	0.607	0.546
Ca	2205	2240	2370	2330
Cr	1.19	2.47	2.4	3.455
Fe	4.655	23.2	17.7	36.45
K	800.5	809.5	869	830
Li	<10.0	<10.0	<10.0	<10.0
Mg	2115	2135	2255	2225
Mn	4525	4895	5265	5405
Na	20300	20100	21650	21350
Ni	547.5	686	798	864
P	<1.00	<1.00	<1.00	<1.00
Pb	1.155	1.93	2.81	2.85
Pd	6.115	5.55	4.94	4.255
Rh	26.2	30.75	31.9	31.8
Ru	32	35.85	36.1	37.75
S	470.5	480	536.5	523
Sr	4.9	4.875	4.84	4.805
Ti	<0.100	<0.100	<0.100	<0.100
Zn	38.8	54.1	61.1	70.7
Zr	0.1475	0.1955	0.148	0.152
Anions (mg/kg)				
NO2	8145	4580	2275	1760
NO3	20170	21770	22300	23950
HCO2	51110	55635	53200	56450
SO4	2700	3045	3250	4225
PO4	<100	<100	<100	<100
C2O4	<100	260	282	<100
Cl	155	132.5	150	148.5
pH Data				
pH	4.65	4.41	3.98	3.67
Solids Data (wt %)				
TS	19.3	19.2	19.3	19.3
IS	10.6	10.3	10.5	10.3
Supernate	9.7	9.9	9.9	10.1
SS	8.7	8.9	8.9	9.1

Process Samples: Hydrogen Onset			
	SB4-62	SB4-63	SB4-64
	150%	160%	170%
Supernate elementals (mg/L)			
Al	12.4	91.85	247.5
B	8.245	7.485	5.325
Ba	1.32	1.095	1.0075
Ca	2465	3045	2495
Cr	<0.100	0.434	1.965
Fe	<0.100	3.83	12.45
K	953.5	1190	933.5
Li	<10.0	<10.0	<10.0
Mg	2395	3000	2390
Mn	5520	7630	6010
Na	23350	29000	22600
Ni	497.5	932.5	944.5
P	<1.00	<1.00	<1.00
Pb	<0.100	3.02	3.225
Pd	0.909	0.9305	0.8865
Rh	4.14	4.905	8.24
Ru	5.47	3.975	27
S	545	676.5	550.5
Sr	5.32	5.49	4.84
Ti	<0.100	<0.100	<0.100
Zn	34.7	74.05	75.85
Zr	0.129	0.159	0.142
Anions (mg/kg)			
NO2	<100	<100	<100
NO3	23895	26100	24850
HCO2	58260	59600	57750
SO4	3485	3485	3915
PO4	<100	<100	<100
C2O4	<100	<100	242
Cl	205	218	160
pH Data			
pH	5.14	nm	nm
Solids Data (wt %)			
TS	21.3	23.6	20.0
IS	11.6	12.6	10.7
Supernate	11.0	12.6	10.5
SS	9.7	11.0	9.4

Process Samples: Hydrogen Peak			
	SB4-62	SB4-63	SB4-64
	150%	160%	170%
Supernate elementals (mg/L)			
Al	2.945	100.2	222
B	8.98	6.67	5.72
Ba	1.73	1.605	1.135
Ca	2965	3435	2975
Cr	<0.100	0.458	1.48
Fe	<0.100	29.7	84.2
K	1190	1335	1140
Li	<10.0	<10.0	<10.0
Mg	2905	3355	2890
Mn	6630	8740	7675
Na	27750	31850	27050
Ni	310.5	1025	1135
P	<1.00	<1.00	<1.00
Pb	<0.100	3.535	5.5
Pd	0.894	0.8935	0.859
Rh	1.53	4.53	7.845
Ru	<0.100	3.55	14.2
S	669.5	767	668
Sr	6.15	6.165	5.52
Ti	<0.100	<0.100	<0.100
Zn	17.05	88.7	101
Zr	0.1375	0.141	0.1505
Anions (mg/kg)			
NO2	<100	<100	<100
NO3	29885	31250	28900
HCO2	67300	66300	65900
SO4	4890	5395	5745
PO4	<100	<100	<100
C2O4	<100	<100	175
Cl	250	240	221
pH Data			
pH	5.91	nm	nm
Solids Data (wt %)			
TS	25.3	26.2	23.5
IS	13.5	14.2	12.4
Supernate	13.6	14.0	12.6
SS	11.8	12.0	11.1

Table A- 2. SRAT Product Results

	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%
Elementals (wt% Calcine Solids)				
Al	15.3	15	15	14.9
B	<0.100	<0.100	<0.100	<0.100
Ba	0.156	0.156	0.154	0.154
Ca	1.81	1.80	1.76	1.79
Cr	0.130	0.124	0.121	0.118
Fe	20.8	20.1	19.45	19.85
K	0.350	0.3530	0.355	0.366
Li	<0.100	<0.100	<0.100	<0.100
Mg	1.461	1.461	1.50	1.54
Mn	4.73	4.63	4.365	4.445
Na	14.85	14.95	14.9	15.35
Ni	1.4	1.395	1.395	1.355
P	0.032	0.037	0.035	0.033
Pb	0.028	0.034	0.051	0.043
Pd	<0.010	<0.010	<0.010	<0.010
Rh	0.0141	<0.010	<0.010	0.0145
Ru	<0.010	<0.010	<0.010	0.0215
S	0.446	0.447	0.438	0.441
Si	2.07	2	2.03	2.005
Ti	0.027	0.027	0.027	0.027
Zn	0.183	0.179	0.178	0.176
Zr	0.200	0.198	0.203	0.203
Anions (mg/kg)				
NO2	260	<100	<100	<100
NO3	23400	26400	28700	30900
HCO2	60800	63700	62900	67050
SO4	1265	1495	1850	3025
PO4	<100	<100	<100	<100
C2O4	<100	<100	<100	<100
Cl	270	265	245	238.5
Density and pH				
pH	8.17	6.83	5.27	4.78
Density	1.2192	1.2172	1.2126	1.2099

	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%
Oxide Results (wt% calcine Solids)				
Al2O3	28.92	28.35	28.35	28.16
B2O3	0.00	0.00	0.00	0.00
BaO	0.174	0.174	0.172	0.172
CaO	2.54	2.52	2.46	2.51
Cr2O3	0.189	0.181	0.176	0.172
Fe2O3	29.74	28.74	27.81	28.39
K2O	0.420	0.424	0.425	0.439
Li2O	0.000	0.000	0.000	0.000
MgO	2.43	2.43	2.49	2.56
MnO2	7.47	7.32	6.90	7.02
Na2O	20.05	20.18	20.12	20.72
NiO	1.78	1.77	1.77	1.72
P2O5	0.073	0.085	0.080	0.076
PbO	0.030	0.036	0.055	0.046
PdO	0.000	0.000	0.000	0.000
RhO2	0.019	0.000	0.000	0.019
RuO2	0.000	0.000	0.000	0.028
SO4	1.34	1.34	1.31	1.32
SiO2	4.43	4.28	4.34	4.29
TiO2	0.045	0.000	0.045	0.000
ZnO	0.226	0.222	0.220	0.218
ZrO2	0.271	0.267	0.273	0.273
Solids Data (wt %)				
TS	26.41	26.58	26.40	26.23
IS	15.44	14.42	14.08	13.62
SS	10.97	12.16	12.32	12.61
CS	16.88	16.52	16.15	15.89

Table A- 3. SRAT Product Supernate Results

	SB4-61	SB4-62	SB4-63	SB4-64		SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%		130%	150%	160%	170%
Supernate Elementals (mg/L)					Supernate Elementals (% soluble)				
Al	1.395	1.33	4.22	101	Al	0.0046	0.0046	0.0150	0.3685
B	1.695	5.82	10.335	12.25	B				
Ba	<0.100	<0.100	<0.100	<0.100	Ba				
Ca	3185	3405	3365	3475	Ca	88.05	98.02	101.70	105.53
Cr	<0.100	<0.100	<0.100	<0.100	Cr				
Fe	<0.100	<0.100	2.25	43	Fe			0.0062	0.1178
K	1050	1045	1290	1280	K	150.2	153.4	193.6	190.4
Li	2.48	2.87	2.94	2.965	Li				
Mg	2695	3305	3245	3260	Mg	92.41	117.17	115.07	115.08
Mn	2135	7240	8635	8855	Mn	22.62	81.01	105.22	108.30
Na	32750	31550	30800	29250	Na	110.5	109.3	110.0	103.6
Ni	0.162	28.45	753	1130	Ni	0.0058	1.0566	28.7116	45.3348
P	1.975	1.585	1.051	1.105	P	3.090	2.225	1.597	1.820
Pb	<0.200	<0.200	0.294	3.5	Pb			0.310	4.477
Pd	<0.500	<0.500	<0.500	<0.500	Pd				
Rh	1.655	1.14	1.425	1.57	Rh	5.87			5.89
Ru	<0.100	<0.100	0.919	3.97	Ru				10.04
S	661	743	789.5	798.5	S	74.32	86.08	95.88	98.54
Si	21.85	28.55	82.55	114.5	Si	0.53	0.74	2.16	3.10
Ti	<0.100	<0.100	<0.100	<0.100	Ti				
Zn	<0.100	0.5725	69.4	120	Zn		0.17	20.80	37.17
Zr	<0.200	<0.200	<0.100	<0.100	Zr				

Table A- 4. SME Product Results

	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%
Elementals (wt% calcined solids)				
Al	5.44	5.44	5.34	5.24
B	2.94	2.83	2.88	2.87
Ba	0.062	0.062	0.062	0.062
Ca	0.626	0.623	0.578	0.615
Cr	0.0488	0.0467	0.0520	0.0525
Fe	7.42	6.68	6.955	6.96
K	0.173	0.169	0.174	0.181
Li	2.40	2.43	2.40	2.36
Mg	0.582	0.579	0.577	0.590
Mn	1.461	1.473	1.480	1.520
Na	7.58	7.56	7.64	7.84
Ni	0.489	0.497	0.486	0.477
P	0.018	0.019	0.016	0.017
Pb	0.045	0.045	0.042	0.041
Pd	<0.010	<0.010	<0.010	<0.010
Rh	<0.010	<0.010	<0.010	<0.010
Ru	<0.010	<0.010	<0.010	<0.010
S	0.138	0.143	0.125	0.151
Si	22.7	22.1	22.3	22.0
Sr	<0.010	<0.010	<0.010	<0.010
Ti	0.013	0.013	0.013	0.013
Zn	0.083	0.069	0.065	0.064
Zr	0.077	0.077	0.076	0.074
Anions (mg/kg)				
NO2	101.5	<100	<100	<100
NO3	20200	22550	24950	23000
HCO2	52450	55500	57450	58600
SO4	2130	2525	1965	2725
PO4	<100	<100	<100	<100
C2O4	130	<100	<100	<100
Cl	220	215	206	203.5
pH and Density				
pH	7.43	6.98	5.72	5.35
Density	1.3996	1.4244	1.4126	1.4044
Solids				
TS	46.64712	47.56768	46.82634	47.13318
IS	37.31269	37.26456	35.9447	36.04549
SS	9.334431	10.30312	10.88163	11.08769
CS	38.80945	39.11076	38.3146	38.16292
Waste Loading				
	35.43469	34.71821	35.63438	36.70938

	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%
Oxides (wt% calcine solids)				
Al2O3	10.28	10.28	10.09	9.90
B2O3	9.47	9.12	9.26	9.23
BaO	0.069	0.070	0.069	0.069
CaO	0.876	0.872	0.809	0.860
Cr2O3	0.071	0.068	0.076	0.077
Fe2O3	10.61	9.55	9.95	9.95
K2O	0.208	0.202	0.208	0.217
Li2O	5.17	5.22	5.15	5.06
MgO	0.966	0.961	0.958	0.979
MnO2	2.31	2.33	2.34	2.40
Na2O	10.23	10.20	10.31	10.58
NiO	0.620	0.631	0.617	0.606
P2O5	0.041	0.043	0.035	0.039
PbO	0.048	0.049	0.045	0.044
PdO	0.000	0.000	0.000	0.000
RhO2	0.000	0.000	0.000	0.000
RuO2	0.000	0.000	0.000	0.000
SO4	0.415	0.429	0.374	0.453
SiO2	48.61	47.19	47.62	47.08
SrO	0.000	0.000	0.000	0.000
TiO2	0.022	0.000	0.022	0.000
ZnO	0.103	0.085	0.081	0.079
ZrO2	0.103	0.104	0.102	0.100

Table A- 5. SME Product Supernate Results

	SB4-61	SB4-62	SB4-63	SB4-64			SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%			130%	150%	160%	170%
Supernate Elementals (mg/L)						Supernate Elementals (% Soluble)				
Al	1.25	1.285	2.11	6.355		Al	0.004	0.004	0.007	0.020
B	768	1060	1015	1185		B	4.220	6.002	5.902	6.931
Ba	<0.100	<0.100	<0.100	<0.100		Ba				
Ca	3560	3650	3900	4085		Ca	91.932	93.977	112.902	111.404
Cr	<0.100	<0.100	<0.100	<0.100		Cr				
Fe	<0.100	<0.100	3.38	11.8		Fe			0.008	0.028
K	993	991	1510	1515		K	92.582	94.272	145.502	140.270
Li	1750	1960	1205	1390		Li	11.766	12.943	8.411	9.891
Mg	3155	3600	3685	3740		Mg	87.570	99.748	106.771	106.230
Mn	2855	6970	9495	9455		Mn	31.560	75.897	107.257	104.243
Na	36500	36000	34700	34850		Na	77.780	76.434	75.932	74.493
Ni	4.895	27.4	558.5	917.5		Ni	0.162	0.885	19.212	32.234
P	2.07	1.775	1.265	0.971		P	1.879	1.520	1.364	0.957
Pb	<0.200	<0.200	<0.200	<0.200		Pb				
Pd	<0.500	<0.500	<0.500	<0.500		Pd				
Rh	1.51	1.105	1.15	1.285		Rh				
Ru	<0.100	<0.100	0.691	2.66		Ru				
S	1010	1030	980.5	971.5		S	118.067	115.569	131.665	107.819
Si	36.4	50.05	69.8	89.6		Si	0.026	0.036	0.052	0.068
Sr	7.35	8.445	8.66	8.815		Sr				
Ti	<0.100	<0.100	<0.100	<0.100		Ti				
Zn	<0.100	<0.100	45.3	97.7		Zn			11.651	25.583
Zr	<0.200	<0.200	<0.100	<0.100		Zr				

Table A- 6. Condensate Sample Results

	SRAT Dewater				**SME Dewater**				**MWWT**				**FAVC**			
	SB4-61	SB4-62	SB4-63	SB4-64	SB4-61	SB4-62	SB4-63	SB4-64	SB4-61	SB4-62	SB4-63	SB4-64	SB4-61	SB4-62	SB4-63	SB4-64
	130%	150%	160%	170%	130%	150%	160%	170%	130%	150%	160%	170%	130%	150%	160%	170%
Elementals (mg/L)																
Al	0.15	0.1435	0.1395	0.151	0.12	0.117	0.1275	0.1235	0.1365	0.124	0.128	0.169	0.3615	0.4365	0.3825	0.226
B	0.0985	0.0765	0.107	0.074	0.3335	0.3095	0.2615	0.3235	0.065	0.0645	0.05	0.0565	0.712	0.2965	0.25	0.297
Ba	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Ca	0.169	0.081	0.1015	0.097	0.059	0.0635	0.116	0.0625	0.057	0.0595	0.061	0.075	0.272	0.1155	0.3805	0.191
Cd	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Cr	0.0335	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	2.18	<0.010	<0.010	<0.010
Fe	0.1405	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	5.4	0.5035	<0.010	<0.010
K	0.2085	0.193	0.195	0.191	0.2025	0.191	0.194	0.1905	0.194	0.189	0.1895	0.1935	0.2715	0.256	0.2875	0.2265
Li	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
Mg	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	0.084	<0.010	0.232	<0.010
Mn	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	0.8185	0.0465	0.7495	0.185
Na	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
Ni	0.1335	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	0.059	<0.010	0.053	<0.010
Pb	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
S	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200	<0.200
Si	62.25	93.05	130	151	21.4	36.5	68.3	73.9	99.6	77.95	55.25	68.45	52.45	73.25	112	144.5
Ti	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Zn	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Zr	<0.100	<0.100	0.0165	0.015	<0.100	<0.100	0.015	0.0145	<0.100	<0.100	0.015	0.015	<0.100	<0.100	0.0295	0.0195
Anions (mg/kg)																
NO2	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
NO3	4135	<100	2250	2595	<100	<100	<100	<100	<100	<100	<100	<100	290500	228000	255500	210000
HCO2	579.5	1085	2185	3410	<100	<100	635.5	1080	<100	<100	828	1820	<100	<100	977	1345
SO4	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	162	157	<100	142.5
PO4	0.116	0.063	<100	<100	0.063	0.078	<100	<100	0.377	0.19	<100	<100	0.096	0	<100	<100
C2O4	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
Cl	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
F	<100	<100	nm	nm	<100	<100	nm	nm	<100	<100	nm	nm	<100	<100	nm	nm
pH and Density																
pH	1.74	1.38	1.59	<1.00	9.27	3.41	2.8	1.35	9.56	3.08	2.97	1.51	2.44	2.32	2.44	2.51
Density	1.01	1	1	1	1	1	1	1	1	1.01	1	1	1.15	1.12	1.14	1.13

Table A- 7. Formate and Nitrate Balance

Inputs	Units	SB4-61	SB4-62	SB4-63	SB4-64
Acid Stiochiometry		130%	150%	160%	170%
Sludge Added	grams	2,845.0	2,845.0	2,845.0	2,845.0
Heel	grams	0	0	0	0
Sludge Nitrite	mg/kg	14,495	14,495	14,495	14,495
Sludge Nitrate	mg/kg	9331	9331	9331	9331
Nitric Added	ml	17.147	28.461	34.087	39.797
Nitric Molarity	molar	10.57	10.57	10.57	10.57
Formic Added	ml	139.084	156.596	165.365	174.097
Formic Molarity	molar	23.6	23.6	23.6	23.6
SRAT Product	grams	2245	2333	2376	2420
SRAT Product Nitrite	mg/kg	260	0	0	0
SRAT Product Nitrate	mg/kg	23400	26400	28700	30900
SRAT Product Formate	mg/kg	60800	63700	62900	67050
Calculations					
SRAT Data					
Nitrite in start of batch	moles	0.896	0.896	0.896	0.896
Nitrate in start of batch	moles	0.428	0.428	0.428	0.428
Formate in start of batch	moles	0.000	0.000	0.000	0.000
Nitrate added	moles	0.181	0.301	0.360	0.421
Formate added	moles	3.282	3.696	3.903	4.109
Nitrate in end of batch	moles	0.847	0.993	1.100	1.206
Formate in end of batch	moles	3.034	3.302	3.321	3.606
Nitrate In	moles	0.609	0.729	0.788	0.849
Nitrite Converted	moles	0.238	0.264	0.311	0.357
Formate In	moles	3.282	3.696	3.903	4.109
Formate Destroyed	moles	0.249	0.394	0.581	0.503
Nitrite Conversion	**%**	**26.55**	**29.47**	**34.74**	**39.85**
Formate Destruction	**%**	**7.58**	**10.66**	**14.90**	**12.24**
SME Product Data					
SRAT Product Sample Size	grams	306	342	314	327
SME product	grams	2424	2478	2506	2533
Nitrate	mg/kg	20200	22550	24950	23000
Formate	mg/kg	52450	55500	57450	58600
Nitrate in end of batch	moles	0.79	0.90	1.01	0.94
Formate in end of batch	moles	2.83	3.06	3.20	3.30
Formate Added	grams	8.766	8.8	8.82	8.84
Nitrate Lost	moles	-0.058	-0.054	-0.054	0.104
Formate Lost	moles	0.384	0.422	0.298	0.484
Nitrate Lost	**%**	**-6.83**	**-5.41**	**-4.92**	**8.58**
Formate Lost	**%**	**12.65**	**12.77**	**8.98**	**13.42**

Table A- 8. SRAT Cycle Run Data

Parameter	Units	SB4-61	SB4-62	SB4-63	SB4-64
		130%	150%	160%	170%
GC Calibration Gas		AL1276	AL1276	AL1276	AL1276
Pre-Run Leak Check In	ml/min	90	90	90	90
Pre-Run Leak Check Out	ml/min	86.8	91.6	84.5	85.8
Post-Run Leak Check In	ml/min	90	90	90	90
Post-Run Leak Check Out	ml/min	95.3	83.6	87.8	91.1
pH Pre-Run Cal: Buffer 4		4.01	4.01	4.01	4.01
pH Pre-Run Cal: Buffer 10		10	10	10	10.03
pH Pre-Run Cal: Buffer 7		7.03	6.97	7.09	6.99
pH Post-Run Cal: Buffer 4		4.4	4.08	4.4	4.08
pH Post-Run Cal: Buffer 10		10	9.49	10.06	9.54
pH Post-Run Cal: Buffer 7		7.31	6.77	7.4	6.82
Air Purge	ml/min	728.3	728.3	728.3	728.3
He Purge	ml/min	3.64	3.64	3.64	3.64
MWWT Water Added	grams	48.5	53.9	60.7	52.3
MWWT Final Mass	grams	50.64	53.93	56.72	58.18
FAVC Final Mass	grams	27.65	25.59	24.32	24.59
Additions					
Sludge Added	grams	2845	2845	2845	2845
AgNO3	grams	0.0882	0.0884	0.088	0.0882
HgO	grams	5.4018	5.4016	5.4015	5.4017
Pd(NO3)2*H2O	grams	0.0492	0.049	0.0493	0.0489
Rh(NO3)3*2H2O	grams	1.0961	1.0958	1.0956	1.0956
RuCl3	grams	0.5908	0.5907	0.591	0.5911
Flush Water	grams	50	50	50	50
Antifoam prior to acid	grams	5.8	5.8	5.8	5.8
Water with 1st antifoam addition	grams	5.78	5.8	5.8	5.8
Additional Antifoam		2.91	0	2.9	0
Water with addition antifoam		2.96	0	2.9	0
Antifoam prior to boiling	grams	14.51	14.51	14.51	14.51
Water with 2nd antifoam addition	grams	14.51	14.51	14.51	14.51
Total	grams	46.47	40.62	46.42	40.62
Ratio: Formic to Nitric Acid		0.9477	0.9247	0.9155	0.9071
Nitric Acid					
Molarity	Molar	10.57	10.57	10.57	10.57
Target Addition	ml	17.147	28.461	34.087	39.797
Start Time		826	810	1824	1810
Starting Weight	grams	1525.9	1228.4	1518.6	1503.5
Target Flowrate	ml/min	0.84	0.84	0.84	0.84
End Time		901	848	1903	1858
Ending Weight	grams	1504	1190	1473.6	1452

Parameter	**Units**	**SB4-61**	**SB4-62**	**SB4-63**	**SB4-64**
		130%	**150%**	**160%**	**170%**
Addition Time	min	35	38	39	48
Addition Weight	grams	21.9	38.4	45	51.5
Addition Volume	ml	16.7	29.3	34.4	39.3
% of Target		-2.5	3.0	0.8	-1.2
Actual Flowrate	ml/min	0.48	0.77	0.88	0.82
Formic Acid					
Molarity	Molar	23.6	23.6	23.6	23.6
Target Addition	ml	139.084	156.596	165.365	174.097
Start Time		910	859	1911	1917
Starting Weight	grams	1391.7	1619.3	1352.7	1296.1
Target Flowrate	ml/min	0.85	0.85	0.85	0.85
End Time		1156	1207	2230	2246
Ending Weight	grams	1225.5	1432.3	1155.5	1088.4
Addition Time	min	166	188	199	209
Addition Weight	grams	166.2	187	197.2	207.7
Addition Volume	ml	137.4	154.5	163.0	171.7
% of Target		-1.2	-1.3	-1.4	-1.4
Actual Flowrate	ml/min	0.83	0.82	0.82	0.82

Table A- 9. SME Cycle Run Data

Parameter	Units	SB4-61	SB4-62	SB4-63	SB4-64
		130%	150%	160%	170%
Air Purge	ml/min	208	209	210	210
He Purge	ml/min	1.04	1.05	1.05	1.05
Boil-Up Rate Target	grams/min	3.78	3.79	3.8	3.81
Antifoam Addition	grams	2	2.08	2.9	2.9
Water with Antifoam	grams	2	2.08	2.9	2.9
Frit Addition 1					
Frit amount	grams	291.9	293.3	293.92	294.5
Formic Amount	grams	4.383	4.4	4.41	4.42
Water Amount	grams	287.5	288.9	289.54	290.1
Start of Boiling		508	1620	1642	
Dewater	grams	292.13	293.3	294	299.4
End of Boiling		629	1829		1555
Time at Boiling	min	81	129		
Boil-Up Rate	g/min	3.61	2.27		
Frit Addition 2					
Frit amount	grams	291.9	293.3	293.88	294.5
Formic Amount	grams	4.383	4.4	4.41	4.42
Water Amount	grams	287.5	258.9	289.45	290.1
Start of Boiling		730	710	1650	1641
Dewater	grams	293.57	293.77	293.9	294.5
End of Boiling		837	827	1742	1755
Time at Boiling	min	67	77	52	74
Boil-Up Rate	g/min	4.38	3.82	5.65	3.98
Total Frit Additions					
Frit amount	grams	583.8	586.6	587.8	589
Formic Amount	grams	8.766	8.8	8.82	8.84
Water Amount	grams	575	547.8	578.99	580.2
Dewater	grams	585.7	587.07	587.9	593.9
Time at Boiling	min	148	206	52	74
Final Dewater Amount	grams	152.22	195.03	214.2	238.2
End of Boiling		957	1011	1845	1855
Time at Boiling	min	39	104	63	60
Boil-Up Rate	g/min	3.90	1.88	3.40	3.97
Final pH		6.82	6.03	5.5	5.1
SME Product Mass	grams				
Time at Boiling (SME Cycle)	hours	3.12	5.17		
Total Time at Boiling (SRAT+SME)	hours	18.63	20.60		
Total Frit Addition	grams	583.8	586.6	587.8	589
SME Sample 1	grams	142.77	143.95	157.09	136.4
SME Sample 2	grams	153.49	145.52	154.03	145.59
Total Dewater (SRAT/SME)		967.12	953.93	945.1	941

Table A- 10. Mercury Results: SRAT and SME Products

Run #	Type	ADS Result	Slurry Basis	Solids Content (wt%)		Hg	Hg Results - Solid	
		μg/g	μg/g	Total	Calcine	grams/kg	Total	Ca
SB4-61	SRAT Product	94.3	943	26.4	16.9	0.943	0.357	0
SB4-61	SRAT Product	40.7	407	26.4	16.9	0.407	0.154	0
SB4-61	SME Product	41.4	414	46.6	38.8	0.414	0.089	0
SB4-61	SME Product	40.4	404	46.6	38.8	0.404	0.087	0
SB4-62	SRAT Product	89.5	895	26.5	16.4	0.895	0.338	0
SB4-62	SRAT Product	90.5	905	26.5	16.6	0.905	0.342	0
SB4-62	SME Product	75.5	755	47.6	39.1	0.755	0.159	0
SB4-62	SME Product	104	1040	47.6	39.1	1.04	0.218	0
SB4-63	SRAT Product	64	640	26.4	16.2	0.64	0.242	0
SB4-63	SRAT Product	65.2	652	26.4	16.1	0.652	0.247	0
SB4-63	SME Product	44.8	448	47.1	38.3	0.448	0.095	0
SB4-63	SME Product	46.3	463	46.5	38.3	0.463	0.100	0
SB4-64	SRAT Product	100	1000	26.2	15.9	1	0.382	0
SB4-64	SRAT Product	102	1020	26.3	15.9	1.02	0.388	0
SB4-64	SME Product	65.6	656	47.2	38.3	0.656	0.139	0
SB4-64	SME Product	64	640	47	38.1	0.64	0.136	0

Table A- 11. Mercury Results: Process Samples

	130%	150%	160%	170%
Feed	1736	1736	1736	1736
H2 Onset		2620	767	1520
H2 Peak		1590	757	2540
SRAT Product	650	900	646	1010
SME Product	409	898	456	648

Appendix B. Sample/Run Results: Graphical Presentations

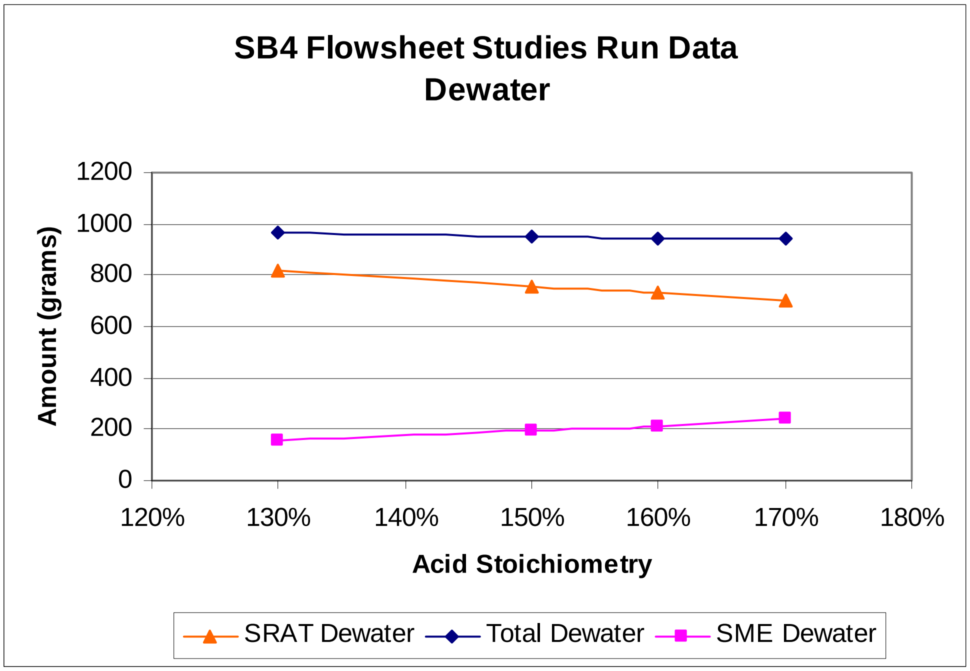

Figure B- 1. Dewater Amounts

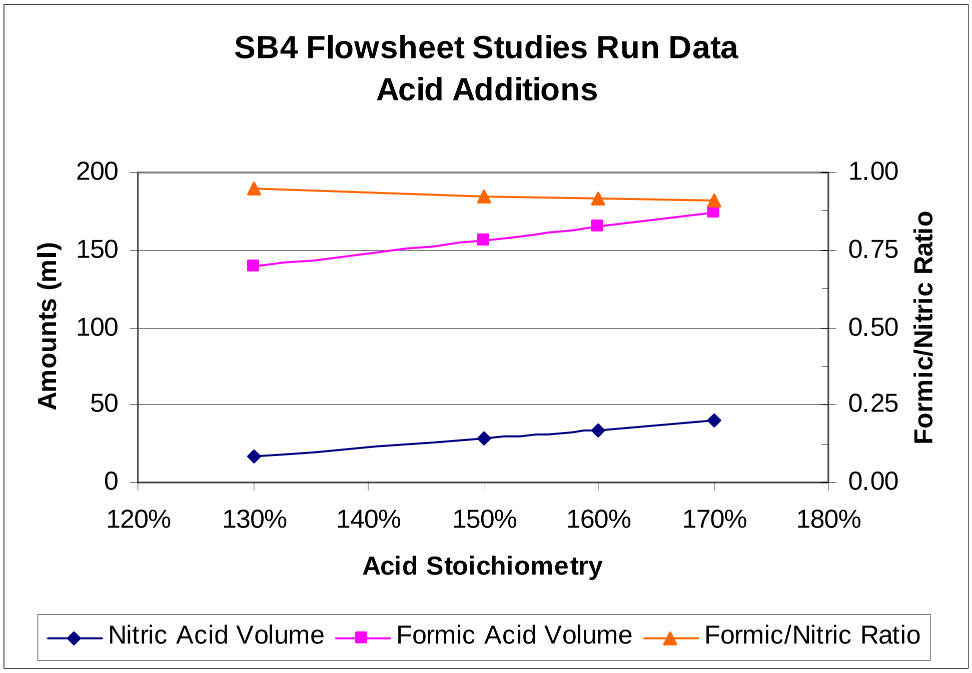

Figure B- 2. Acid Addition Volumes and Ratios

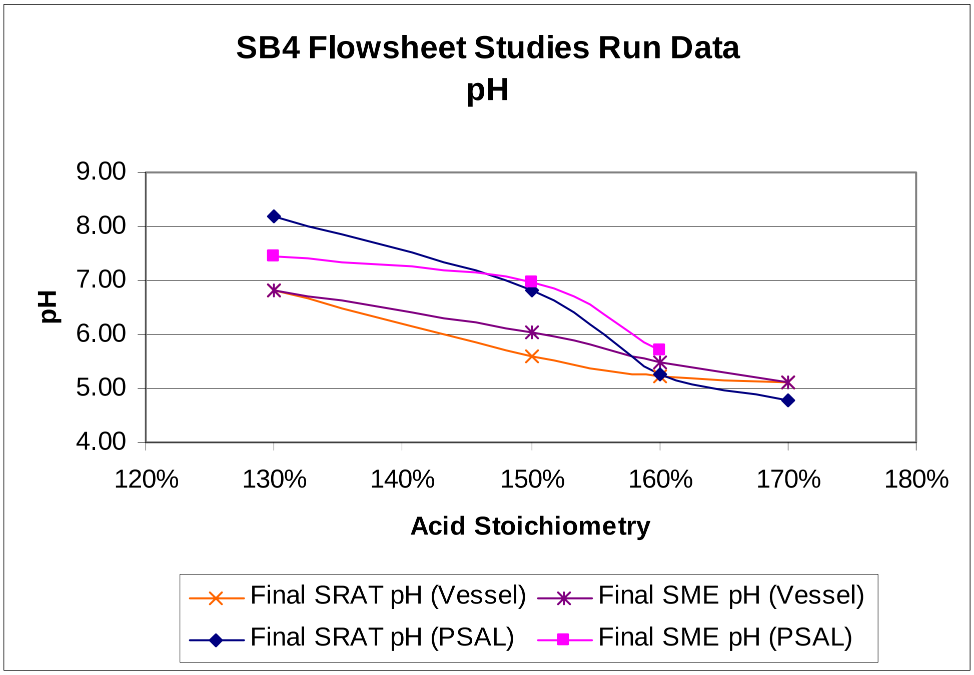

Figure B- 3. Final SRAT and SME pH

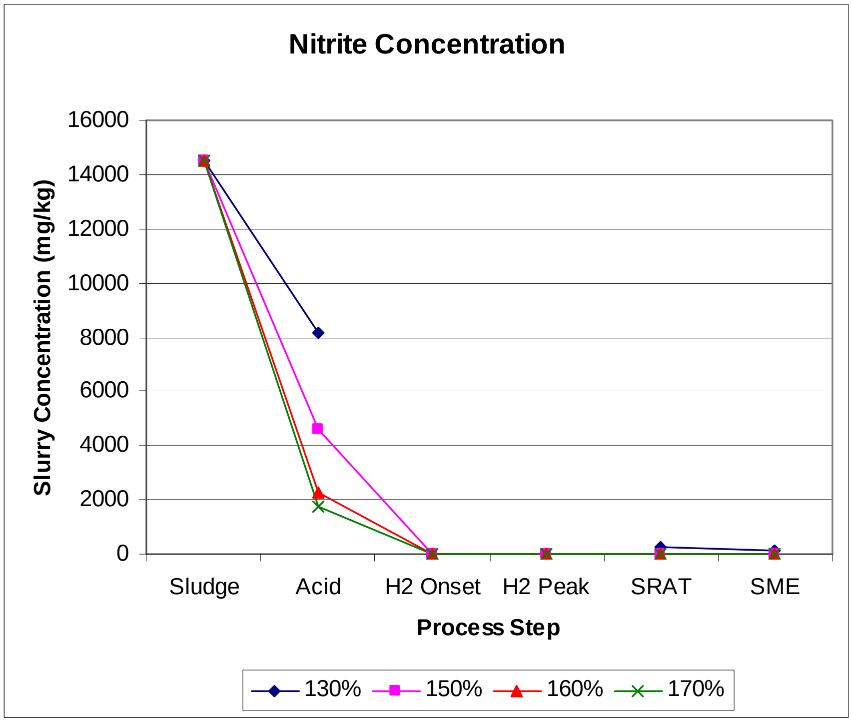

Figure B- 4. Nitrite Concentration Profile

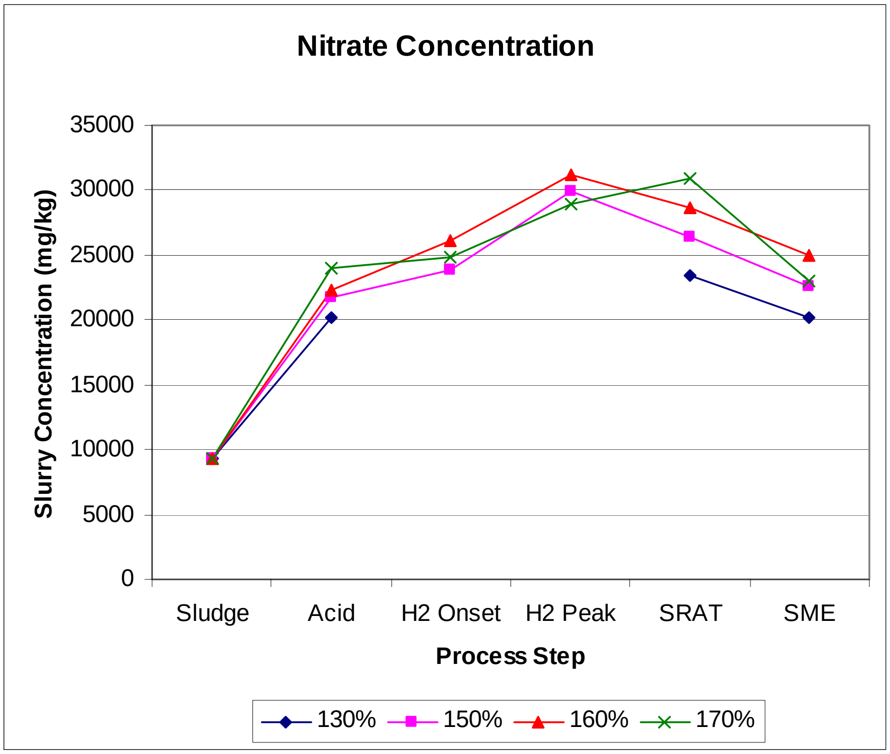

Figure B- 5. Nitrate Concentration Profile

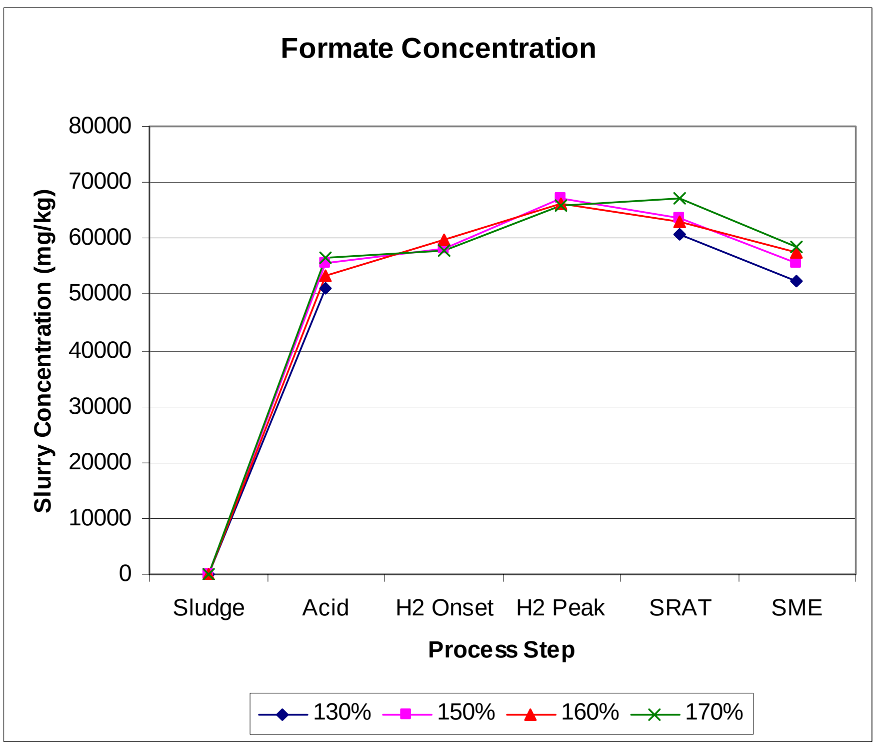

Figure B- 6. Formate Concentration Profile

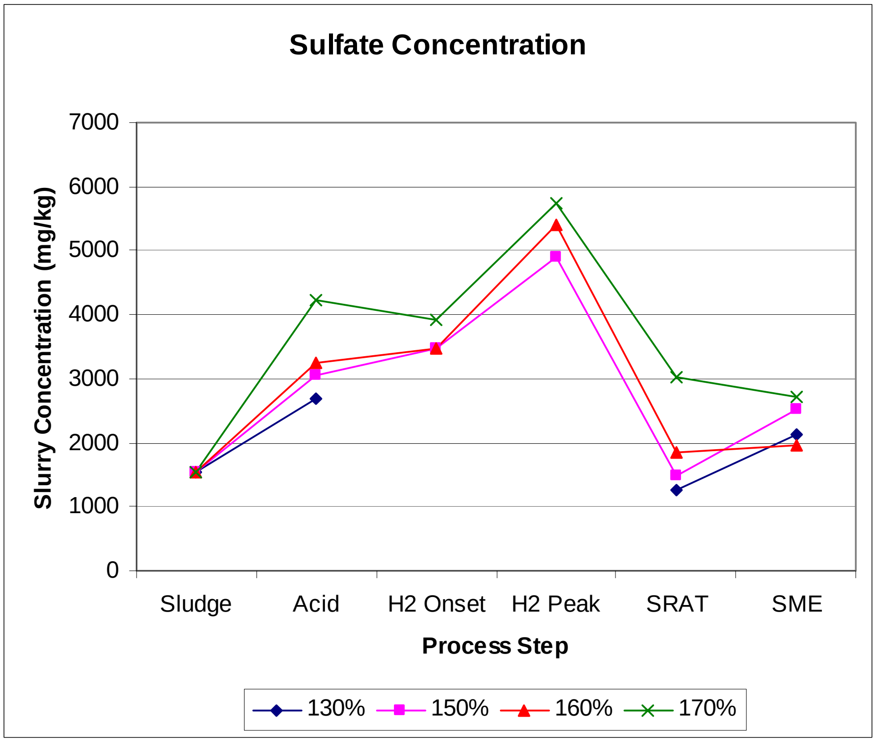

Figure B- 7. Sulfate Concentration Profile

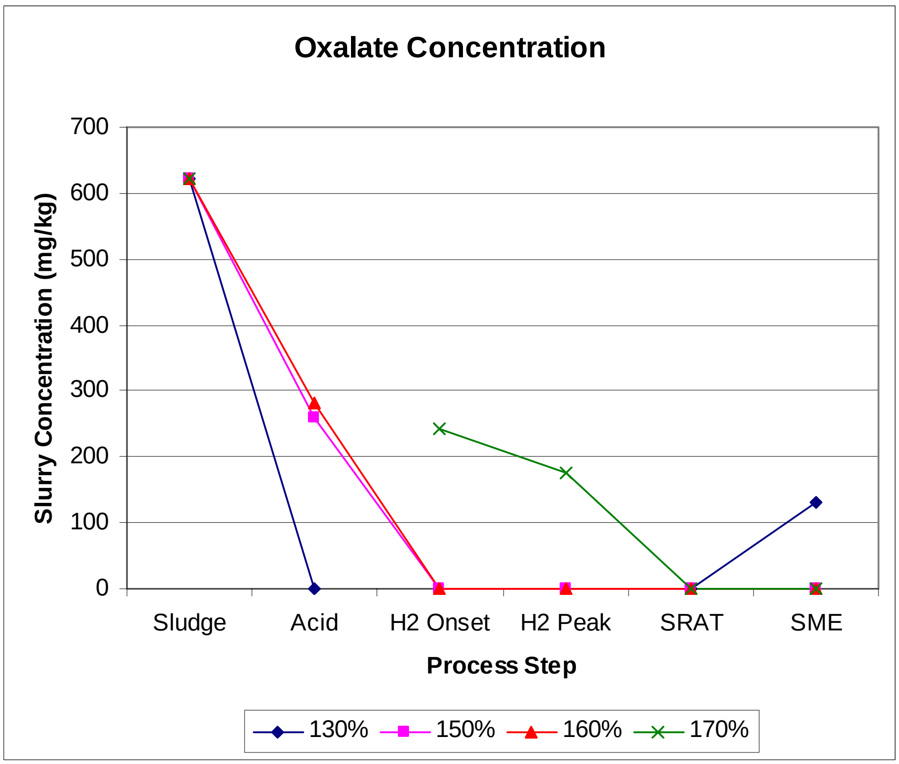

Figure B- 8. Oxalate Concentration Profile

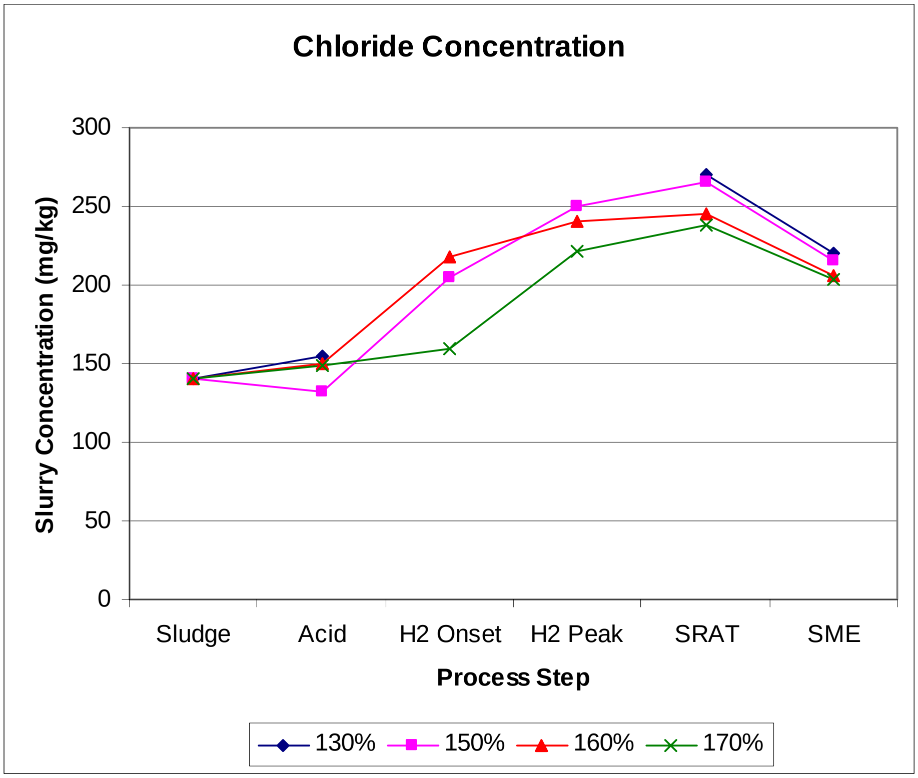

Figure B- 9. Chloride Concentration Profile

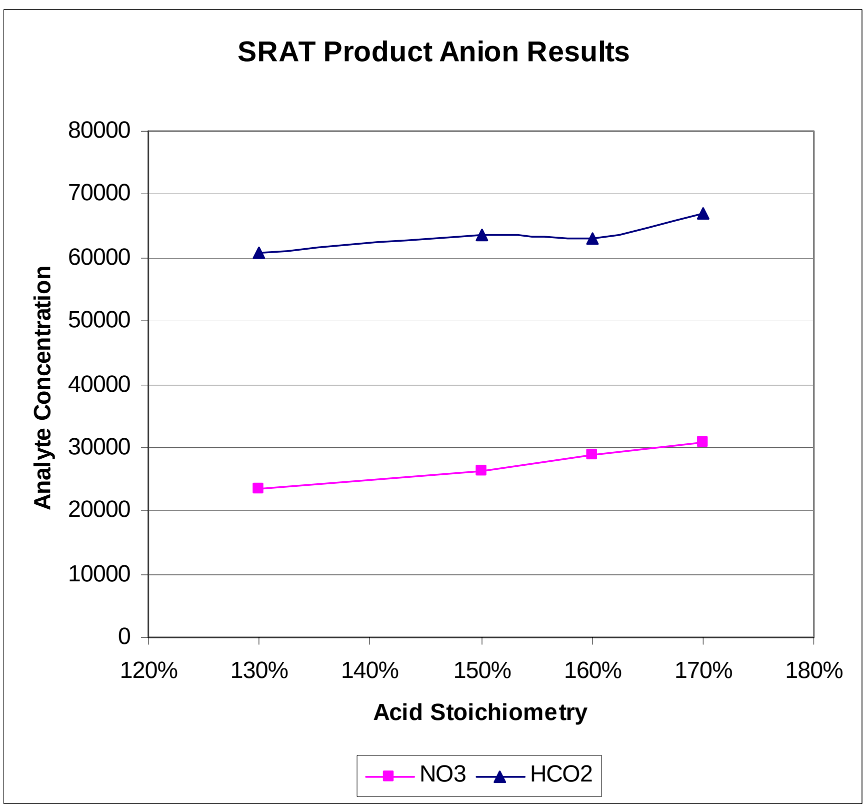

Figure B- 10. Nitrate and Formate Concentration versus Acid Stoichiometry

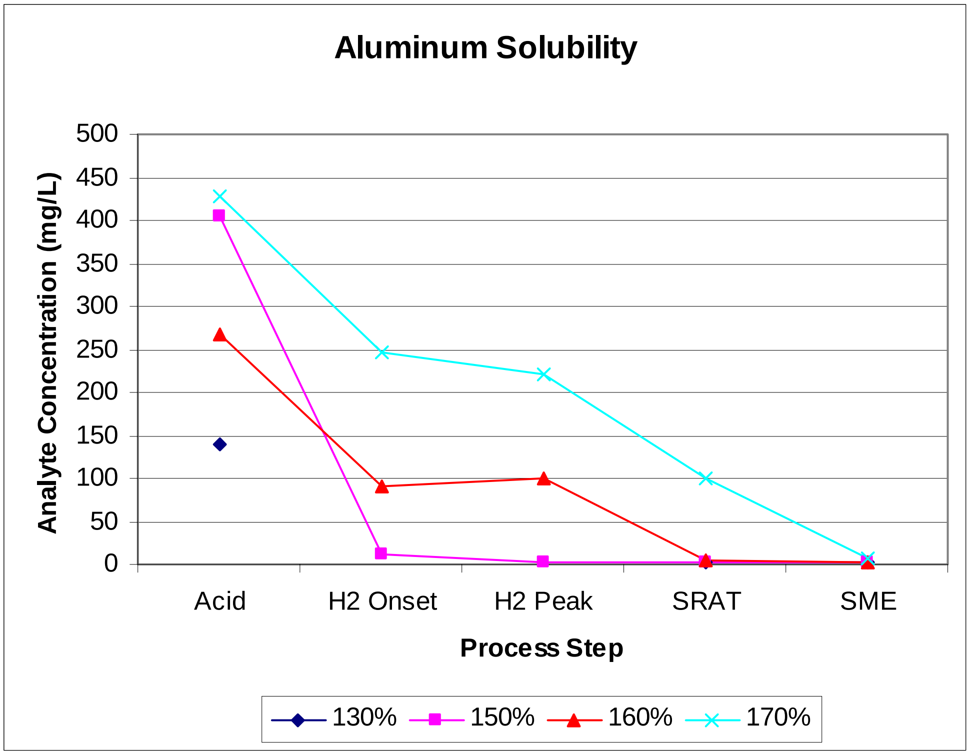

Figure B- 11. Aluminum Solubility

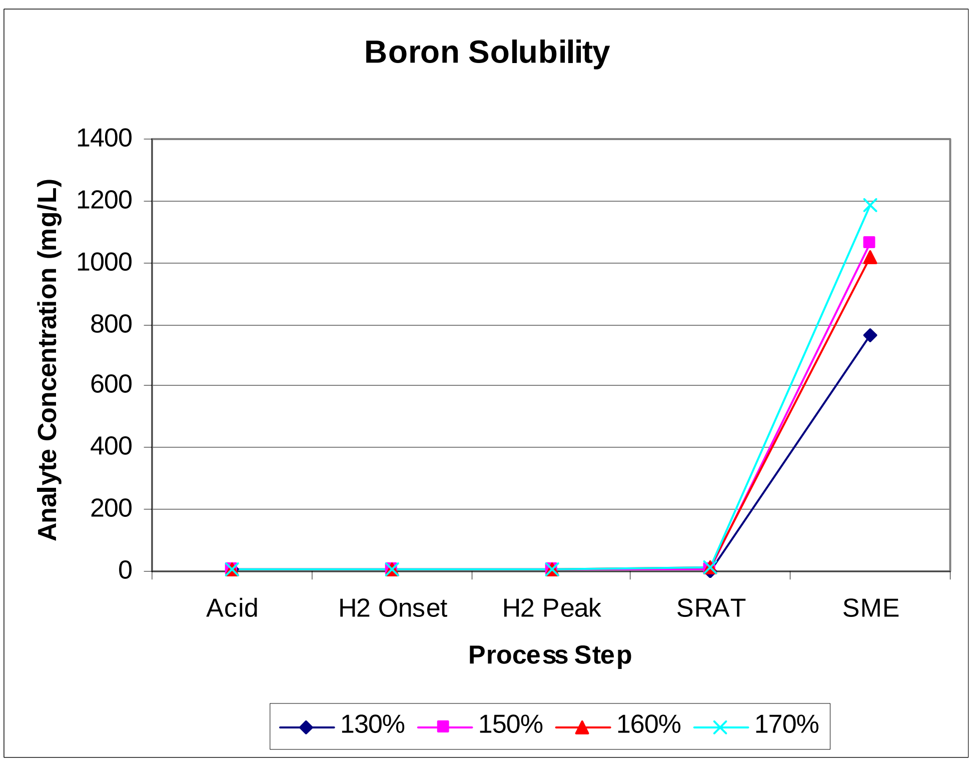

Figure B- 12. Boron Solubility

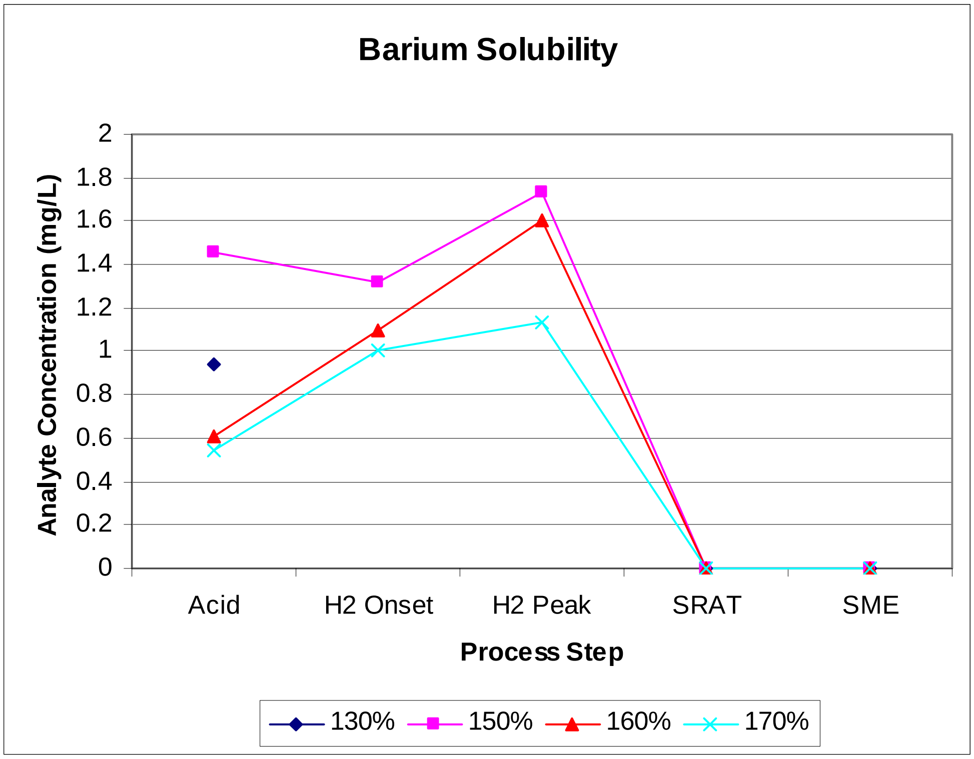

Figure B- 13. Barium Solubility

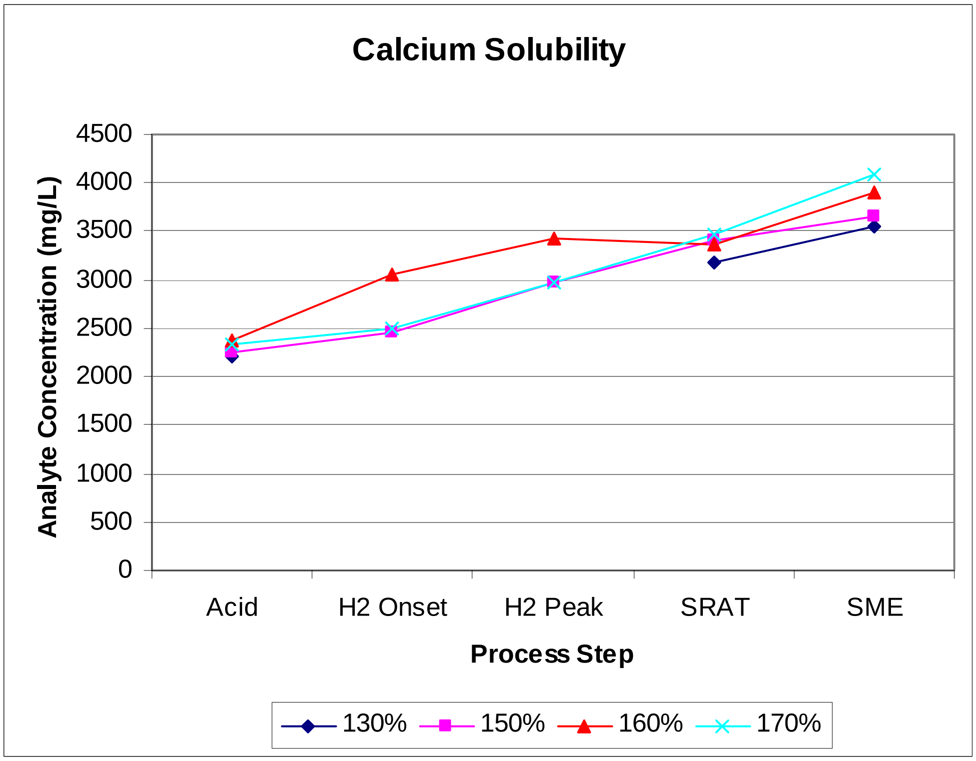

Figure B- 14. Calcium Solubility

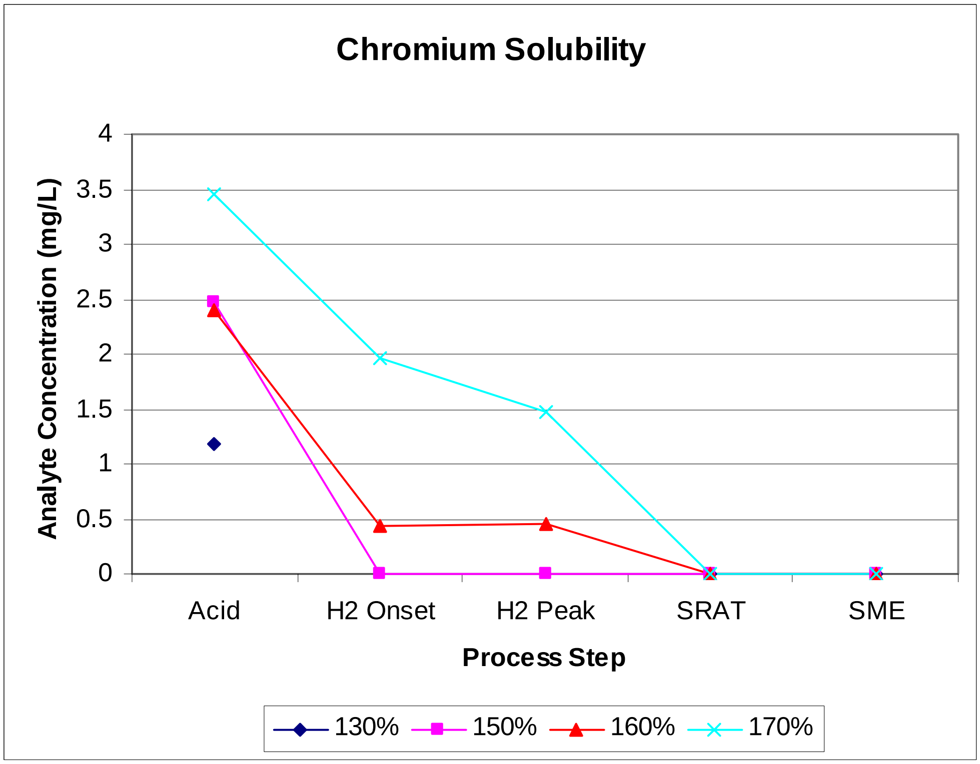

Figure B- 15. Chromium Solubility

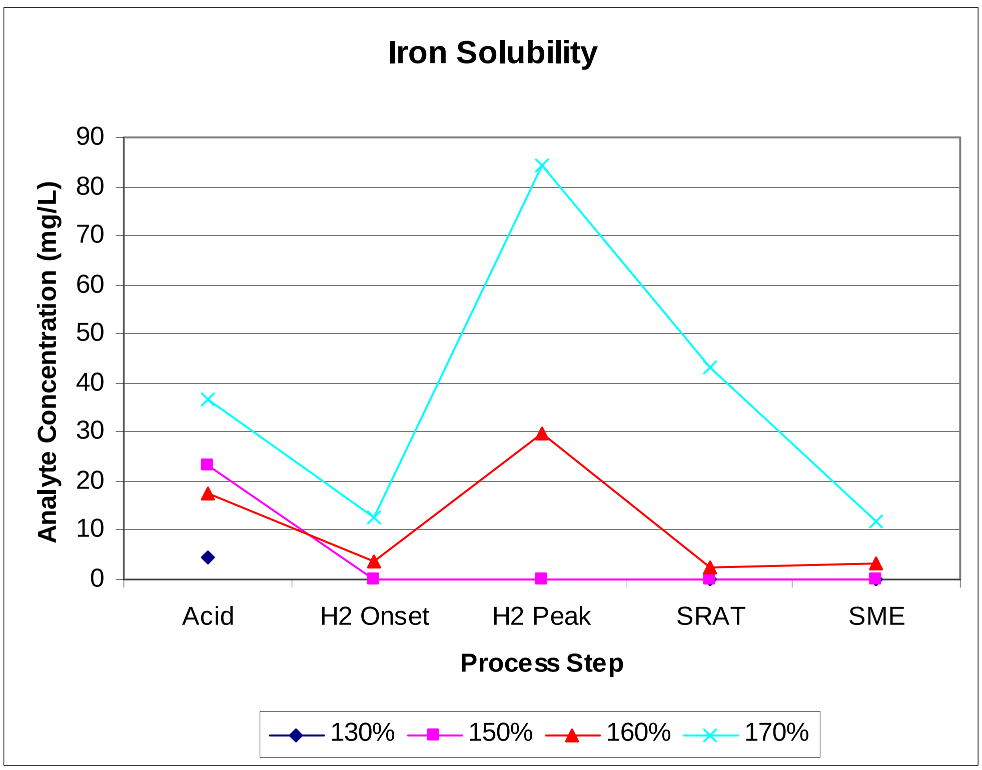

Figure B- 16. Iron Solubility

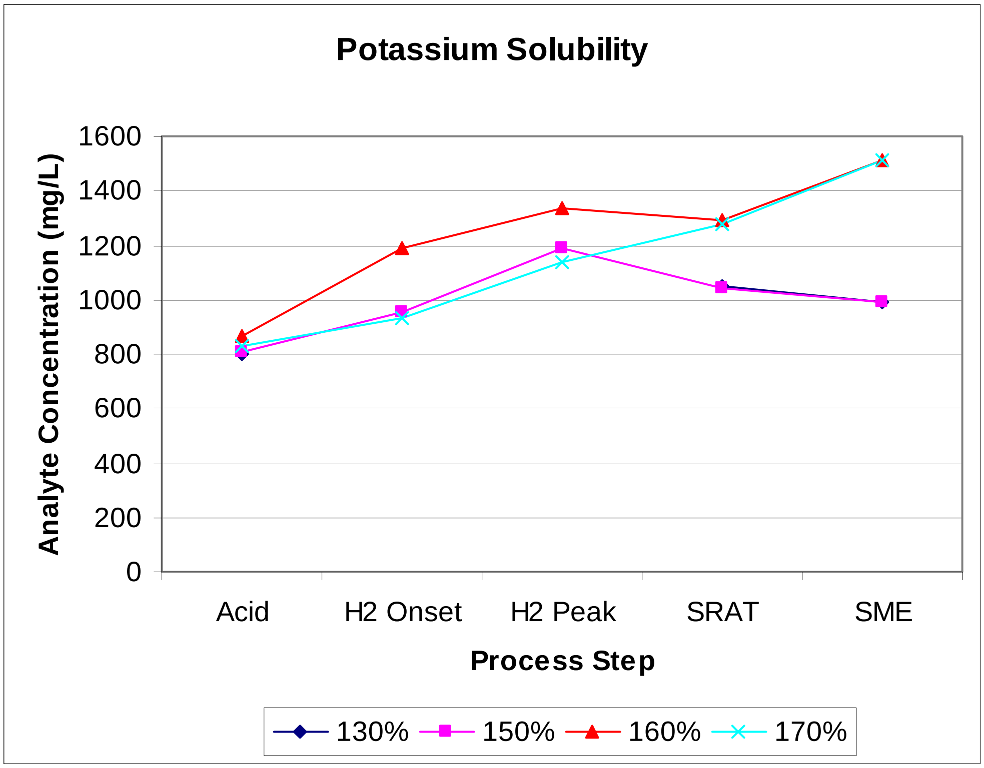

Figure B- 17. Potassium Solubility

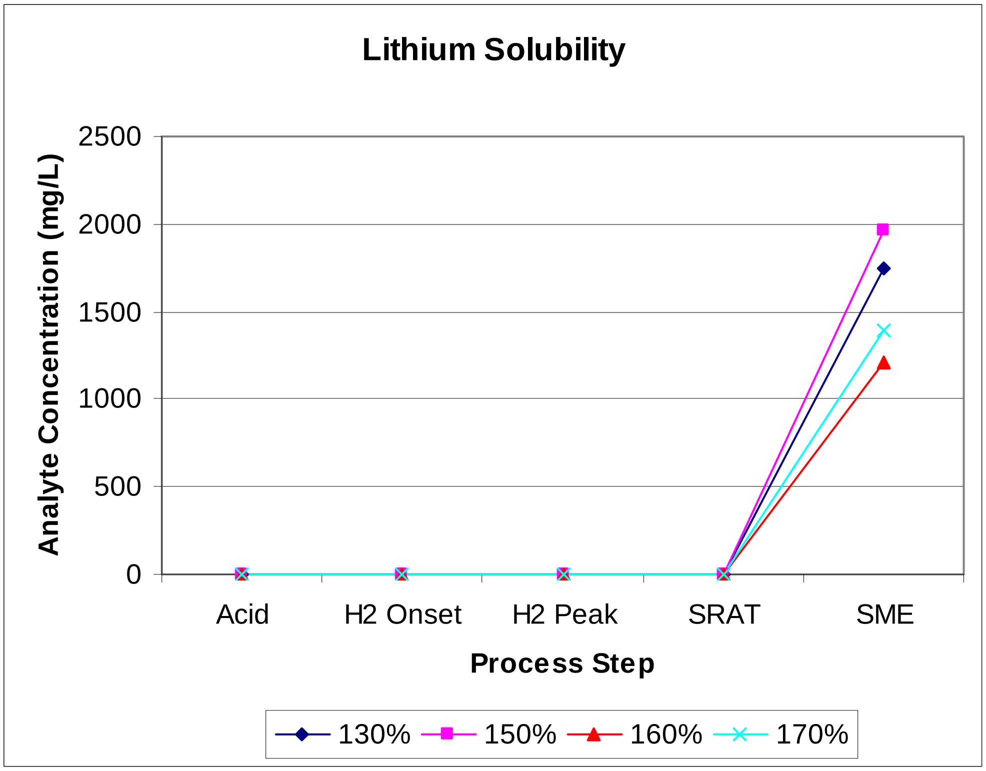

Figure B- 18. Lithium Solubility

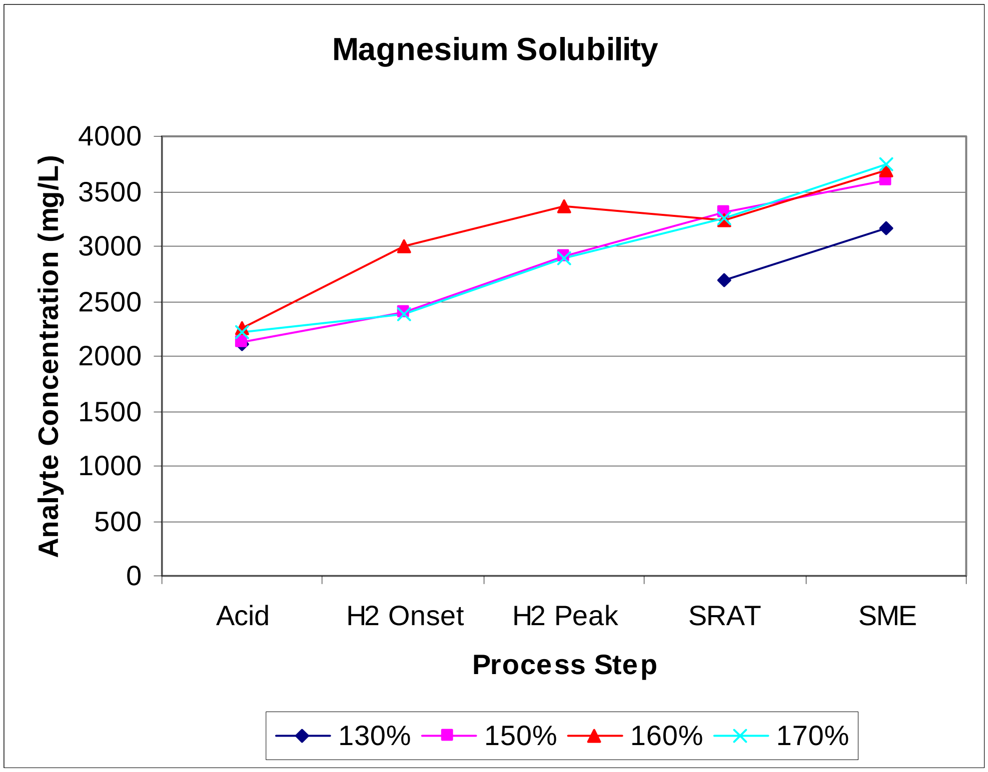

Figure B- 19. Magnesium Solubility

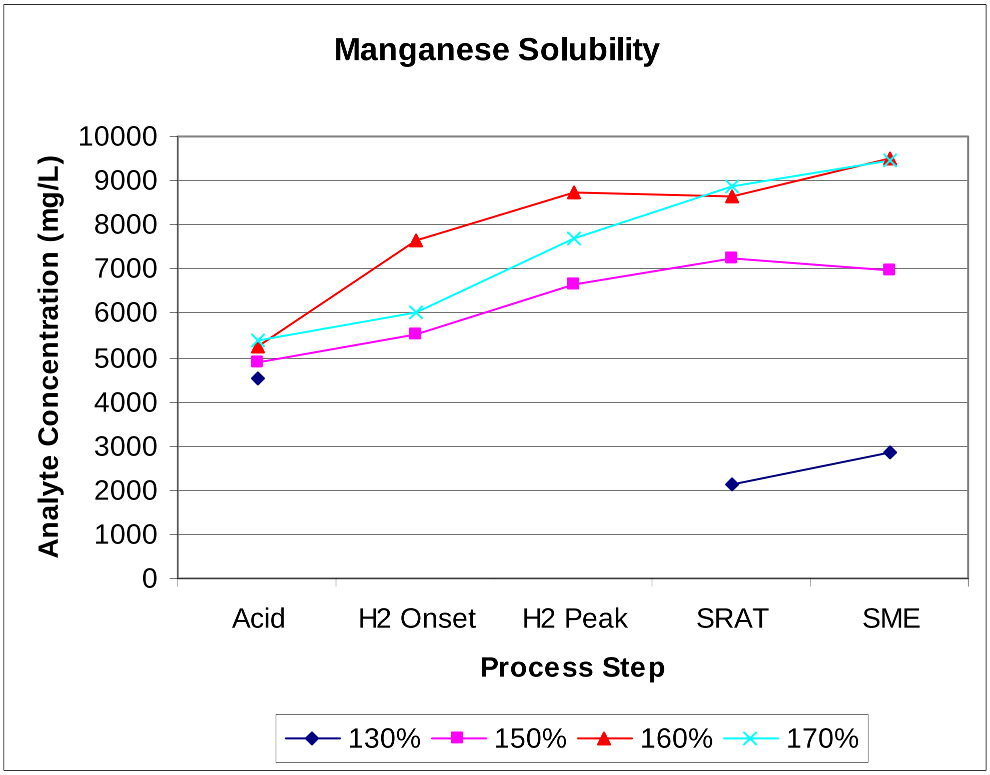

Figure B- 20. Manganese Solubility

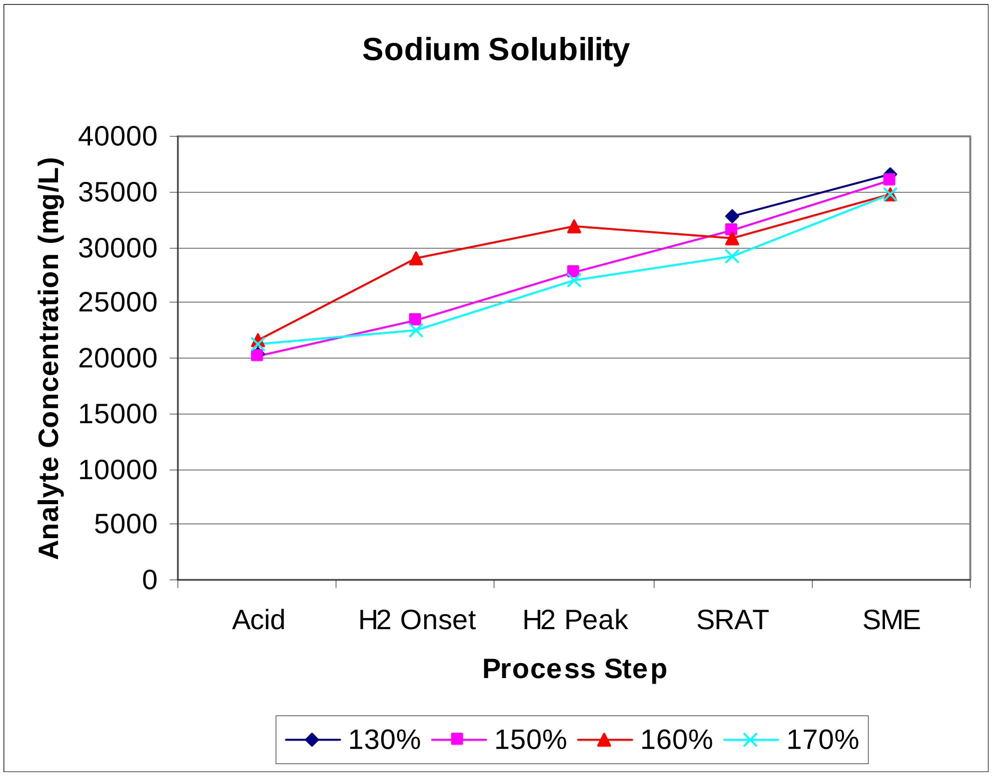

Figure B- 21. Sodium Solubility

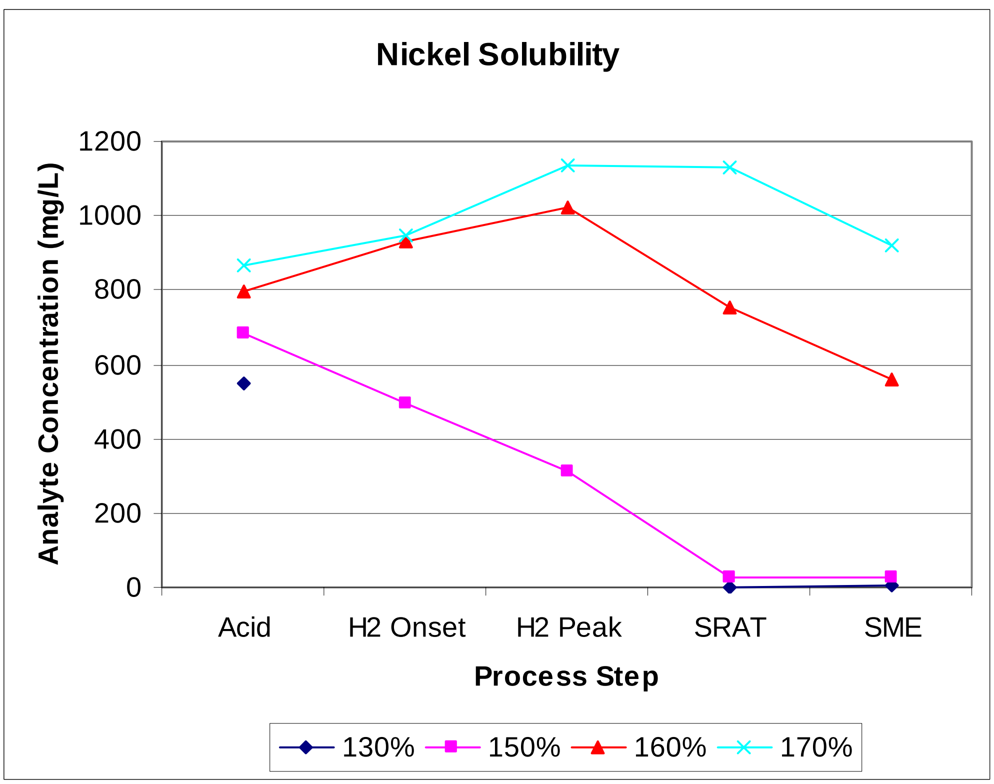

Figure B- 22. Nickel Solubility

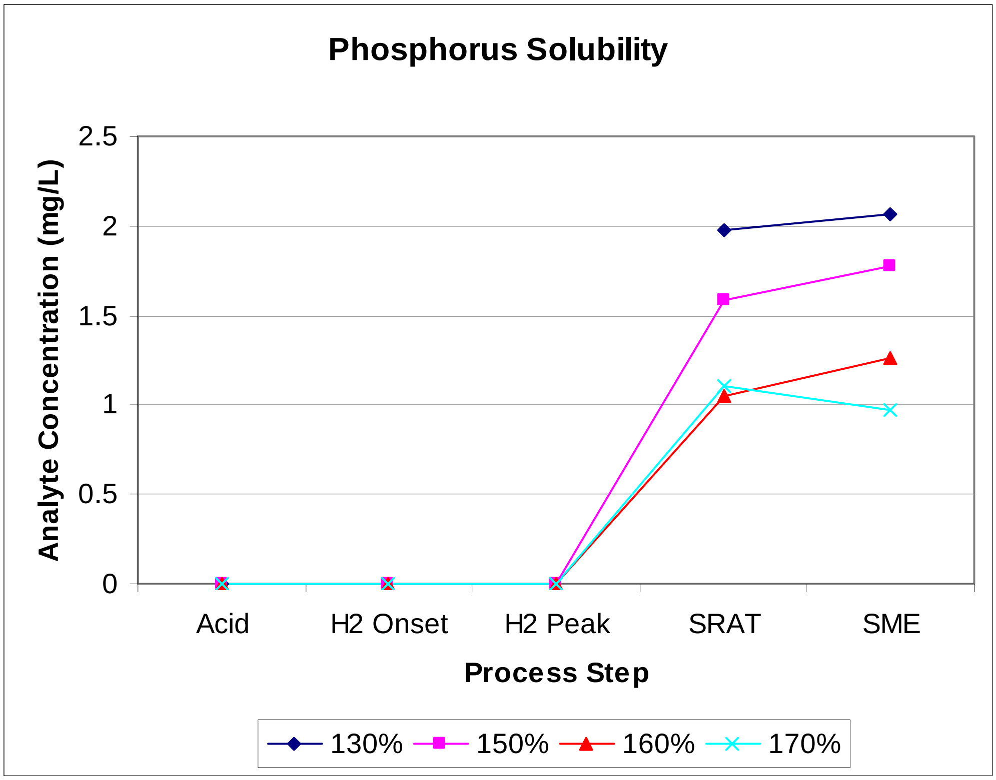

Figure B- 23. Phosphorus Solubility

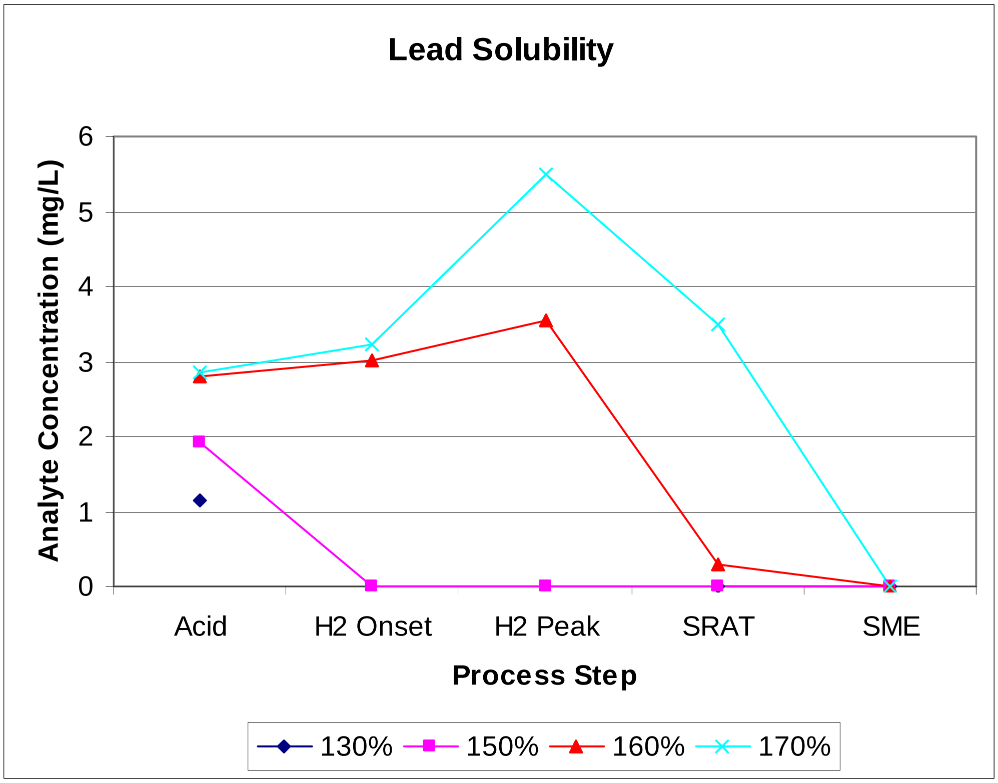

Figure B- 24. Lead Solubility

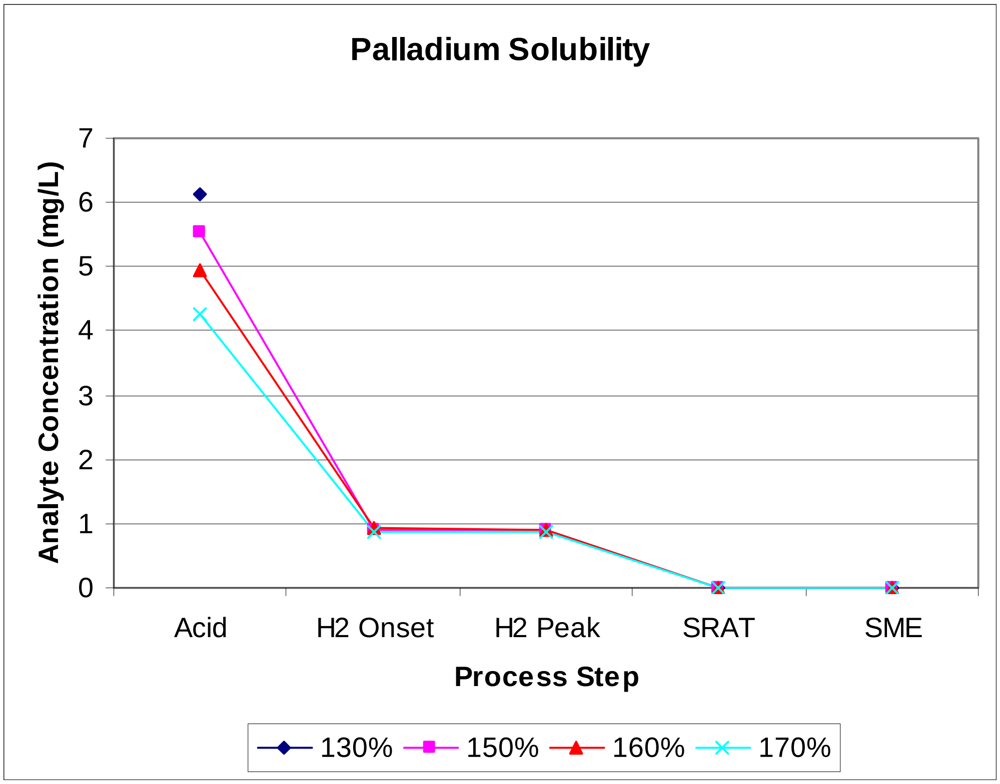

Figure B- 25. Palladium Solubility

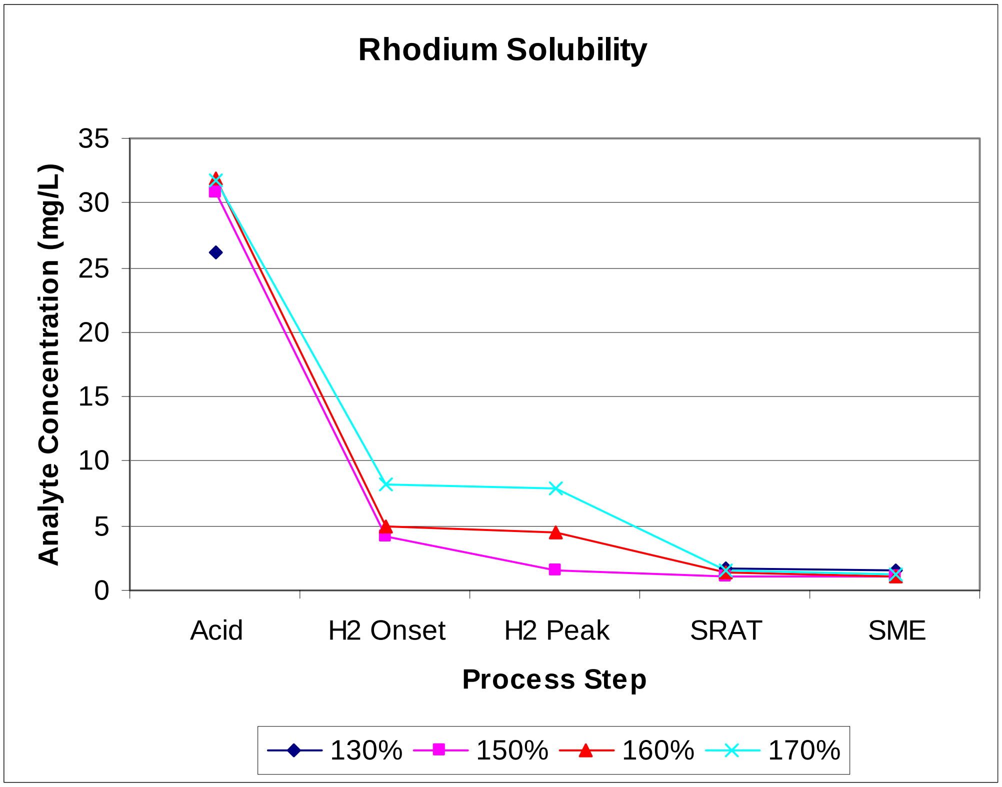

Figure B- 26. Rhodium Solubility

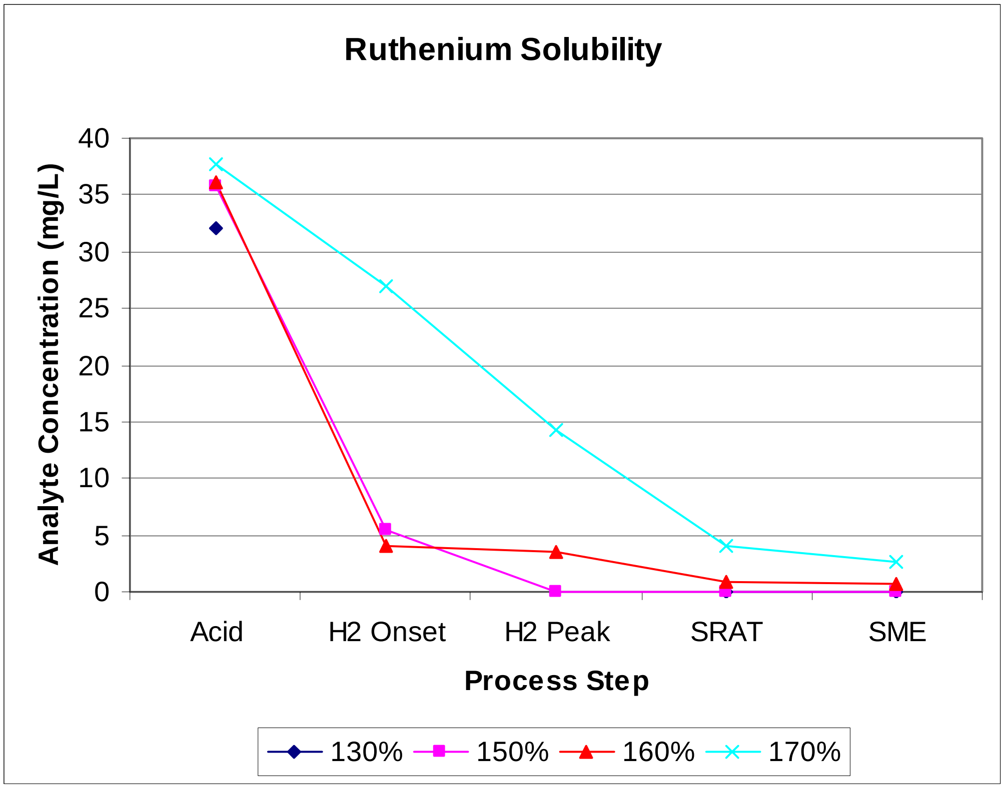

Figure B- 27. Ruthenium Solubility

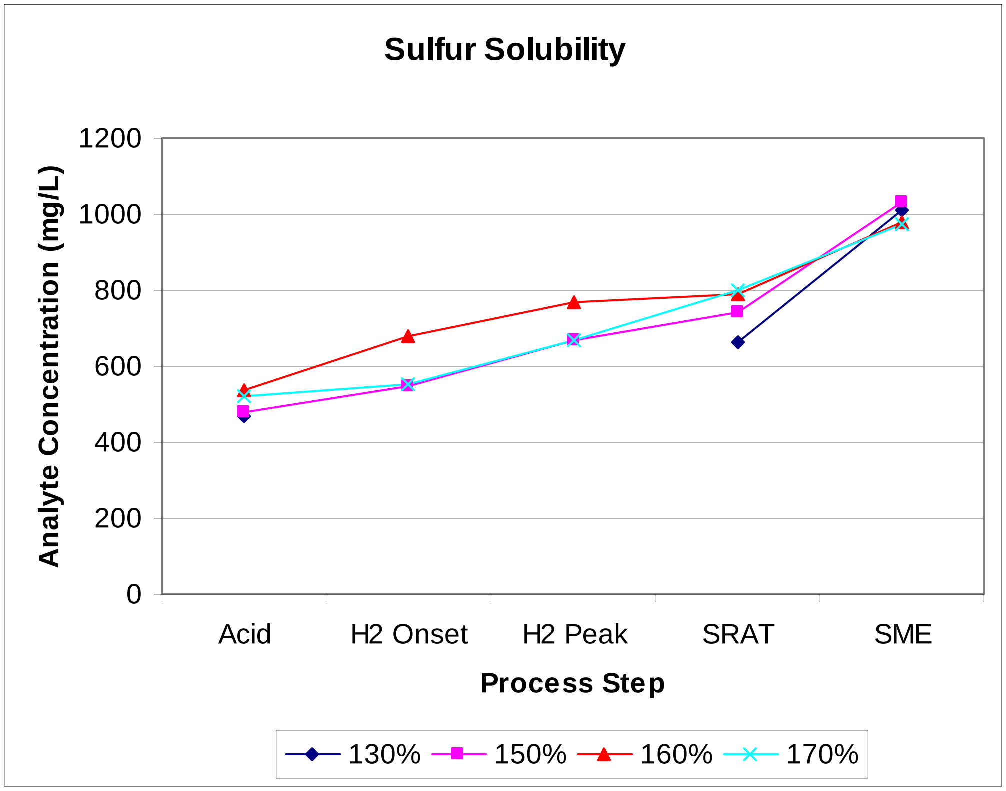

Figure B- 28. Sulfur Solubility

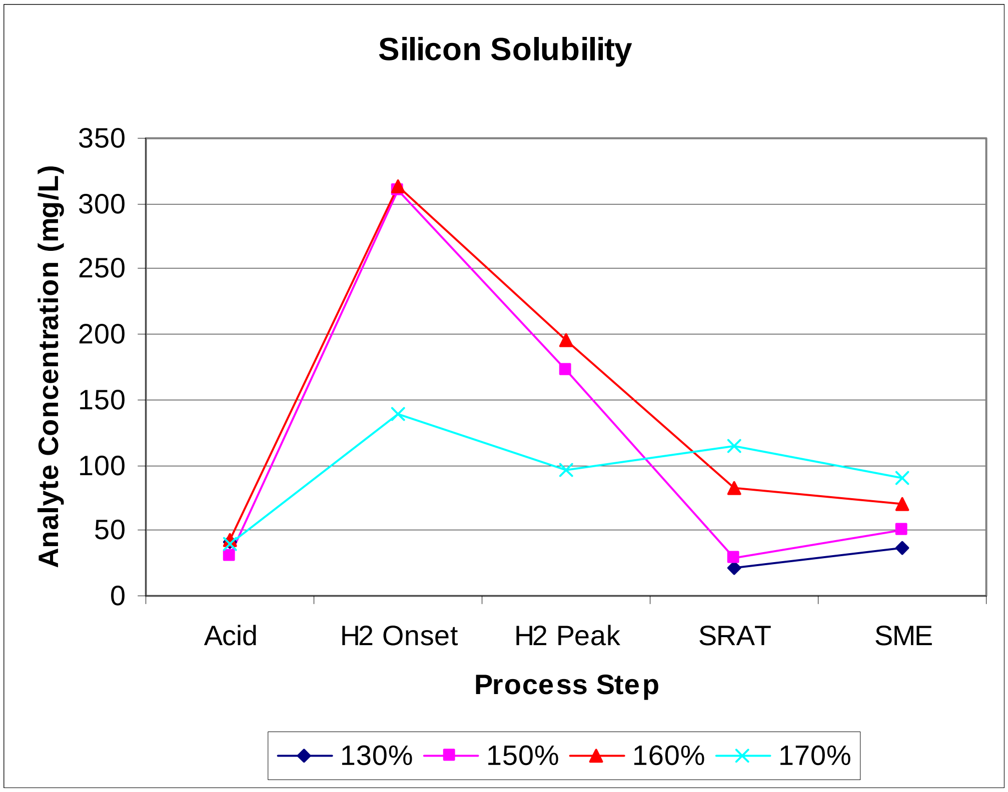

Figure B- 29. Silicon Solubility

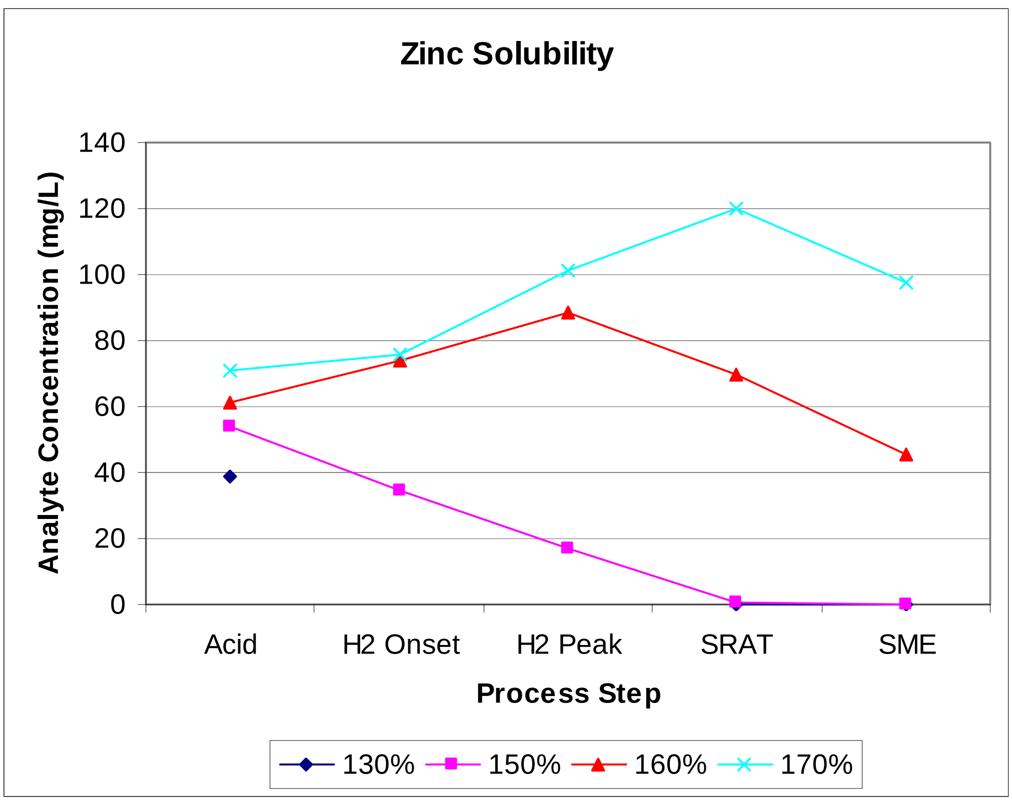

Figure B- 30. Zinc Solubility

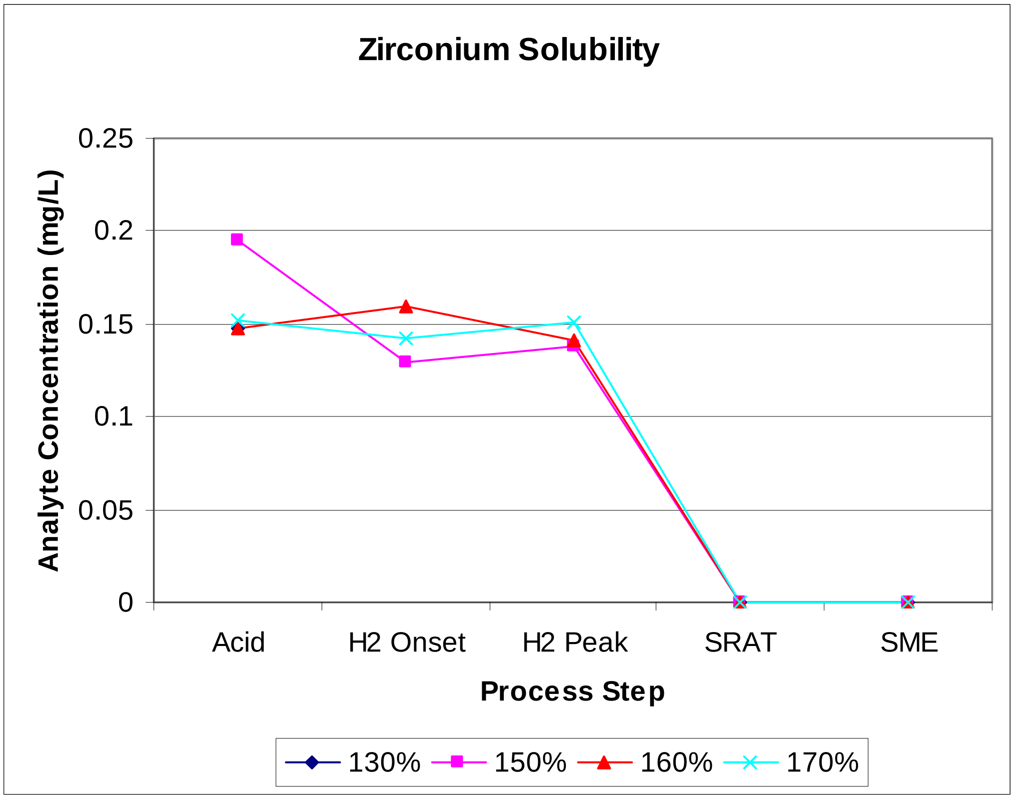

Figure B- 31. Zirconium Solubility

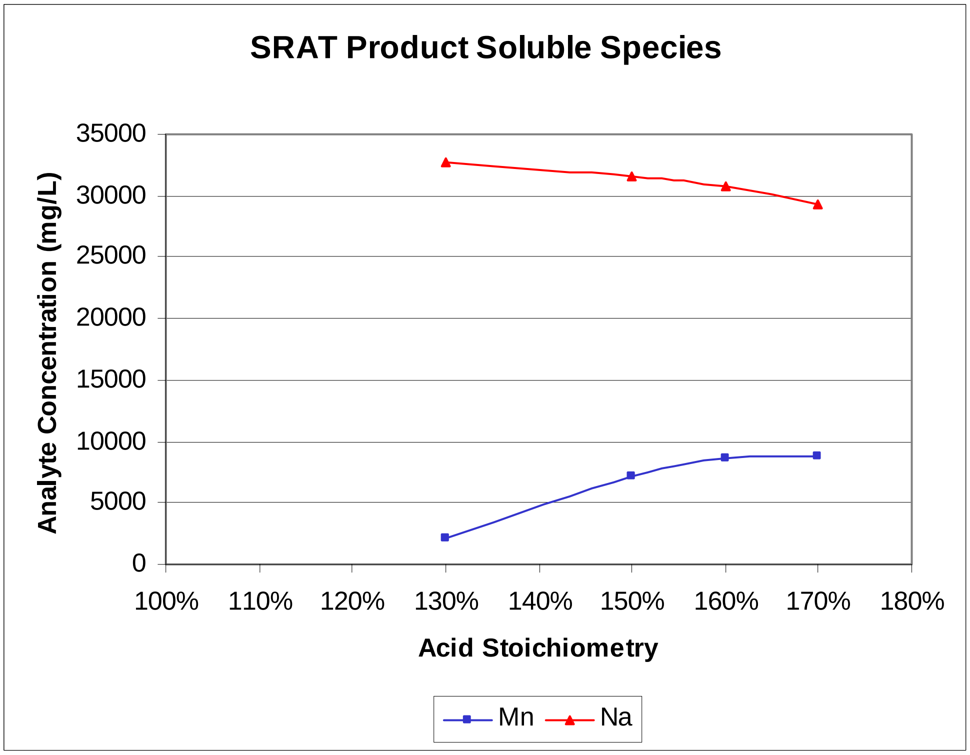

Figure B- 32. SRAT Product Soluble Species: High Concentrations

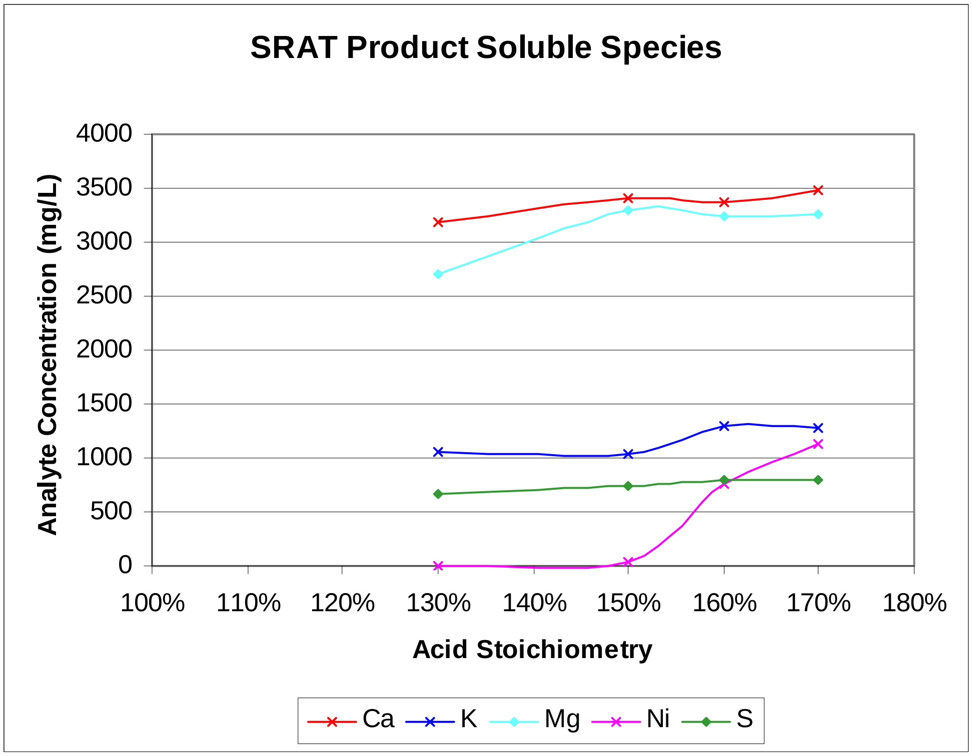

Figure B- 33. SRAT Product Soluble Species: Medium Concentrations

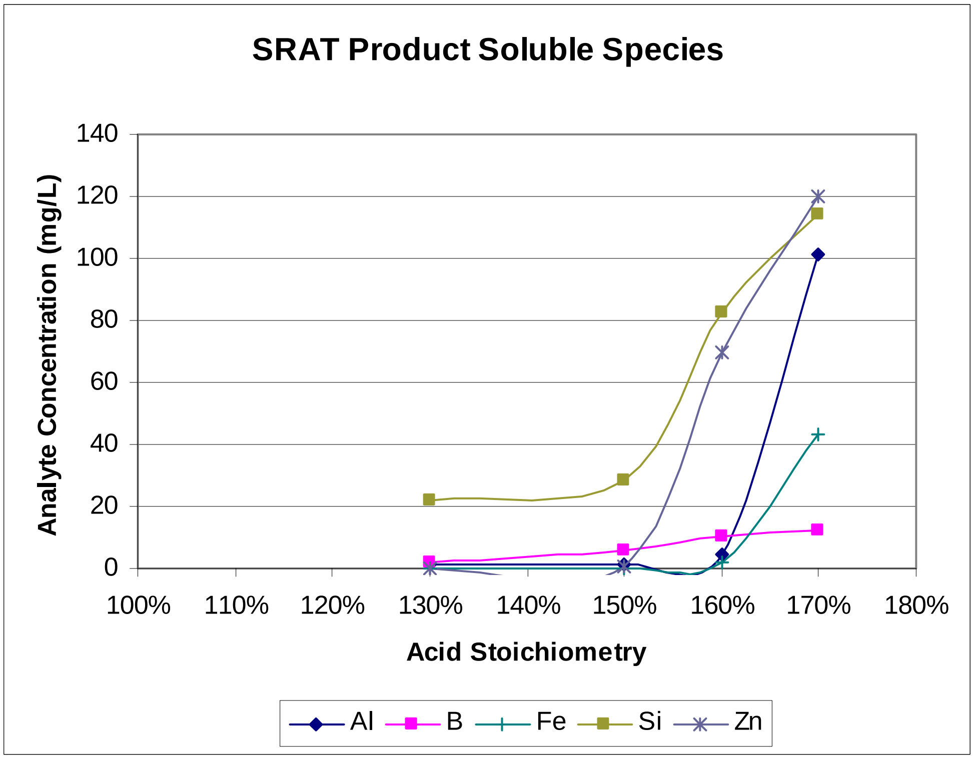

Figure B- 34. SRAT Product Soluble Species: Low Concentrations

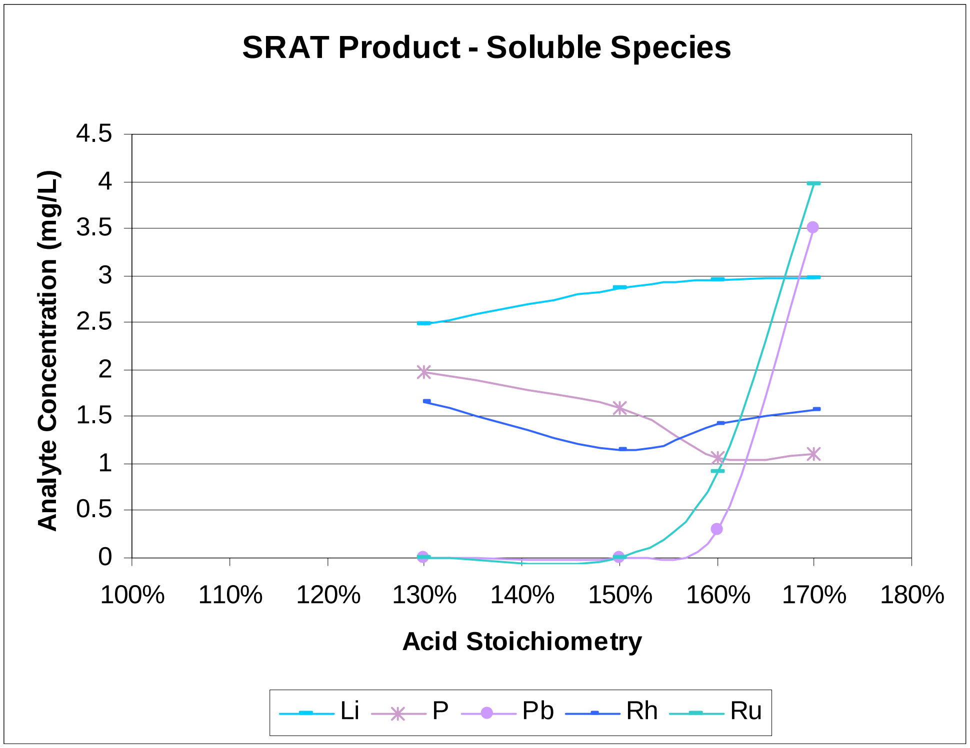

Figure B- 35. SRAT Product Soluble Species: Very Low Concentrations

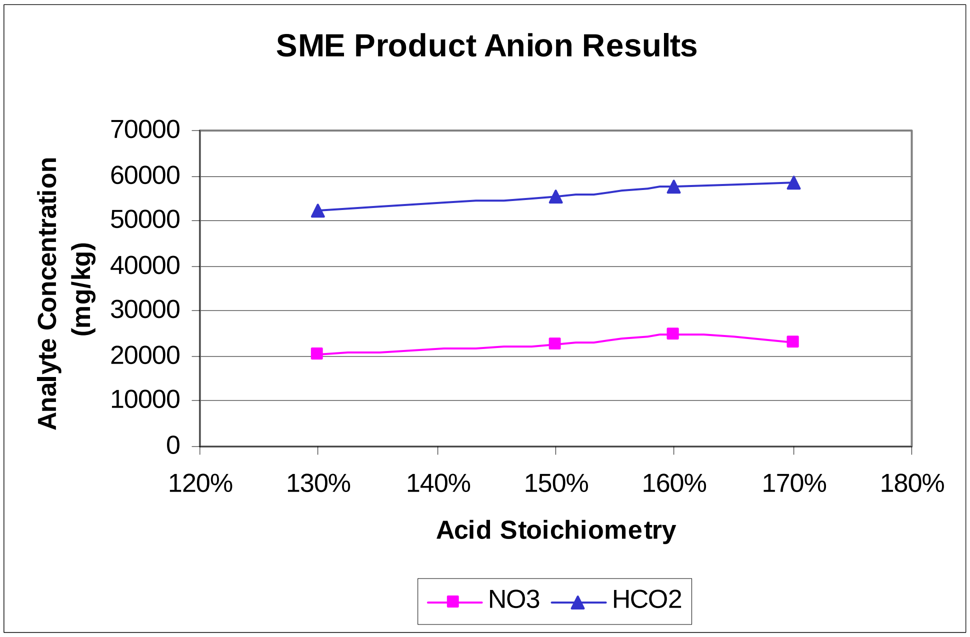

Figure B- 36. SME Product Nitrate and Formate versus Acid Stoichiometry

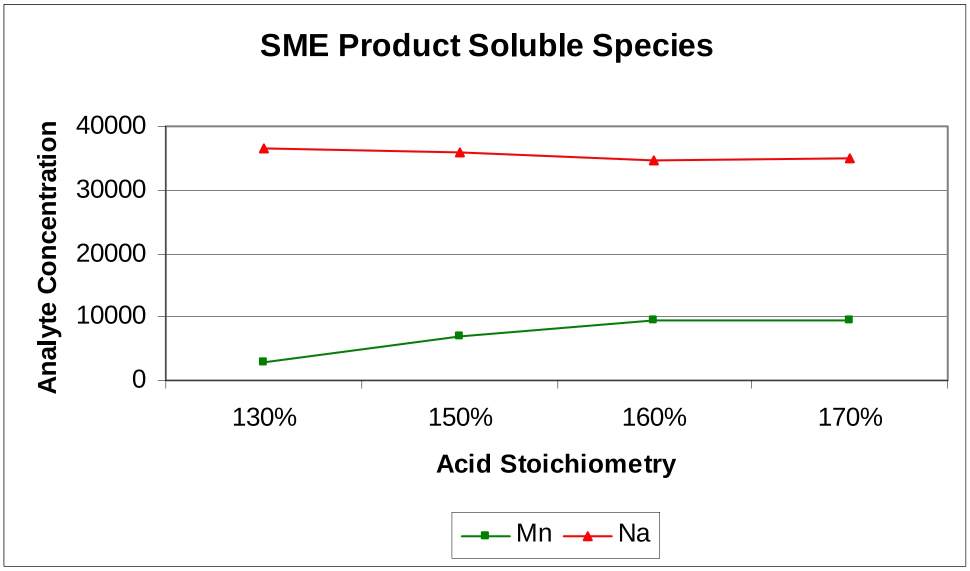

Figure B- 37. SME Product Soluble Species: Very High Concentrations

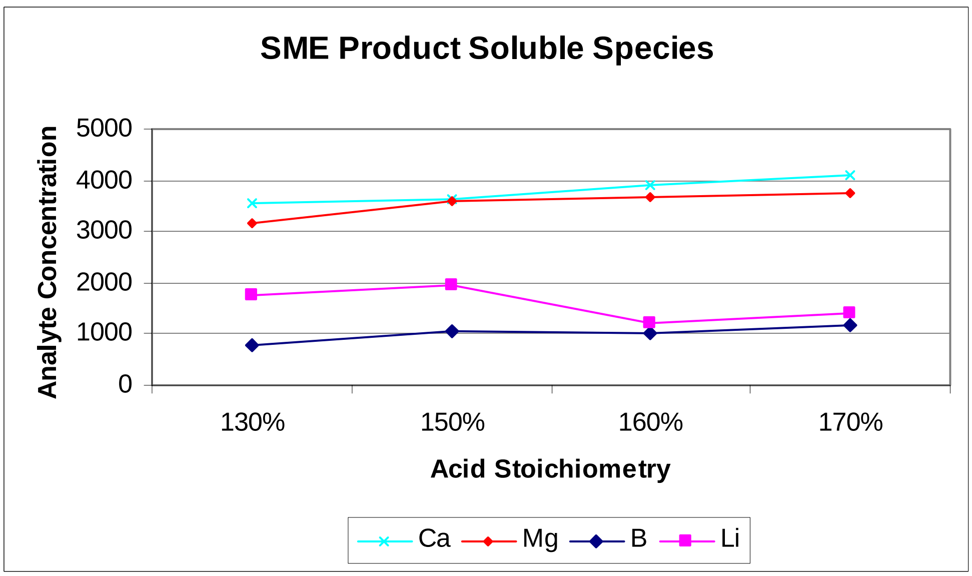

Figure B- 38. SME Product Soluble Species: High Concentrations

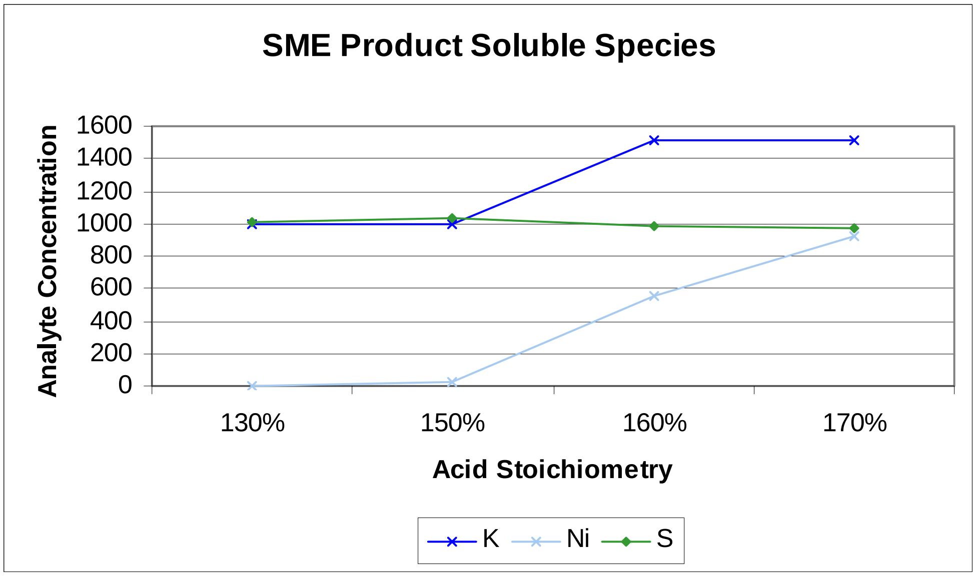

Figure B- 39. SME Product Soluble Species: Medium Concentrations

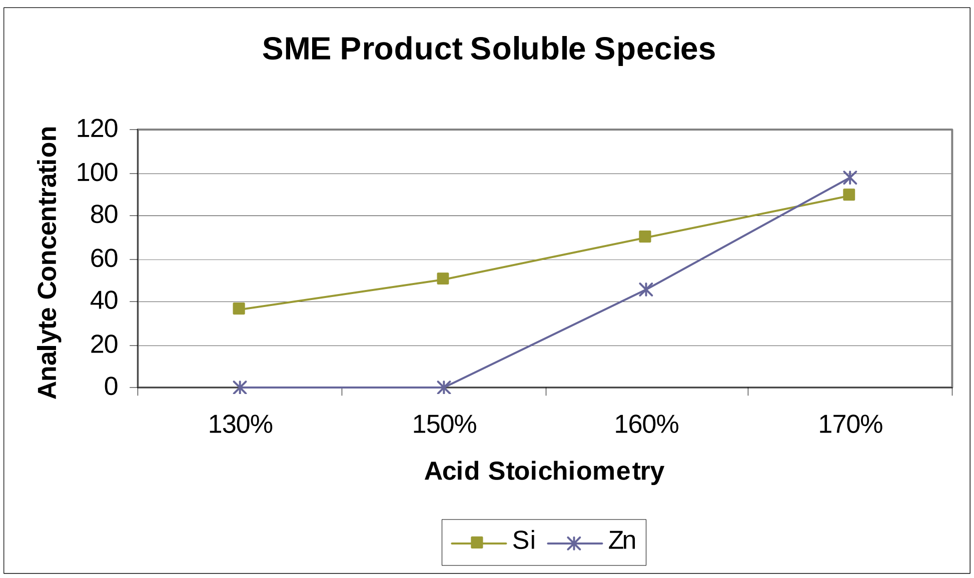

Figure B- 40. SME Product Soluble Species: Low Concentrations

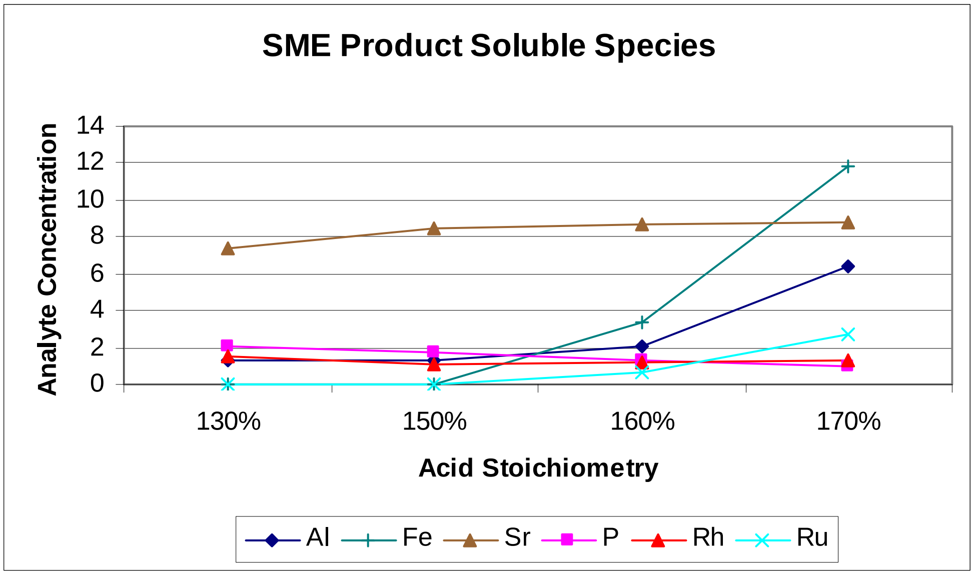

Figure B- 41. SME Product Soluble Species: Very Low Concentrations

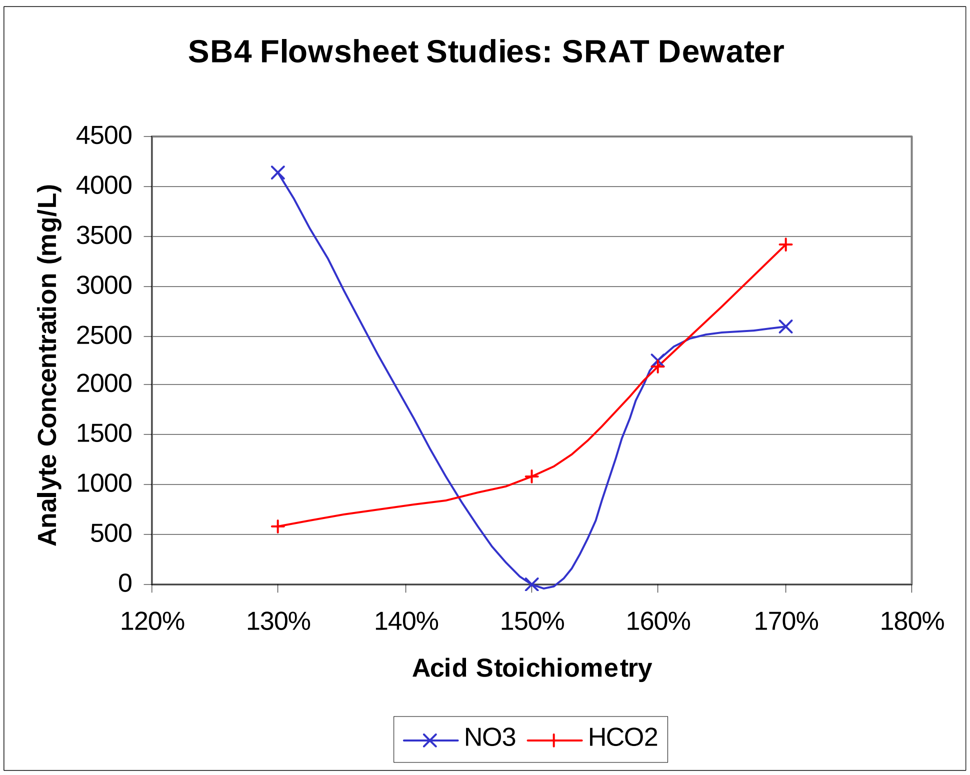

Figure B- 42. SRAT Dewater Anion Concentrations

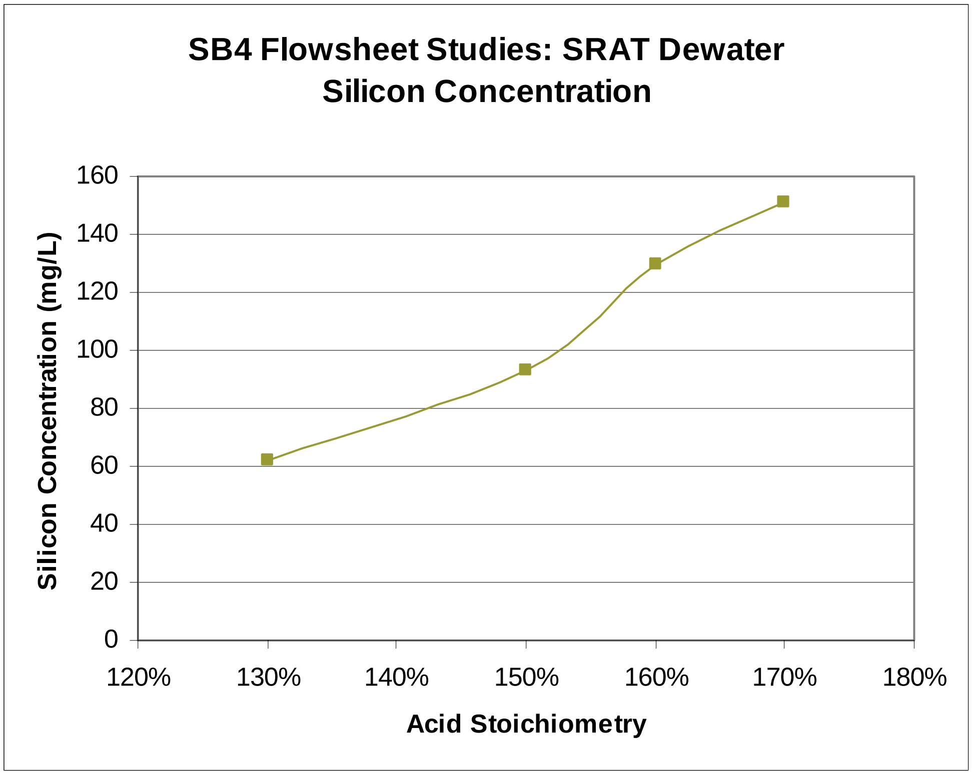

Figure B- 43. SRAT Dewater Silicon Concentration

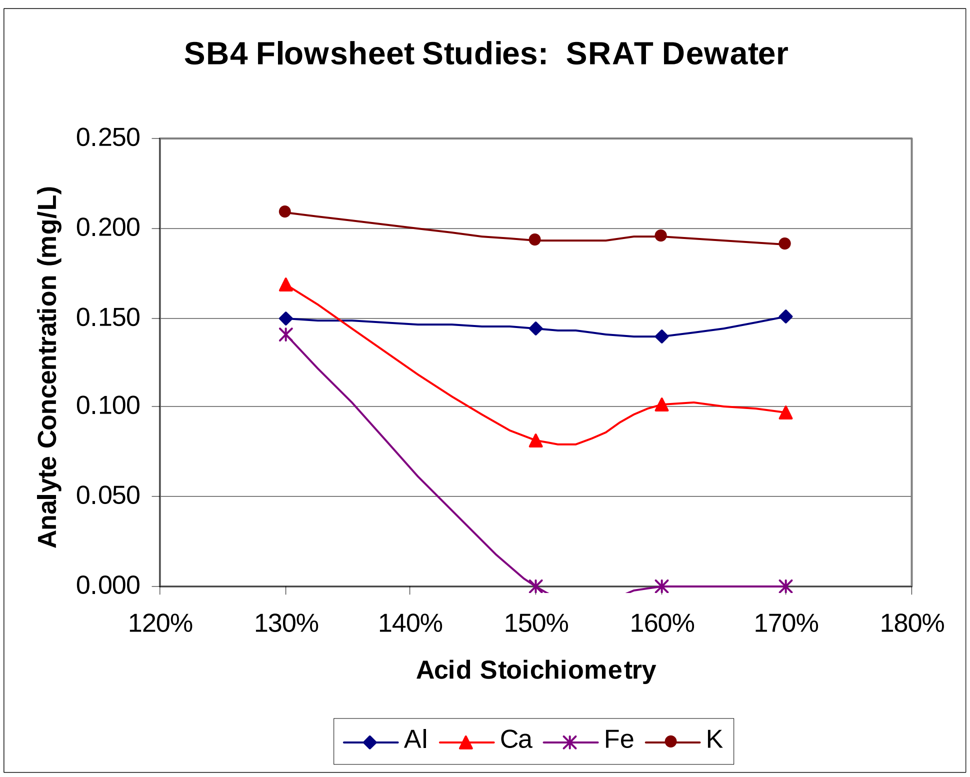

Figure B- 44. SRAT Dewater Elementals

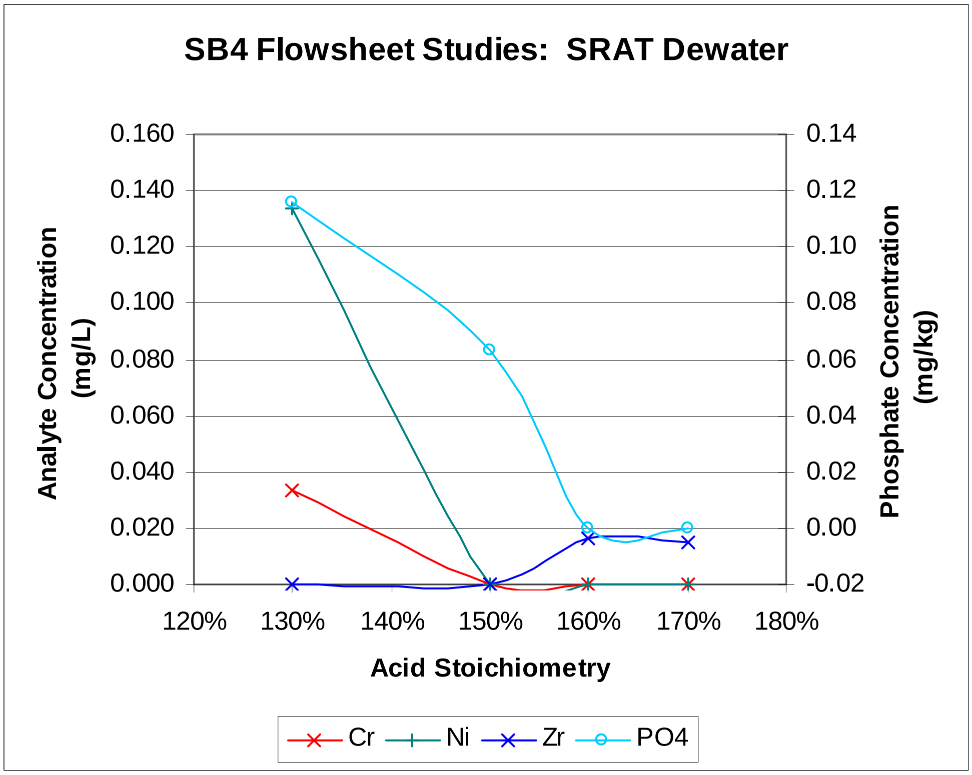

Figure B- 45. SRAT Dewater Trace Elementals

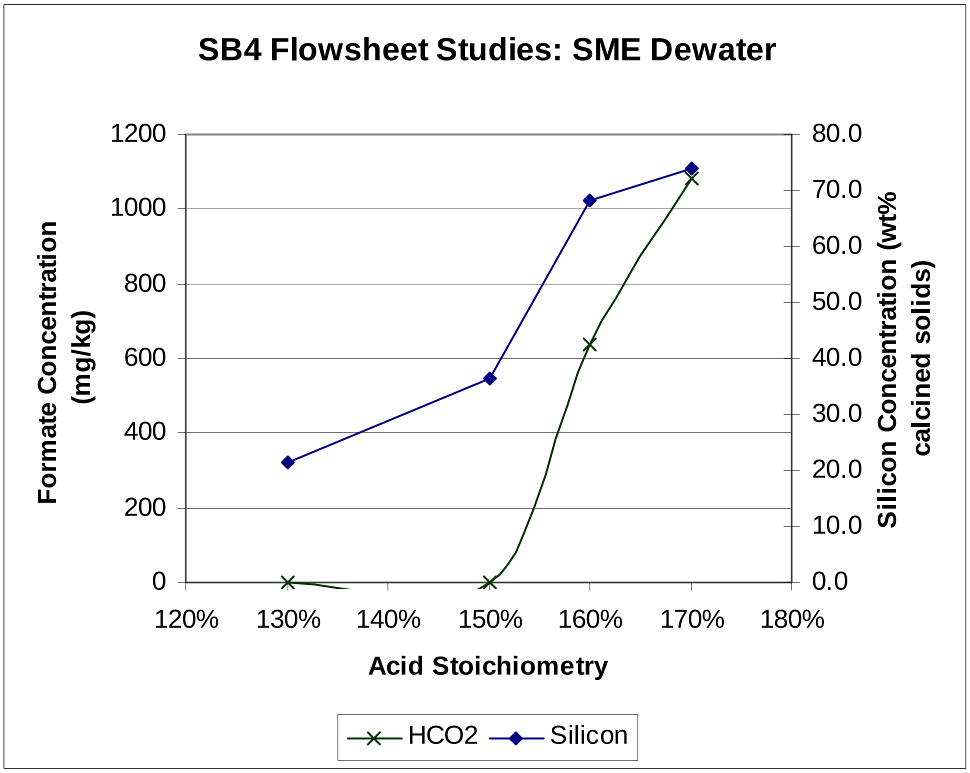

Figure B- 46. SME Dewater Formate and Silicon Concentrations

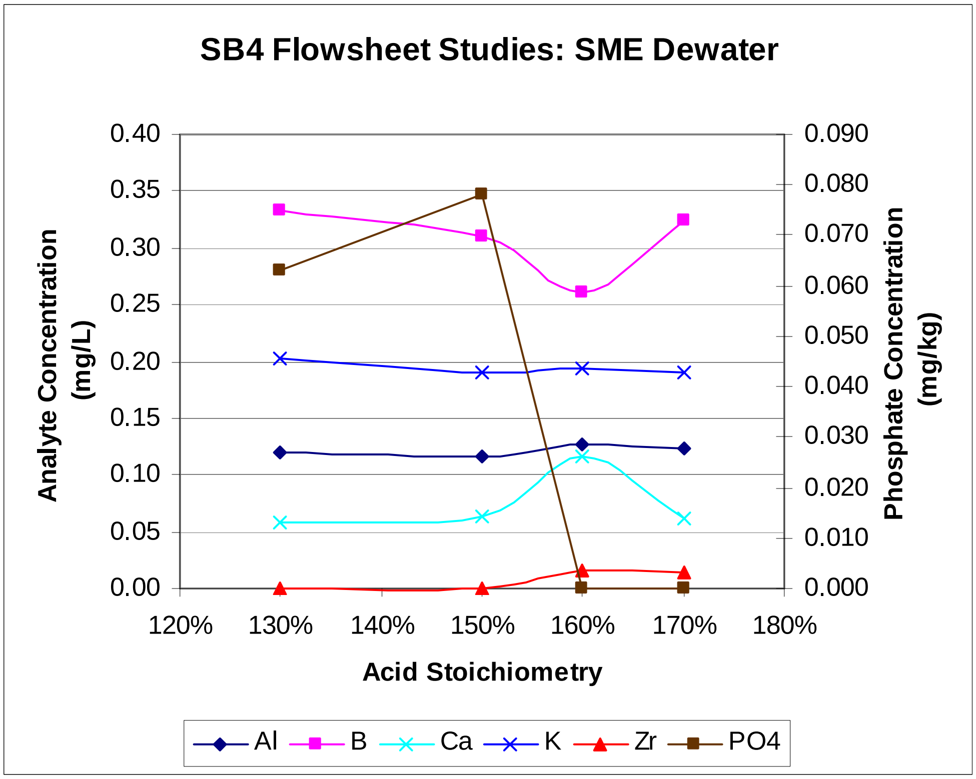

Figure B- 47. SME Dewater Elementals

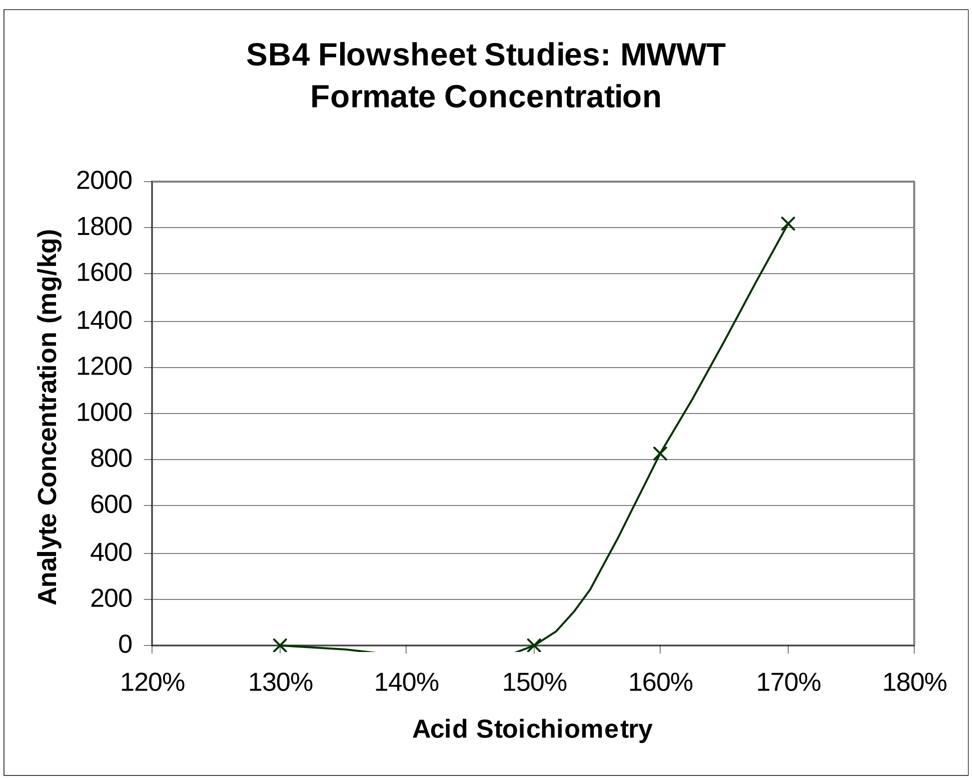

Figure B- 48. MWWT Formate Concentration

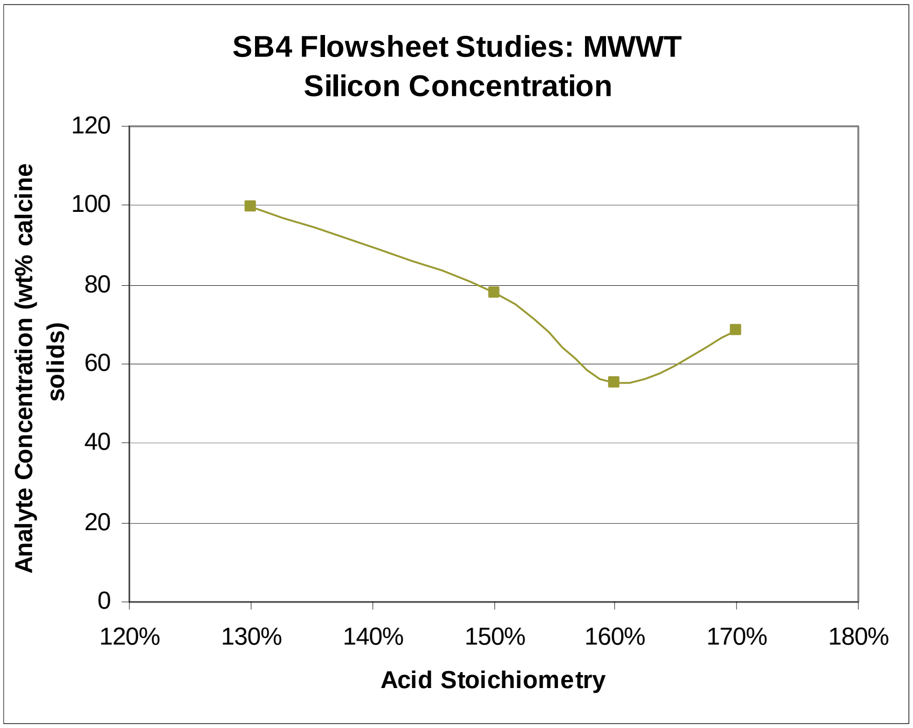

Figure B- 49. MWWT Silicon Concentration

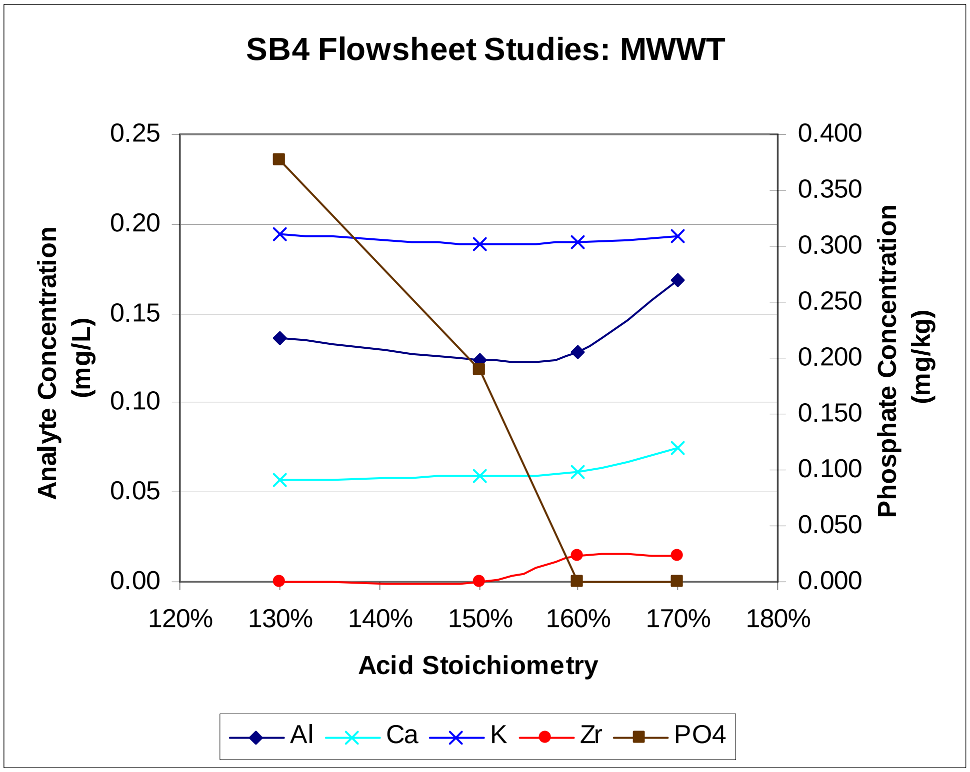

Figure B- 50. MWWT Elementals

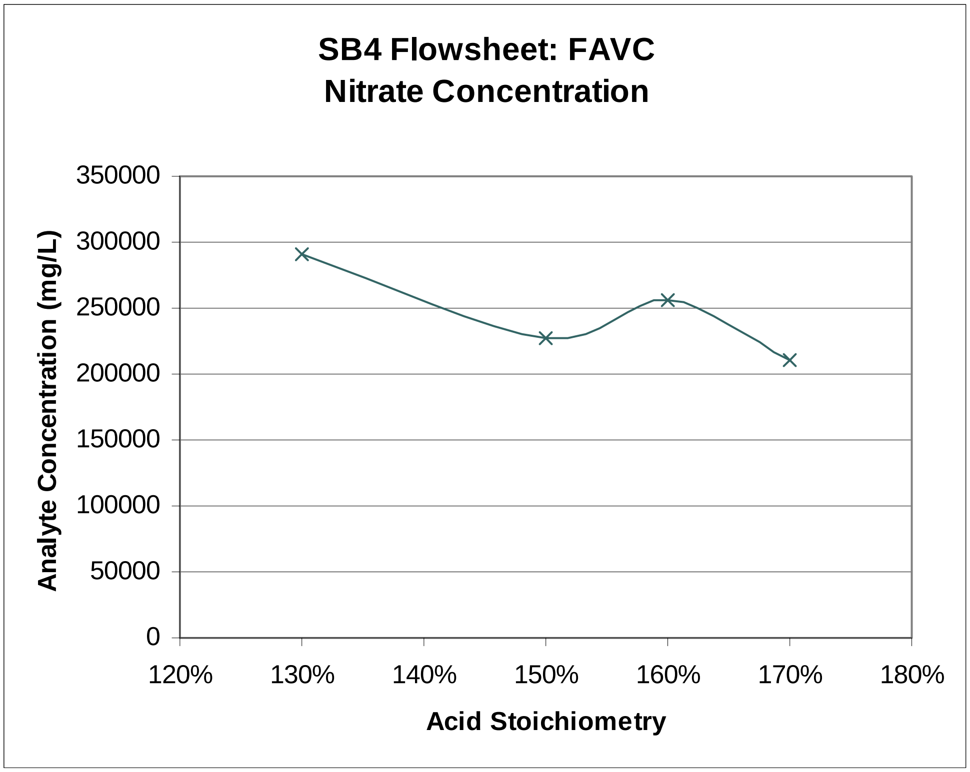

Figure B- 51. FAVC Nitrate Concentration

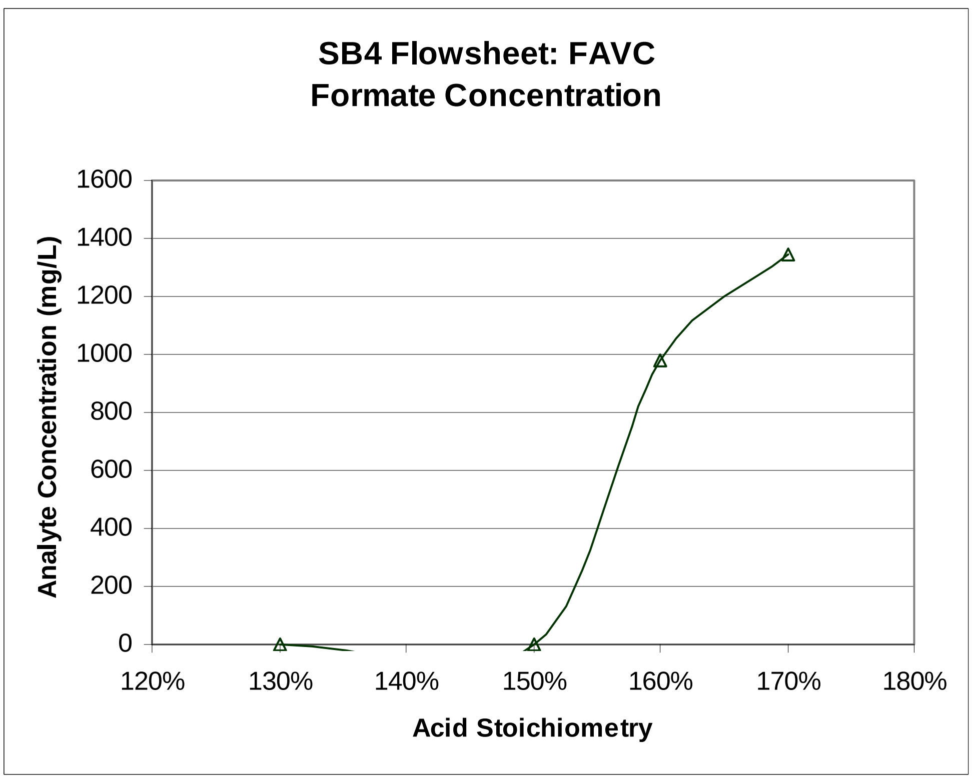

Figure B- 52. FAVC Formate Concentration

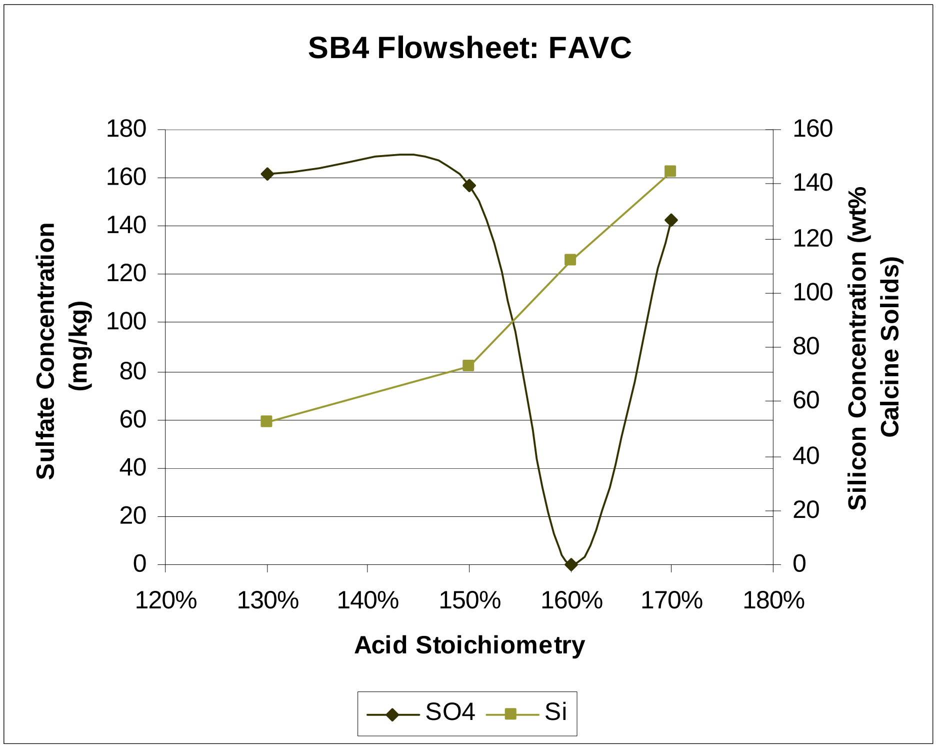

Figure B- 53. FAVC Sulfate and Silicon Concentration

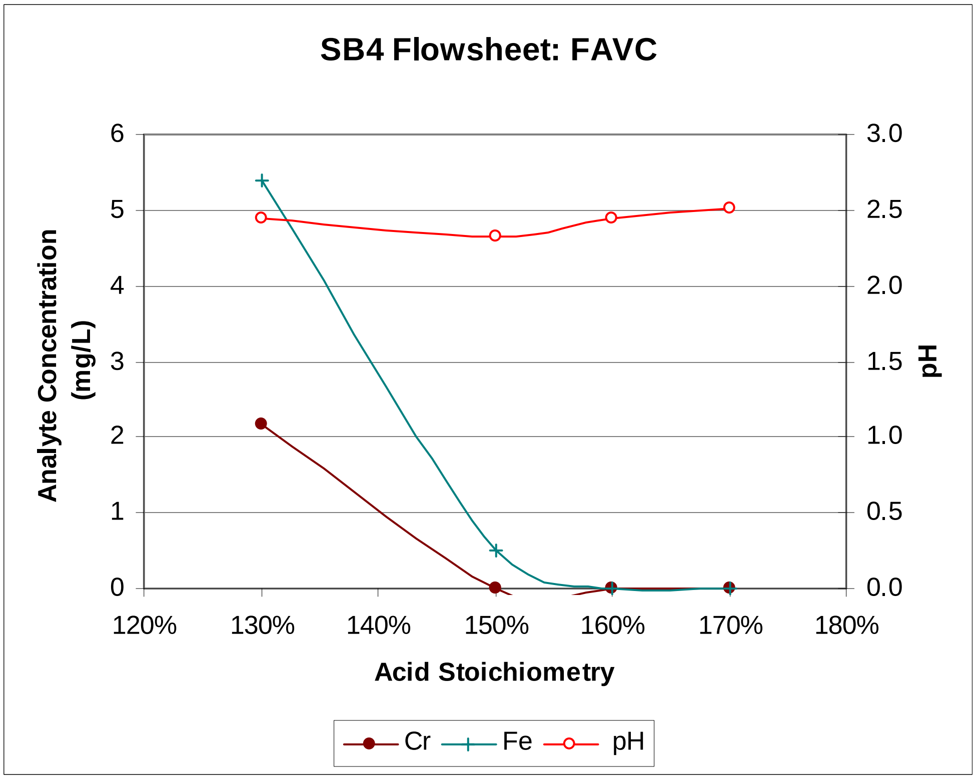

Figure B- 54. FAVC pH and Elementals

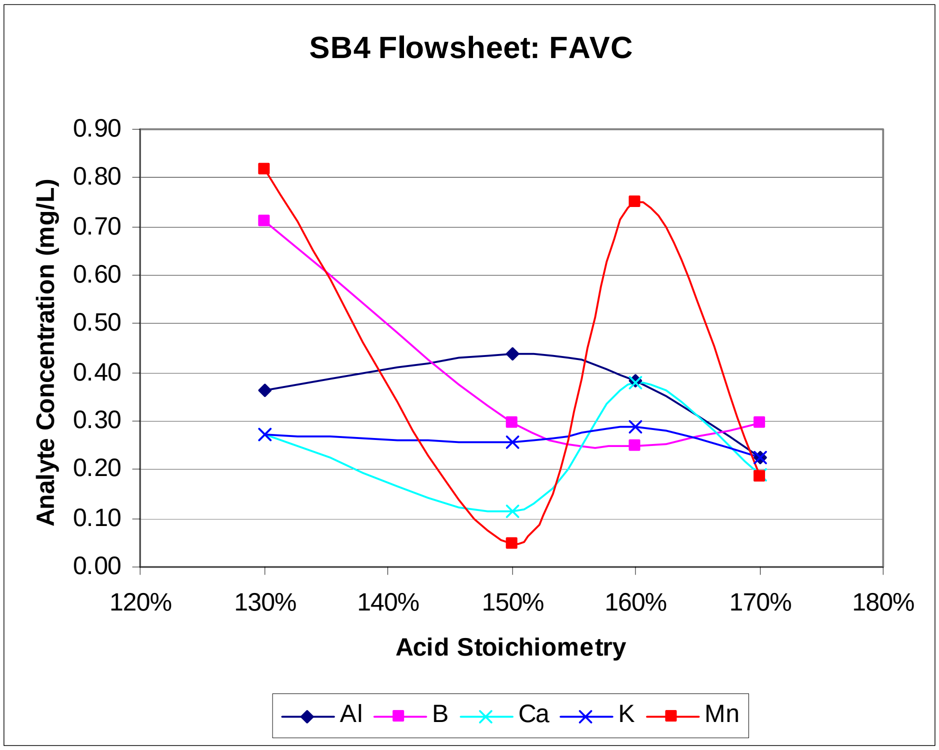

Figure B- 55. FAVC Elementals

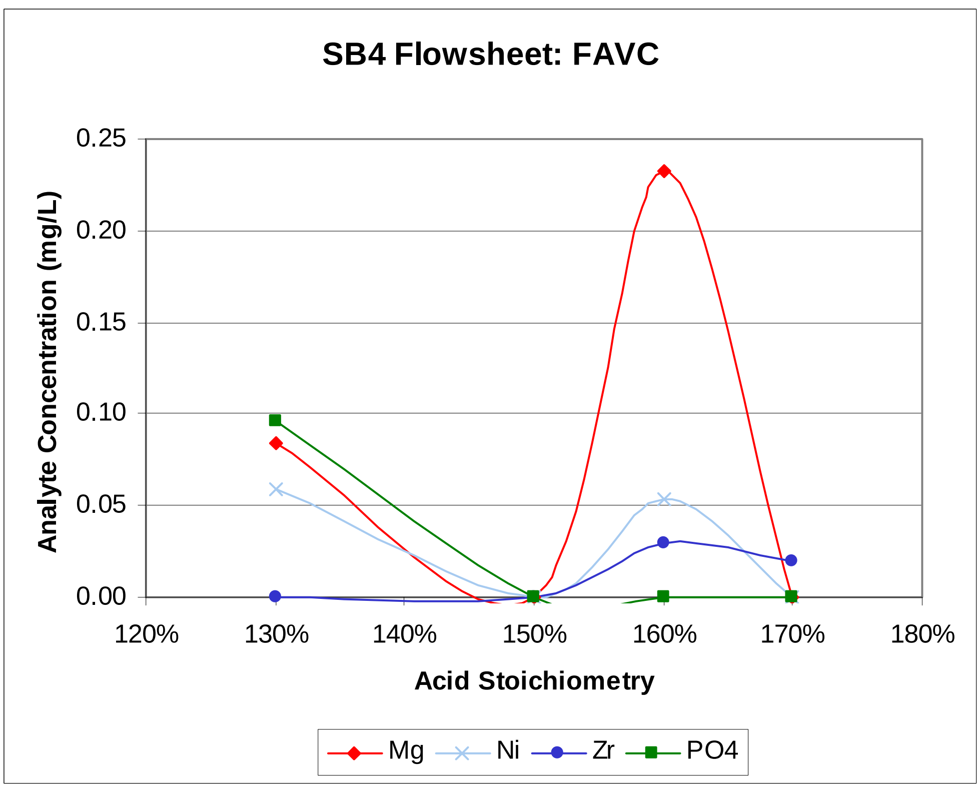

Figure B- 56. FAVC Trace Elementals

Appendix C. Offgas Composition Data

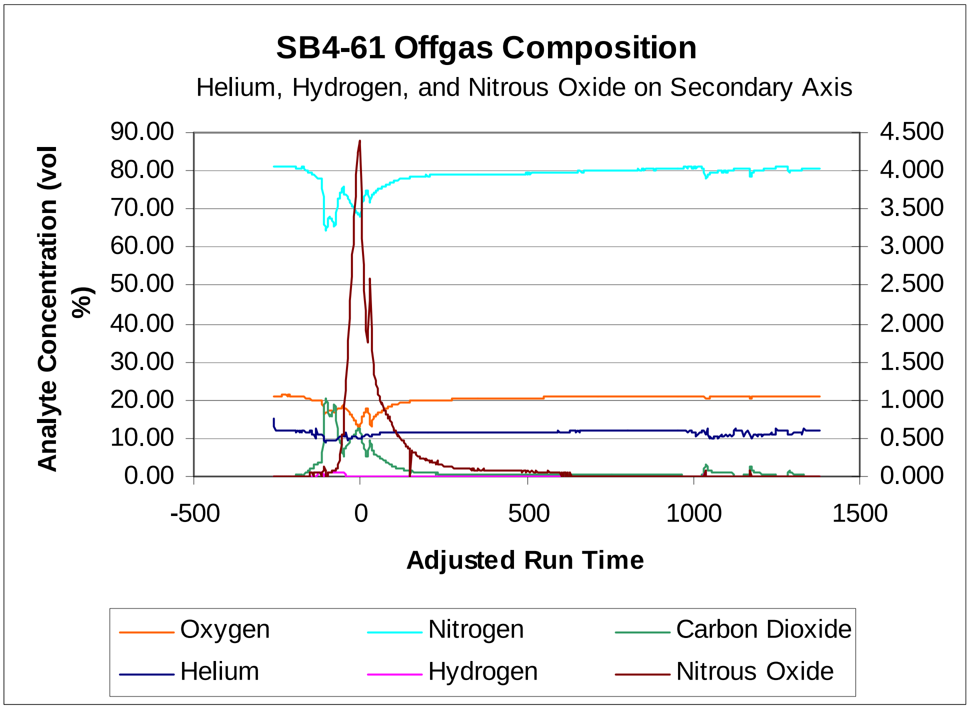

Figure C- 1. SB4-61 (130% Acid) Offgas Data

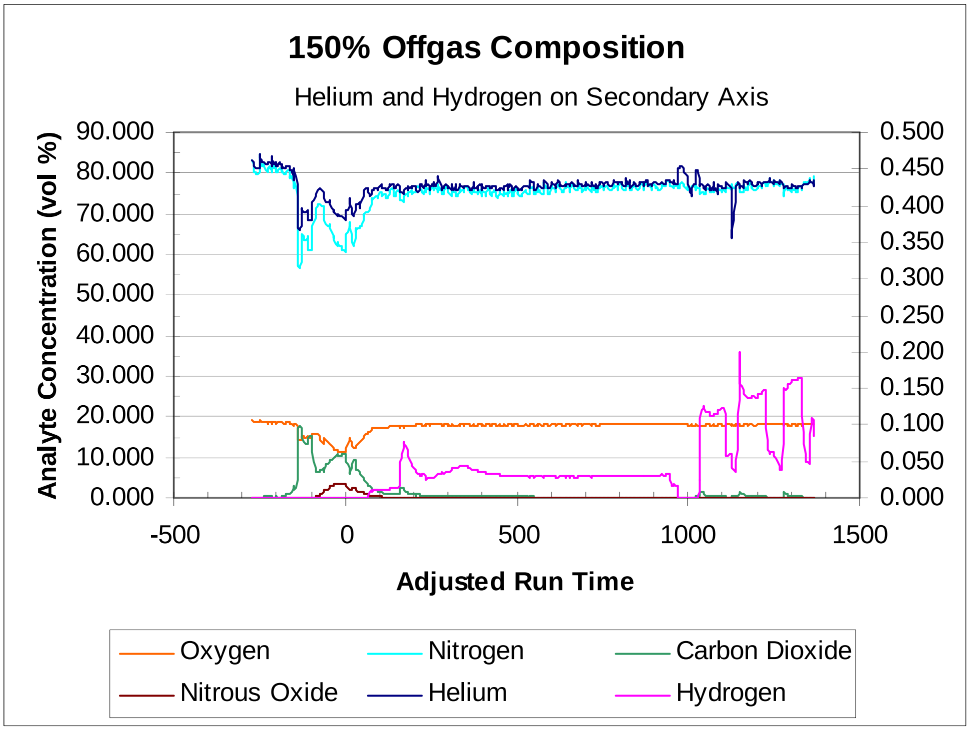

Figure C- 2. SB4-62 (150% Acid) Offgas Data

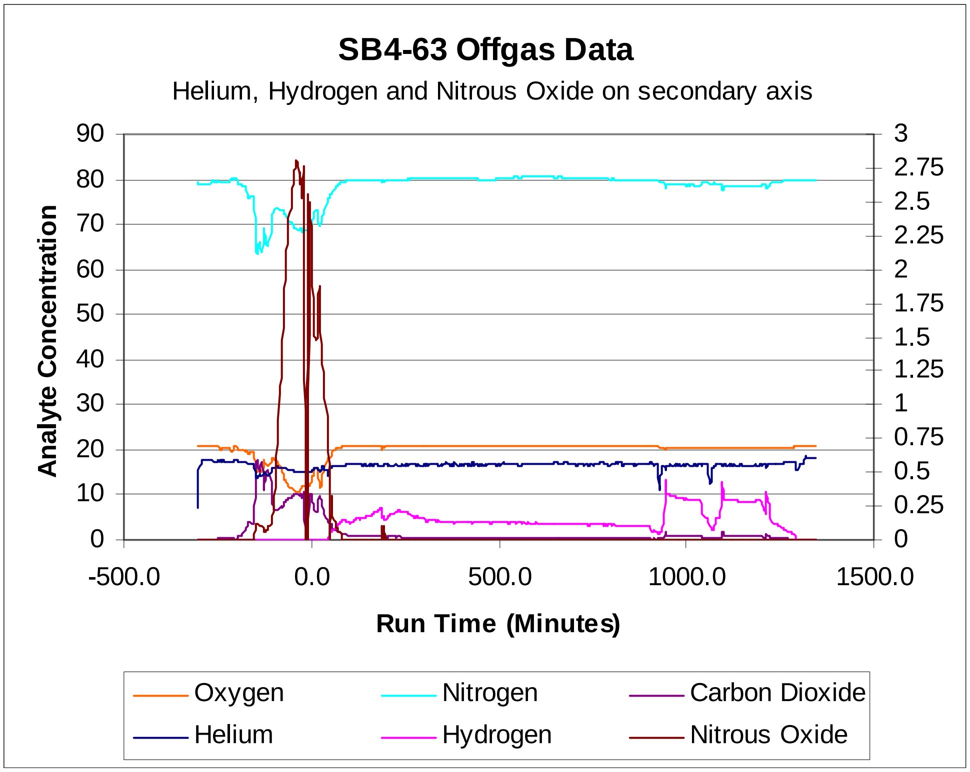

Figure C- 3. SB4-63 (160% Acid) Offgas Data

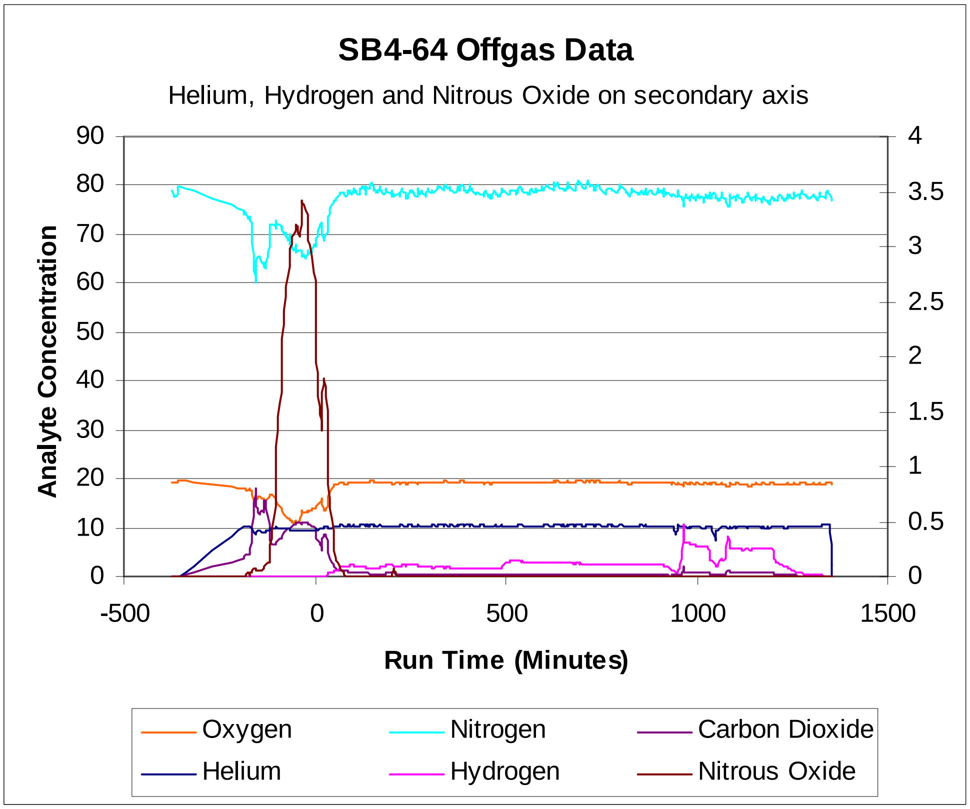

Figure C- 4. SB4-64 (170% Acid) Offgas Data

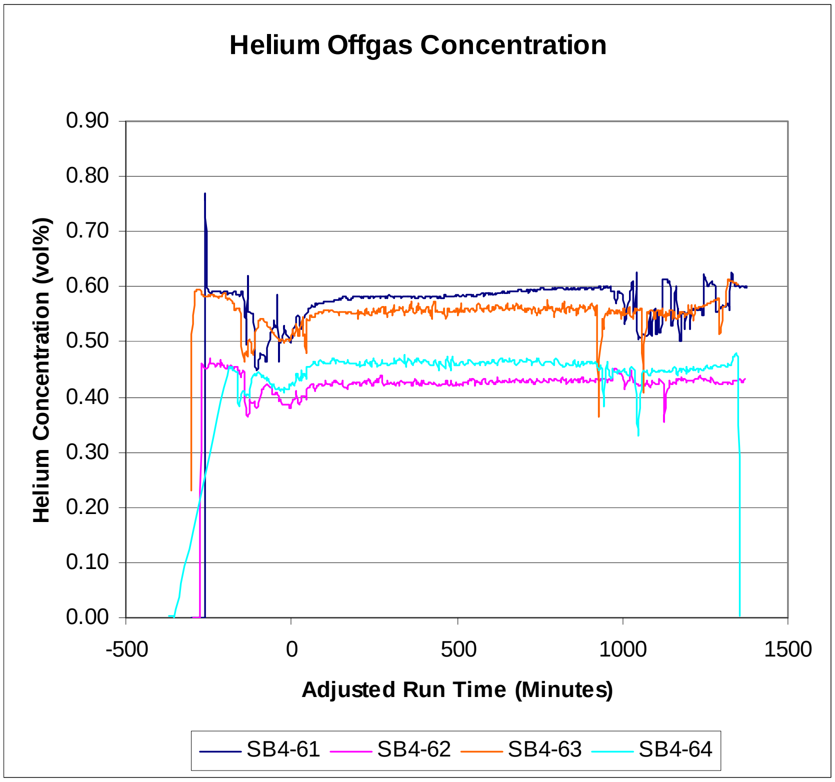

Figure C- 5. Helium Profiles

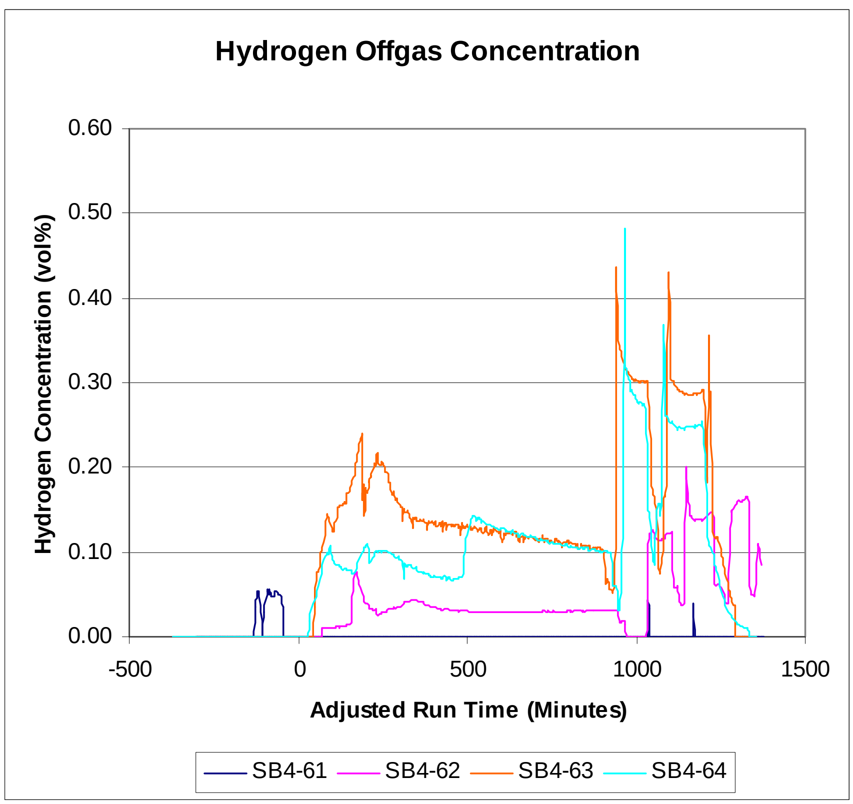

Figure C- 6. Hydrogen Profiles

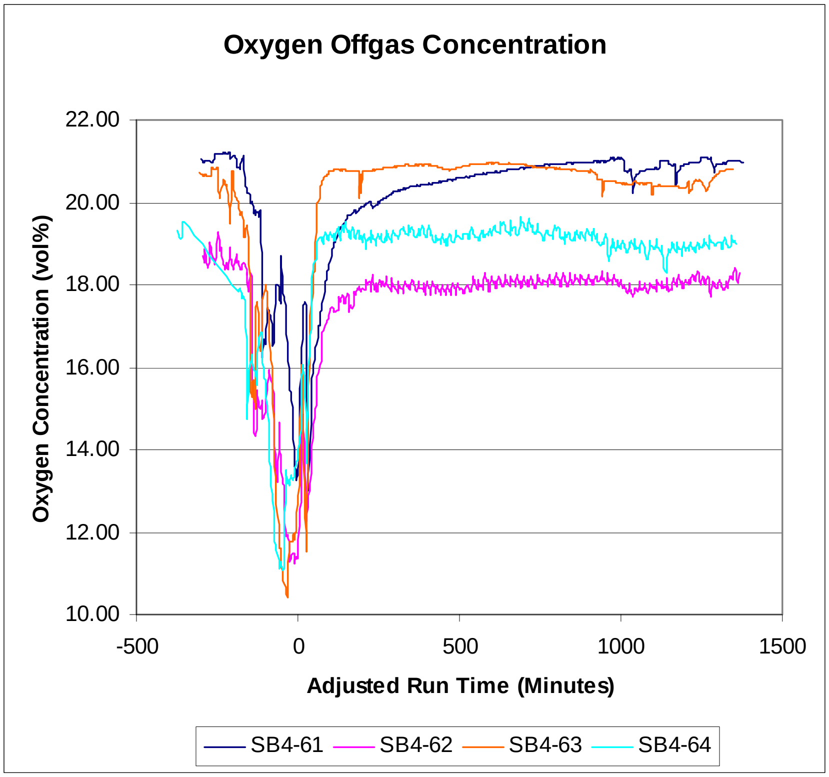

Figure C- 7. Oxygen Profiles

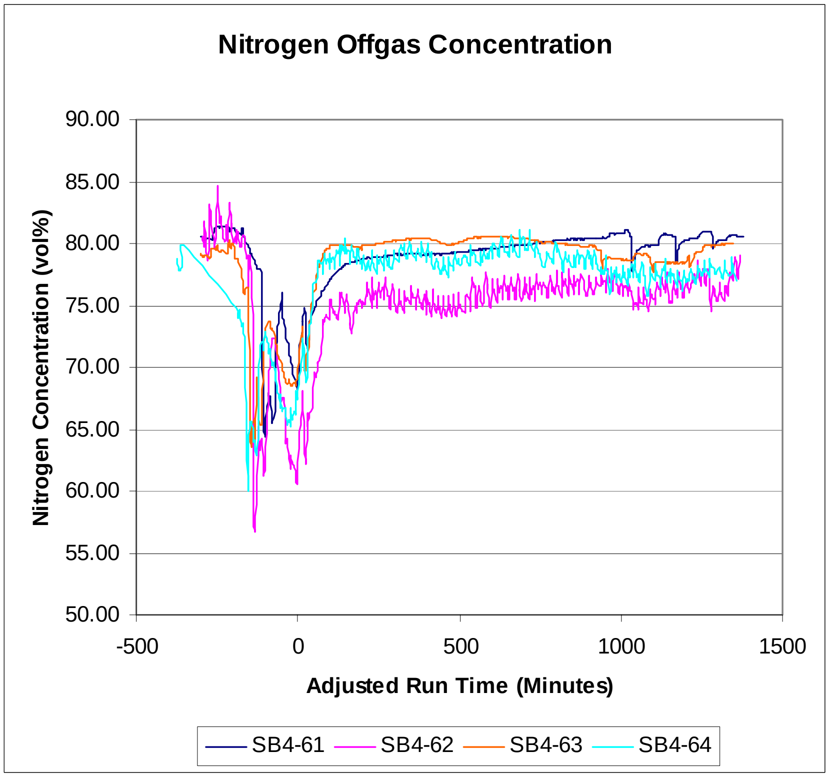

Figure C- 8. Nitrogen Profiles

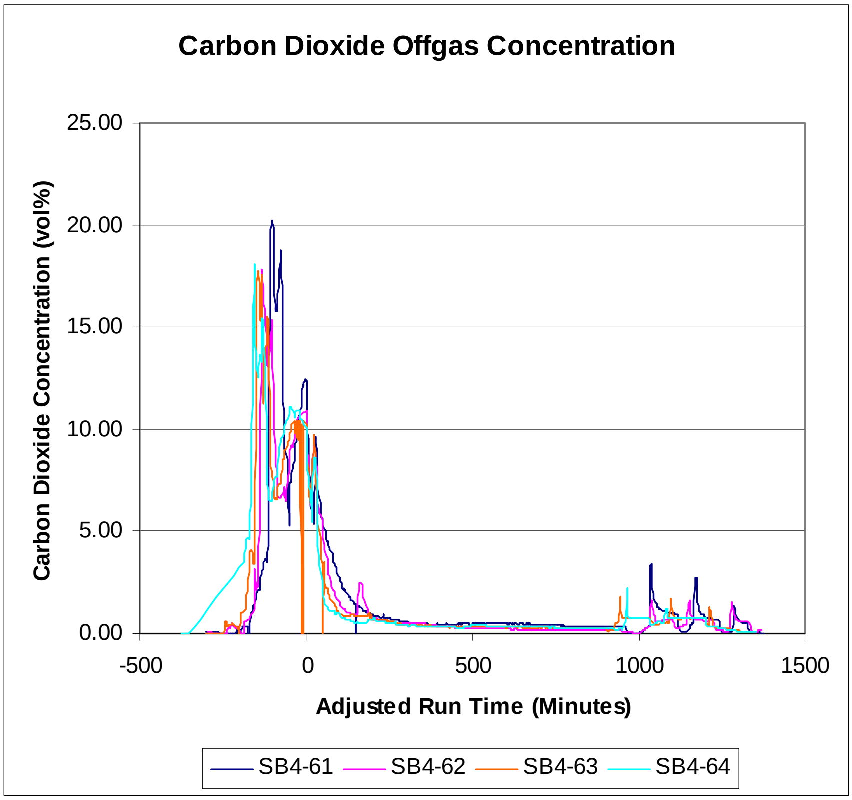

Figure C- 9. Carbon Dioxide Profiles

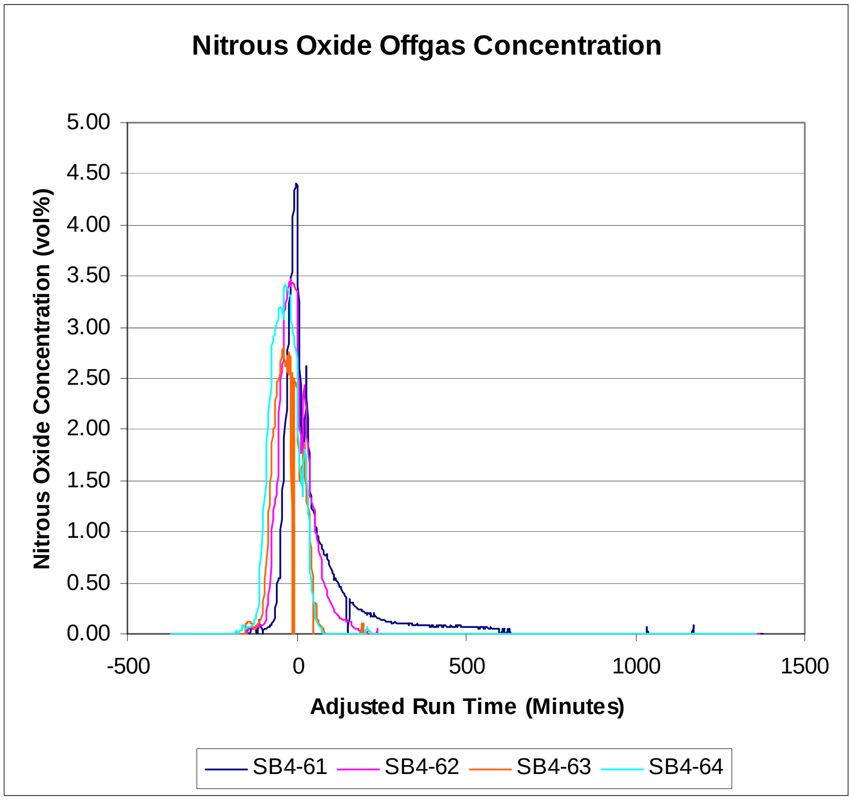

Figure C- 10. Nitrous Oxide Profiles

Appendix D. Rheological Results Charts and Flow Curves

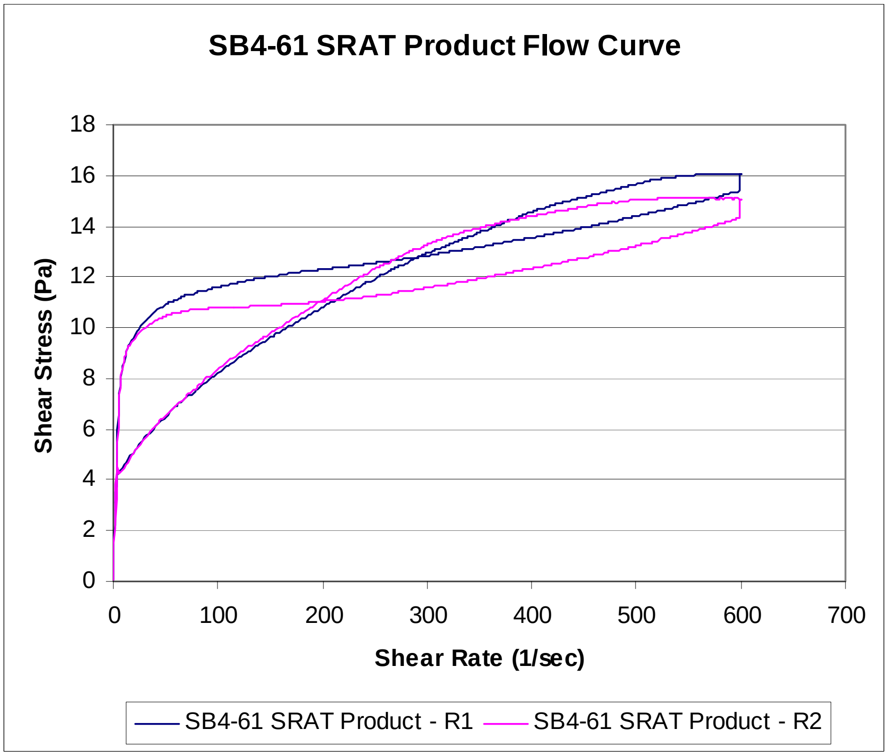

Figure D- 1. SB4-61 (130% Acid) Flow Curves

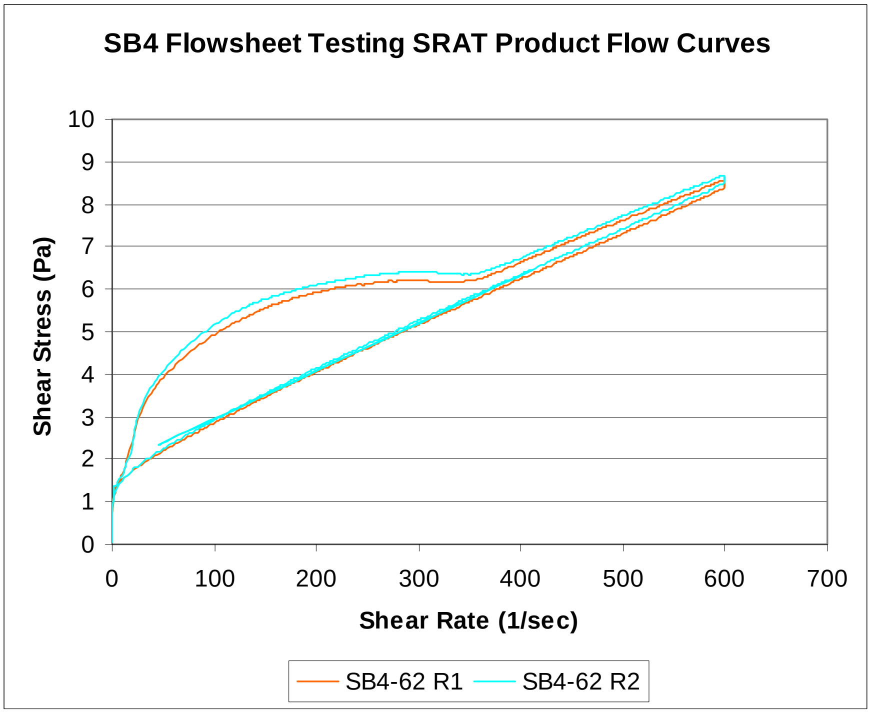

Figure D- 2. SB4-62 (150% Acid) Flow Curves

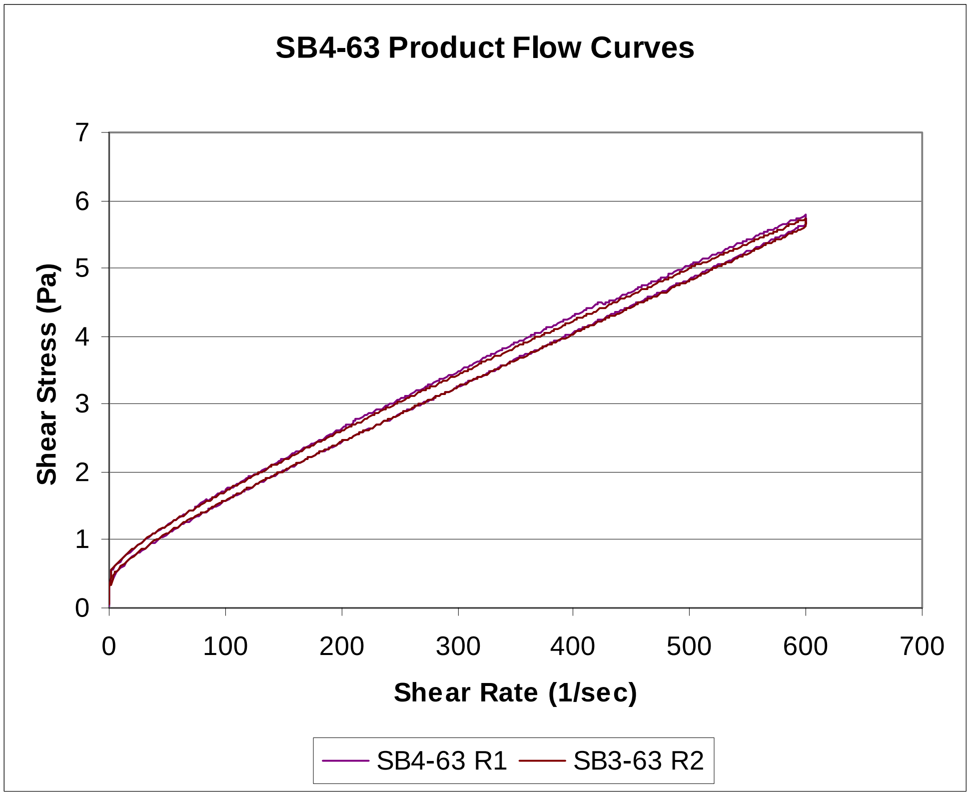

Figure D- 3. SB4-63 (160% Acid) Flow Curves

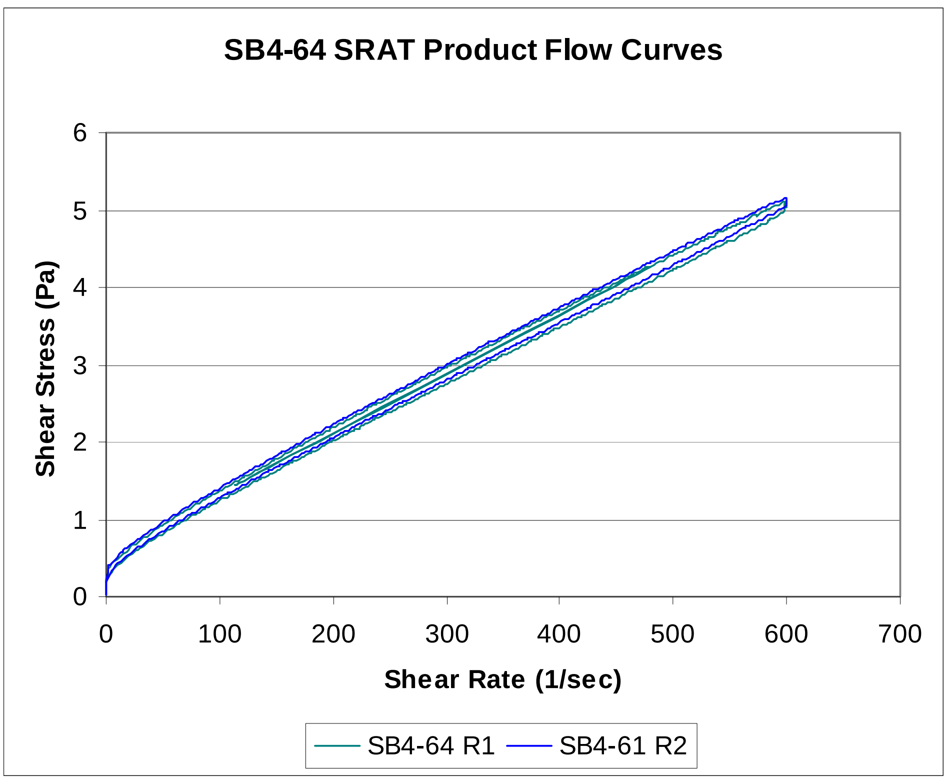

Figure D- 4. SB4-64 (170% Acid) Flow Curves

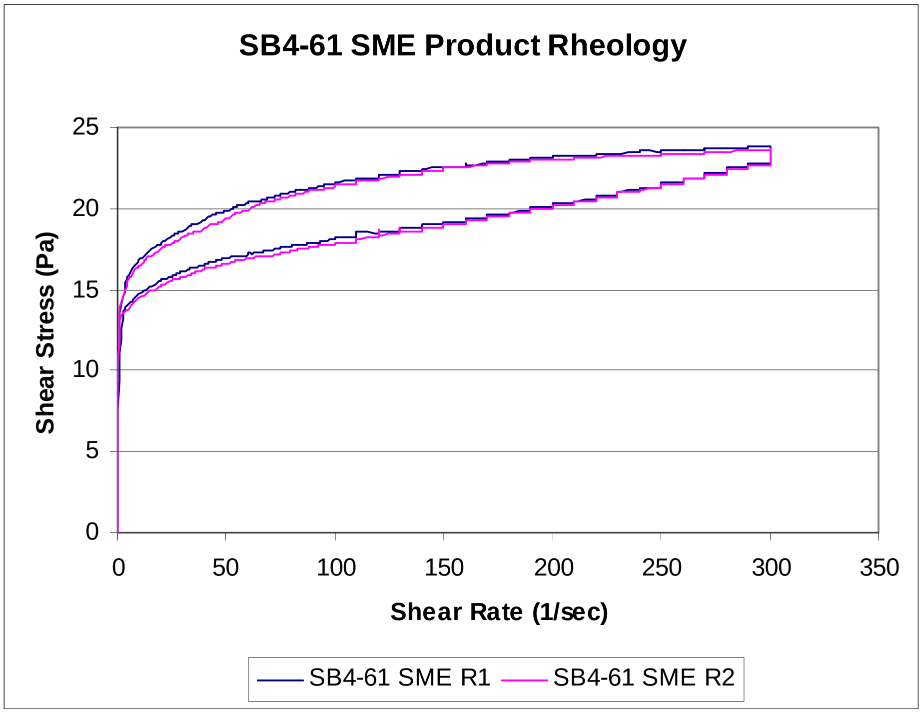

Figure D- 5. SB4-61 (130% Acid) SME Product Flow Curves

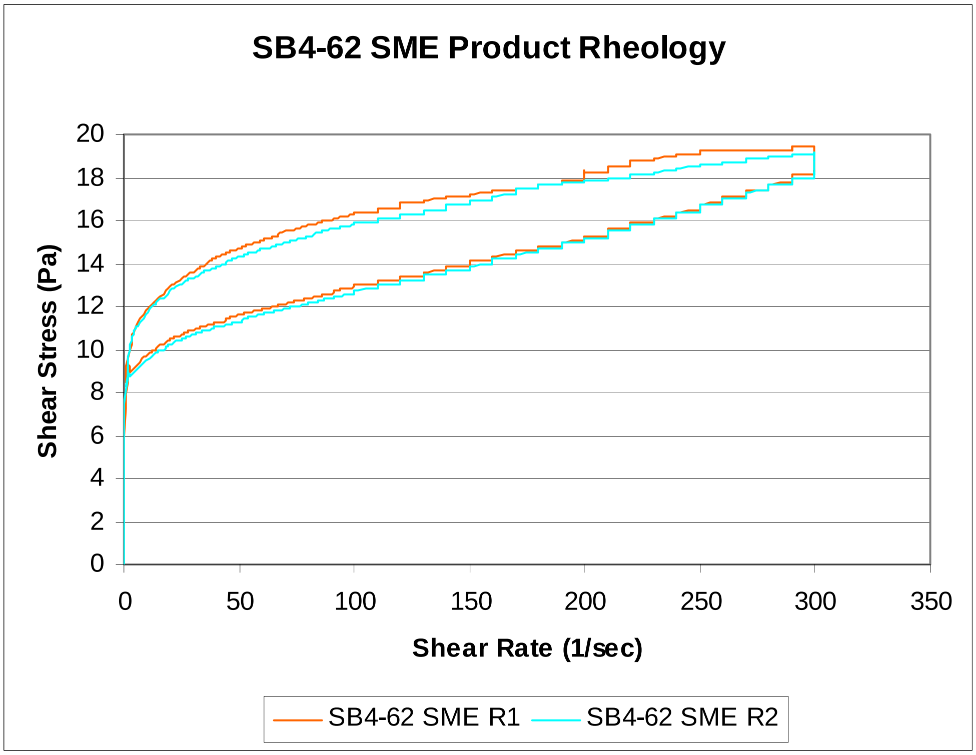

Figure D- 6. SB4-62 (150% Acid) SME Product Flow Curves

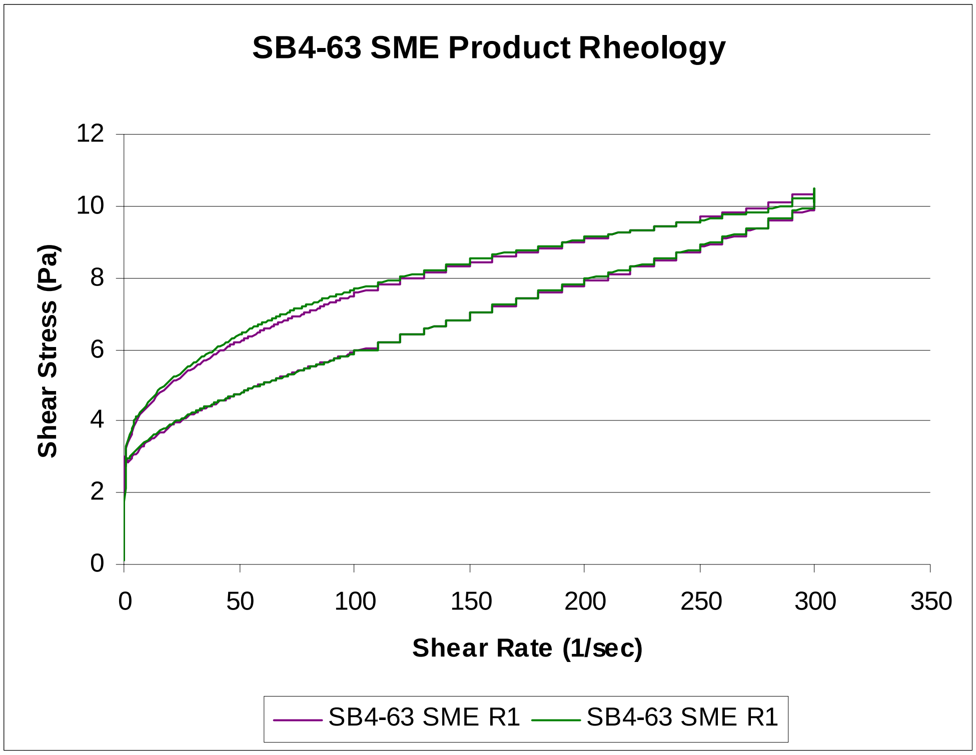

Figure D- 7. SB4-63 (160% Acid) SME Product Flow Curves

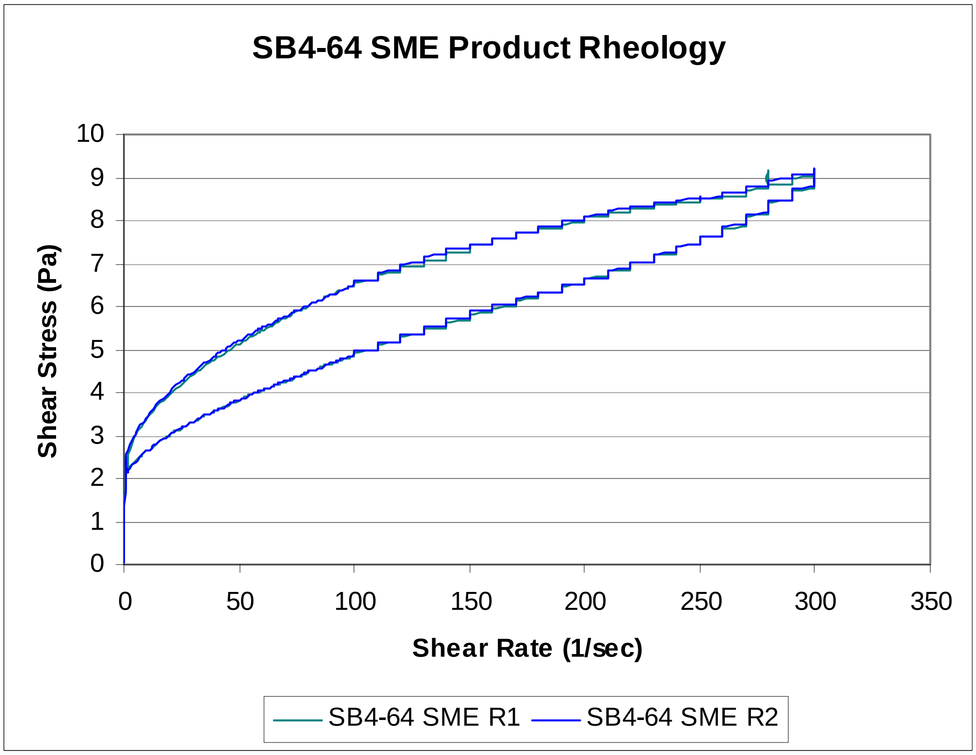

Figure D- 8. SB4-64 (170% Acid) SME Product Flow Curves

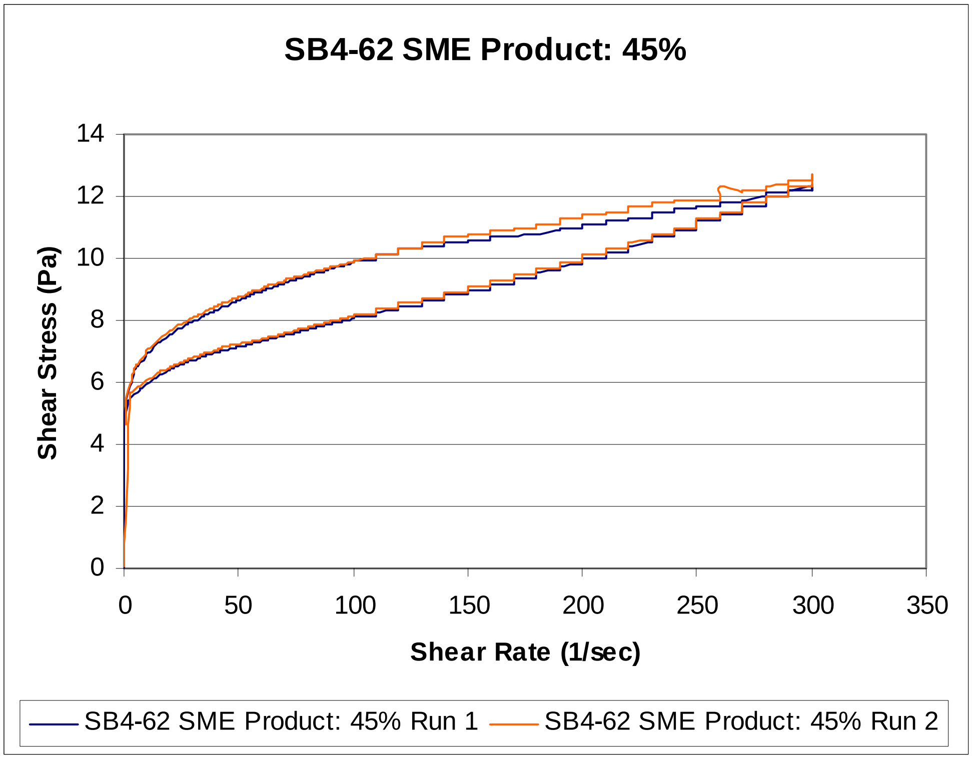

Figure D- 9. SB4-62 SME Product Adjusted to 45 wt% Solids Flow Curves

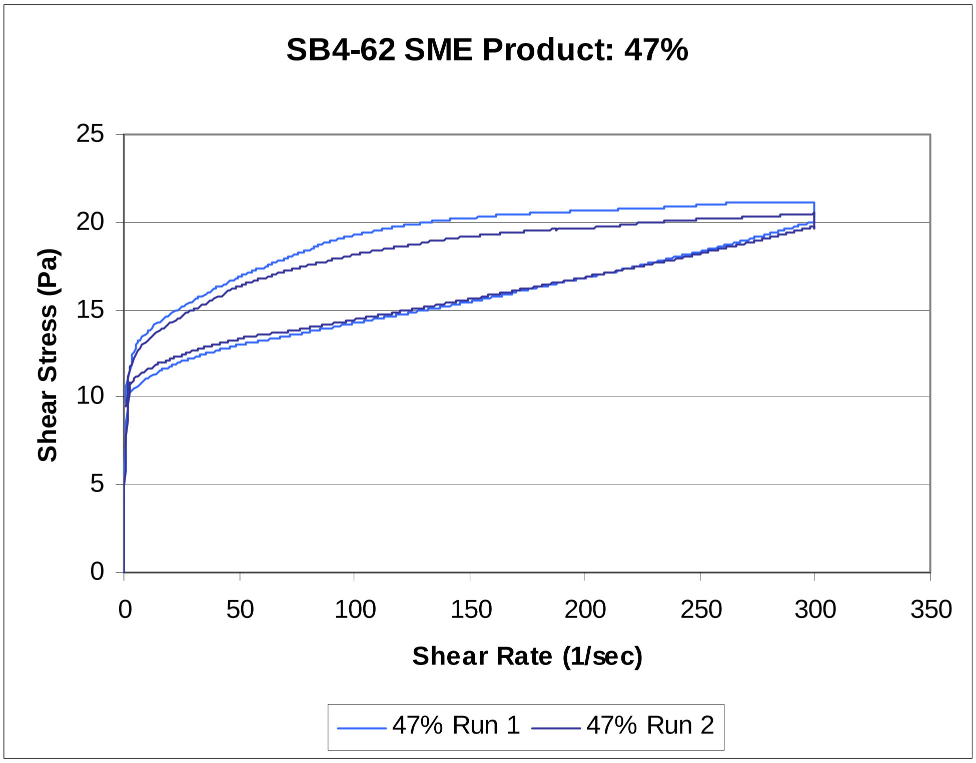

Figure D- 10. SB4-62 SME Product Flow Curves After Centrifuging

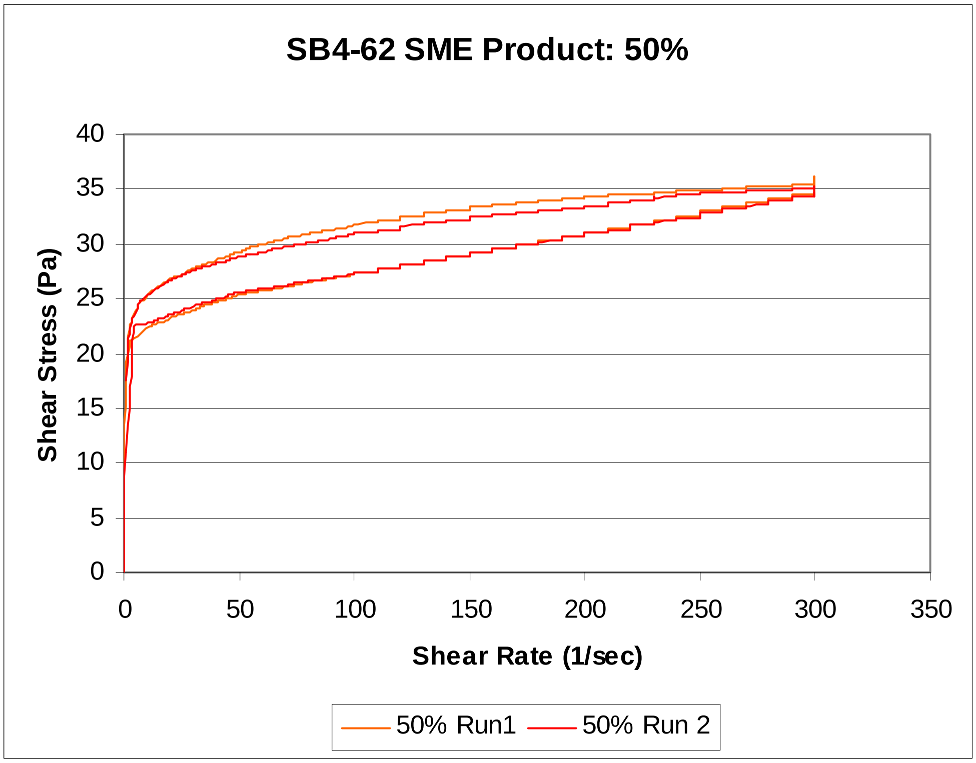

Figure D- 11. SB4-62 SME Product Flow Curves after Adjusting to 50 wt% Solids

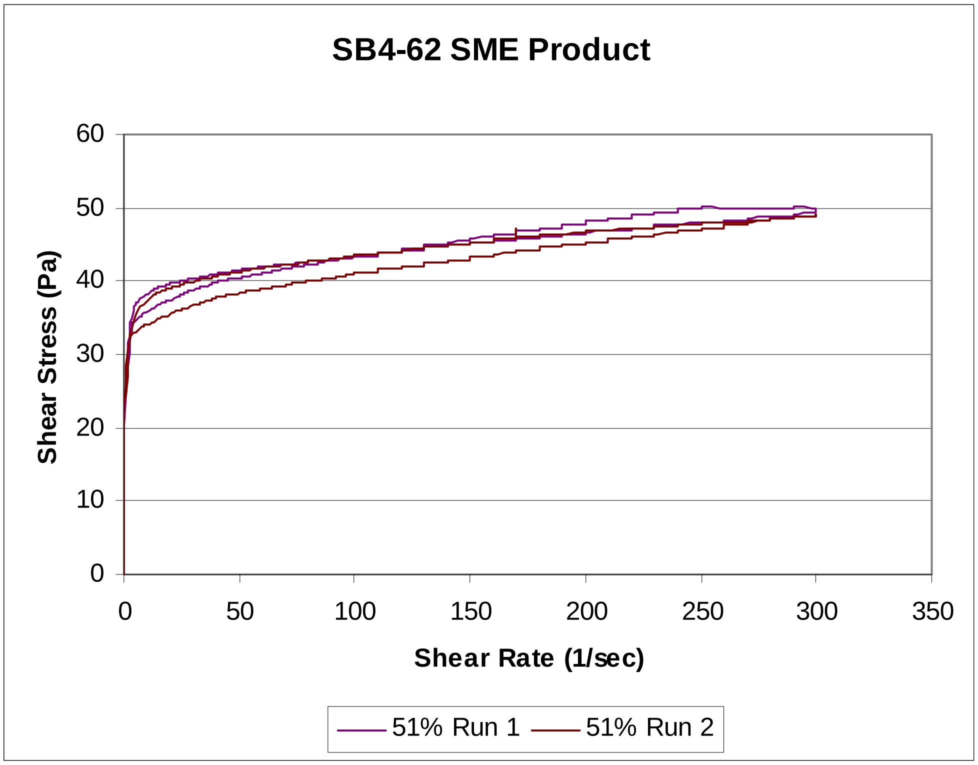

Figure D- 12. SB4-62 SME Product Flow Curves after Adjusting to 51 wt% Solids

Distribution:

J.E. Marra, SRNL
R.E. Edwards, SRNL
D.A. Crowley, 999-W
T.B. Calloway, 999-W
D.B. Burns, 786-1A
N.E. Bibler, SRNL
C.M. Jantzen, SRNL
J.R. Harbour, 773-42A
G.G. Wicks, SRNL
M. J. Barnes, 999-W
C.C. Herman, 773-42A
M.E. Smith, 999-W
M.E. Stone, 999-W
M.J. Barnes, 999-W
C.J. Bannochie, 773-42A
W.E. Daniel, 999-W
R.E. Eibling, 999-W
D.C. Koopman, 999-W
D.P Lambert, 999-W
J.M. Pareizs, SRNL
M.S. Miller, 704-S
J.E. Occhipinti, 704-S
R.M. Hoeppel, 704-27S
J.F. Iaukea, 704-30S
J.W. Ray, 704-S
B.A. Davis, 704-27S
M.A. Rios-Armstrong, 766-H
W.B. Van-Pelt, 704-S
H.B. Shah, 766-H
J.M. Gillam, 766-H
P.M. Patel, 704-27S