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Revision #0

FEED PREPARATION FOR SOURCE OF ALKALI MELT RATE TESTS

M. E. Stone
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Immobilization Technology Section
Savannah River National Laboratory
Aiken, SC 29808

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EXECUTIVE SUMMARY

The purpose of the Source of Alkali testing was to prepare feed for melt rate testing in order to determine the maximum melt-rate for a series of batches where the alkali was increased from 0% Na_2O in the frit (low washed sludge) to 16% Na_2O in the frit (highly washed sludge). This document summarizes the feed preparation for the Source of Alkali melt rate testing. The Source of Alkali melt rate results will be issued in a separate report.[3]

Five batches of Sludge Receipt and Adjustment Tank (SRAT) product and four batches of Slurry Mix Evaporator (SME) product were produced to support Source of Alkali (SOA) melt rate testing. Sludge Batch 3 (SB3) simulant and frit 418 were used as targets for the 8% Na_2O baseline run. For the other four cases (0% Na_2O, 4% Na_2O, 12% Na_2O, and 16% Na_2O in frit), special sludge and frit preparations were necessary. The sludge preparations mimicked washing of the SB3 baseline composition, while frit adjustments consisted of increasing or decreasing Na and then re-normalizing the remaining frit components. For all batches, the target glass compositions were identical.

The five SRAT products were prepared for testing in the dry fed melt-rate furnace and the four SME products were prepared for the Slurry-fed Melt-Rate Furnace (SMRF). At the same time, the impacts of washing on a baseline composition from a Chemical Process Cell (CPC) perspective could also be investigated.

Five process simulations (0% Na_2O in frit, 4% Na_2O in frit, 8% Na_2O in frit or baseline, 12% Na_2O in frit, and 16% Na_2O in frit) were completed in three identical 4-L apparatus to produce the five SRAT products. The SRAT products were later dried and combined with the complementary frits to produce identical glass compositions. All five batches were produced with identical processing steps, including off-gas measurement using online gas chromatographs.

Two slurry-fed melter feed batches, a 4% Na_2O in frit run (less washed sludge combined with frit with less Na) and a 12% Na_2O in frit run (more washed sludge combined with frit with more Na), were produced for the SMRF targeting glasses that were identical in composition. These batches were duplicates of two smaller batches which were prepared for the dry fed melt-rate testing. Four process simulations were completed in two identical experimental 22-L apparatus to produce these two melter feed batches. Both melter feed batches were produced as planned. The targeted solids content for both batches was 50-wt%.

Significant results from these 22-L feed preparations runs are listed below:

- The more washed (12% Na_2O in frit) melter feed, at a target of 50-wt % total solids, had a higher yield stress and was not pumpable at room temperature. The less washed melter feed (4% Na_2O in frit) had a lower yield stress and was easily pumped. The more washed melter feed was 42-wt % insoluble solids approximately 6-wt% higher than the less washed melter feed. The more washed melter feed was diluted down to an insoluble solid target of 36-wt % and was easily pumped. The resulting yield stress approximately

matched the undiluted, less washed melter feed after dilution to the same insoluble solids content.

- The generation of nitric oxide, nitrous oxide, and carbon dioxide were generally at the maximum for the 0% Na_2O runs (least washed sludge) and at the minimum for the 16% Na_2O runs (most washed sludge). The highest hydrogen generation occurred in the run with the least washed sludge (0% Na_2O in frit) and the lowest highest hydrogen generation occurred in the run with the most washed sludge (16% Na_2O in frit). The peak hydrogen concentration was 0.944 volume % in the 0% Na_2O in frit run.
- The least washed sludge had the highest hydrogen generation. The runs with the highest washing levels (16% Na_2O in frit) had very low hydrogen generation and no detectable formate destruction.
- Less washed sludge requires more acid than more washed sludge, leading to longer processing times. The acid addition time varied from 2.2 hours for the most washed sludge to 7.2 hours for the least washed sludge.
- The lowest pH SRAT product was the 8% Na_2O in frit run (SB3 baseline) at 5.7. The lowest and higher Na_2O in frit runs both had a SRAT product final pH of >6. The 12% Na_2O in frit run had a lower pH SME product at 5.8 and 6.2 respectively for runs 1 and 2. The 4% Na_2O in frit run had a higher pH SME product at 6.1 and 6.7 respectively for runs 1 and 2.
- The chemical processing in the 22-L and 4-L rigs are similar, based on the data that was collected at both scales.

Recommendations

The following recommendations result from this testing.

1. Evaluate targeting the insoluble solids of the melter feed, not the total solids as insoluble solids control the resulting slurry rheology.
2. Additional control of the experiments can be accomplished by improving the consistency of insulating the vessels. Insulation of the vessels with a vacuum wrap or other reproducible insulating material should be considered in future experiments.

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
CETL	Clemson Environmental Technologies Laboratory
CPC	Chemical Process Cell
DWPF	Defense Waste Processing Facility
GC	Gas Chromatograph
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
MDL	Minimum Detection Limit
MRF	Melt Rate Furnace – Experimental Test Facility at ACTL
MWWT	Mercury Water Wash Tank
REDOX	Reduction/Oxidization Potential of Melter Feed Slurry
SB2	Sludge Batch 2
SB3	Sludge Batch 3
SME	Slurry Mix Evaporator
SMRF	Slurry Fed Melt Rate Furnace – Experimental Test Facility at ACTL
SOA	Source of Alkali
SRAT	Sludge Receipt and Adjustment Tank

1.0 INTRODUCTION

The melt rate impact of the Source of Alkali (SOA) in the glass was evaluated using the dry-fed Melt Rate Furnace (MRF) and Slurry-fed Melt Rate Furnace (SMRF). The amount of sodium in the incoming sludge feed can be reduced by washing the sludge batch prior to processing at the Defense Waste Processing Facility (DWPF), while the amount of sodium in the glass frit can be increased or decreased as desired. One factor impacting the DWPF production rate is the total alkali in the melter feed. The SOA tests were designed to determine if the source of the alkali (sludge or frit) impacts the melt rate of the DWPF.

This task was initiated by DWPF Engineering via a Task Technical Request[1]. The testing plan was documented in a Task Technical and Quality Assurance Plan[2]. The results of the melt rate tests have been reported in a separate document[3].

2.0 EXPERIMENTAL METHODS

The tests were conducted using the current SB3 sludge simulants and processing strategy (155% acid stoichiometry, Frit 418, 35% waste loading, etc.) as the baseline[4]. Frit 418 contains 8% sodium oxide and 8% lithia as well as 8% boron oxide and 76% silica. Frits were produced at approximately 0%, 4%, 12%, and 16% sodium oxide. All other species in the frit were renormalized. The amount of sodium in the sludge, as well as the waste loading, was adjusted to maintain a constant glass composition throughout the tests. The targeted frit compositions are shown in Attachment 1.

Sludge was produced for each test by blending the existing SB2 and SB3 simulants (produced by Clemson Environmental Technologies Laboratory) and then adding trim species as required to meet composition requirements.[5] Sodium is present in the sludge as a number of species, including carbonate, fluoride, hydroxide, nitrate, nitrite, phosphate, and sulfate. In order to maintain a constant glass composition and since they have limited solubility in glass, the fluoride, phosphate, and sulfate were held constant during the tests. The amounts of carbonate, hydroxide, nitrate, and nitrite were adjusted in constant relative proportion to meet the required sodium concentrations in the sludge. The targeted sludge compositions are shown in Attachment 1.

Waste loading (defined as the mass fraction of the glass that comes from the sludge) adjustment was required since removal of sodium from the frit requires less frit in the glass to maintain constant boron oxide, lithia, and silica concentrations. Therefore, waste loading was decreased as the sodium concentration of the sludge was decreased which corresponds to additional washing. Correspondingly, waste loading was increased as the sludge washing was decreased and less sodium was added to the frit. The targeted waste loadings are shown in Attachment 1.

The sludge was processed through lab-scale simulations of the Chemical Process Cell (CPC) process to provide feed for the melt rate program. Five tests, Sludge Receipt and Adjustment Tank (SRAT) cycles only, were performed at 0%, 4%, 8%, 12% and 16% frit sodium oxide in a

4-L vessel to provide five 2.5-L batches of slurry to be dried and then used as feed for the MRF tests. These feeds were mixed with the matching frit before performing the melt rate test. Four tests were performed with both SRAT and Slurry Mix Evaporator (SME) cycles in a 22-L vessel. There were two tests each at 4% and 12% frit sodium oxide to provide a total of four 14-L batches of slurry for the SMRF tests. The SRAT cycles were performed at 155% acid stoichiometry and targeted a Reduction/Oxidation (REDOX) potential to yield a ratio of 0.2 $Fe^{+2}/\Sigma Fe$ in the glass. Other parameters utilized in the acid calculation include estimates of the amount of formate destroyed during processing and conversion of nitrite to nitrate. The basis for the conversions and destructions is data from previous SB2/3 experiments.[6] These parameters are shown in Table 1 and in the acid calculation sheets shown in Attachment 2.

Table 1– Targets for Acid Calculations

Fresh Sludge Analysis	SOA 0% Na2O Frit 4L SRAT Run	SOA 4% Na2O Frit 4L SRAT Run	SOA 8% Na2O Frit 4L SRAT Run	SOA 12% Na2O Frit 4L SRAT Run	SOA 16% Na2O Frit 4L SRAT Run	Units
Conversion of Nitrite to Nitrate	30	30	30	30	30	%
Destruction of Nitrite including SME cycle	100	100	100	100	100	%
Destruction of Formic acid charged	13	13	13	13	13	%
Destruction of oxalate charged	10	10	10	10	10	%
Percent Acid in Excess Stoichiometric Ratio	155	155	155	155	155	%
Reduction/Oxidation (REDOX)	0.2	0.2	0.2	0.2	0.2	$Fe^{+2}/\Sigma Fe$

The SRAT/SME apparatus used during the runs consisted of a SRAT/SME vessel, SRAT condenser, Mercury Water Wash Tank (MWWT), vent condenser, temperature indication and controller, pH probe, antifoam addition funnel, mixer, acid addition lines and pumps, air purge lines with flow controllers and a blowout manometer, and recirculating water baths for the condensers. A diagram and pictures of the apparatus (4-L and 22-L) are shown in Attachment 3.

During the 4-L tests, an online gas chromatograph (GC) was utilized to measure the hydrogen, helium, carbon dioxide, nitrous oxide, oxygen, nitrogen, and nitric oxide concentrations in the offgas. Helium was added to the air purge as a tracer and monitored during these runs to give an estimation of overall flowrate.

The SRAT cycles in the 4-L apparatus were performed in the same manner for all runs by utilizing the steps shown in Attachment 4 for the 4-L 4% Na_2O SOA run. These steps mimic the DWPF process as closely as possible, but do not include the SRAT process heels. The SRAT and SME cycles in the 22-L apparatus were performed in the same manner for all runs by utilizing the steps shown in Attachment 4 for the 22-L 4% Na_2O SOA run. These steps mimic the DWPF process as closely as possible, but do not include the SRAT or SME process heels or the decontamination frit additions of the DWPF process. Samples were taken of the sludge simulants and the SRAT product for each run and analyzed for solids content (total, soluble, insoluble, and calcined solids), density, pH, metal content via Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES), anion content via Ion Chromatography (IC), and rheological properties. The 4-L runs were stopped after the SRAT cycle was completed. Data sheets were utilized to record temperature, pH, and offgas composition profiles during the run. All data were collected in a laboratory notebook[7].

Analytical

Analyses for this task used guidance of an Analytical Study Plan[8]. Sample request forms were used for samples to be analyzed, and analyses followed the guidelines and means of sample control stated in the Analytical Study Plan for the task. A unique ITS, Immobilization Technology Section - Mobile Lab (Mobile Lab), and/or Analytical Development Section (ADS) lab identification number was assigned to each sample for tracking purposes. Analyses were performed using approved analytical and QA procedures.

Samples were taken of the simulant before the runs were initiated and of the SRAT product at the end of the cycles for analyses. The samples were analyzed by the Mobile Lab, the ITS, and the ADS. The Mobile Lab performed analyses on the sludge slurries 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 lithium metaborate fusion. The preparations were then analyzed using ICP-AES to measure the cations present. 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 initial and product samples. ITS performed the titration on the starting sludge samples to provide the necessary input for the acid calculation. A manual titration was performed at ACTL using a 1M HNO_3 solution and 10:1 dilution of the sample. The calibration curve was performed to a pH of 4 and was performed in duplicate at a minimum. Finally, the ADS measured the total inorganic carbon (TIC) of the sludge simulant using the ITS Acid Demand TIC method. The total inorganic carbon information was needed as an input in the acid calculation.

Gases were monitored during the 4-L runs using a high-speed micro GC to provide insight into the reactions occurring during processing and to determine whether a flammable mixture is formed. As mentioned above, helium was used as a GC internal standard. The GC is self-contained and is designed specifically for fast and accurate analysis. The GCs had five main components. The first is the carrier gas (argon for this testing) to transport the sample through the molecular sieve and poraplot Q columns. The second is the injector, which introduces a measured amount of sample into the inlet of the analytical columns where it is separated. 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 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. 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 single calibration standard was used in each run to bound upper quantities of the expected gases. The concentrations of these calibration standards were specified based upon previous work. These concentrations are re-evaluated when new calibration gases are ordered. Calibration checks are performed before and after each run. The calibration standards are balanced in nitrogen because helium is used as an internal standard and is also used to detect leakage during the actual runs.

3.0 RESULTS

3.1 4-L Runs

The 4-L SRAT runs were performed the week of 12/13/04. All runs were completed without incident and no processing issues, such as excessive foaming or thickening of the slurry, were noted during any of the runs. The formic acid addition on the 0% Na_2O in Frit run did not meet the targeted amount when the sample tray was weighed. This result may have been due to an air bubble in the titrator pump syringe.

3.1.1 Run Data

The SRAT cycle dewater amounts and pH of the vessel was monitored during each run. These data are shown in Figure 1 and Attachment 5. The dewater rates during acid addition varied considerably during the runs. The insulation on each of the rigs was custom-made and may have led to different heat losses from each kettle, causing more internal reflux during selected runs depending on how well the insulation blanket insulated the kettle and how tightly the blanket was secured to the vessel. The two runs with the lowest acid consumption also had the lowest dewater rates, leading to speculation that the vessel did not have time to fully heat up prior to the conclusion of acid addition. All runs had acid addition times greater than one hour, and the condensate generation rates of other runs were much higher at equivalent times during the runs.

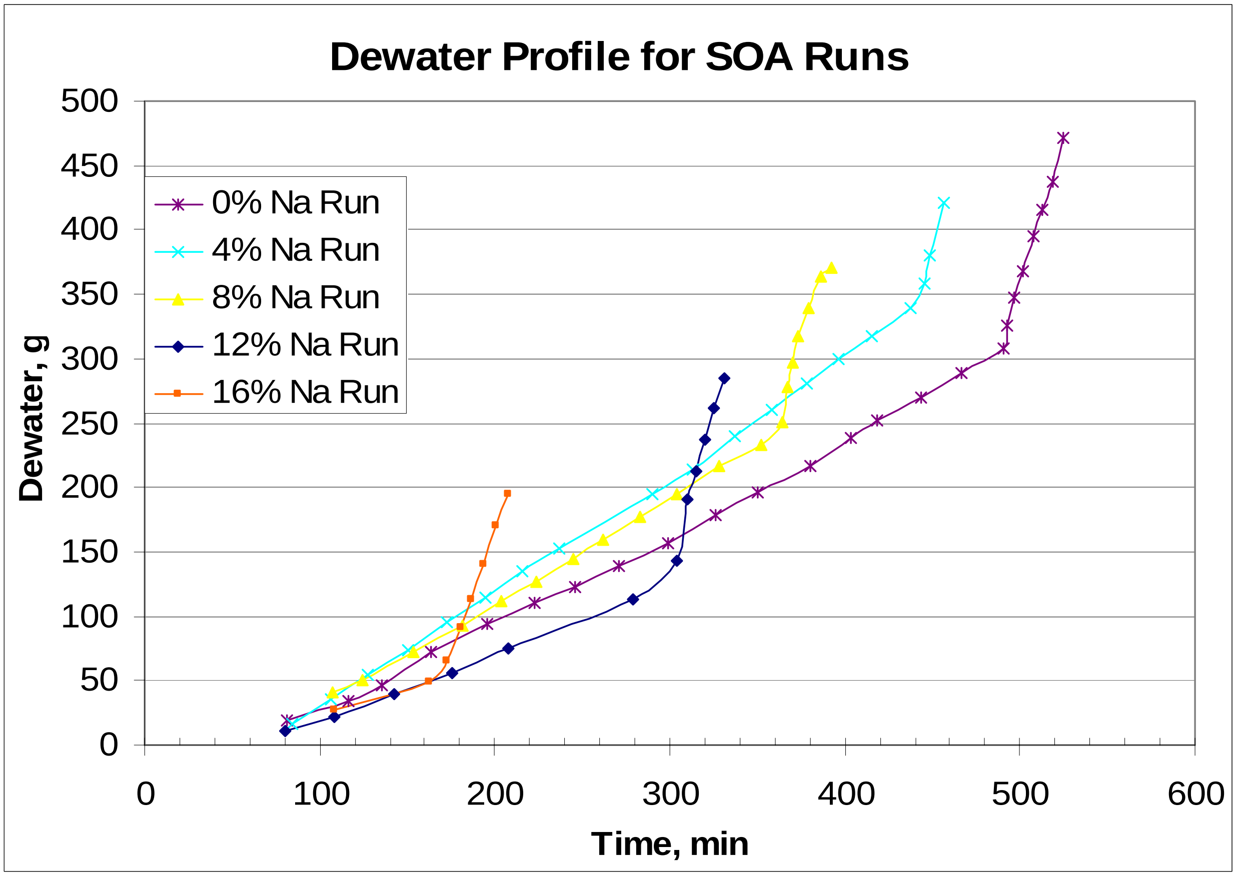


Figure 1 – Dewater Profile of 4-L runs

The pH profiles for all runs are shown in Attachment 5. The run with the least amount of acid addition (16% Na_2O in Frit) had the highest pH after acid addition (pH = 5.0), but only rose 1.15 pH points during boiling. The run with the largest acid addition (0% Na_2O in Frit) had the largest pH shift during boiling: 2.12 pH. The baseline run had the lowest initial pH and a small pH rise during boiling in the SRAT. This was likely due to a slight overaddition of acid. The baseline run had the highest calculated H^+ concentration increase of all the runs. In general, the runs with the lowest washing had the highest pH at the end of the SRAT cycle, likely due to the consumption of formic acid and the production of hydrogen.

The major acid consumers are the neutralization of hydroxide, carbonate decomposition, manganese reduction, and nitrite decomposition. Table 2 below summarizes the concentrations of these acid consumers for each of the runs.

Table 2 – Acid Consumers Present in 4-L Runs

	Less Washed			More Washed		
	0%	4%	8%	12%	16%	Units
Nitrite (NO_2^-)	1.452	1.132	0.803	0.466	0.032	moles
Mn	0.384	0.403	0.398	0.410	0.357	moles
Carbonate	0.770	0.690	0.553	0.501	0.383	moles
OH^-	1.899	1.635	1.456	0.857	0.372	moles

3.1.2 Offgas Data

The data from the on-line GC's was regressed to compare the gas generation from the runs as a function of the frit sodium oxide concentration. The data was not shifted to account for the start of each acid addition since each run had acid additions of different lengths and only the start of nitric acid addition would have been synchronized. The offgas data are shown in Attachment 6.

Carbon dioxide can be produced from manganese reduction, carbonate decomposition or formic acid decomposition. Carbon dioxide generation was highest for the runs with highest acid addition (0% Na_2O in Frit) and much lower in the runs with lowest acid addition amounts (16% Na_2O in Frit) as shown in Figure 2. It is also noted in section 3.1.3 that there was no measured formate destruction in the 12% and 16% experiments, suggesting that all of the carbon dioxide generation in these two runs was due solely to manganese reduction and carbonate decomposition.

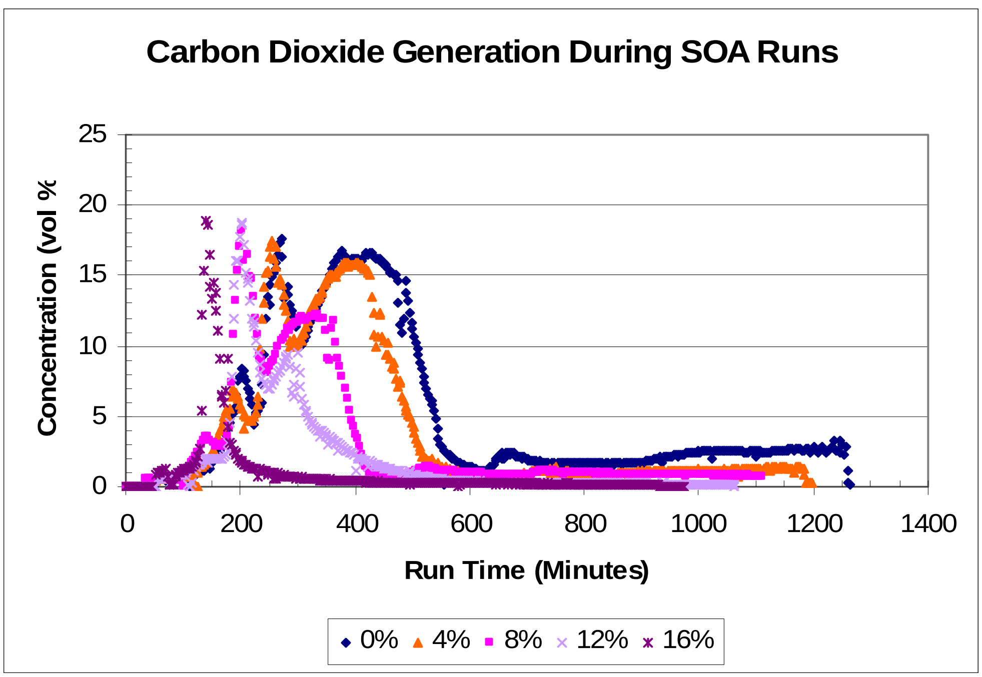


Figure 2 – Carbon Dioxide Profile of 4-L runs

In general, the runs with the highest nitrite had the highest nitric oxide production. The exceptions were the 4% and 8% runs. During the 4 and 8% runs, the nitric oxide was not accurate, and was likely underestimated. However, the trends from the other runs followed the nitrite concentration of the feed, as did the nitrous oxide concentration.

Nitrogen, oxygen and helium concentrations dipped during the runs when large quantities of gases were being emitted from the batch as shown in Figure 3. During the 8% SRAT cycle, the nitrogen and helium continued to drop throughout the batch. This is not reflective of normal processing but is indicative of quantification problems with the GC. For all five runs, the dip in concentration followed a general trend in relation to the amount of acid added to each batch.

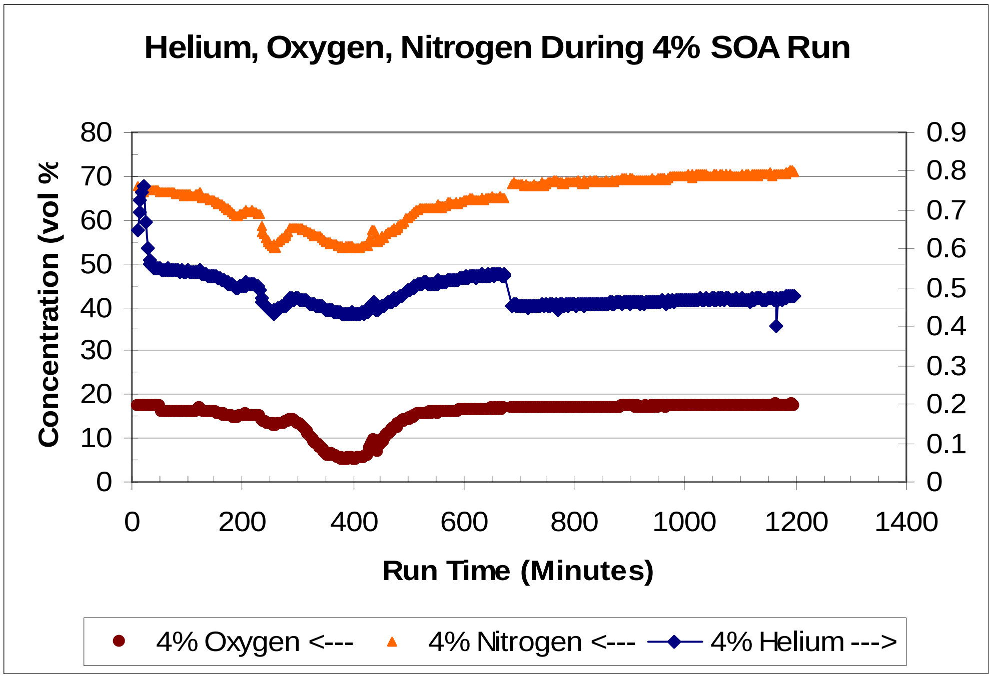


Figure 3 – Oxygen, Nitrogen and Helium Profile of 4% SOA 4-L run

The bulk of the hydrogen is produced from rhodium catalyzed decomposition of formic acid.[9] The 0% SRAT cycle had the highest hydrogen generation with a hydrogen peak of 0.937 volume % as shown in Figure 4. The 8% SRAT cycle had the second highest hydrogen generation with a hydrogen peak of 0.351 volume %. The 4% SRAT cycle had the second highest hydrogen generation with a hydrogen peak of 0.240 volume %. The maximum hydrogen in the 12% and 16% SRAT cycles was <0.01 volume %. The 8% SRAT cycle was expected to have a lower hydrogen generation than the 4% SRAT cycle. This reversal may have been due to the slight overaddition of acid for the 8% SRAT cycle.

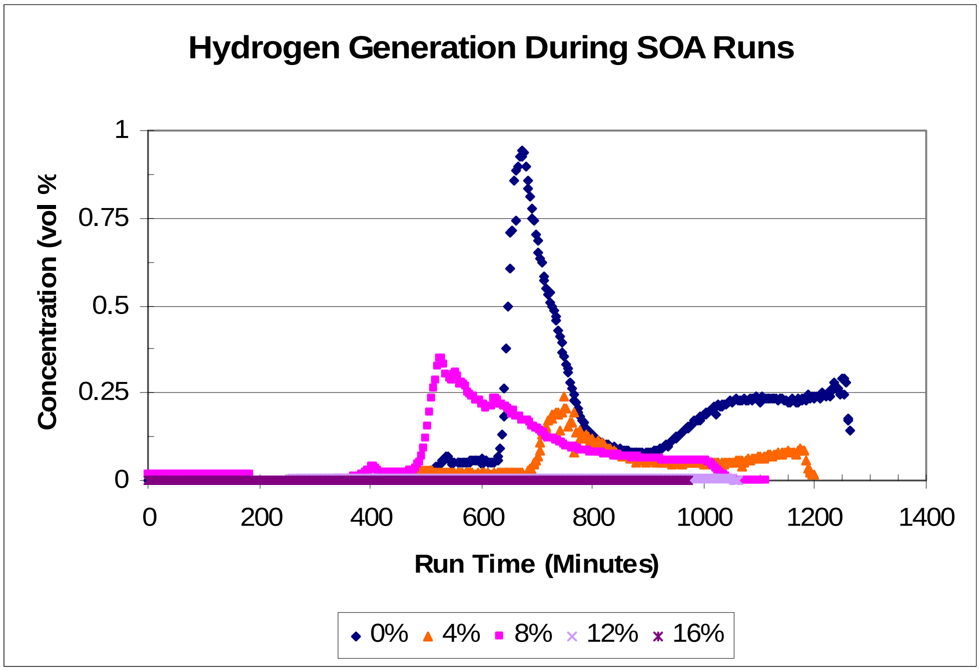


Figure 4 – Hydrogen Profile of 4-L runs

3.1.3 Anion Balance

The amount of nitrite, nitrate, and formate in the sludge, acid additions, and final product was determined and utilized to perform a mass balance to determine the actual nitrite to nitrate conversion and the formic acid destruction during the runs. A summary of this data is shown in Table 3. The analytical results used to calculate the conversion and destruction efficiency are in Attachment 7. Overall, formic acid destruction for the 0%, 4%, and 8% runs was similar at 9-13%. The 12% and 16% runs had values indicating that no formic acid destruction occurred. Overall, the nitrite conversion was 16-25% on a SRAT product basis.

Table 3 - Nitrite to Nitrate Conversion and Formic Acid Destruction in 4-L Runs

%Na_2O in frit	0	4	8	12	16[1]
Formate Destruction (%)	10.31	9.21	12.90	-0.12	-1.58
% nitrite conversion (remaining in product)	20.63	16.40	25.04	16.89	77.53
Overall nitrite to nitrate conversion %	38.60	31.53	44.92	23.84	86.54

3.1.4 Metals Content of 4-L SRAT Products

The SRAT products were calcined and analyzed for metals content and calcine factors. The frit additions determined for each run as well as the targeted frit compositions were utilized to calculate glass compositions for each of the runs from the measured SRAT compositions as shown in Table 4. The complete data set is shown in Attachment 7. It can be concluded that the runs were successful in generating melter feeds that will result in the same glass compositions. It

[1] Nitrite concentration in the 16% SRAT Cycle was so low that the conversions involving nitrite are meaningless.

should be noted that the Mn was consistently lower than the target and the Li, SO_4 and ZrO_2 were consistently higher than the target. Also, the 8% Fe value is lower than the target.

Table 4 –Projected Glass Composition in 4-L Runs from SRAT Product Data

Oxide	Target Composition	Projected Glass Compositions, wt % by Target Na_2O in Frit				
		0	4	8	12	16
Ag_2O	0.00	<MDL	<MDL	<MDL	<MDL	<MDL
Al_2O_3	5.991	6.133	6.143	6.303	6.152	6.02
B_2O_3	5.200	5.152	5.297	4.846	5.361	5.474
BaO	0.00	0.036	0.036	0.038	0.036	0.035
CaO	1.291	0.969	0.935	0.977	0.911	0.88
CdO	0.050	<MDL	<MDL	<MDL	<MDL	<MDL
Cr_2O_3	0.082	0.096	0.091	0.085	0.082	0.093
CuO	0.068	0.075	0.066	0.072	0.074	0.083
Fe_2O_3	14.6	14.5	14.3	13.7	14.3	14.3
K2O	0.015	0.061	0.059	0.073	0.060	0.067
MgO	1.500	1.645	1.575	1.622	1.562	1.726
MnO	2.283	1.883	1.864	1.845	1.864	1.812
Gd_2O_3	0.027	0.024	0.024	0.025	0.025	0.026
Li_2O	5.200	5.526	5.923	5.633	5.751	5.880
Na_2O	12.5	12.9	13.0	12.9	12.8	13.2
NiO	0.448	0.420	0.404	0.377	0.383	0.418
P_2O_5	0.078	0.074	0.071	0.073	0.071	0.070
PbO	0.023	<MDL	0.038	0.039	0.039	0.037
PdO	0.00	<MDL	<MDL	<MDL	<MDL	<MDL
RuO_2	0.00	<MDL	<MDL	<MDL	<MDL	<MDL
SO_4	0.342	0.477	0.477	0.479	0.453	0.524
SiO_2	50.1	50.0	51.6	51. 3	51.9	52.8
TiO_2	0.00	0.017	0.017	0.038	0.018	0.017
ZnO	0.132	0.151	0.144	0.144	0.143	0.142
ZrO_2	0.145	0.240	0.247	0.252	0.258	0.237
Totals	100.00	100.382	102.331	100.864	102.273	103.829

3.1.5 Rheological Properties

The rheological properties of the sludge simulants and SRAT products were measured to determine the impact of washing on rheology. The yield stress and consistency of the simulants and SRAT products are shown in Table 5, along with the solids content of the samples. Note that the insoluble solids were relatively constant (13.5-14.5 wt% solids) while the soluble solids varied from 4.5-17.8 wt % due to the degree of sludge washing. The yield stress of the five SRAT products is relatively consistent and well below DWPF limits for processing. The complete data set including the flow curves are shown in Attachment 8.

Table 5 – SRAT Product Rheological Properties in 4-L Runs

Run	Total Solids (wt %)	Insoluble Solids (wt %)	Soluble Solids (wt %)	Consistency (cP)	Yield Stress (Pa)
0%	32.13%	14.31%	17.82%	9.22	1.75
4%	29.96%	13.96%	16.01%	8.49	1.21
8%	27.50%	13.94%	13.57%	9.07	1.87
12%	23.77%	14.48%	9.28%	5.33	4.57
16%	17.98%	13.46%	4.52%	5.47	1.51

3.1.6 Acid Demand

The acid demand and SRAT product pH of the sludge simulants were measured and are shown in Table 6. The least washed sludge, the 0% Na_2O in frit run, required the most acid to neutralize the extra sodium hydroxide nitrite and other soluble acid consumers. However, its SRAT product pH was highest, due to the consumption of formic acid and production of hydrogen. The 16% Na_2O in frit run required the least acid to neutralize since most of the sodium hydroxide and other soluble acid consumers were washed out. However, its SRAT product pH was lower, due to the low consumption of formic acid and low hydrogen generation.

Table 6 – Acid Added and Final SRAT pH in 4-L Runs

Run	Acid Added, M	SRAT Product pH
0%	2.98	6.66
4%	2.55	6.45
8%	2.12	5.72
12%	1.47	6.11
16%	0.755	6.15

3.2 22-L SRAT/SME Runs

The 4% and 12% Na_2O in Frit runs were repeated in the 22-L vessel to prepare feeds for the SMRF testing. Two separate batches, each targeting approximately 14-L of product slurry, were completed to prepare 28-L of 4% melter feed and 28-L of 12% melter feed. A SRAT/SME cycle was performed on each run with a targeted total solids content of 50 wt% for the SME products. Online GC's were not utilized during the tests, but the SRAT cycle of each run was otherwise conducted in the same manner as the 4-L runs. The first 4% run is labeled 4-1 and the second is labeled 4-2 in the following sections. The first 12% run is labeled 12-1 and the second is labeled 12-2 in the following sections.

3.2.1 Run Data

The dewater amounts and pH of the vessel were monitored during each run. This data is shown in Attachment 5. The dewater rates during acid addition varied considerably during the runs. The insulation on each of the rigs was custom-made. This may have led to different heat losses from each kettle, causing more internal reflux during selected runs depending on how well the insulation blanket insulated the kettle and how tightly the blanket was secured to the vessel.

The pH profiles for all runs are shown in Attachment 5.

3.2.2 Metals Content of 22-L SME Products

After the SME products were calcined, they were analyzed for metals content. Based on these results, the calculated oxides present in the calcined SME product are shown in Table 7. It can be concluded that the runs were successful in generating melter feeds that will result in the same glass compositions. It should be noted that Ca and Mn were consistently lower than the target and the ZrO_2 was consistently higher than the target. The complete data set is shown in Attachment 7.

Table 7 – Measured Glass Composition in 22-L Runs

		Measured Glass Composition			
Oxide	**Target**	**4-1**	**4-2**	**12-1**	**12-2**
Ag_2O	0	0.017	0.013	0.000	0.000
Al2O3	5.99	6.44	6.70	6.55	6.58
B2O3	5.2	5.07	4.96	5.17	5.09
BaO	0	0.0515	0.0526	0.0498	0.0493
CaO	1.291	0.936	0.975	0.920	0.979
CdO	0.05	0.000	0.000	0.000	0.000
Cr_2O_3	0.082	0.079	0.082	0.077	0.077
CuO	0.068	0.071	0.099	0.079	0.074
Fe_2O_3	14.6	14.4	14.7	14.4	14.4
Gd_2O_3	0.027	0.020	0.018	0.019	0.017
K2O	0.015	0.055	0.059	0.056	0.061
Li_2O	5.2	5.1	5.0	5.2	5.2
MgO	1.5	1.4	1.4	1.4	1.4
MnO	2.283	1.800	1.858	1.800	1.793
Na_2O	12.5	12.6	12.7	12.3	12.7
NiO	0.448	0.507	0.512	0.498	0.526
P_2O_5	0.078	0.063	0.077	0.074	0.055
PbO	0.023	0.036	0.037	0.034	0.033
SO_4	0.342	0.246	0.348	0.254	0.293
SiO_2	50.1	50.2	48.9	50.9	50.0
TiO_2	0	0.017	0.017	0.017	0.017
ZnO	0.132	0.143	0.151	0.137	0.137
ZrO_2	0.145	0.254	0.259	0.269	0.246
Totals	100	99.6	99.0	100.2	99.7

3.2.3 Rheological Properties

The rheological properties of the SME products were measured to determine the impact of washing on rheology. Flow curves are shown in Attachment 8. The yield stress and consistency of the SME products are shown in Table 8, along with the solids content of the samples. The yield stress of the 12% SME product was 2.25 times higher than the yield stress of the 4%

product. After cooling, the 12% slurry was very difficult to mix in the kettle and pump using a peristaltic pump. The last 10% of the 12% SME product had to be removed from the 22-L vessel using a spatula. Note that slurry with higher insoluble solids would be expected to have a higher yield stress.

Table 8 – Rheological Properties of SME product in 22-L Runs

Run	Total Solids (wt %)	Insoluble Solids (wt %)	Soluble Solids (wt %)	Consistency (cP)	Yield Stress (Pa)
4-1	49.33	35.52	13.81	56.0	27.5
4-2	48.90	36.12	12.78	94.0	32.7
12-1	50.17	41.73	8.43	193	71.4
12-2	49.32	40.55	8.77	93.9	64.1

The DWPF design basis is from 2.5 to 15 Pa (25 to 150 dynes/cm^2) for the yield stress of melter feed. The DWPF design basis is from 10 cP to 40 cP for the consistency.[10] All of these products were more viscous than the DWPF design basis. Even though both products are above design basis, the 4% SME product was pumpable and easily mixed?

Since the 12% slurry was very difficult to pump, the sample was diluted to the same insoluble solids concentration as the 4% slurry. The diluted sample had a yield stress of 37.1 Pa, approximately the same yield stress as the 4% SME product. After dilution, this slurry was easily pumped, and all melt rate testing was done with diluted 12% slurry.

3.2.4 Separate Phase in Drain-leg Between Condenser and MWWT

A separate organic layer was noticed to have accumulated in the 90° bend of the drain-leg between the SRAT Condenser and the MWWT. This has not been noticed in previous runs and is likely due to a change in the condenser and MWWT design, which added this drain-leg (added to make equipment more prototypic and easier to assemble). It is likely that the organic accumulation is the result of antifoam degradation products related to the large quantify of antifoam that is added to minimize foaming. A photo of the accumulation is included in Figure 3-3 (Attachment 3). This accumulation occurred during each of the four 22-L experiments. This organic layer was sampled, but the sample was discarded with the condensate from the run prior to analysis. The same type of accumulation has been noted in subsequent runs. A sample has been submitted from these runs to determine if the antifoam is the source of the organic layer.

3.3 Comparison between the 4-L and 22-L Runs

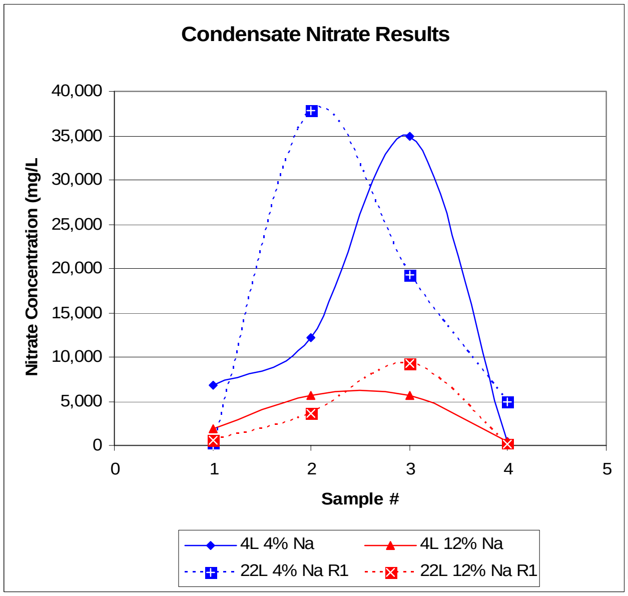
The SRAT simulations completed in the 4-L and 22-L vessels were designed to be identical, except for scale. The acid addition flowrates, boilup flux, reflux time, offgas purge rates and other parameters were all scaled to ensure the process times were identical. The 22-L Runs were completed with a SME cycle but SRAT product was not sampled and the GC was not online during the runs. The 4-L runs were SRAT cycles only. Comparisons were made based on the data available and estimates of the 22L SRAT product composition determined from the SME product samples.

3.3.1 Acid requirement and final pH

In both the 4-L runs and 22-L runs, the batching for the runs was calculated based on analysis of the sludge simulant. For the 4% Na_2O experiments, the acid requirement was 2.55 M for the 4-L run and 2.59 for the 22-L runs. The final SRAT pH was 6.45 for the 4-L run and 6.36 and 6.43 for the two 22-L runs. For the 12% Na_2O experiments, the acid requirement was 1.47 M for the 4-L run and 1.49 M for the 22-L runs. The final SRAT pH was 6.11 for the 4-L run and 5.88 and 6.27 (an average of 6.08) for the two 22-L runs. Based on this comparison, there was excellent agreement between the two scales.

3.3.2 Condensate collection and composition

The condensate was collected throughout the run (Dewater samples 1, 2 and 3) and the condensate collected in the FAVC and MWWT were both collected and analyzed. The Dewater samples were generally higher in formate and nitrate concentration at the end of the SRAT cycle (Dewater 3) than at the beginning of the SRAT cycle. In addition, many of the FAVC samples were extremely high in nitrate and low in pH. The results are summarized in Attachment 5. The profiles for the nitrate and formate concentration are similar in the 4-L and 22-L rigs as shown below in Figures 5 A and 5B. Note also that the profiles are similar when comparing the two 4% Na_2O runs to each other, but are distinctly different from the 12% Na_2O runs.



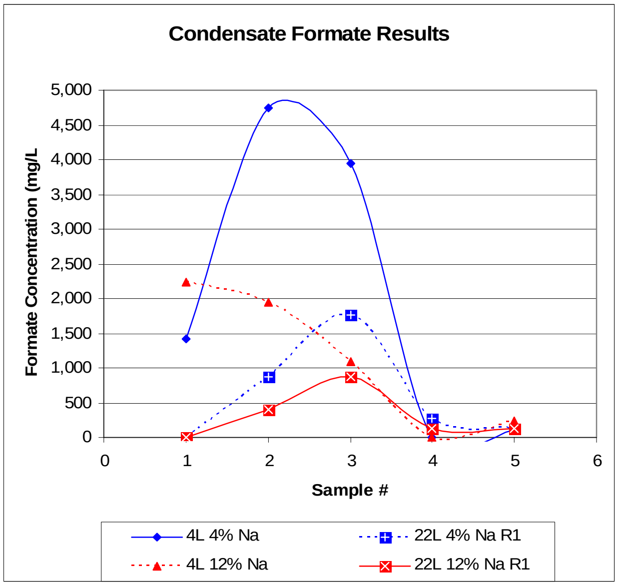


Figure 5A and 5B – Nitrate and Formate Profile during SRAT Cycle

3.3.3 Dewater Rate

Much of the dewatering occurred during acid addition. The runs with the least washing had the longest acid addition times and led to the most dewatering during acid addition. In the 22-L runs, most of the dewater mass was collected before boiling was initiated. Table 9 summarizes the dewater rate during acid addition. The dewater rate was approximately double in the 22-L rigs versus the 4-L rigs. A big factor in the variability from run to run is the quality of insulation.

Improvements could be made in reproducibility by improving the consistency of insulating the rigs.

Table 9 – SRAT Dewater Rate during Acid Addition

Run	Acid Feed Time, hrs	Condensate Collection, g	Calculated Dewater rate, g/min/L
4-L 0% Na_2O in frit	7.2	288.6	0.27
4-L 4% Na_2O in frit	6.25	268.49	0.34
4-L 8% Na_2O in frit	5.27	233.1	0.29
4-L 12% Na_2O in frit	3.8	112.5	0.20
4-L 16% Na_2O in frit	2.2	65.4	0.20
22-L 4% Na_2O in frit	6.28	2256.2	0.50
22-L 4% Na_2O in frit	6.35	2652.2	0.58
22-L 12% Na_2O in frit	3.52	1631.6	0.64
22-L 12% Na_2O in frit	3.58	958.9	0.37

3.3.4 Anion Comparison

Since no SRAT product samples were pulled in the 22-L experiments, the SME product samples were corrected to SRAT product values by multiplying the appropriate SME product concentration by the SRAT sludge:SME sludge ratio. The SRAT:SME ratio was 1.22-1.25 for the 4% Na_2O in frit run and 1.15-1.16 for the 12% Na_2O in frit run. A comparison of the data is shown in Table 10. Note that the formate concentration in the 22-L results is about 3-9% lower than the comparable 4-L result. Also, the nitrate concentration in the 22-L results is about 11% higher than the comparable 4-L result. These results are within the expected error of the analytical and estimation methods used. Note that the 22L SME product contained less formate than the 4L SRAT products in spite of the formic acid additions during frit additions. The lower formate results are consistent with continued destruction of formate during the SME cycle from noble metal catalyzed reactions.

Table 10 –SRAT Product Anion Comparison Corrected From 22-L SME Product Data

Analyte	Sludge Content	Calculated Sludge Solids	Target Sludge Solids	SRAT:SME Ratio	Cl^-	NO_2^-	NO_3^-	$HCOO^-$
Run	g/1000g	wt%	wt%		mg/kg	mg/kg	mg/kg	mg/kg
4% 22L #1*	751.49	32.71	30	1.22	0	0	42,800	75,700
4% 22L #2*	754.00	31.88	30	1.25	125	0	41,800	73,600
4% 4L	No Correction Needed				113	0	37,400	81,200
12% 22L #1*	706.98	29.30	23.8	1.15	118	200	22,900	49,500
12% 22L #2*	713.72	28.75	23.8	1.16	122	0	24,100	50,500
12% 4L	No Correction Needed				113	478	21,000	51,300

* Calculated values from SME product samples.

3.4 Processing Conclusions

Testing was completed with a series of sludges and frits that spanned the extreme from unwashed (0% Na_2O in frit) to overwashed (16% Na_2O in frit). The following processing conclusions can be made based on this testing:

- The more washed (12% Na_2O in frit) melter feed, at a target of 50-wt % total solids had a higher yield stress and was not pumpable at room temperature. The less washed melter feed (4% Na_2O in frit) had a lower yield stress and was easily pumped. The more washed melter feed was 42-wt % insoluble solids approximately 6-wt% higher than the less washed melter feed. The more washed melter feed was diluted down to an insoluble solid target of 36-wt % and was easily pumped. The resulting yield stress approximately matched the undiluted, less washed melter feed after dilution to the same insoluble solids content.
- The generation of nitric oxide, nitrous oxide, and carbon dioxide were generally maximum for the 0% Na_2O runs (least washed sludge) and minimum for the 16% Na_2O runs (most washed sludge).
- The least washed sludge had the highest hydrogen generation. The runs with the highest washing levels (16% Na_2O in frit) had very low hydrogen generation and no detectable formate destruction. The highest hydrogen generation occurred in the run with the least washed sludge (0% Na2O in frit) and the lowest highest hydrogen generation occurred in the run with the most washed sludge (16% Na2O in frit). The peak hydrogen concentration was 0.944 volume % in the 0% Na2O in frit run.
- The less washed sludge requires more acid than more washed sludge, leading to longer processing times. The acid addition time varied from 2.2 hours for the most washed sludge to 7.2 hours for the least washed sludge.
- The lowest pH SRAT product was the 8% Na_2O in frit run (SB3 baseline) at 5.7. The lowest sodium and higher sodium runs both had a SRAT product final pH of >6. The 12% Na_2O in frit run had a lower pH SME product at 5.8 and 6.2 respectively for runs 1 and 2. The 4% Na_2O in frit run had a higher pH SME product at 6.1 and 6.7 respectively for runs 1 and 2.
- The chemical processing in the 22-L and 4-L rigs are similar, based on the data that was collected at both scales.

3.5 Recommendations

The following recommendations result from this testing.

1. Evaluate targeting the insoluble solids of the melter feed, not the total solids as insoluble solids control the resulting slurry rheology.
2. Additional control of the experiments can be accomplished by improving the consistency of insulating the vessels. Insulation of the vessels with a vacuum wrap or other reproducible insulating material should be considered in future experiments.

4.0 REFERENCES

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5.0 ATTACHMENTS

5.1 Attachment 1: Sludge Composition Targets
5.2 Attachment 2: Run Preparation: Acid Calculations
5.3 Attachment 3: Diagram and Pictures of the Apparatus (4-L and 22-L)
5.4 Attachment 4: Run Execution: Processing Steps for 22-L Baseline SOA Run
5.5 Attachment 5. Run Data: pH and Dewater Profiles
5.6 Attachment 6. Run Data: Offgas Composition Charts
5.7 Attachment 7: Sample Results
5.8 Attachment 8: Rheology Results

5.1 Attachment 1 -- Sludge and Frit Composition Targets

Table 1- 1 SB2/3 Sludges for SOA Tests

Add Mat'ls below to Partly Trim:	0% Na_2O Target (g)	4% Na_2O Target (g)	8% Na_2O Target (g)	12% Na_2O Target (g)	16% Na_2O Target (g)
CETL SB2 Simulant	2102.8	2152.7	2209.8	2275.2	2349.2
CETL SB3 Simulant	3154.2	3229.0	3314.7	3412.8	3523.8
Cr_2O_3	2.28	2.34	2.40	2.47	2.55
$Gd(NO_3)_3$•$6H_2O$	1.88	1.92	1.97	2.03	2.10
$Mg(OH)_2$	56.74	58.09	59.63	61.39	63.39
Na_3PO_4	3.09	3.16	3.24	3.34	3.45
Na_2CO_3	44.67	35.64	25.29	13.45	0.05
NaF	4.06	4.16	4.27	4.39	4.53
NaOH	117.09	93.40	66.29	35.25	0.13
$NaNO_3$	181.3	144.6	102.65	54.58	0.20
$NaNO_2$	286.9	228.8	162.4	86.36	0.31
Na_2SO_4	13.67	13.99	14.36	14.79	15.27
$PbSO_4$	0.87	0.89	0.92	0.94	0.97
Rinse Water	30.55	31.27	32.10	33.05	34.13
Total	**6000.0**	**6000.0**	**6000.0**	**6000.0**	**6000.0**

Table 1- 2 SB2/3 Frits for SOA Tests

Targeted Frit Compositions, wt %					
	0%	4%	Baseline	12%	16%
B_2O_3	8.69	8.35	8.00	7.66	7.32
Li_2O	8.69	8.35	8.00	7.66	7.32
Na_2O	0.05	4.02	8.00	11.95	15.80
SiO_2	82.57	79.29	76.00	72.73	69.55
Waste Loading	40.17	37.69	35.00	32.08	28.97

5.2 Attachment 2 -- Run Preparation: Acid Calculations

Table 2- 1 4L Baseline Run Acid Calculations

Fresh Sludge Analysis	**SOA 0% Na_2O Frit 4L SRAT Run**	**SOA 4% Na_2O Frit 4L SRAT Run**	**SOA 8% Na_2O Frit 4L SRAT Run**	**SOA 12% Na_2O Frit 4L SRAT Run**	**SOA 16% Na_2O Frit 4L SRAT Run**	**Units**
Mass of Fresh Sludge without trim chemicals	2801	2762	2655.4	2671.6	2553	gms
Weight % Total Solids	25.5	24.4	22.7	20.6	16.3	wt%
Weight % Insoluble Solids	15.350	15.350	15.350	15.350	15.350	wt%
Density	1.213	1.196	1.150	1.157	1.106	kg / L
Nitrite	31800	25150	18550	10700	766	mg/Kg
Nitrate	23000	18500	14550	8680	2155	mg/Kg
Manganese (% of Total solids)	2.460	2.740	3.023	3.408	3.925	wt %
TIC (Carbonate) Analysis	1476	1476	1476	1476	1476	microgram/ml
TIC (Carbonate) Analysis converted to mg/Kg	1217	1234	1284	1307	1335	mg/Kg
TIC (Carbonate) ALL CALCS BELOW BASED ON THIS ENTRY	1650	1500	1250	1126	901	mg/Kg
Hydroxide (Base Equivalents) pH = 7.0	0.822	0.708	0.630	0.371	0.161	Molar
Mercury (% of total solids)	0.0000	0.0000	0.0000	0.0000	0.0000	wt% dry basis
Oxalate	1900	1948	2000	2060	2126	mg/Kg
Formate	0	0	0	0	0	mg/Kg
Coal	0	0	0	0	0	wt% dry basis
Sand	0	0	0	0	0	wt% dry basis
Supernate manganese	0	0	0	0	0	mg/L supernate
Supernate density	1.0635	1.0635	1.0635	1.0635	1.06	gm/ml
Fresh Sludge Calcine Factor (1100C), gm oxide/gm dry solids	0.706	0.717	0.718	0.752	0.791	gm/gm
Fresh feed nitrite	1.936	1.510	1.071	0.621	0.043	moles
Fresh feed Mn minus soluble Mn	0.320	0.336	0.332	0.341	0.297	moles
Fresh feed carbonate	0.385	0.345	0.276	0.250	0.192	moles
Fresh feed hydroxide	1.899	1.634	1.456	0.857	0.372	moles
Fresh feed mercury	0.000	0.000	0.000	0.000	0.000	moles
Fresh feed oxalate	0.060	0.061	0.060	0.063	0.062	moles
Fresh feed grams of calcined oxides	504.264	483.350	432.830	414.098	329.337	gm
Trim Chemicals						
Target Wt% Coal dry basis	0.067	0.068	0.070	0.072	0.074	total wt% dry basis
Mass of Coal to add (CF =.08)	0.48	0.49	0.50	0.52	0.53	gms
Calculated wt% coal after trim additions	0.067	0.072	0.083	0.093	0.127	wt%
Target wt% sand dry basis	0.284	0.290	0.298	0.307	0.317	total wt% dry

Fresh Sludge Analysis	**SOA 0% Na_2O Frit 4L SRAT Run**	**SOA 4% Na_2O Frit 4L SRAT Run**	**SOA 8% Na_2O Frit 4L SRAT Run**	**SOA 12% Na_2O Frit 4L SRAT Run**	**SOA 16% Na_2O Frit 4L SRAT Run**	**Units** basis
Mass of sand to add (CF=1.0)	2.04	2.08	2.14	2.20	2.27	gms
Calculated wt% sand after trim additions	0.284	0.307	0.353	0.398	0.541	wt%
Target sodium oxalate per gm total solids	0.000	0.000	0.000	0.000	0.000	total wt% dry basis
Sodium Oxalate to add (CF=0.463)	0.00	0.00	0.00	0.00	0.00	gms
Calculated oxalate conc. After trim additions	0.7416	0.7943	0.8759	0.9934	1.2925	total wt% dry basis
Target Ag	0.0109	0.0112	0.011500	0.0118	0.0122	total wt% dry basis
AgNO3 to add (CF=0.682)	0.12371	0.12657	0.12995	0.13385	0.13814	gms
Target Gd	0.000	0.000	0.000	0.000	0.000	total wt% dry basis
Gd(NO3)3*6H2O to add (CF=0.401)	0.000	0.000	0.000	0.000	0.000	gms
Target wt% Hg dry basis	0.000	0.000	0.000	0.000	0.000	total wt% dry basis
HgO to add	0.000	0.000	0.000	0.000	0.000	gms
Calculated total wt% Hg dry basis	0.0000	0.0000	0.0000	0.0000	0.0000	wt% dry basis
Target Pd metal content	0.0013	0.0014	0.0014	0.0014	0.0015	total wt% dry basis
Wt % Pd in reagent solution	15.2700	15.2700	15.2700	15.2700	15.2700	wt% in solution
Pd(NO3)2*H2O to add (CF=1.150 gm metal oxide/gm metal)	0.063	0.064	0.066	0.068	0.070	gms of solution
Target Rh metal content	0.0072	0.0074	0.0076	0.0078	0.0081	total wt% dry basis
Wt% Rh in reagent solution	4.93	4.93	4.93	4.93	4.93	wt% in solution
Rh(NO3)3*2H2O (CF=1.311gm metal oxide/gm metal)	1.053	1.078	1.106	1.140	1.176	gms of solution
Target Ru	0.0343	0.0351	0.0360	0.0371	0.0383	total wt% dry basis
Wt% Ru RuCl3 reagent solids	41.74	41.74	41.74	41.74	41.74	wt% in solids
RuCl3 to Add (CF=1.0)	0.589	0.603	0.619	0.638	0.658	gms solid
Trim Chemical calcine oxides	2.83	2.89	2.97	3.06	3.16	gms
Total Calcine solids	507.1	486.2	435.8	417.2	332.5	gms
Total solids before trim addition	714.3	673.9	602.8	550.3	416.1	gms
Total solids after trim addition	717.65	677.40	606.34	554.02	419.93	gms

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Total mass of trim chemicals added	4.34	4.44	4.56	4.70	4.85	gms
Water to dilute trim chemicals	40.00	40.00	40.00	40.00	40.00	gms
DWPF sludge transfer flush water (ASSUMED ALREADY IN SLUDGE)						
Trim chemical water	40.00	40.00	40.00	40.00	40.00	gms
Mass of trimmed sludge	2845.34	2806.44	2699.96	2716.30	2597.85	gms
Calculated wt% Total solids in trimmed sludge	25.22	24.14	22.46	20.40	16.16	wt%
Sample Mass of Trimmed sludge	0.00	0.00	0.00	0.00	0.00	gms
Mass of trimmed sludge reacted	2845.34	2806.44	2699.96	2716.30	2597.85	gms
Sample Removal Ratio at start of SRAT	1.0000	1.0000	1.0000	1.0000	1.0000	
Calcine solids at start of SRAT	507.09	486.24	435.80	417.16	332.49	gms
Assumed Parameters						
Percent of TRIM sodium oxalate which generates Base Equivalents	0.00	0.00	0.00	0.00	0.00	moles OH- /100 moles Oxalate
Conversion of Nitrite to Nitrate	30.00	30.00	30.00	30.00	30.00	moles NO3- /100 moles NO2-
Destruction of Nitrite including SME cycle	100.00	100.00	100.00	100.00	100.00	%
Destruction of Formic acid charged	13.00	13.00	13.00	13.00	13.00	%
Destruction of oxalate charged	10.00	10.00	10.00	10.00	10.00	%
Destruction of Nitrate charged "NOT USED or DETERMINED"	0.00	0.00	0.00	0.00	0.00	%
Percent Acid in Excess Stoichiometric Ratio	155.00	155.00	155.00	155.00	155.00	%
Nitric Acid Molarity	10.52	10.52	10.52	10.52	10.52	Molar
Formic Acid Molarity	23.71	23.71	23.71	23.71	23.71	Molar
Nitric Acid Density at 20 deg C	1.3128	1.3128	1.3128	1.3128	1.3128	gms/ml
Formic Acid Density at 20 deg C	1.2054	1.2054	1.2054	1.2054	1.2054	gms/ml
Stoichiometric Acid Ratios Used						
Acid Requirement per mole of Nitrite	0.75	0.75	0.75	0.75	0.75	mole H+/ mole NO2-
Acid Requirement per mole of Mn	1.20	1.20	1.20	1.20	1.20	mole H+/ mole Mn
Acid Requirement per mole of Carbonate	2.00	2.00	2.00	2.00	2.00	mole H+/ mole CO3=

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Acid Requirement per mole of Hydroxide	1.00	1.00	1.00	1.00	1.00	mole H+/ mole OH-
Acid Requirement per mole of Hg	1.00	1.00	1.00	1.00	1.00	mole H+/ mole Hg++
Acid Required per mole of Oxalate	0.00	0.00	0.00	0.00	0.00	mole H+/ mole C2O4=
STOICHIOMETRIC ACID CALCULATION						
Fresh feed NO2-	1.4521	1.1324	0.8030	0.4660	0.0319	moles
Fresh feed Mn	0.38379	0.40334	0.39802	0.40974	0.35680	moles
Fresh feed Carbonate	0.7696	0.6899	0.5527	0.5010	0.3831	moles
Fresh feed OH-	1.8987	1.6345	1.4556	0.8570	0.3717	moles
Hg from trim	0.000000	0.000000	0.000000	0.000000	0.000000	moles
Hg from fresh sludge	0.00000	0.00000	0.00000	0.00000	0.00000	moles
Trim oxalate	0.0000	0.0000	0.0000	0.0000	0.0000	moles
Fresh feed oxalate	0.0000	0.0000	0.0000	0.0000	0.0000	moles
Base generated from Trim sodium oxalate	0.0000	0.0000	0.0000	0.0000	0.0000	moles
Total Stoichiometric Acid required	4.5042	3.8602	3.2094	2.2337	1.1435	moles
Percent in Excess Stoichiometric Ratio	155.000	155.000	155.000	155.000	155.000	%
Actual Acid	6.9816	5.9833	4.9746	3.4622	1.7724	moles
Acid required in moles per liter of starting sludge	2.9753	2.5507	2.1188	1.4742	0.7544	
REDOX CALCULATION (SME PRODUCT REDOX PREDICTION)						
Redox Target	0.200	0.200	0.200	0.200	0.200	Fe+2 / Fe
Ratio of Formic Acid to Total Acid	0.9222	0.9106	0.8950	0.8807	0.8148	mole / mole
Formic Acid Amount	6.438	5.448	4.452	3.049	1.444	moles
Nitric Acid Amount	0.544	0.535	0.522	0.413	0.328	moles
Total Manganese in fresh feed	0.320	0.336	0.332	0.341	0.297	moles
Manganese removed with SRAT product sample	0.000	0.000	0.000	0.000	0.000	moles
Total manganese after SME	0.320	0.336	0.332	0.341	0.297	moles
Formate moles with fresh sludge	0.000	0.000	0.000	0.000	0.000	moles
Formate moles added with formic acid	6.438	5.448	4.452	3.049	1.444	moles
Formate moles reacted in SRAT (% of acid Charged)	0.837	0.708	0.579	0.396	0.188	moles
Formate moles removed with SRAT product sample	0.000	0.000	0.000	0.000	0.000	moles

Fresh Sludge Analysis	**SOA 0% Na_2O Frit 4L SRAT Run**	**SOA 4% Na_2O Frit 4L SRAT Run**	**SOA 8% Na_2O Frit 4L SRAT Run**	**SOA 12% Na_2O Frit 4L SRAT Run**	**SOA 16% Na_2O Frit 4L SRAT Run**	**Units**
Formate Moles after SME	5.601	4.740	3.874	2.653	1.256	moles
Frit slurry formate	0.000	0.000	0.000	0.000	0.000	moles
Final SME formate	5.601	4.740	3.874	2.653	1.256	moles
Oxalate in fresh feed	0.060	0.061	0.060	0.063	0.062	moles
Oxalate from trim	0.000	0.000	0.000	0.000	0.000	moles
Oxalate destroyed during reaction	0.006	0.006	0.006	0.006	0.006	moles
Oxalate removed with SRAT product sample	0.000	0.000	0.000	0.000	0.000	moles
Total Oxalate Moles remaining after trimmed,sample,reacted, sampled	0.054	0.055	0.054	0.056	0.055	moles
Carbon from Coal in fresh feed	0.000	0.000	0.000	0.000	0.000	moles
Carbon from Trim coal	0.040	0.041	0.042	0.043	0.044	moles
Carbon removed in SRAT product Sample	0.000	0.000	0.000	0.000	0.000	moles
Total moles of Carbon from coal remaining after trimmed sludge sample,reacted,sampled	0.040	0.041	0.042	0.043	0.044	moles
Nitrate moles from fresh sludge	1.039	0.824	0.623	0.374	0.089	moles
Nitrate moles from nitric acid	0.544	0.535	0.522	0.413	0.328	moles
Nitrate from conversion of nitrite to nitrate	0.581	0.453	0.321	0.186	0.013	moles
Nitrate from minor trim chemicals	0.00242	0.00248	0.00254	0.00262	0.00270	moles
Nitrate destroyed in the reactions	0.00000	0.00000	0.00000	0.00000	0.00000	moles
Nitrate removed with SRAT product sample	0.00000	0.00000	0.00000	0.00000	0.00000	moles
Total Nitrate Moles (Sum of inputs - destroyed)	2.166	1.814	1.469	0.976	0.432	moles
Nitrite Moles remaining after SME cycle	0.0000	0.0000	0.0000	0.0000	0.0000	moles
Assumed SME density	1.450	1.450	1.450	1.450	1.450	gm/ml
Final SME mass	3.2831191	3.2635604	3.0962890	3.0690393	2.5897552	Kg
Manganese Fraction in final melter feed	0.097	0.103	0.107	0.111	0.115	gmol/Kg of SME Slurry
Formate concentration in final SME	1.706	1.452	1.251	0.864	0.485	gmol/Kg of SME Slurry
Oxalate Concentration in final SME	0.017	0.017	0.018	0.018	0.021	gmol/Kg of SME Slurry
Carbon from Coal concentration in final SME	0.012	0.012	0.014	0.014	0.017	gmol/Kg of SME Slurry
Nitrate Concentration in	0.660	0.556	0.474	0.318	0.167	gmol/Kg of

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
final SME						SME Slurry
Nitrite Concentration in final SME	0.000	0.000	0.000	0.000	0.000	gmol/Kg of SME Slurry
Predicted Redox	0.200	0.200	0.201	0.202	0.205	
Formic Acid	271.534	229.791	187.783	128.604	60.907	ml
Nitric Acid	51.663	50.848	49.642	39.257	31.206	ml
BENCH SCALE CALCULATIONS						
Sample Mass of trimmed sludge	0.00	0.00	0.00	0.00	0.00	gms
Mass of SRAT Batch after trim and sample	2845.34	2806.44	2699.96	2716.30	2597.85	gms
Formic Acid Required	271.534	229.791	187.783	128.604	60.907	ml
Nitric Acid Required	51.663	50.848	49.642	39.257	31.206	ml
Bench Scale Operational Setting						
Scaled formic acid feed rate based on nominal 23.551 M	0.7768	0.7766	0.7773	0.7775	0.7778	ml/min
Scaled Nitric Acid Feed Rate based on nominal 10.395 M	0.7728	0.7725	0.7732	0.7735	0.7738	ml/min
Formic Acid Feed Time	349.5	295.9	241.6	165.4	78.3	min
Nitric Acid Feed Time	66.9	65.8	64.2	50.8	40.3	min
Wt% Active Agent In Antifoam Solution	10	10	10	10	10	%
Target Conc. based on Sludge slurry Mass after trim	100	100	100	100	100	ppm
Antifoam charge at 1:10	2.85	2.81	2.70	2.72	2.60	gms
Number of antifoam additions	7	7	7	7	7	
Dewatering Calc for Target Wt. % Total Solids in SRAT Product						
Final SRAT Total Solids (UNDER TOOLS USE SOLVER)	32.77	30.86	28.47	24.75	18.88	%
Water in Starting SRAT Slurry	2127.80	2129.15	2093.73	2162.39	2178.04	gms
Solids in Starting SRAT Slurry	717.54	677.29	606.23	553.91	419.81	gms
Mass 1:20 antifoam added	39.83	39.29	37.80	38.03	36.37	gms
Mass of pure formic acid (HCOOH) added	296.27	250.72	204.89	140.32	66.46	gms
Mass of pure nitric acid (HNO3) added	34.25	33.71	32.91	26.02	20.69	gms
Mass of formic acid	31.04	26.27	21.46	14.70	6.96	gms

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
dilution water added						
Mass of nitric acid dilution water added	33.58	33.05	32.26	25.51	20.28	gms
Solids Lost, and Water Made, during base equiv neutralization	34.21	29.45	26.22	15.44	6.70	gms
Solids Lost in TIC destruction	23.09	20.70	16.59	15.03	11.49	gms
Water Made in TIC destruction	6.93	6.21	4.98	4.51	3.45	gms
Solids Lost in Nitrite Destruction	62.35	48.63	34.48	20.01	1.37	gms
Water Made in Nitrite Destruction	34.88	27.20	19.29	11.19	0.77	gms
Formate Converted to CO2	37.67	31.88	26.05	17.84	8.45	gms
Revised Water Mass in slurry	2306.27	2288.65	2233.86	2269.87	2250.74	gms
Revised Solids Mass in slurry	892.73	833.03	742.57	653.83	480.76	gms
Target Final Water Mass in slurry to hit total solids target	1831.30	1865.98	1865.72	1987.51	2066.10	gms
Total Water to Remove	474.97	422.67	368.14	282.36	184.65	gms
Boiling Time to Remove Water at Scaled Rate	121.63	108.27	94.22	72.24	47.22	min
Mass of carbonate lost as CO2	16.93	15.18	12.16	11.02	8.43	gms
Mass of nitrite lost as NO	40.66	31.71	22.49	13.05	0.89	gms
Formate converted to CO2	37.67	31.88	26.05	17.84	8.45	gms
Final Sludge Mass in SRAT after acid addition and dewater	2704.38	2682.42	2595.04	2631.17	2540.99	gms
Mass of Final SRAT Samples	0.00	0.00	0.00	0.00	0.00	gms
Mass of treated sludge going into SME cycle	2704.38	2682.42	2595.04	2631.17	2540.99	gms
SME sample ratio	1.0000	1.0000	1.0000	1.0000	1.0000	
Calcined Solids going to SME	507.09	486.24	435.80	417.16	332.49	gms
DWPF SCALE TO BENCH SCALE						
DWPF Scale SRAT cycle						
Volume based scale factor 6000 gal starting SRAT	9679.4	9682.4	9674.0	9671.0	9666.9	
SRAT Air purge	230	230	230	230	230	scfm
SRAT Boil-up Rate	5000	5000	5000	5000	5000	lbs/hr
SRAT Total Boil-Up	60,000	60,000	60,000	60,000	60,000	lbs
Indicated SRAT Boiling Time	720	720	720	720	720	min

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Bench Scale SRAT cycle						
99.5% of Scaled Air Purge	669.5	669.3	669.9	670.1	670.4	sccm
Scaled Boil-up Rate	3.91	3.90	3.91	3.91	3.91	gm/min
Water to remove in SRAT	474.97	422.67	368.14	282.36	184.65	gms
Required Boiling Time at above Rate	121.6	108.3	94.2	72.2	47.2	min
Helium Purge rate at 0.5 vol%	3.3	3.3	3.4	3.4	3.4	sccm
DWPF Scale SME cycle						
Water Flush Volume After Frit Slurry Addition	0.0	0.0	0.0	0.0	0.0	gal
SME air purge	74.0	74.0	74.0	74.0	74.0	scfm
SME Boil-up Rate	5000	5000	5000	5000	5000	lbs/hr
SME Total Boil-Up	30,000	30,000	30,000	30,000	30,000	lbs
SME Boiling Time	360	360	360	360	360	min
Bench Scale SME cycle						
SME scale factor (SAME AS SRAT,BUT ADJUSTED FOR SAMPLE)	9679.4	9682.4	9674.0	9671.0	9666.9	
99.5% Scaled SME Air Purge	215.4	215.3	215.5	215.6	215.7	sccm
Helium Purge rate at 0.5 vol%	1.08	1.08	1.08	1.08	1.08	sccm
Solids Remaining at Start of SME	886.3	827.9	738.8	651.3	479.7	gms
SRAT product Calcine Factor (Calculated)	0.568	0.584	0.587	0.638	0.692	gms oxide/gms dry SRAT Product
Sludge calcined solids - based on SRAT product	503.43	483.25	433.59	415.55	331.73	gms
Sludge Oxide Contribution in SME	40.17	37.69	35.00	32.08	28.97	%
Frit Oxide Contribution	59.83	62.31	65.00	67.92	71.03	%
Frit Calcine Factor	1.00	1.00	1.00	1.00	1.00	gms oxide/gms frit
Frit Slurry Density	1.50	1.50	1.50	1.50	1.50	gms/ml
Frit Slurry wt % solids	50.00	50.00	50.00	50.00	50.00	wt%
Frit Slurry Formic Acid Ratio	0.00	0.00	0.00	0.00	0.00	gms 90 wt% FA/100 gms Frit
Frit Solids	755.3	803.9	809.3	883.2	815.2	gms
90 wt% Formic Acid (corrections necessary for other concentrations)	0.00	0.00	0.00	0.00	0.00	gms
Water in Frit Slurry	0.0	0.0	0.0	0.0	0.0	gms
Scaled Transfer Water	0.00	0.00	0.00	0.00	0.00	gms

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Total Frit Slurry Water	0.0	0.0	0.0	0.0	0.0	gms
Total Mass of Frit Slurry	755.3	803.9	809.3	883.2	815.2	gms
SME Frit Addition #1	251.8	268.0	269.8	294.4	271.7	gms
SME 90-wt% Formic Addition #1	0.00	0.00	0.00	0.00	0.00	gms
SME Water Addition #1	0.0	0.0	0.0	0.0	0.0	gms
SME Frit Addition #2	251.8	268.0	269.8	294.4	271.7	gms
SME 90-wt% Formic Addition #2	0.00	0.00	0.00	0.00	0.00	gms
SME Water Addition #2	0.0	0.0	0.0	0.0	0.0	gms
SME Frit Addition #3	251.8	268.0	269.8	294.4	271.7	gms
SME 90-wt% Formic Addition #3	0.0	0.0	0.0	0.0	0.0	gms
SME Water Addition #3	0.0	0.0	0.0	0.0	0.0	gms
SME Water to remove after each frit addition:	0.0	0.0	0.0	0.0	0.0	gms
Scaled Boil-up Rate	3.91	3.90	3.91	3.91	3.91	gms/min
Approximate Time to remove water:	0.0	0.0	0.0	0.0	0.0	min
Final Solids Content in SME	1641.6	1631.8	1548.1	1534.5	1294.9	gms
Target SME Solids total Wt%	50.0	50.0	50.0	50.0	50.0	%
Mass of Water to Boil Off for Final SME Concentration	176.5	222.7	308.1	445.3	766.5	gms
Scaled Boil-up Rate	3.91	3.90	3.91	3.91	3.91	gms/min
Approximate Time to remove water:	45.2	57.1	78.9	113.9	196.0	min
Run Plan Table 1 - Batch Make-up Sheet						
Mass of Fresh Sludge without trim chemicals	2801	2762	2655.4	2671.6	2553	gms
Mass of Coal to add	0.48	0.49	0.50	0.52	0.53	gms
Mass of sand to add	2.04	2.08	2.14	2.20	2.27	gms
Sodium Oxalate to add	0.00	0.00	0.00	0.00	0.00	gms
AgNO3	0.1237	0.1266	0.1300	0.1339	0.1381	gms
Gd(NO3)3*6H2O	0.00	0.00	0.00	0.00	0.00	gms
HgO to add	0.000	0.000	0.000	0.000	0.000	gms
Pd(NO3)2*H2O 15.27% Pd solution	0.0626	0.0641	0.0658	0.0678	0.0699	gms
Rh(NO3)3*2H2O 4.93% Rh solution	1.0532	1.0776	1.1063	1.1395	1.1760	gms
RuCl3	0.5892	0.6029	0.6190	0.6375	0.6580	gms solid
DI Water to dilute trim chemicals	40.00	40.00	40.00	40.00	40.00	gms
Sample Mass of Trimmed sludge	0.00	0.00	0.00	0.00	0.00	gms

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Run Plan Table #2 SRAT Cycle Operating Parameters						
SRAT CYCLE						
SRAT Scaled Air Purge (99.5% of prototypical)	669.5	669.3	669.9	670.1	670.4	sccm
SRAT Helium Purge (0.5% of prototypical air purge)	3.3	3.3	3.4	3.4	3.4	sccm
Target Antifoam Conc. based on sludge slurry mass after trim	100	100	100	100	100	ppm
747 Antifoam charge at 1:10	2.85	2.81	2.70	2.72	2.60	gms
Nitric Acid Molarity	10.52	10.52	10.52	10.52	10.52	Molar
Nitric Acid Required	51.663	50.848	49.642	39.257	31.206	ml
Scaled Nitric Acid Feed Rate based on nominal 10.395M, 2 gpm	0.7728	0.7725	0.7732	0.7735	0.7738	ml/min
Nitric Acid Feed Time	66.9	65.8	64.2	50.8	40.3	min
Formic Acid Molarity	23.71	23.71	23.71	23.71	23.71	Molar
Formic Acid Required	271.534	229.791	187.783	128.604	60.907	ml
Scaled formic acid feed rate based on nominal 23.551M, 2 gpm	0.7768	0.7766	0.7773	0.7775	0.7778	ml/min
Formic Acid Feed Time	349.5	295.9	241.6	165.4	78.3	min
SRAT Scaled Boil-up Rate	3.91	3.90	3.91	3.91	3.91	gm/min
SRAT Dewater Mass	475.0	422.7	368.1	282.4	184.6	gms
SRAT Dewater Time	121.63	108.27	94.22	72.24	47.22	min
Mass of SRAT Product Samples	0.00	0.00	0.00	0.00	0.00	gms
Run Plan Table #3 SME Cycle Operating Parameters						
SME CYCLE						
SME Frit Addition #1	251.8	268.0	269.8	294.4	271.7	gms
Frit Addition #1 Water	0.0	0.0	0.0	0.0	0.0	gms
Frit Addition #1 Formic Acid	0.00	0.00	0.00	0.00	0.00	gms
SME Frit Addition #2	251.8	268.0	269.8	294.4	271.7	gms
Frit Addition #2 Water	0.0	0.0	0.0	0.0	0.0	gms
Frit Addition #2 Formic Acid	0.00	0.00	0.00	0.00	0.00	gms
SME Frit Addition #3	251.8	268.0	269.8	294.4	271.7	gms
Frit Addition #3 Water	0.0	0.0	0.0	0.0	0.0	gms

Fresh Sludge Analysis	SOA 0% Na_2O Frit 4L SRAT Run	SOA 4% Na_2O Frit 4L SRAT Run	SOA 8% Na_2O Frit 4L SRAT Run	SOA 12% Na_2O Frit 4L SRAT Run	SOA 16% Na_2O Frit 4L SRAT Run	Units
Frit Addition #3 Formic Acid	0.00	0.00	0.00	0.00	0.00	gms
Frit Addition Dewater (Times 3)	0.0	0.0	0.0	0.0	0.0	gms
Final Dewater mass	176.5	222.7	308.1	445.3	766.5	gms
SME Air Purge	0.215	0.215	0.216	0.216	0.216	slm
SME He Purge	1.08	1.08	1.08	1.08	1.08	sccm

Table 2- 2 22L Acid Calculations

Fresh Sludge Analysis	SOA 4% Na_2O Frit 22L SRAT/SME Run	SOA 12% Na_2O Frit 22L SRAT/SME Run	
Mass of Fresh Sludge without trim chemicals	16800.000	16240.000	gms
Weight % Total Solids	24.400	21.340	wt%
Weight % Insoluble Solids	15	0	wt%
Density	1	1	kg / L
Nitrite	26050	10650	mg/Kg
Nitrate	18350	9435	mg/Kg
Manganese (% of Total solids)	2.758	3.420	wt %
TIC (Carbonate) Analysis	1476	0	microgram/ml
TIC (Carbonate) Analysis converted to mg/Kg	1230.	0.000	mg/Kg
TIC (Carbonate) ALL CALCS BELOW BASED ON THIS ENTRY	1500	1126.	mg/Kg
Hydroxide (Base Equivalents) pH = 7	0.708	0.371	Molar
Mercury (% of total solids)	0	0	wt% dry basis
Oxalate	1948	2060	mg/Kg
Formate	0	0	mg/Kg
Coal	0	0	wt% dry basis
Sand	0	0	wt% dry basis
Supernate manganese	0.000	0.000	mg/L supernate
Supernate density	1.0635	1.0635	gm/ml
Fresh Sludge Calcine Factor (1100C), gm oxide/gm dry solids	0.722	0.755	gm/gm
Fresh feed nitrite	9.513	3.759	moles
Fresh feed Mn minus soluble Mn	2.058	2.158	moles
Fresh feed carbonate	2.098	1.523	moles
Fresh feed hydroxide	9.912	5.194	moles
Fresh feed mercury	0.000	0.000	moles
Fresh feed oxalate	0.371806408	0.380077255	moles
Fresh feed grams of calcined oxides	2959.62	2616.54	gm
Trim Chemicals			
Target Wt% Coal dry basis	0.068	0.072	total wt% dry basis
Mass of Coal to add (CF =.08)	2.808	2.512	gms
Calculated wt% coal after trim additions	0.07	0.07	wt%
Target wt% sand dry basis	0.290	0.307	total wt% dry basis
Mass of sand to add (CF=1.0)	11.956	10.692	gms
Calculated wt% sand after trim additions	0.29	0.31	wt%
Target sodium oxalate per gm total solids	0.0000	0.0000	total wt% dry basis
Sodium Oxalate to add (CF=0.463)	0.0000	0.0000	gms
Calculated oxalate conc. After trim additions	0.79450	0.96038	total wt% dry basis
Target Ag	0.011	0.012	total wt% dry basis

Fresh Sludge Analysis	**SOA 4% Na_2O Frit 22L SRAT/SME Run**	**SOA 12% Na_2O Frit 22L SRAT/SME Run**	
AgNO3 to add (CF=0.682)	0.726	0.650	gms
Target Gd	0.000	0.000	total wt% dry basis
Gd(NO3)3*6H2O to add (CF=0.401)	0.000	0.000	gms
Target wt% Hg dry basis	0.0000	0.0000	total wt% dry basis
HgO to add	0.0000	0.0000	gms
Calculated total wt% Hg dry basis	0.0000	0.0000	wt% dry basis
Target Pd metal content	0.001	0.001	total wt% dry basis
Wt % Pd in reagent solution	15.2700	15.2700	wt% in solution
Pd(NO3)2*H2O to add (CF=1.150 gm metal oxide/gm metal)	0.37	0.33	gms of solution
Target Rh metal content	0.007	0.008	total wt% dry basis
Wt% Rh in reagent solution	4.9300	4.9300	wt% in solution
Rh(NO3)3*2H2O (CF=1.311gm metal oxide/gm metal)	6.18	5.53	gms of solution
Target Ru	0.035	0.037	total wt% dry basis
Wt% Ru RuCl3 reagent solids	41.74	41.74	wt% in solids
RuCl3 to Add (CF=1.0)	3.46	3.09	gms solid
Trim Chemical calcine oxides	16.60064075	14.84587714	gms
Total Calcine solids	2976.2	2631.4	gms
Total solids before trim addition	4099.20	3465.62	gms
Total solids after trim addition	4119.13	3483.44	gms
Total mass of trim chemicals added	25.50	22.81	gms
Water to dilute trim chemicals	200.00	200.00	gms
DWPF sludge transfer flush water (ASSUMED ALREADY IN SLUDGE)			
Trim chemical water	200.00	200.00	gms
Mass of trimmed sludge	17025.50	16462.81	gms
Calculated wt% Total solids in trimmed sludge	24.19	21.16	wt%
Sample Mass of Trimmed sludge	0.0000	0.0000	gms
Mass of trimmed sludge reacted	17025.5038	16462.8079	gms
Sample Removal Ratio at start of SRAT	1.00	1.00	
Calcine solids at start of SRAT	2976.223041	2631.385957	gms
Assumed Parameters			
Percent of TRIM sodium oxalate which generates Base Equivalents	0.00	0.00	moles OH-/100 moles Oxalate
Conversion of Nitrite to Nitrate	30.00	30.00	moles NO3-/100 moles NO2-
Destruction of Nitrite including SME cycle	100.00	100.00	%
Destruction of Formic acid charged	13.00	13.00	%
Destruction of oxalate charged	10.00	10.00	%
Destruction of Nitrate charged "NOT USED or DETERMINED"	0.00	0.00	%
Percent Acid in Excess Stoichiometric Ratio	155.00	155.00	%
Nitric Acid Molarity	10.4457	10.4457	Molar

Fresh Sludge Analysis	**SOA 4% Na_2O Frit 22L SRAT/SME Run**	**SOA 12% Na_2O Frit 22L SRAT/SME Run**	
Formic Acid Molarity	23.7100	23.7100	Molar
Nitric Acid Density at 20 deg C	1.311	1.311	gms/ml
Formic Acid Density at 20 deg C	1.20539096	1.20539096	gms/ml
Stoichiometric Acid Ratios Used			
Acid Requirement per mole of Nitrite	0.75	0.75	mole H+/ mole NO2-
Acid Requirement per mole of Mn	1.20	1.20	mole H+/ mole Mn
Acid Requirement per mole of Carbonate	2.00	2.00	mole H+/ mole CO3=
Acid Requirement per mole of Hydroxide	1.00	1.00	mole H+/ mole OH-
Acid Requirement per mole of Hg	1	1	mole H+/ mole Hg++
Acid Required per mole of Oxalate	0	0	mole H+/ mole C2O4=
STOICHIOMETRIC ACID CALCULATION			
Fresh feed NO2-	7.1345	2.8196	moles
Fresh feed Mn	2.4695	2.5890	moles
Fresh feed Carbonate	4.196503	3.045169	moles
Fresh feed OH-	9.91200	5.19400	moles
Hg from trim	0.0000	0.0000	moles
Hg from fresh sludge	0.0000	0.0000	moles
Trim oxalate	0.0000	0.0000	moles
Fresh feed oxalate	0.0000	0.0000	moles
Base generated from Trim sodium oxalate	0.0000	0.0000	moles
spare			moles
spare			moles
Total Stoichiometric Acid required	23.713	13.648	moles
Percent in Excess Stoichiometric Ratio	155.0000	155.0000	%
Actual Acid	36.75437755	21.15400883	moles
Acid required in moles per liter of starting sludge	2.5905	1.4906	
REDOX CALCULATION (SME PRODUCT REDOX PREDICTION)			
Redox Target	0.2	0.2	Fe+2 / Fe
Ratio of Formic Acid to Total Acid	0.899	0.865	mole / mole
Formic Acid Amount	33.025	18.295	moles
Nitric Acid Amount	3.730	2.859	moles
Total Manganese in fresh feed	2.058	2.158	moles
Manganese removed with SRAT product sample	0.000	0.000	moles

Fresh Sludge Analysis	SOA 4% Na_2O Frit 22L SRAT/SME Run	SOA 12% Na_2O Frit 22L SRAT/SME Run	
Total manganese after SME	2.058	2.158	moles
Formate moles with fresh sludge	0.000	0.000	moles
Formate moles added with formic acid	33.025	18.295	moles
Formate moles reacted in SRAT (% of acid Charged)	4.293	2.378	moles
Formate moles removed with SRAT product sample	0.000	0.000	moles
Formate Moles after SME	28.732	15.917	moles
Frit slurry formate	1.452	1.644	moles
Final SME formate	30.183	17.560	moles
Oxalate in fresh feed	0.372	0.380	moles
Oxalate from trim	0.000	0.000	moles
Oxalate destroyed during reaction	0.037	0.038	moles
Oxalate removed with SRAT product sample	0.000	0.000	moles
Total Oxalate Moles remaining after trimmed,sample,reacted, sampled	0.335	0.342	moles
Carbon from Coal in fresh feed	0.000	0.000	moles
Carbon from Trim coal	0.234	0.209	moles
Carbon removed in SRAT product Sample	0.000	0.000	moles
Total moles of Carbon from coal remaining after trimmed sludge sample,reacted,sampled	0.234	0.209	moles
Nitrate moles from fresh sludge	4.972	2.471	moles
Nitrate moles from nitric acid	3.72955	2.85909	moles
Nitrate from conversion of nitrite to nitrate	2.85380	1.12783	moles
Nitrate from minor trim chemicals	0.01422	0.01272	moles
Nitrate destroyed in the reactions	0.000	0.000	moles
Nitrate removed with SRAT product sample	0.000	0.000	moles
Total Nitrate Moles (Sum of inputs - destroyed)	11.5694	6.4708	moles
Nitrite Moles remaining after SME cycle	0.000	0.000	moles
Assumed SME density	1.450	1.450	gm/ml
Final SME mass	20.072	19.468	Kg
Manganese Fraction in final melter feed	0.103	0.111	gmol/Kg of SME Slurry
Formate concentration in final SME	1.504	0.902	gmol/Kg of SME Slurry
Oxalate Concentration in final SME	0.017	0.018	gmol/Kg of SME Slurry
Carbon from Coal concentration in final SME	0.012	0.011	gmol/Kg of SME Slurry
Nitrate Concentration in final SME	0.576	0.332	gmol/Kg of SME Slurry
Nitrite Concentration in final SME	0.000	0.000	gmol/Kg of SME Slurry

Fresh Sludge Analysis	SOA 4% Na_2O Frit 22L SRAT/SME Run	SOA 12% Na_2O Frit 22L SRAT/SME Run	
Predicted Redox	0.200	0.200	
Formic Acid	1392.86488	771.6120078	ml
Nitric Acid	357.0430268	273.7105368	ml
BENCH SCALE CALCULATIONS			
Sample Mass of trimmed sludge	0.000	0.000	gms
Mass of SRAT Batch after trim and sample	17025.504	16462.808	gms
Formic Acid Required	1392.86488	771.6120078	ml
Nitric Acid Required	357.0430268	273.7105368	ml
Bench Scale Operational Setting			
Scaled formic acid feed rate based on nominal 23.551 M	4.7	4.7	ml/min
Scaled Nitric Acid Feed Rate based on nominal 10.395 M	4.7	4.7	ml/min
Formic Acid Feed Time	296.538277	164.2266194	min
Nitric Acid Feed Time	75.87202173	58.14674674	min
Wt% Active Agent In Antifoam Solution	10.00	10.00	%
Target Conc. based on Sludge slurry Mass after trim	100	100	ppm
Antifoam charge at 1:10	17	16	gms
Number of antifoam additions	7	7	
Dewatering Calc for Target Wt. % Total Solids in SRAT Product			
Final SRAT Total Solids (UNDER TOOLS USE SOLVER)	30.97	25.58	%
Water in Starting SRAT Slurry	12906.98	12979.92	gms
Solids in Starting SRAT Slurry	4118.52	3482.89	gms
Mass 1:20 antifoam added	238.36	230.48	gms
Mass of pure formic acid (HCOOH) added	1519.74	841.90	gms
Mass of pure nitric acid (HNO3) added	235.01	180.16	gms
Mass of formic acid dilution water added	159.21	88.20	gms
Mass of nitric acid dilution water added	233.07	178.67	gms
Solids Lost, and Water Made, during base equiv neutralization	178.57	93.58	gms
Solids Lost in TIC destruction	125.92	91.37	gms
Water Made in TIC destruction	37.80	27.43	gms
Solids Lost in Nitrite Destruction	306.35	121.07	gms
Water Made in Nitrite Destruction	171.37	67.72	gms
Formate Converted to CO2	193.24	107.05	gms
Revised Water Mass in slurry	13913.45	13654.47	gms
Revised Solids Mass in slurry	5081.11	4103.41	gms
Target Final Water Mass in slurry to hit total solids target	11328.06	11935.06	gms
Total Water to Remove	2585.39	1719.41	gms
Boiling Time to Remove Water at Scaled Rate	109.49	72.80	min

Fresh Sludge Analysis	SOA 4% Na_2O Frit 22L SRAT/SME Run	SOA 12% Na_2O Frit 22L SRAT/SME Run	
Mass of carbonate lost as CO2	92.34	67.00	gms
Mass of nitrite lost as NO	199.79	78.96	gms
Formate converted to CO2	193.24	107.05	gms
Final Sludge Mass in SRAT after acid addition and dewater	16306.08	15976.87	gms
Mass of Final SRAT Samples	0.0000	0.0000	gms
Mass of treated sludge going into SME cycle	16306.08	15976.87	gms
SME sample ratio	1	1	
Calcined Solids going to SME	2976.223041	2631.385957	gms
DWPF SCALE TO BENCH SCALE			
DWPF Scale SRAT cycle			
Volume based scale factor 6000 gal starting SRAT	1600.830867	1600.362084	
SRAT Air purge	230	230	scfm
SRAT Boil-up Rate	5000	5000	lbs/hr
SRAT Total Boil-Up	60000	60000	lbs
Indicated SRAT Boiling Time	720	720	min
Bench Scale SRAT cycle			
99.5% of Scaled Air Purge	4048.09	4049.28	sccm
Scaled Boil-up Rate	23.61	23.62	gm/min
Water to remove in SRAT	2585.4	1719.4	gms
Required Boiling Time at above Rate	109.5	72.8	min
Helium Purge rate at 0.5 vol%	20.2505844	20.25651627	sccm
DWPF Scale SME cycle			
Water Flush Volume After Frit Slurry Addition	0	0	gal
SME air purge	74	74	scfm
SME Boil-up Rate	5000	5000	lbs/hr
SME Total Boil-Up	30000.0	30000.0	lbs
SME Boiling Time	360.0	360.0	min
Bench Scale SME cycle			
SME scale factor (SAME AS SRAT,BUT ADJUSTED FOR SAMPLE)	1600.83	1600.36	
99.5% Scaled SME Air Purge	1302.4	1302.8	sccm
Helium Purge rate at 0.5 vol%	6.5	6.5	sccm
Solids Remaining at Start of SME	5049.19	4087.65	gms
SRAT product Calcine Factor (Calculated)	0.59	0.64	gms oxide/gms dry SRAT Product
Sludge calcined solids - based on SRAT product	2957.53	2621.28	gms

Fresh Sludge Analysis	SOA 4% Na_2O Frit 22L SRAT/SME Run	SOA 12% Na_2O Frit 22L SRAT/SME Run	
Sludge Oxide Contribution in SME	37.69	32.08	%
Frit Oxide Contribution	62.31	67.92	%
Frit Calcine Factor	1.00	1.00	gms oxide/gms frit
Frit Slurry Density	1.50	1.50	gms/ml
Frit Slurry wt % solids	50	50	wt%
Frit Slurry Formic Acid Ratio	1.50	1.50	gms 90 wt% FA/100 gms Frit
Frit Solids	4920.4	5571.2	gms
90 wt% Formic Acid (corrections necessary for other concentrations)	73.81	83.57	gms
Water in Frit Slurry	4846.6	5487.6	gms
Scaled Transfer Water	0.0	0.0	gms
Total Frit Slurry Water	4846.556933	5487.620891	gms
Total Mass of Frit Slurry	9840.7	11142.4	gms
SME Frit Addition #1	1640.1	1857.1	gms
SME 90-wt% Formic Addition #1	24.60181184	27.85594361	gms
SME Water Addition #1	1615.5	1829.2	gms
SME Frit Addition #2	1640.1	1857.1	gms
SME 90-wt% Formic Addition #2	24.6	27.9	gms
SME Water Addition #2	1615.5	1829.2	gms
SME Frit Addition #3	1640.1	1857.1	gms
SME 90-wt% Formic Addition #3	24.60181184	27.85594361	gms
SME Water Addition #3	1615.5	1829.2	gms
SME Water to remove after each frit addition:	1640.1	1857.1	gms
Scaled Boil-up Rate	23.61221753	23.61913409	gms/min
Approximate Time to remove water:	69.5	78.6	min
Final Solids Content in SME	10036.0	9734.0	gms
Target SME Solids total Wt%	50.00	50.00	%
Mass of Water to Boil Off for Final SME Concentration	1154.5	2080.0	gms
Scaled Boil-up Rate	23.61221753	23.61913409	gms/min
Approximate Time to remove water:	48.89383568	88.06246384	min

5.3 Attachment 3 -- Diagram and Pictures of the Apparatus (4-L and 22-L)

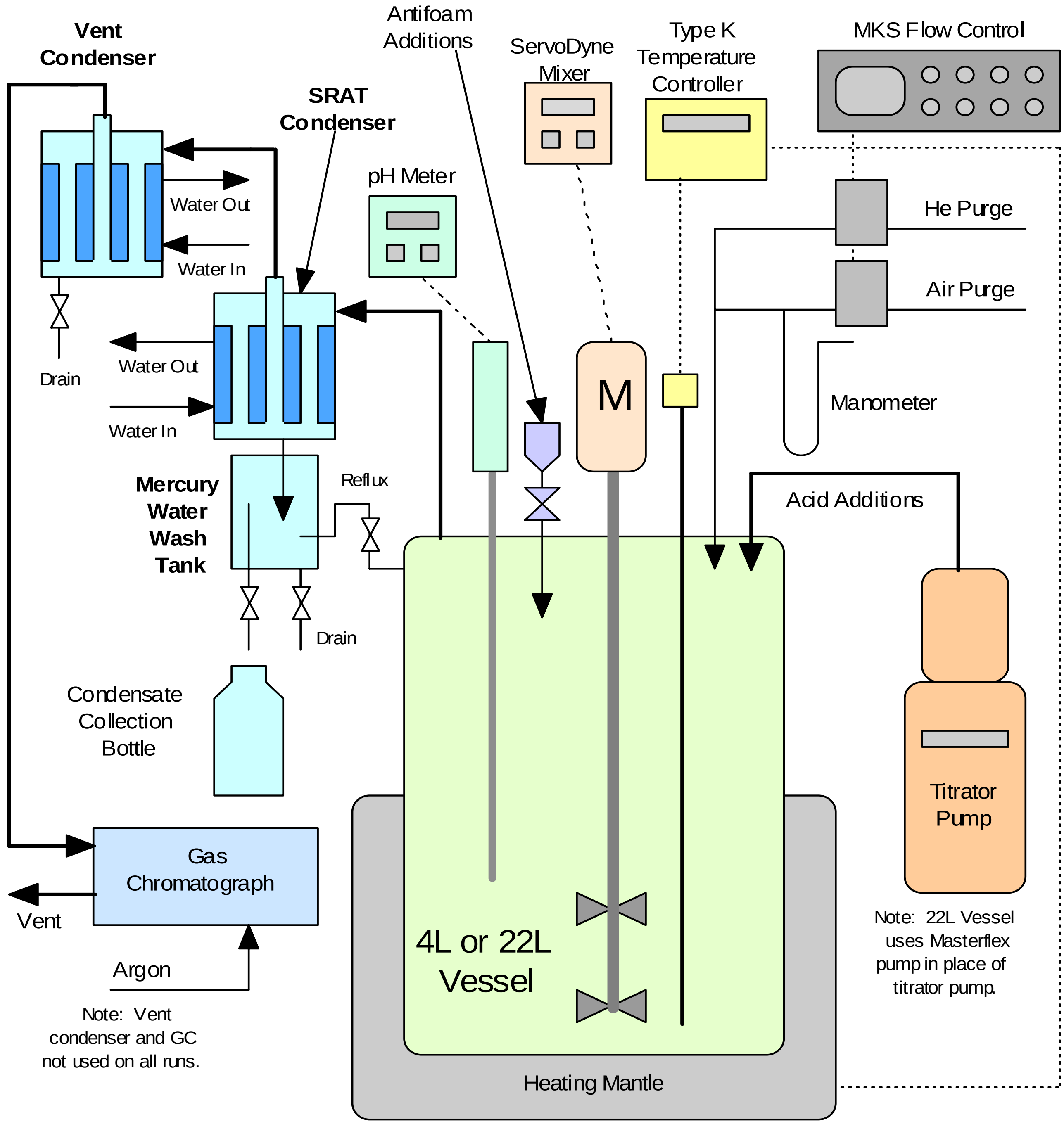


Figure 3-1– Sketch of 4L and 22L SRAT/SME Apparatus

Figure 3-2 – Photograph of 22-L Experimental Apparatus

Figure 3-3– Unidentified Organic Accumulation in Condensate Return Elbow between SRAT Condenser and MWWT

Figure 3-4–Formic Acid Vent Condenser and MWWT

Figure 3-5–SRAT Condenser, stirrer and MWWT

Figure 3-6– 22L kettle and Kettle Head with penetrations for pH probe, antifoam, sampling , thermocouple, agitator, offgas, etc.

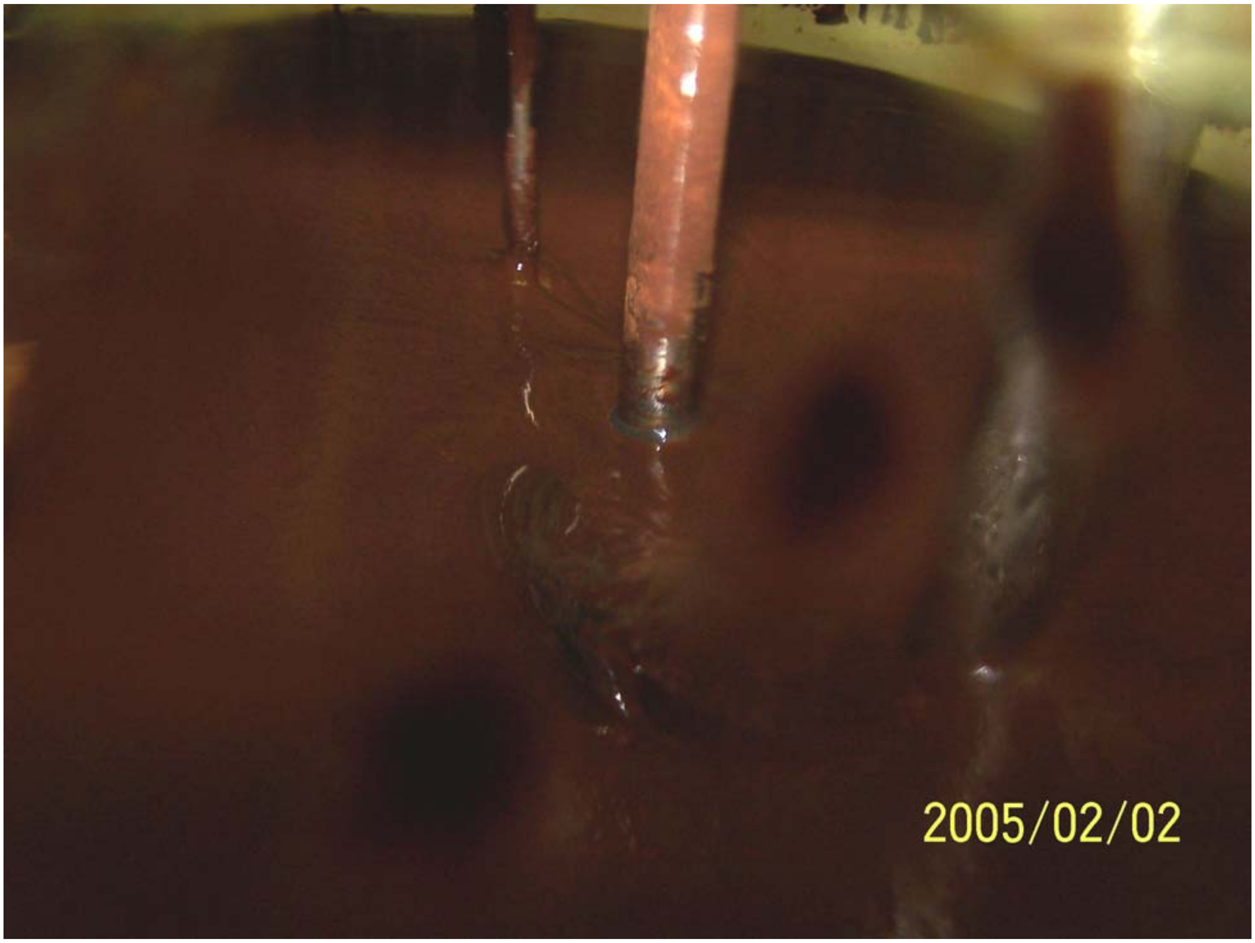


Figure 3-7– 22L Mixing Slurry from 12% Na_2O – No Vortex

Figure 3-8– 22L Mixing Slurry from 4% Na2O – Large Vortex

5.4 Attachment 4: Run Execution: Processing Steps for 22-L Baseline SOA Run

Four 22-L runs were completed, two with 4% sludge and two with 12% sludge. The Run details are summarized below:

4% Run Details

The following steps will be used to complete the run:

1. The equipment configuration (Figure 1) used for bench-scale 22-L vessel testing should be used for this run. Components include the 22-L kettle, heating mantle, variable speed mixer and controller, FAVC, MWWT, SRAT condenser, pH meter and probe, temperature controller and thermocouple, manometer, water chillers (10°C and 40°C), MKS flow controllers, and Masterflex pump for acid addition. However, no GC will be used for this run.
2. Record all M&TE utilized in the appropriate usage log and on the run preparation datasheet.
3. Ensure that the equipment is leak-checked and that the final outlet flow is within 90% of the inlet flow. Final leak check flowrates should be recorded on the run preparation datasheet.
4. Fill the MWWT with DI water to the overflow back to the SRAT. Do not let the water return to the SRAT. Record the mass added to the MWWT on the run preparation datasheet
5. Once the vessel and the equipment are set-up, the sludge and trim chemicals should be added per the batch make-up sheet, Table 1. Record the actual amounts added and scales utilized in the run preparation datasheet.
6. Agitate for 20 minutes. There is **no SRAT Receipt sample** this run.
7. Check the calibration of the pH probe to be used at pH 4, 10 and 7 and record readings on run preparation datasheet. Record the initial pH of the sludge once all trim chemicals have been added on the run preparation datasheet.
8. Insulate the entire SRAT vessel, allowing a small window for observing the slurry level.
9. Set the air purge to **4,068 sccm** (4.068 sL/m).
10. Turn on the cooling water to the SRAT condenser (40°C) and the FAVC (10°C).
11. Turn on the mixer in the vessel to obtain uniform mixing with a small vortex (nominally **230 rpm**). Adjust the setting as necessary to obtain thorough mixing.
12. Start heating up the kettle. Set the temperature setting to 93°C and load to 100% power.
13. Ensure reflux stopcock from MWWT is closed.
14. When the kettle temperature reaches 40°C, add **34.06 g** IIT747 antifoam solution and an equal amount of water directly to the kettle. Record the antifoam solution addition amount ____________, water addition amount __________, and time of addition _______.
15. If the kettle foams at anytime, add **17.03 g** of IIT747 antifoam solution and an equal amount of water. Record any additional amounts ________ g solution, _________ g water, and the time of addition ___________.

    Amounts _________, _________ Time ________________

    Amounts _________, _________ Time ________________
16. When the kettle reaches 93°C, add **357.0 ml** of ~50wt% nitric acid **(10.47 M)** at **4.706 ml/min**.

    Nitric Acid start ____________ Nitric Acid end __________ pH _________
17. Add 25 mL of water to flush nitric acid line.
18. Add **1392.9 ml** of ~90wt% formic acid **(23.71 M)** at **4.697 ml/min** (~297 minutes).

    Formic Acid start ____________Formic Acid end __________ pH _________
19. Add 25 mL of water to flush formic acid line.

20. Record amount of condensate collected during acid addition.
21. Add **85.15 g** of IIT747 antifoam solution and an equal amount of water directly to the kettle. Record the antifoam solution addition amount ____________, water addition amount __________, and time of addition _______.
22. Change the set point on the temperature controller to 110°C.
23. Once the kettle has started to boil, record the time and switch the temperature controller to control by load instead of temperature. Adjust the load setting to obtain a boil-up rate of **23.6 g/min**. Check the boil-up rate continuously during decanting.

    Time boiling starts __________

    Boil-up rate _______________ Boil-up rate _______________

    Boil-up rate _______________ Boil-up rate _______________

    Boil-up rate _______________ Boil-up rate _______________

    Boil-up rate _______________ Boil-up rate _______________
24. De-water until **2,635.4 g** of water is removed. Collect the de-water mass as three samples. The first ~880 g should be labeled **FPMR-0082 for Run 1 and FPMR-0095 for Run 2**,, the second ~880 g should be labeled **FPMR-0083 for Run 1 and FPMR-0096 for Run 2**, and the remainder (~880 g) should be labeled **FPMR-0084 for Run 1 and FPMR-0097 for Run 2**. Samples should also be labeled with the date and name of PI.
25. Record the actual total weight removed (sum of the three de-water samples) and the completion time. These three samples should be checked for pH and for anion concentrations by IC.

    Total de-water mass _____________ Time de-water complete __________
26. Switch the SRAT to reflux. Reflux the SRAT for **12 hours**. Check the boil-up rate intermittently during the run.

    Boil-up rate _______________ Time _______________
27. Continue to monitor the boil-up rate intermittently during the run and attempt to hold **23.6 g/min**.

    Boil-up rate _______________ Time _______________

    Boil-up rate _______________ Time _______________

    Boil-up rate _______________ Time _______________

    Boil-up rate _______________ Time _______________

    Boil-up rate _______________ Time _______________

    Boil-up rate _______________ Time _______________
28. At the end of boiling (reflux), stop the SRAT cycle by turning off the heating mantle. Record the SRAT completion time _________.
29. Record the pH of the SRAT product _________.

SME Cycle Sequence

1. Set the air purge to **1,310 sccm** (1.302 sL/m) as specified in Table 3 for SME process.
2. Add **17.03 g** antifoam charge in amount shown in Table 3 followed by equal mass of water.
3. Add **1640.1 g** 4% Frit to the kettle as shown in Table 3 (1st frit addition).
4. Add **24.60 g** formic acid to the kettle.
5. Add **1615.5 g** water to the kettle.
6. Perform first dewater by removing **1640.1 g** as specified in Table 3.
7. Add **1640.1 g** 4% Frit to the kettle as shown in Table 3 (2nd frit addition).

8. Add **24.60 g** formic acid to the kettle.
9. Add **1615.5 g** water to the kettle.
10. Add **1640.1 g** 4% Frit to the kettle as shown in Table 3 (3rd frit addition).
11. Add **24.60 g** formic acid to the kettle.
12. Add **1615.5 g** water to the kettle.
13. Perform final dewater by removing **2794.6 g** as specified in Table 3.
14. Turn off mantle and allow material to cool.
15. Turn off remaining equipment when vessel cools below boiling.
16. Record the FAVC condensate mass collected __________. Label the sample as **FPMR-0087 for Run 1 and FPMR-0099 for Run 2**, along with the date and name of PI. This sample should be checked for pH and for anion concentrations by IC.
17. Collect the MWWT contents in a sample bottle and record the mass collected _________. Label the sample as **FPMR-0088 for Run 1 and FPMR-0098 for Run 2**, along with the date and name of PI. This sample should be checked for pH and for anion concentrations by IC.
18. Transfer SME product to polybottles labeled as follows plus the date and time the bottle is filled.

4% Na 22L SME Product	Run 1: FPMR-89 Run 2: FPMR-101

19. Pull two 125 gm sample of SME product from the polybottle upon transfer.

4% Na 22L SME Product	Run 1: FPMR-89A, FPMR-89B Run 2: FPMR-101A, FPMR-101B

20. Install the outlet flowmeter to the purge gas. Check the flow with 100.0 – 500 sccm air. Record the inlet flow _________ and outlet flow ________.
21. Perform a post-run calibration check of the pH probe with buffers of pH 4 _______, pH 10 ________ and pH 7 ________.
22. Housekeep after completion of run as specified by researcher.

Table 4.1. Batch Make-up Sheet

Mass of Fresh Sludge without trim chemicals	16800	gms
Mass of Coal to add	2.81	gms
Mass of sand to add	11.96	gms
Sodium Oxalate to add	0.00	gms
AgNO3	0.7265	gms
Gd(NO3)3*6H2O	0.00	gms
HgO to add	0.000	gms
Pd(NO3)2*H2O 15.27% Pd solution	0.3678	gms
Rh(NO3)3*2H2O 4.93% Rh solution	6.1849	gms
RuCl3	3.4603	gms solid
DI Water to dilute trim chemicals	200.00	gms

Table 4.2. SRAT Cycle Operating Parameters

SRAT Scaled Air Purge	4068.4	sccm
Target Antifoam Conc. based on sludge slurry mass after trim	100	ppm
747 Antifoam charge at 1:10	17.03	gms
Nitric Acid Molarity	10.45	Molar
Nitric Acid Required	357.0	ml
Scaled Nitric Acid Feed Rate based on nominal 10.395M, 2 gpm	4.706	ml/min
Nitric Acid Feed Time	75.9	min
Formic Acid Molarity	23.71	Molar
Formic Acid Required	1392.9	ml
Scaled formic acid feed rate based on nominal 23.551M, 2 gpm	4.697	ml/min
Formic Acid Feed Time	296.5	min
SRAT Scaled Boil-up Rate	23.61	gm/min
SRAT Dewater Mass	2635.2	gms
SRAT Dewater Time	111.6	min

Table 4.3. SME Cycle Operating Parameters

SME Frit Addition #1	1640.1	gms
Frit Addition #1 Water	1615.5	gms
Frit Addition #1 Formic Acid	24.60	gms
SME Frit Addition #2	1640.1	gms
Frit Addition #2 Water	1615.5	gms
Frit Addition #2 Formic Acid	24.60	gms
SME Frit Addition #3	1640.1	gms
Frit Addition #3 Water	1615.5	gms
Frit Addition #3 Formic Acid	24.60	gms
Frit Addition Dewater (Times 3)	1640.1	gms
Final Dewater mass	1154.5	gms
SME Air Purge	1.310	slm

12% Run Details

The following steps will be used to complete the run:

1. The equipment configuration (Figure 1) used for bench-scale 22-L vessel testing should be used for this run. Components include the 22-L kettle, heating mantle, variable speed mixer and controller, FAVC, MWWT, SRAT condenser, pH meter and probe, temperature controller and thermocouple, manometer, water chillers (10°C and 40°C), MKS flow controllers, and Masterflex pump for acid addition. However, no GC will be used for this run.
2. Record all M&TE utilized in the appropriate usage log and on the run preparation datasheet.
3. Ensure that the equipment is leak-checked and that the final outlet flow is within 90% of the inlet flow. Final leak check flowrates should be recorded on the run preparation datasheet.
4. Fill the MWWT with DI water to the overflow back to the SRAT. Do not let the water return to the SRAT. Record the mass added to the MWWT on the run preparation datasheet
5. Once the vessel and the equipment are set-up, the sludge and trim chemicals should be added per the batch make-up sheet, Table 1. Record the actual amounts added and scales utilized in the run preparation datasheet.
6. Agitate for 20 minutes. There is **no SRAT Receipt sample** this run.
7. Check the calibration of the pH probe to be used at pH 4, 10 and 7 and record readings on run preparation datasheet. Record the initial pH of the sludge once all trim chemicals have been added on the run preparation datasheet.
8. Insulate the entire SRAT vessel, allowing a small window for observing the slurry level.
9. Set the air purge to **4,070 sccm** (4.070 sL/m).
10. Turn on the cooling water to the SRAT condenser (40°C) and the FAVC (10°C).
11. Turn on the mixer in the vessel to obtain uniform mixing with a small vortex (nominally **230 rpm**). Adjust the setting as necessary to obtain thorough mixing.
12. Start heating up the kettle. Set the temperature setting to 93°C and load to 100% power.
13. Ensure reflux stopcock from MWWT is closed.
14. When the kettle temperature reaches 40°C, add **32.92 g** IIT747 antifoam solution and an equal amount of water directly to the kettle. Record the antifoam solution addition amount ____________, water addition amount __________, and time of addition _______.
15. If the kettle foams at anytime, add **16.46 g** of IIT747 antifoam solution and an equal amount of water. Record any additional amounts ________ g solution, _________ g water, and the time of addition ___________.

    Amounts _________, _________ Time ________________

    Amounts _________, _________ Time ________________

16. When the kettle reaches 93°C, add **273.71 ml** of ~50wt% nitric acid **(10.47 M)** at **4.707 ml/min**.

    Nitric Acid start ____________ Nitric Acid end __________ pH _________

17. Add 25 mL of water to flush nitric acid line.
18. Add **771.61 ml** of ~90wt% formic acid **(23.71 M)** at **4.699 ml/min** (~164 minutes).

    Formic Acid start ____________ Formic Acid end __________ pH _________

19. Add 25 mL of water to flush formic acid line.
20. Record amount of condensate collected during acid addition.

21. Add **82.30 g** of IIT747 antifoam solution and an equal amount of water directly to the kettle. Record the antifoam solution addition amount ____________, water addition amount __________, and time of addition _______.

22. Change the set point on the temperature controller to 110°C.

23. Once the kettle has started to boil, record the time and switch the temperature controller to control by load instead of temperature. Adjust the load setting to obtain a boil-up rate of **23.6 g/min**. Check the boil-up rate continuously during decanting.

Time boiling starts __________

Boil-up rate _______________ Boil-up rate _______________

Boil-up rate _______________ Boil-up rate _______________

Boil-up rate _______________ Boil-up rate _______________

Boil-up rate _______________ Boil-up rate _______________

24. De-water until **1,769.4 g** of water is removed. Collect the de-water mass as three samples. The first ~590 g should be labeled **FPMR-0105 for Run 1 and FPMR-0114 for Run 2**,, the second ~590 g should be labeled **FPMR-0106 for Run 1 and FPMR-0115 for Run 2**, and the remainder (~590 g) should be labeled **FPMR-0107 for Run 1 and FPMR-0116 for Run 2**. Samples should also be labeled with the date and name of PI.

25. Record the actual total weight removed (sum of the three de-water samples) and the completion time. These three samples should be checked for pH and for anion concentrations by IC.

Total de-water mass _____________ Time de-water complete __________

26. Switch the SRAT to reflux. Reflux the SRAT for **12 hours**. Check the boil-up rate intermittently during the run.

Boil-up rate _______________ Time _______________

27. Continue to monitor the boil-up rate intermittently during the run and attempt to hold **23.6 g/min**.

Boil-up rate _______________ Time _______________

Boil-up rate _______________ Time _______________

Boil-up rate _______________ Time _______________

Boil-up rate _______________ Time _______________

Boil-up rate _______________ Time _______________

Boil-up rate _______________ Time _______________

28. At the end of boiling (reflux), stop the SRAT cycle by turning off the heating mantle. Record the SRAT completion time _________.

29. Record the pH of the SRAT product _________.

SME Cycle Sequence

1. Set the air purge to **1,310 sccm** (1.310 sL/m) as specified in Table 3 for SME process.
2. Add **16.46 g** antifoam charge in amount shown in Table 3 followed by equal mass of water.
3. Add **1,857.1 g** 12% Frit to the kettle as shown in Table 3 (1st frit addition).
4. Add **27.86 g** formic acid to the kettle.
5. Add **1,829.2 g** water to the kettle.
6. Add **1,857.1 g** 12% Frit to the kettle as shown in Table 3 (2nd frit addition).
7. Add **27.86 g** formic acid to the kettle.
8. Add **1,829.2 g** water to the kettle.
9. Add **1,857.1 g** 12% Frit to the kettle as shown in Table 3 (3rd frit addition).

10. Add **27.86 g** formic acid to the kettle.
11. Add **1,829.2 g** water to the kettle.
12. Perform final dewater by removing **3937.1 g** as specified in Table 3.
13. Turn off mantle and allow material to cool.
14. Turn off remaining equipment when vessel cools below boiling.
15. Record the FAVC condensate mass collected __________. Label the sample as **FPMR-0108 for Run 1 and FPMR-0117 for Run 2**, along with the date and name of PI. This sample should be checked for pH and for anion concentrations by IC.
16. Collect the MWWT contents in a sample bottle and record the mass collected _________. Label the sample as **FPMR-0110 for Run 1 and FPMR-0118 for Run 2**, along with the date and name of PI. This sample should be checked for pH and for anion concentrations by IC.
17. Transfer SME product to polybottles labeled as follows plus the date and time the bottle is filled.

    12% Na 22L SME Product **Run 1: FPMR-0111**
    Run 2: FPMR-0119

18. Pull two 125 gm sample of SME product from the polybottle upon transfer.

    12% Na 22L SME Product **Run 1: FPMR-0111A, FPMR-0111B**
    Run 2: FPMR-119A, FPMR-119B

19. Install the outlet flowmeter to the purge gas. Check the flow with 100.0 – 500 sccm air. Record the inlet flow _________ and outlet flow ________.
20. Perform a post-run calibration check of the pH probe with buffers of pH 4 _______, pH 10 ________ and pH 7 ________.
21. Housekeep after completion of run as specified by researcher.

Table 4.4. Batch Make-up Sheet

Mass of Fresh Sludge without trim chemicals	16240	gms
Mass of Coal to add	2.51	gms
Mass of sand to add	10.69	gms
Sodium Oxalate to add	0.00	gms
AgNO3	0.6497	gms
Gd(NO3)3*6H2O	0.00	gms
HgO to add	0.000	gms
Pd(NO3)2*H2O 15.27% Pd solution	0.3290	gms
Rh(NO3)3*2H2O 4.93% Rh solution	5.5311	gms
RuCl3	3.0945	gms solid
DI Water to dilute trim chemicals	200.00	gms
Sample Mass of Trimmed sludge	0.00	gms

Table 4.5. SRAT Cycle Operating Parameters

SRAT Scaled Air Purge	4069.6	sccm
Target Antifoam Conc. based on sludge slurry mass after trim	100	ppm
747 Antifoam charge at 1:10	16.46	gms
Nitric Acid Molarity	10.47	Molar
Nitric Acid Required	273.7	ml
Scaled Nitric Acid Feed Rate based on nominal 10.395M, 2 gpm	4.707	ml/min
Nitric Acid Feed Time	58.1	min
Formic Acid Molarity	23.71	Molar
Formic Acid Required	771.6	ml
Scaled formic acid feed rate based on nominal 23.551M, 2 gpm	4.698	ml/min
Formic Acid Feed Time	164.2	min
SRAT Scaled Boil-up Rate	23.62	gm/min
SRAT Dewater Mass	1769.4	gms
SRAT Dewater Time	74.91	min

Table 4.6. SME Cycle Operating Parameters

SME Frit Addition #1	1857.1	gms
Frit Addition #1 Water	1829.2	gms
Frit Addition #1 Formic Acid	27.86	gms
SME Frit Addition #2	1857.1	gms
Frit Addition #2 Water	1829.2	gms
Frit Addition #2 Formic Acid	27.86	gms
SME Frit Addition #3	1857.1	gms
Frit Addition #3 Water	1829.2	gms
Frit Addition #3 Formic Acid	27.86	gms
Frit Addition Dewater (Times 3)	1857.1	gms
Final Dewater mass	2080.0	gms
SME Air Purge	1.310	slm

5.5 Attachment 5. Run Data: pH and Dewater Profiles

Figure 5.1 is a graph summarizing the dewater profile for the five SRAT batches. No dewater data was collected during the 22L batches. Time zero is defined as the start of nitric acid addition.

Figures 5.2-5.6 are graphs summarizing the pH profile for the five SRAT batches. Figures 5.7 and 5.8 are graphs summarizing the pH profile for the four 22-L SRAT/SME batches.

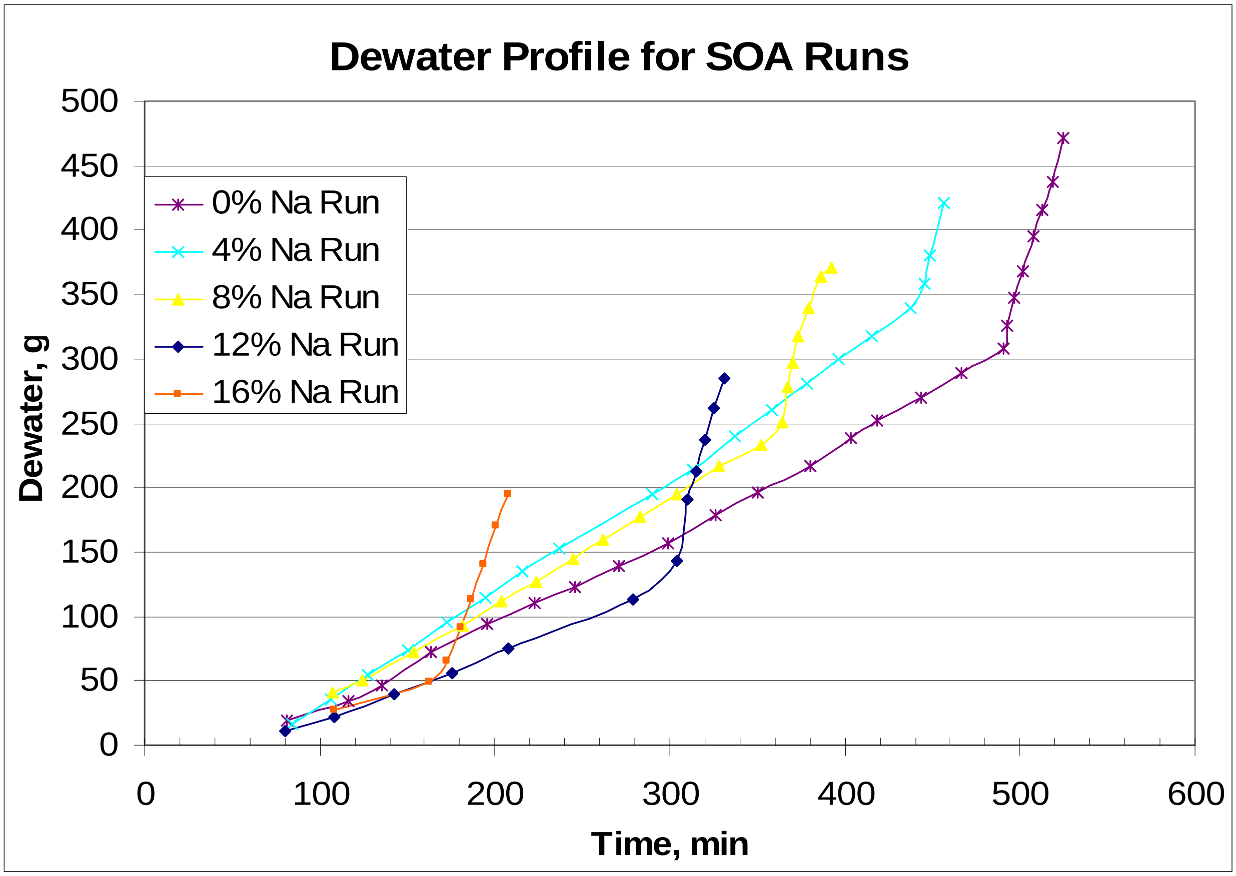


Figure 5.1 – Dewater Profile for 4L SOA Runs

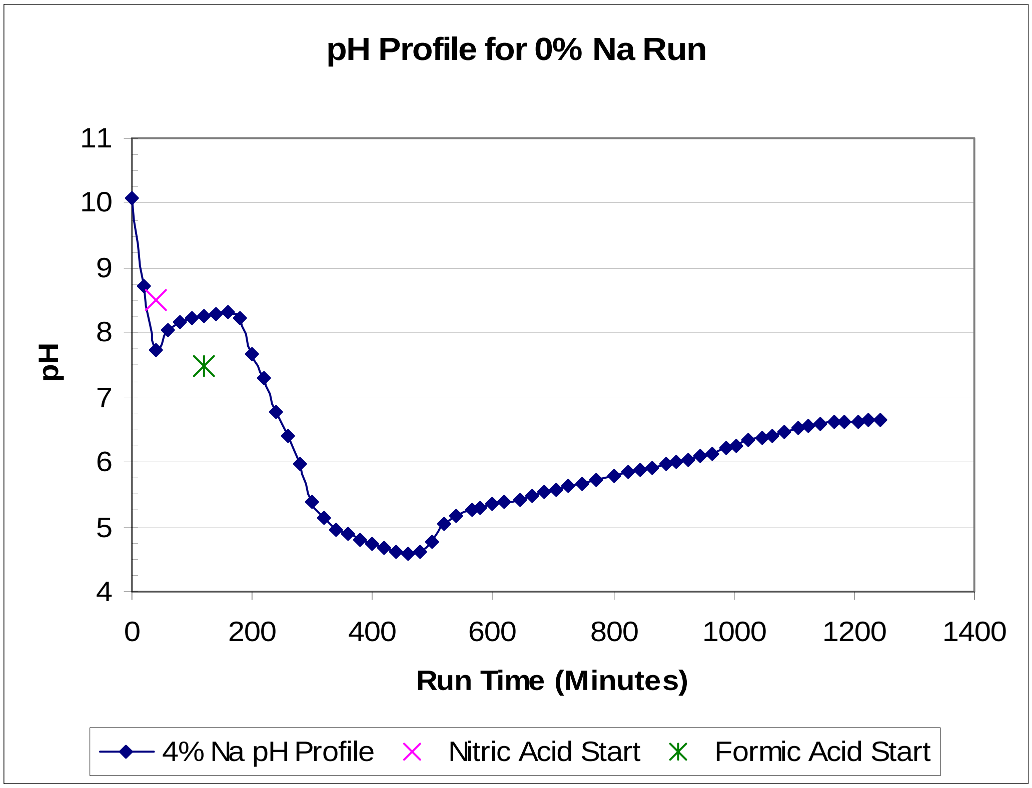


Figure 5.2 – pH Profile for 4L 0% Na_2O SOA Run

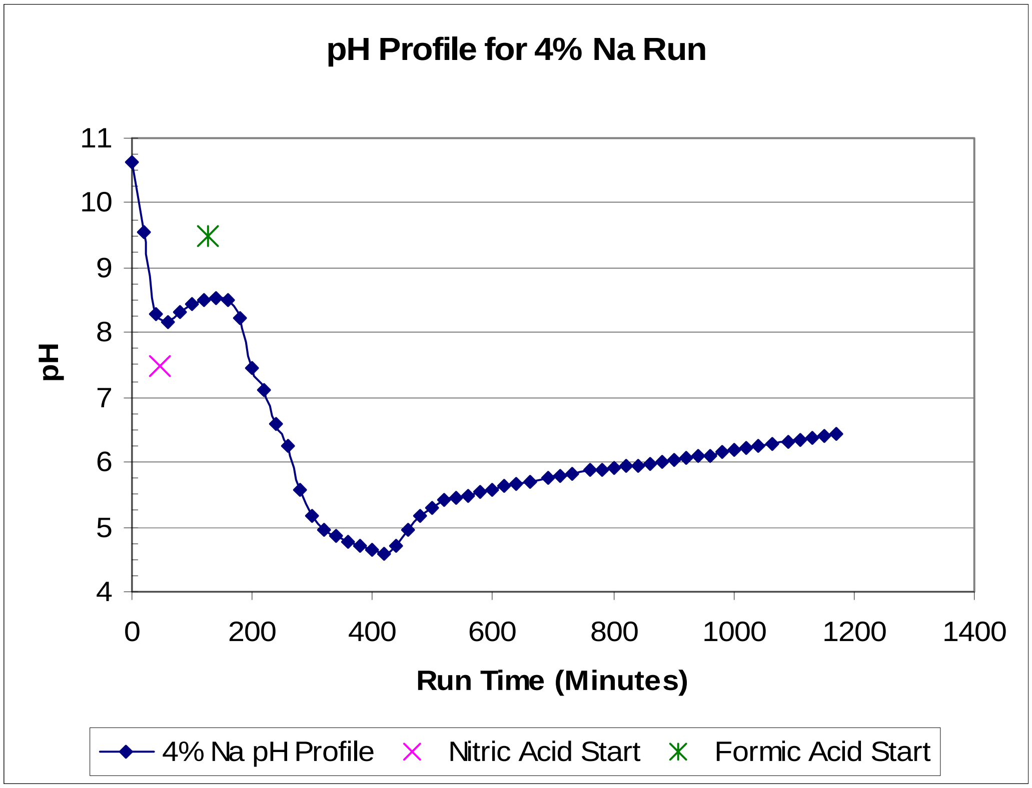


Figure 5.3 – pH Profile for 4L 4% Na_2O SOA Run

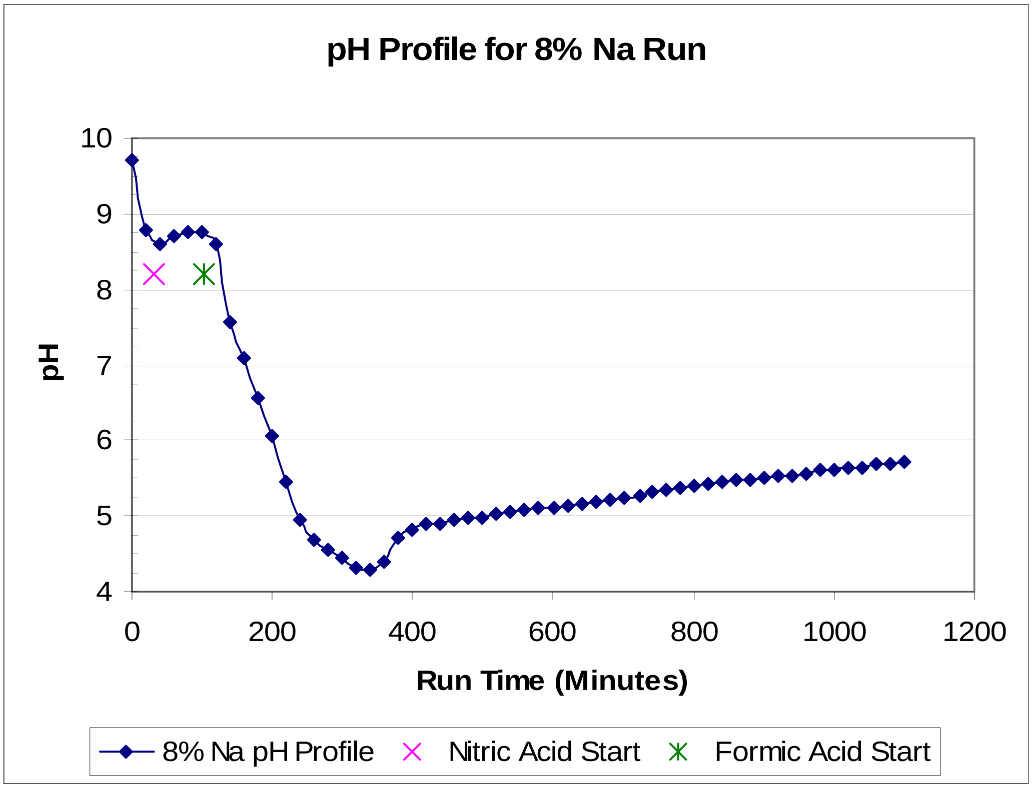


Figure 5.4 – pH Profile for 4L 8% Na_2O SOA Run

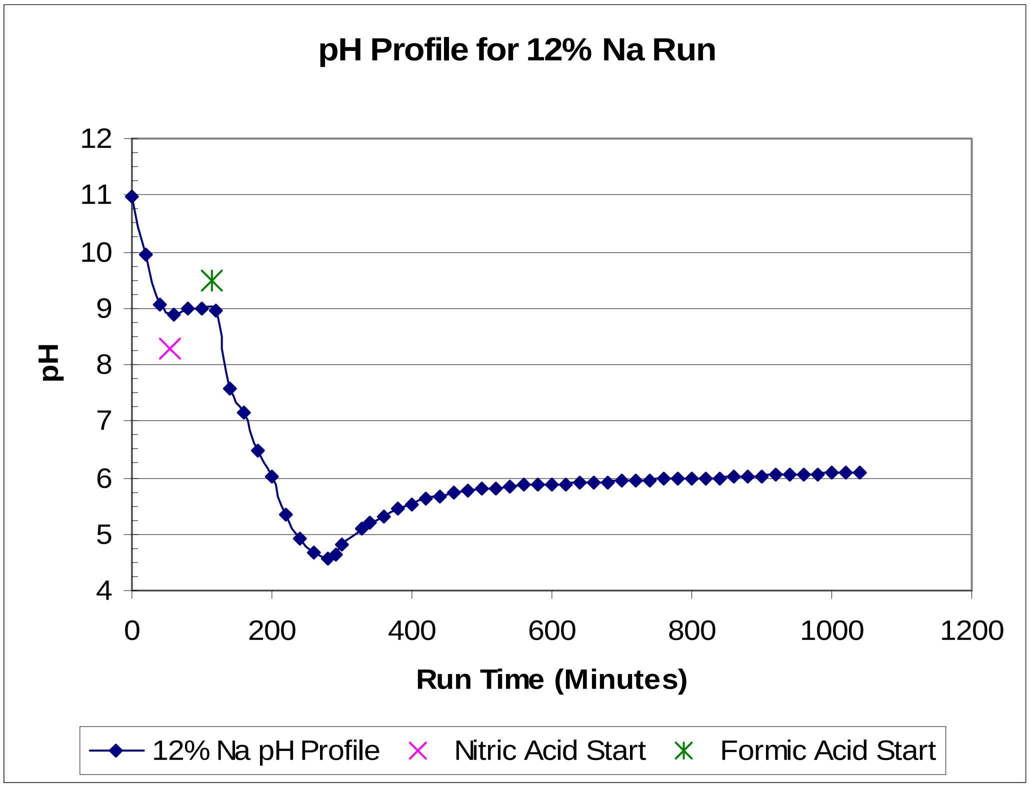


Figure 5.5 – pH Profile for 4L 12% Na_2O SOA Run

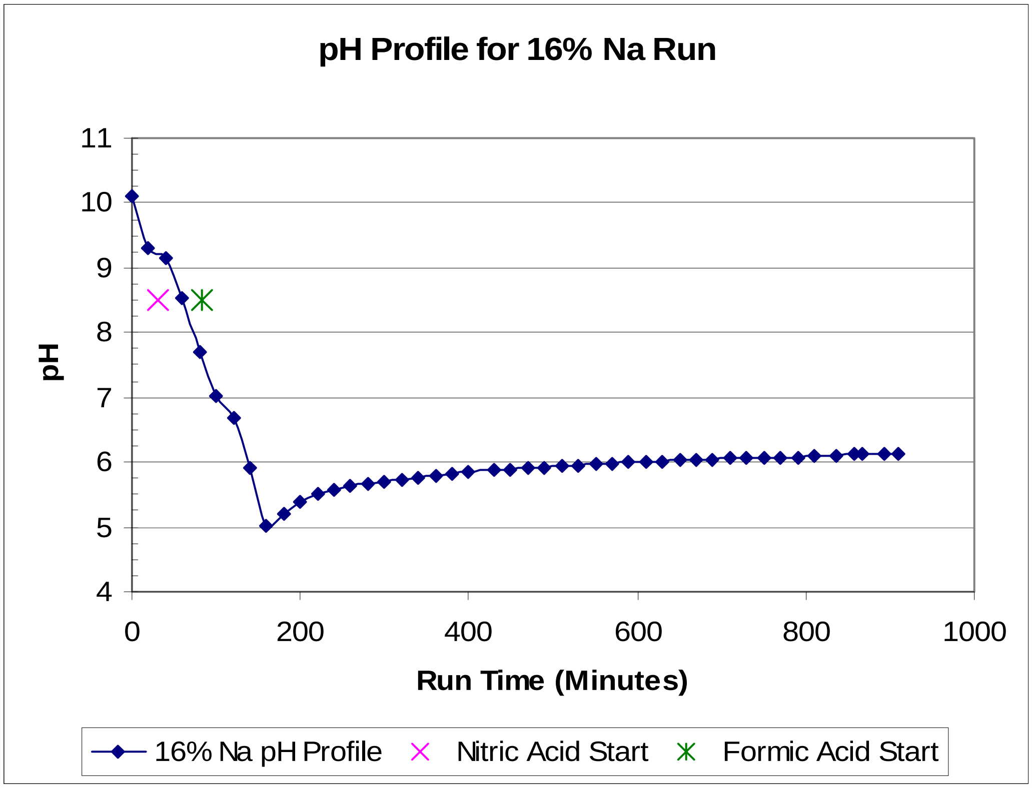


Figure 5.6 – pH Profile for 4L 16% Na_2O SOA Run

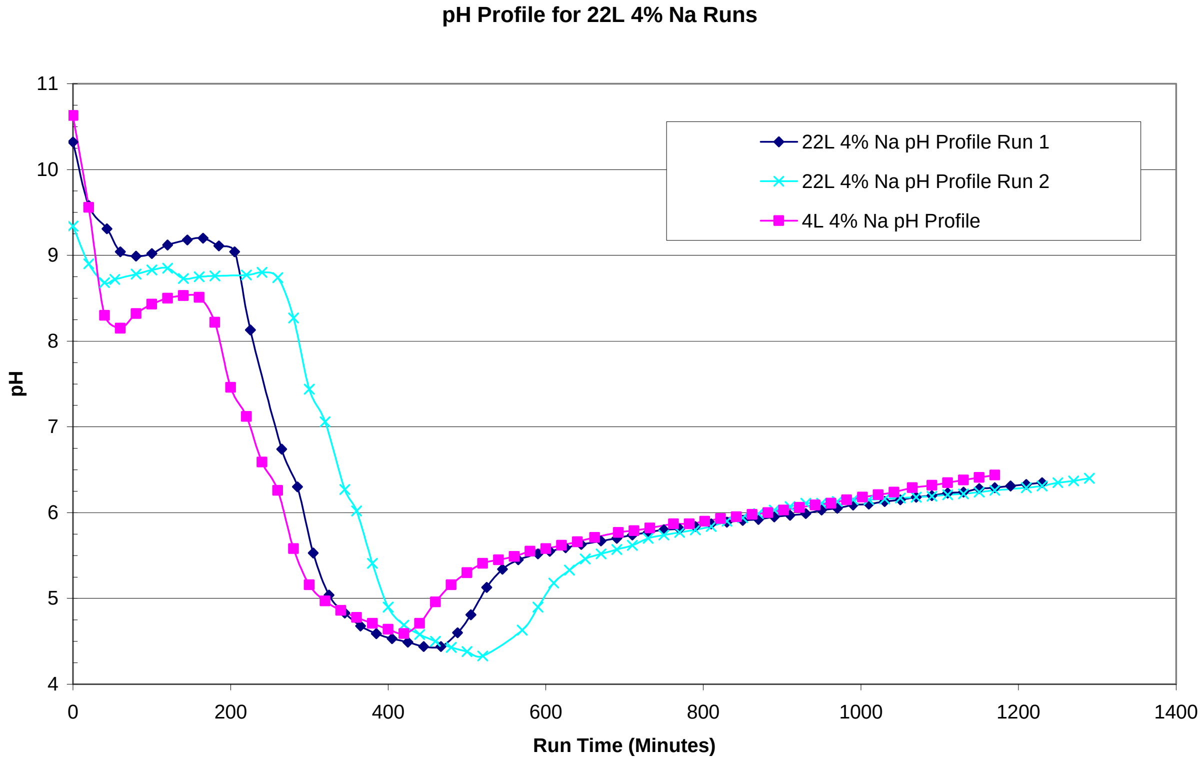


Figure 5.7 – pH Profile for 22L 4% Na_2O SOA Runs

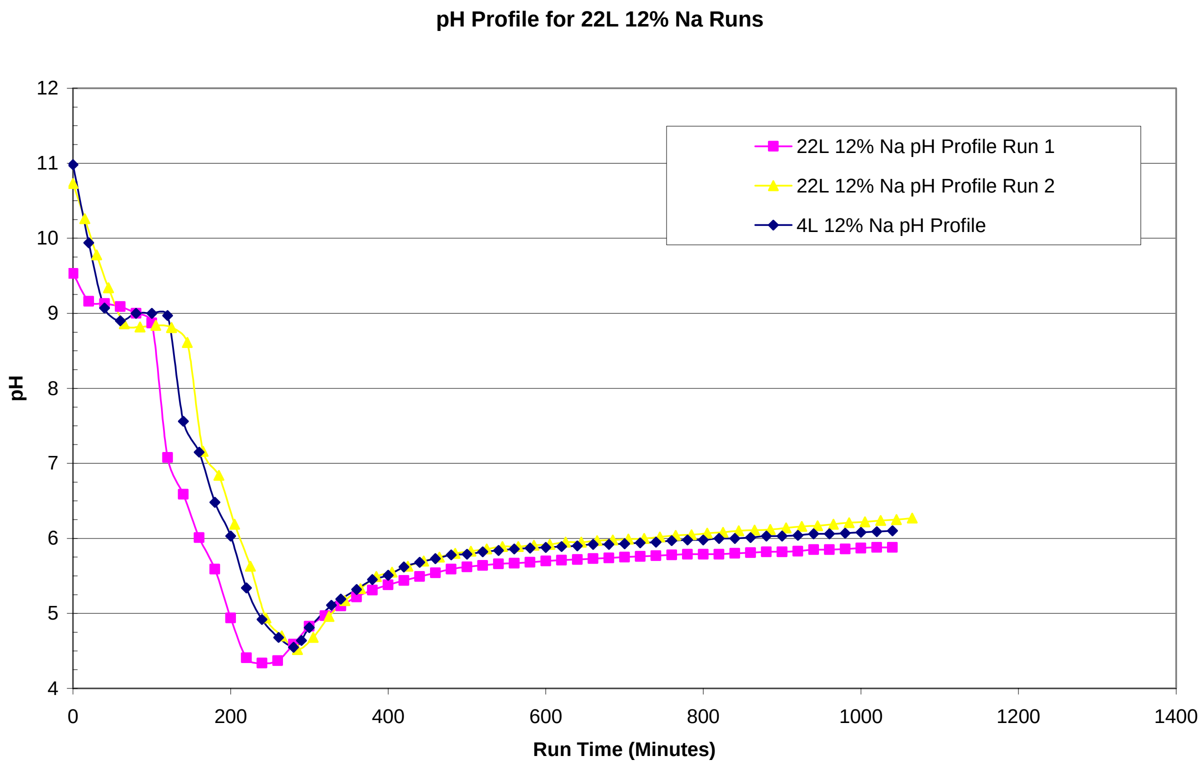


Figure 5.8 – pH Profile for 22L 12% Na_2O SOA Runs

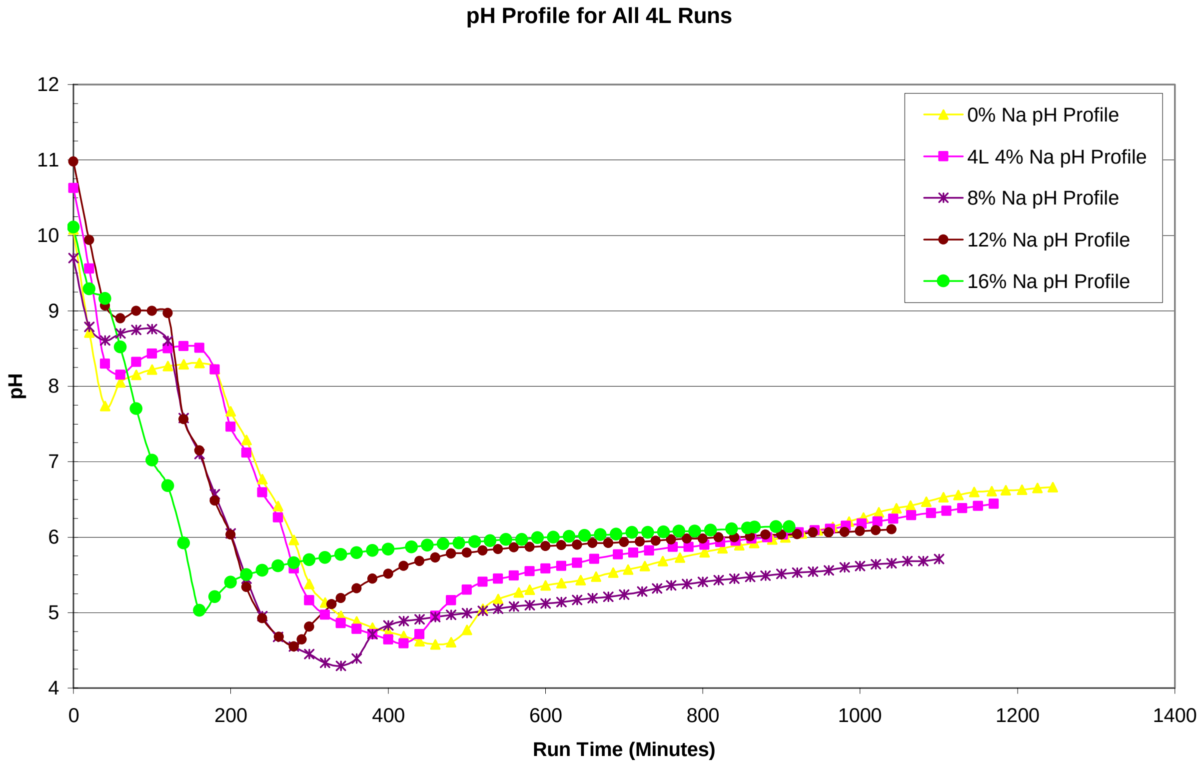


Figure 5.9 – pH Profile for all 4L Na_2O SOA Runs

Table 5- 1 summarizes the pH and dewater profiles for the five SRAT batches. Table 5- 2 summarizes the condensate anion and pH data for all the runs.

Table 5- 1 – Run Data: pH and Dewater Profiles

Parameter	Units	0%	4%	8%	12%	16%
Run Location		129-S	129-S	132-S	129-N	129-N
Total Mass of Sludge Simulant	grams	2845	2806	2699	2715	2596
Time Required to reach 93 degrees	hr:min	0:53	0:50	0:48	0:58	1:00
Condensate Rate during Acid Addition	g/min	0.72	0.93	0.85	0.50	0.39
Nitric Acid Added (by weight)	ml	51.80	51.08	49.44	39.40	32.30
Formic Acid Added (by weight)	ml	239.3	229.8	189.4	129.0	59.5
Total Acid Added	molar	2.49	2.39	2.00	1.39	0.70
Formic Acid Added	molar	2.27	2.18	1.80	1.22	0.56
pH at End of Acid Addition		4.54	4.58	4.27	4.55	5
Time at Boiling	hrs	12.60	12.25	12.50	12.52	12.57
pH at End of SRAT Cycle		6.66	6.45	5.72	6.11	6.15
pH Delta during Boiling		2.12	1.87	1.45	1.56	1.15
Calculated H^+ at end of acid addition	M	2.88E-05	2.63E-05	5.37E-05	2.82E-05	1.00E-05
Calculated H^+ at End of SRAT Cycle	M	2.19E-07	3.55E-07	1.91E-06	7.76E-07	7.08E-07
Delta H^+ concentration during boiling	M	2.86E-05	2.59E-05	5.18E-05	2.74E-05	9.29E-06
Target Nitric addition	ml	51.66	50.85	49.64	39.26	31.21
Delta from Actual	%	0.27	0.45	-0.41	0.36	3.51
Target formic acid Addition	ml	271.53	229.79	187.78	128.60	60.91
Delta from Actual	%	-11.86	0.00	0.86	0.31	-2.34
MWWT Sample 1 Wt	grams	122	115.12	126.12	75.31	75.2
MWWT Sample 2 Wt	grams	129.7	124.84	125.12	100.49	75.10
MWWT Sample 3 Wt	grams	220.3	181.4	118.95	48.16	54.4
MWWT Final Wt	grams	48.1	47.5	54.5	54.7	56.2
FAVC Sample Wt	grams	20.9	15	23.7	5.3	4.2

Note: Experiments were performed at ACTL, Laboratory Rooms 129 and 132, in the north and south hoods.

Table 5- 2 – Run Data: Condensate Anion and pH Data

Batch	Dewater 1			Dewater 2			Dewater 3		
	HCO_2^- (mg/L)	NO_3^- (mg/L)	pH	HCO_2^- (mg/L)	NO_3^- (mg/L)	pH	HCO_2^- (mg/L)	NO_3^- (mg/L)	pH
4L 0% Na_2O	1,735	8,515	0.2	3,230	28,000	2.92	2,630	32,500	2.84
4L 4% Na_2O	1,410	6,785	0.8	4,750	12,150	0.43	3,950	35,000	2.97
4L 8% Na_2O	2,535	3,865	1.23	4,790	23,100	0.17	2,795	20,100	0.17
4L 12% Na_2O	2,240	1,858	1.31	1,950	5,685	0.91	1,095	5,685	0.88
4L 16% Na_2O	570	188	0.91	415	114	1.16	230	<100	1.50
22L 4%Na_2O R1	<100	<100	1.97	865	37,800	0.40	1,760	19,250	2.67
22L 12%Na_2O R1	<100	586	1.14	400	3,650	0.76	878	9,270	0.57

Batch	MWWT			FAVC		
	HCO_2^- (mg/L)	NO_3^- (mg/L)	pH	HCO_2^- (mg/L)	NO_3^- (mg/L)	pH
4L 0% Na_2O	<100	2,855	9.57	<100	<100	1.46
4L 4% Na_2O	<100	437	1.99	107	381,500	1.62
4L 8% Na_2O	213	307	2.13	245	<100	1.67
4L 12% Na_2O	<100	447	1.79	245	301,000	2.45
4L 16% Na_2O	<100	<100	2.01	154	26,500	0.18
22L 4%Na_2O R1	270	4,895	1.14	133	203,000	0.53
22L 12%Na_2O R1	122	177	1.36	121	75,450	0.05

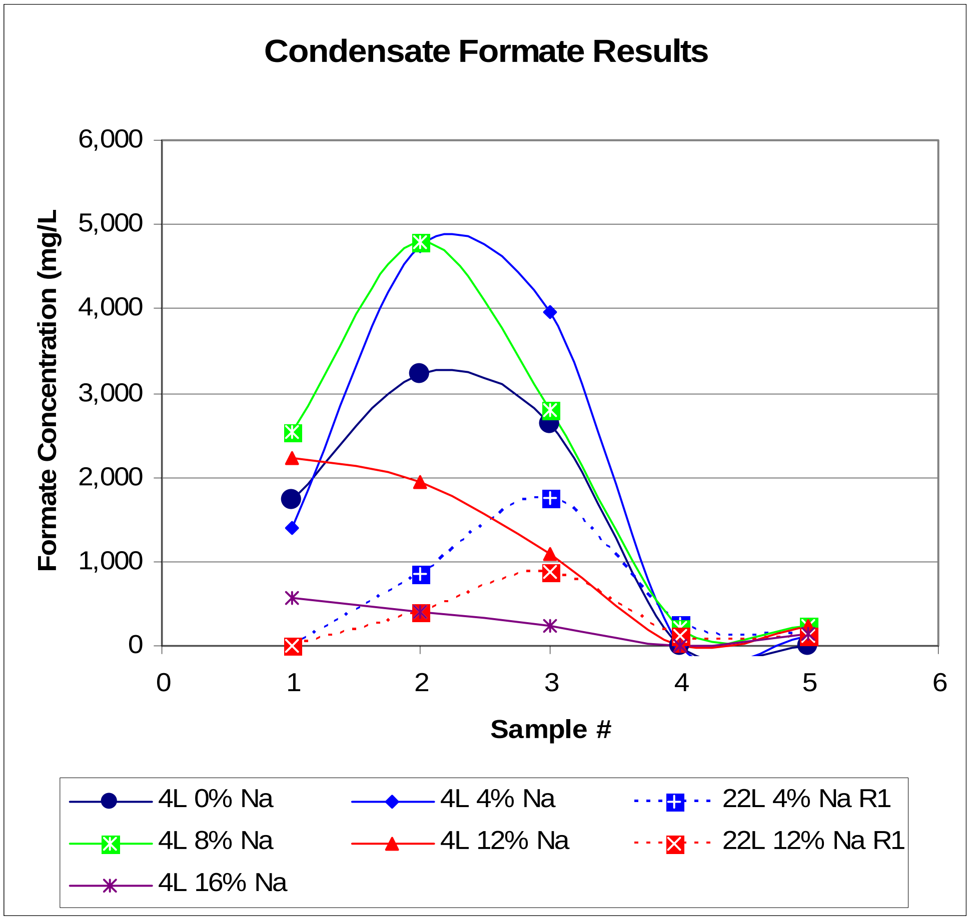


Figure 5.10 – Formate Profile in Condensate during SRAT Cycles

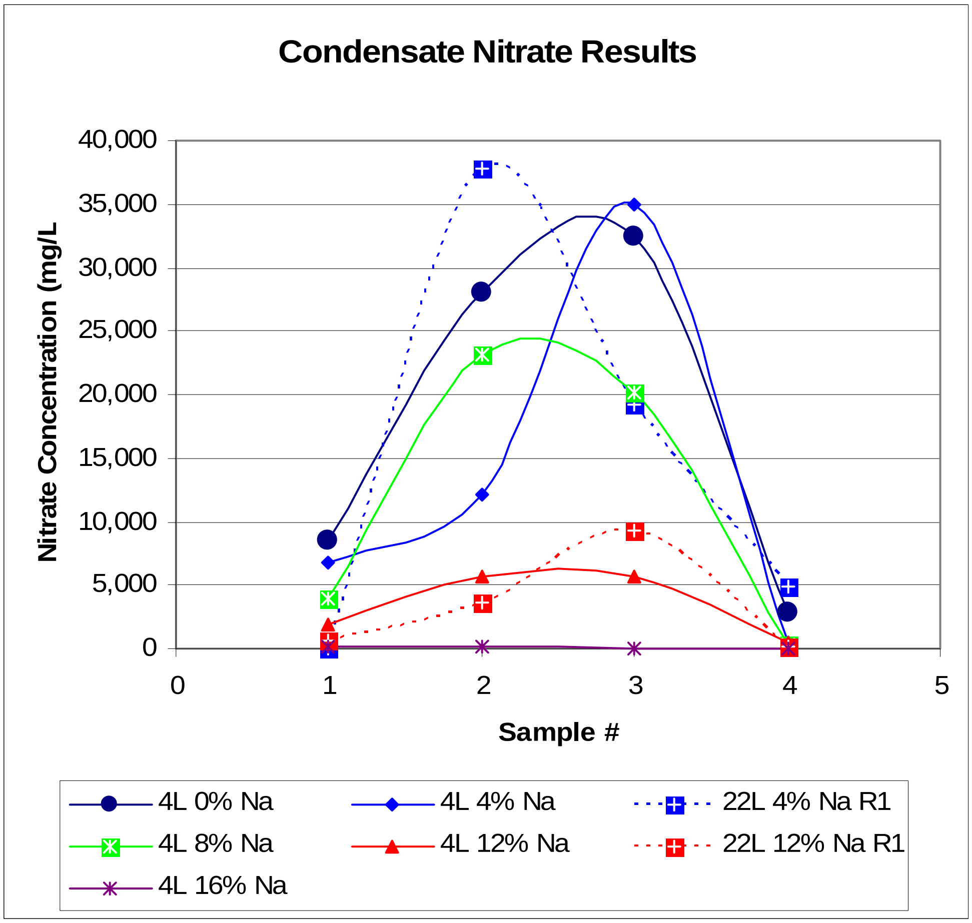


Figure 5.11 – Nitrate Profile in Condensate during SRAT Cycles

5.6 Attachment 6. Run Data: Offgas Composition Charts

The measured offgas composition is summarized in the following graphs which combine the results for the five SRAT cycles.

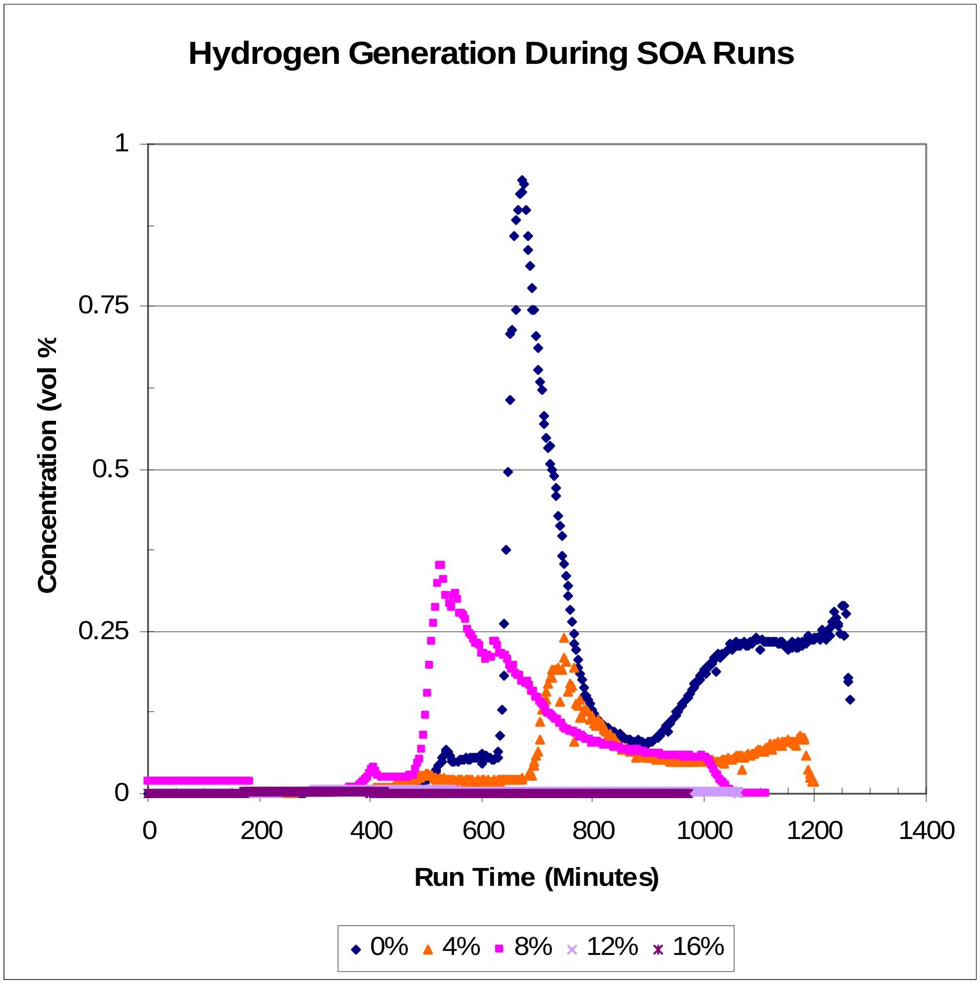


Figure 6.1 – Hydrogen Profile during SRAT Cycles

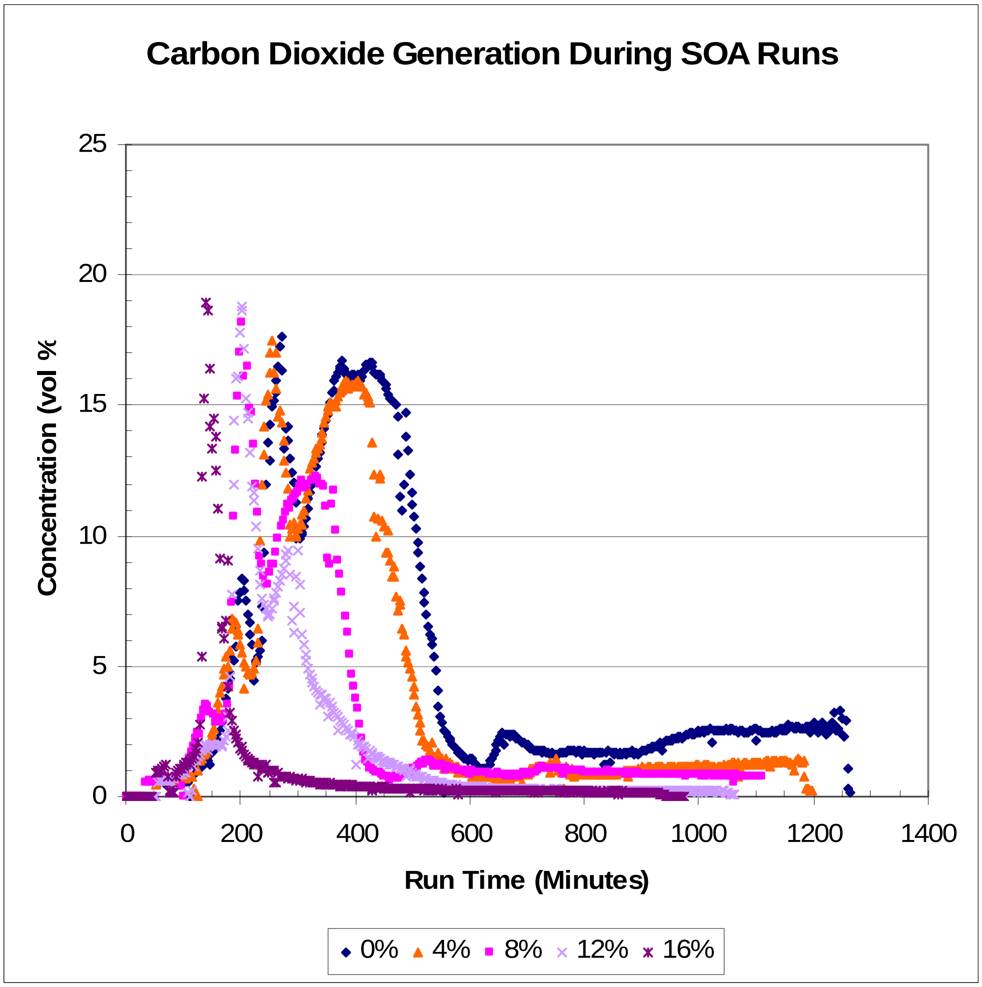


Figure 6.2 – Carbon Dioxide Profile during SRAT Cycles

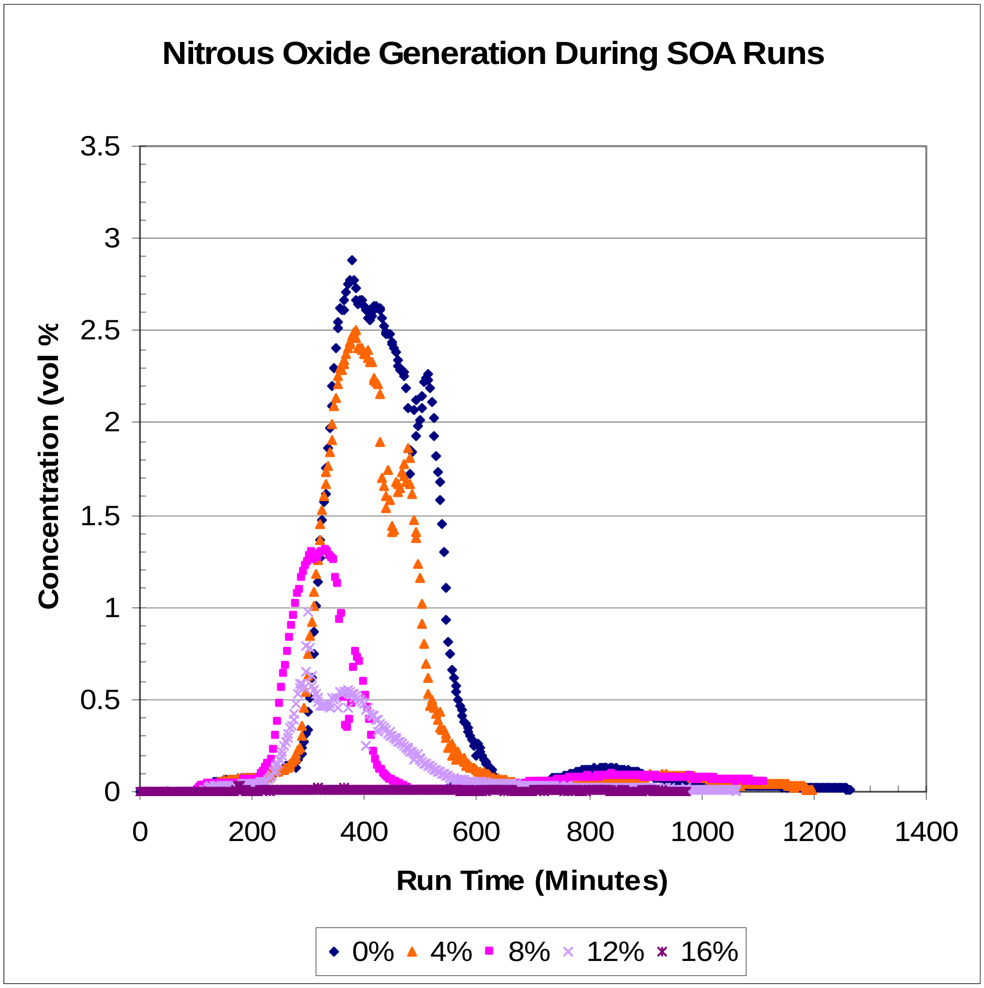


Figure 6.3 – Nitrous Oxide Profile during SRAT Cycles

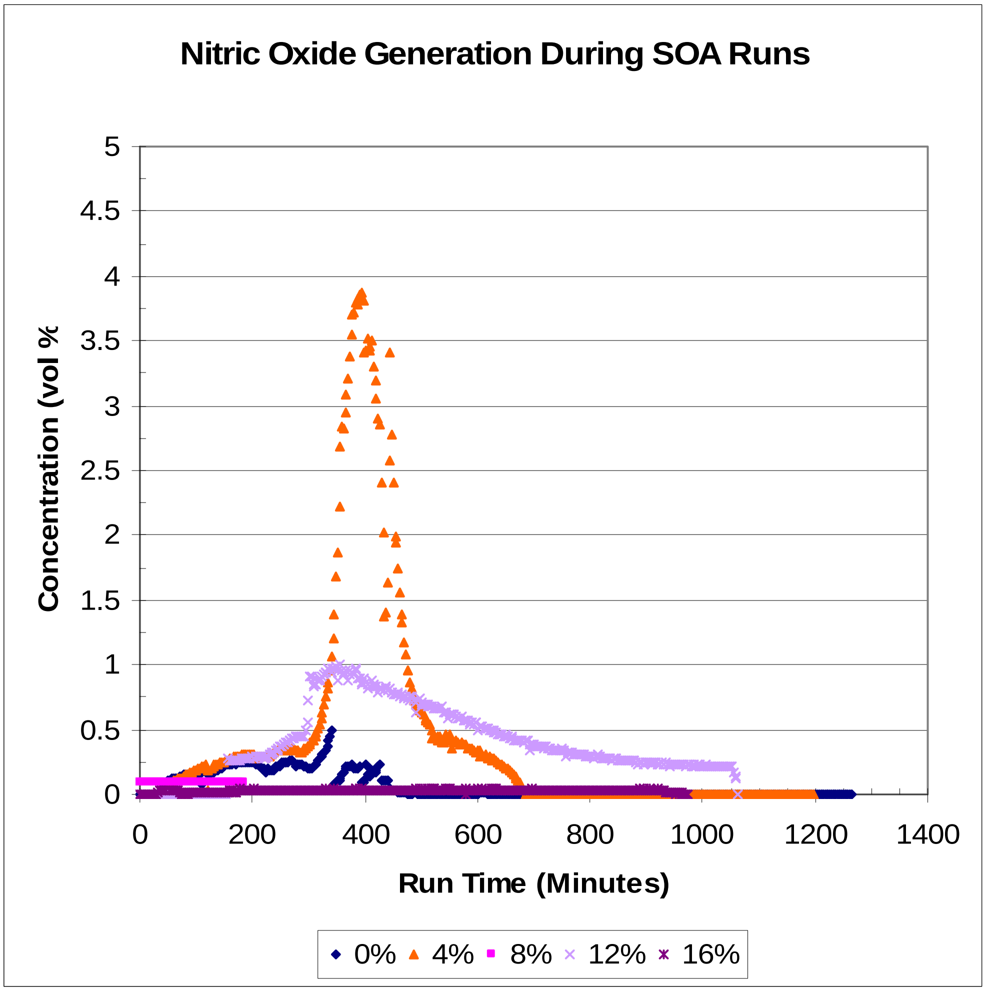


Figure 6.4 – Nitric Oxide Profile during SRAT Cycles

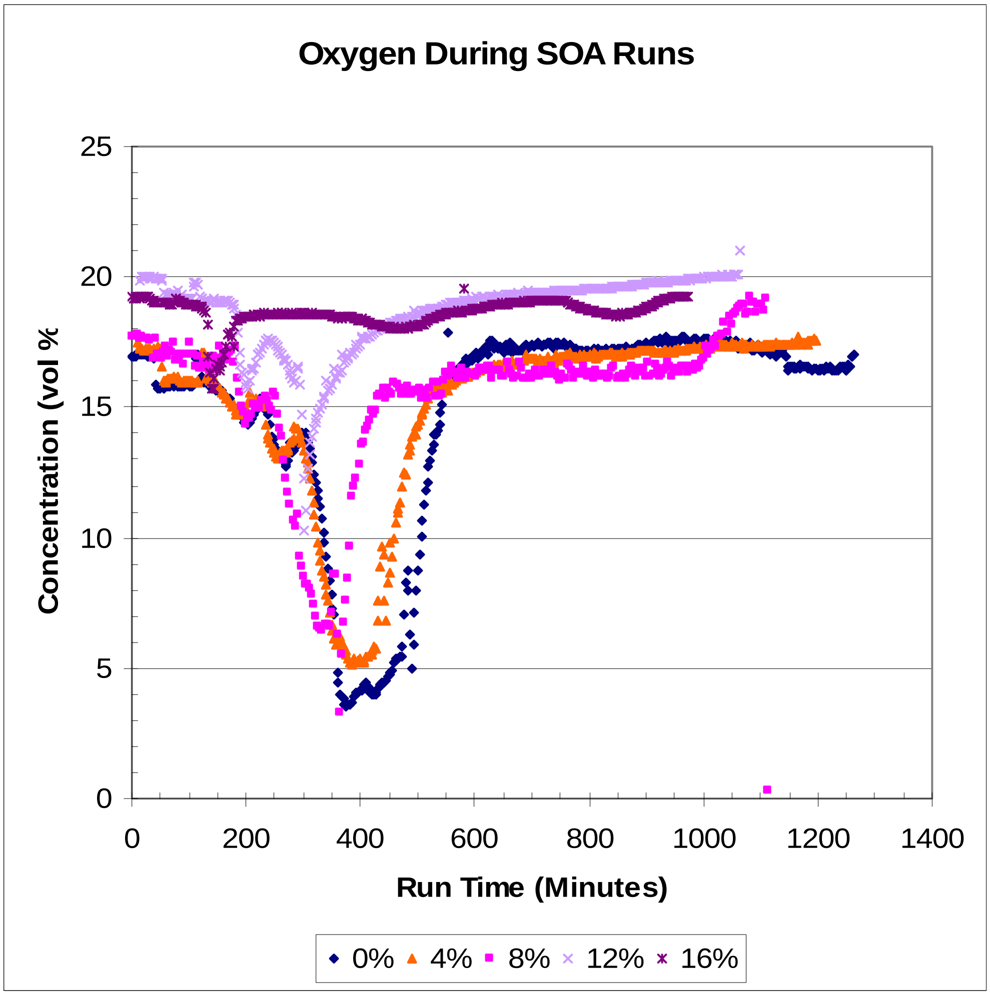


Figure 6.5 – Oxygen Profile during SRAT Cycles

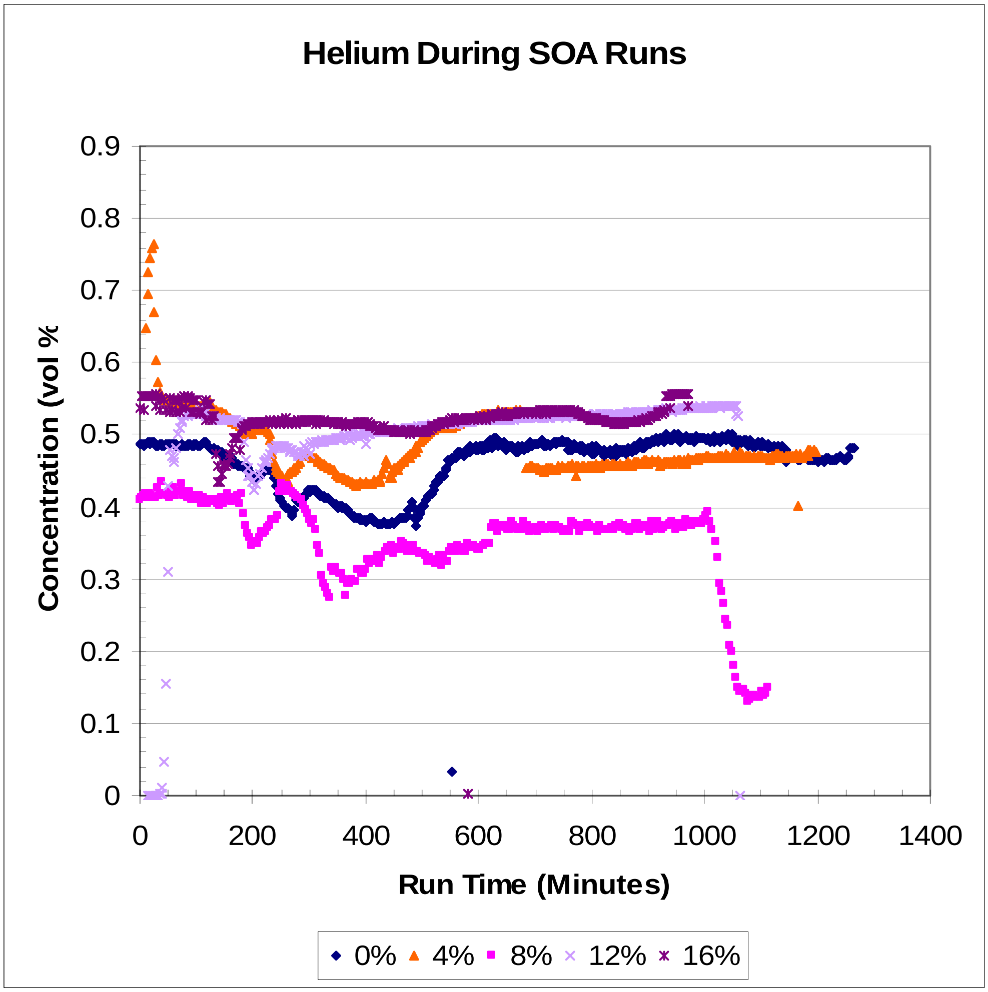


Figure 6.6 – Helium Profile during SRAT Cycles

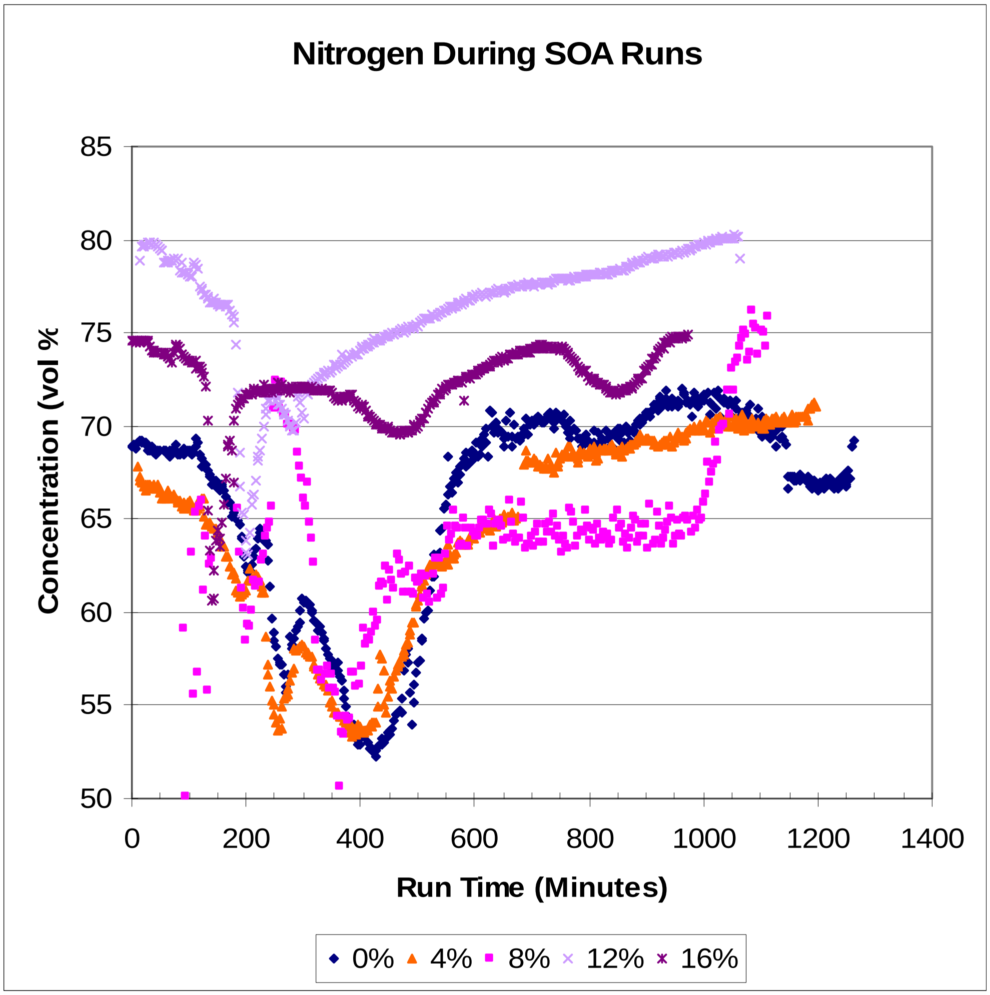


Figure 6.7 – Nitrogen Profile during SRAT Cycles

5.7 Attachment 7: Sample Results

Table 7- 1 – SRAT Product ICP-ES Metals, wt% solids

Run #	0%	4%	8%	12%	16%
Sample ID	FPRM-0046-A	FPRM-0052-A	FPRM-0040-A	FPRM-0058-A	FPRM-0064-A
ML ID	05-0009	04-2188	05-0011	04-2189	05-0010
Ag	<0.010	<0.010	<0.010	<0.010	<0.010
Al	8.37	8.73	9.82	10.35	11.75
Ba	0.111	0.121	0.1335	0.1475	0.162
Ca	1.85	2.02	2.16	2.23	2.58
Cd	<0.010	<0.010	<0.010	<0.010	<0.010
Cr	0.13	0.14	0.16	0.15	0.18
Cu	0.13	0.11	0.14	0.18	0.19
Fe	24.7	27.0	28.5	32.0	34.2
K	0.08	<0.010	0.112	0.127	0.163
Mg	2.395	2.52	2.87	2.775	3.68
Mn	3.45	3.74	4.00	4.40	4.80
Gd	0.061	nm	0.0735	nm	0.0975
Na	22.35	18.15	15.8	9.955	3.895
Ni	0.926	0.979	1.070	1.195	1.220
P	0.040	0.038	0.039	0.038	0.037
Pb	0.0195	<0.010	0.013	0.031	0.038
Pd	<0.010	0.019	<0.010	0.015	<0.010
Ru	0.0295	0.0195	0.0235	0.0150	0.0235
S	0.412	0.416	0.494	0.465	0.637
Si	1.00	1.13	1.17	1.42	1.31
Ti	0.019	0.022	0.0225	0.024	0.027
Zn	0.292	0.306	0.332	0.377	0.399
Zr	0.335	0.354	0.378	0.405	0.384

Table 7- 2 – SRAT Product Anions, mg/kg

Run #	0%	4%	8%	12%	16%
Sample ID	FPRM-0046-A	FPRM-0052-A	FPRM-0040-A	FPRM-0058-A	FPRM-0064-A
ML ID	05-0009	04-2188*	05-0011	04-2189*	05-0010
HCO2	82850	81200	66400	51300	24900
NO2	<100	<100	<100	477.5	349
NO3	45800	37400	33900	21000	10850
Cl	107	113	107	113.5	<100
PO4	<100	<100	<100	<100	<100
SO4	1115	1245	1420	1393	1505

Table 7- 3 – SRAT Product Total, Insoluble, Soluble and Calcined Solids, wt %, density, pH

Run #	0%	4%	8%	12%	16%
Sample ID	FPRM-0046-A	FPRM-0052-A	FPRM-0040-A	FPRM-0058-A	FPRM-0064-A
ML ID	05-0009	04-2188*	05-0011	04-2189*	05-0010
Total Solids	32.1	30.0	27.5	23.8	18.0
Soluble Solids	14.31	13.96	13.94	14.48	13.46
Insoluble Solids	19.35	18.48	17.34	16.12	12.96
Density	1.31	1.29	1.22	1.19	1.09
pH	8.12	7.66	6.64	7.16	7.49
Calcine Solids/Dry Solids	0.602	0.617	0.631	0.678	0.721

Table 7- 4 – Sludge ICP-ES Metals, wt% solids

	0% Frit	4% Frit	8% Frit - Baseline	12 % Frit	16% Frit
	FPMR-0036 (A)	FPMR-0037 (A)	SB3-11-18-04-ML (A)	FPMR-0038 (A)	FPMR-0039 (A)
Al	8.265	8.98	9.865	10.7	11.7
Ba	0.1145	0.122	0.1305	0.145	0.157
Ca	2.03	2.19	2.145	2.64	2.93
Cr	0.1315	0.137	0.167	0.165	0.184
Cu	0.1265	0.146	0.153	0.1765	0.1865
Fe	26.05	27.65	29.7	32.45	35.8
K	0.056	0.1005	0.116	0.1345	0.162
Mg	2.68	2.825	2.945	3.285	3.905
Mn	3.48	3.82	4.26	4.53	4.955
Gd	0.06	0.062	0.0725	0.075	0.092
Na	18.75	16.8	14.9	8.77	3.065
Ni	0.919	1.005	0.964	1.175	1.31
P	0.0295	0.0315	0.045	0.034	0.037
Pb			0.005	0.027	0.02
S	0.3585	0.37	0.393	0.439	0.446
Si	0.9215	0.975	1.16	1.165	1.24
Zn	0.2865	0.305	0.336	0.343	0.3425
Zr	0.3145	0.3555	0.404	0.4015	0.39

Table 7- 5 – Sludge Anions, mg/kg

	0% Frit	4% Frit	8% Frit - Baseline	12 % Frit	16% Frit
	FPMR-0036 (A)	FPMR-0037 (A)	SB3-11-18-04-ML (A)	FPMR-0038 (A)	FPMR-0039 (A)
NO2	31800	25150	18550	10700	766
NO3	23000	18500	14550	8680	2155
SO4	2010	2075	2120	2135	2195
C2O4	1904	1948	2000	2060	2126
TIC (calc)	1650	1500	1324	1125	901

Table 7- 6 – Sludge Total and Calcined Solids, wt %, density, pH

	0% Frit	4% Frit	8% Frit - Baseline	12 % Frit	16% Frit
	FPMR-0036 (A)	FPMR-0037 (A)	SB3-11-18-04-ML (A)	FPMR-0038 (A)	FPMR-0039 (A)
Total Solids	25.49	24.37	22.71	20.60	16.29
Calcined Solids	17.97	17.47	16.34	15.51	12.90
Density	1.21	1.20	1.15	1.16	1.11
pH	13.2	13.4	13.4	13.2	12.9
Calcine Solids/Dry Solids	0.705	0.717	0.720	0.753	0.792

Table 7- 7 – Frit Preparation for 22-L Batches, composition, wt %

Sample ID	Lab ID	B	Cr	Fe	Li	Na	Ni	Si
12%Na_2OFrit 1 (A)	05-0079	2.42	0.012	0.058	3.34	9.02	<0.010	34.2
12%Na_2OFrit 1 (B)	05-0079	2.43	0.013	0.055	3.41	8.76	<0.010	34.2
12%Na_2OFrit 2 (A)	05-0080	2.39	0.014	0.068	3.29	8.61	<0.010	33.2
12%Na_2OFrit 2 (B)	05-0080	2.40	0.014	0.064	3.30	8.70	<0.010	33.9
12%Na_2OFrit 3 (A)	05-0081	2.40	0.014	0.048	3.39	8.84	<0.010	33.8
12%Na_2OFrit 3 (B)	05-0081	2.34	0.013	0.059	3.31	8.80	<0.010	33.1
4%Na_2OFrit 1 (A)	05-0082	2.62	0.015	0.055	3.67	3.08	<0.010	37.3
4%Na_2OFrit 1 (B)	05-0082	2.62	0.015	0.052	3.66	2.93	<0.010	37.2
4%Na_2OFrit 1 (A)	05-0083	2.85	0.015	0.083	3.66	2.96	<0.010	37.7
4%Na_2OFrit 1 (B)	05-0083	2.77	0.014	0.090	3.59	2.94	<0.010	37.4
4%Na_2OFrit 1 (A)	05-0084	2.76	0.015	0.052	3.51	3.02	<0.010	36.6
4%Na_2OFrit 1 (B)	05-0084	2.78	0.014	0.051	3.52	2.93	<0.010	37.4

Sample ID	Lab ID	B2O3	Cr2O3	Fe2O3	Li2O	Na_2O	NiO	SiO2	Total
12%Na_2OFrit 1 (A)	05-0079	7.79	0.018	0.083	7.18	12.2	0.000	73.2	100
12%Na_2OFrit 1 (B)	05-0079	7.82	0.019	0.079	7.33	11.8	0.000	73.2	100
12%Na_2OFrit 2 (A)	05-0080	7.70	0.020	0.097	7.07	11.6	0.000	71.0	97.6
12%Na_2OFrit 2 (B)	05-0080	7.73	0.020	0.092	7.10	11.7	0.000	72.5	99.2
12%Na_2OFrit 3 (A)	05-0081	7.73	0.020	0.069	7.29	11.9	0.000	72.3	99.4
12%Na_2OFrit 3 (B)	05-0081	7.53	0.019	0.084	7.12	11.9	0.000	70.8	97.5
12%Na_2OFrit Average		7.72	0.02	0.08	7.18	11.86	0.00	72.19	99.06
12%Na_2OFrit Target		7.66	0.00	0.00	7.66	11.95	0.00	72.73	100.00
Sample ID	Lab ID	B2O3	Cr2O3	Fe2O3	Li2O	Na_2O	NiO	SiO2	Total
4%Na_2OFrit 1 (A)	05-0082	8.44	0.022	0.079	7.89	4.16	0.000	79.8	100
4%Na_2OFrit 1 (B)	05-0082	8.44	0.022	0.074	7.87	3.96	0.000	79.6	100
4%Na_2OFrit 1 (A)	05-0083	9.18	0.022	0.119	7.87	4.00	0.000	80.7	102
4%Na_2OFrit 1 (B)	05-0083	8.92	0.020	0.129	7.72	3.97	0.000	80.0	101
4%Na_2OFrit 1 (A)	05-0084	8.89	0.022	0.074	7.55	4.08	0.000	78.3	98.9
4%Na_2OFrit 1 (B)	05-0084	8.95	0.020	0.073	7.57	3.96	0.000	80.0	101
4%Na_2OFrit Average		8.80	0.02	0.09	7.74	4.02	0.00	79.75	100.43
4%Na_2OFrit Target		8.35	0.00	0.00	8.35	4.02	0.00	79.29	100.00

5.8 Attachment 8: Rheology Results

Sample	Run	Sample	Total Solids (wt %)	Insoluble Solids (wt %)	Soluble Solids (wt %)	Consistency (cP)	Yield Stress (Pa)	Curve Fit	Shear rate range (sec^{-1})
Sludge	0%	FPMR-0036A	25.49	Not Measured	Not Measured	7.87	2.02	up/down	50 - 600
Sludge	4%	FPMR-0037A	24.37	Not Measured	Not Measured	8.30	2.37	up/down	50 - 600
Sludge	12%	FPMR-0038A	20.60	Not Measured	Not Measured	8.66	3.22	up/down	50 - 600
Sludge	16%	FPMR-0039A	16.29	Not Measured	Not Measured	5.65	1.82	up/down	50 - 600
SRAT Product	0%	FPMR-0046B	32.13%	14.31%	17.82%	9.22	1.75	up/down	50 - 600
SRAT Product	4%	FPMR-0052B	29.96%	13.96%	16.01%	8.49	1.21	up/down	50 - 600
SRAT Product	8%	FPMR-0040B	27.50%	13.94%	13.57%	9.07	1.87	down	150 - 600
SRAT Product	12%	FPMR-0058B	23.77%	14.48%	9.28%	5.33	4.57	down	150 - 600
SRAT Product	16%	FPMR-0064B	17.98%	13.46%	4.52%	5.47	1.51	up/down	150 - 600

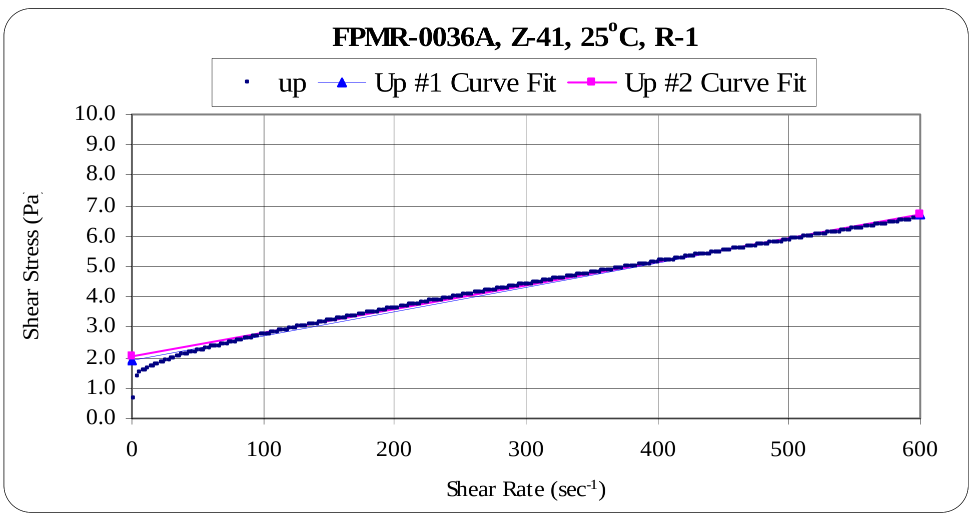


Figure 8.1a – Rheology Profile, 0% Na_2O Sludge

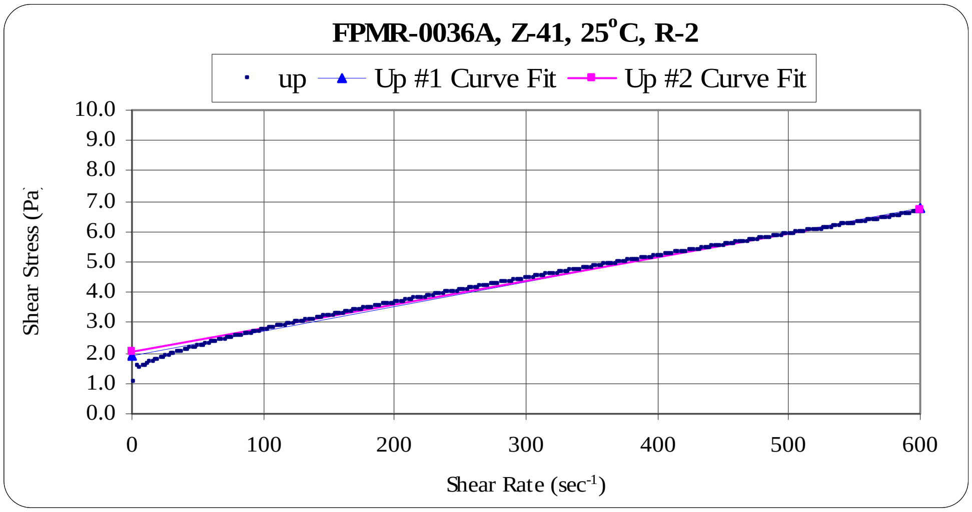


Figure 8.1b – Rheology Profile, 0% Na_2O Sludge

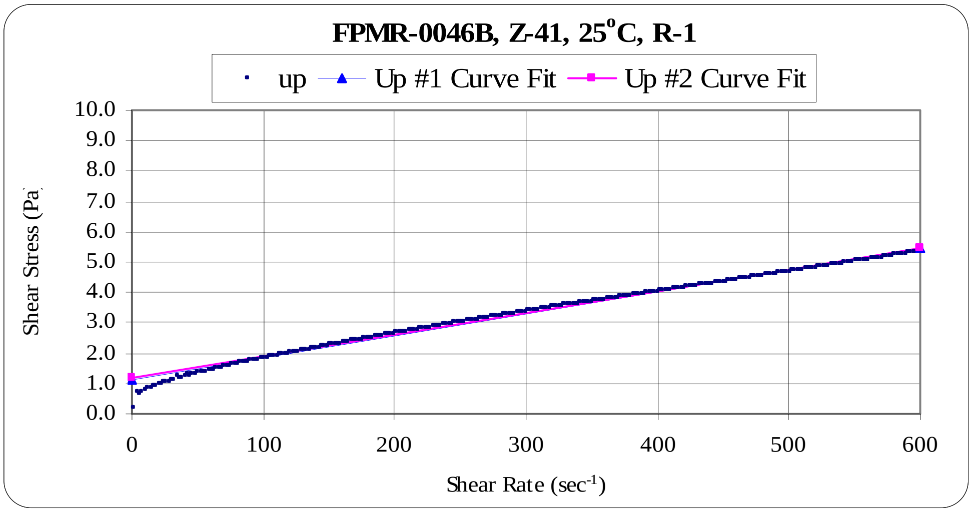


Figure 8.1c – Rheology Profile, 0% Na_2O SRAT Product

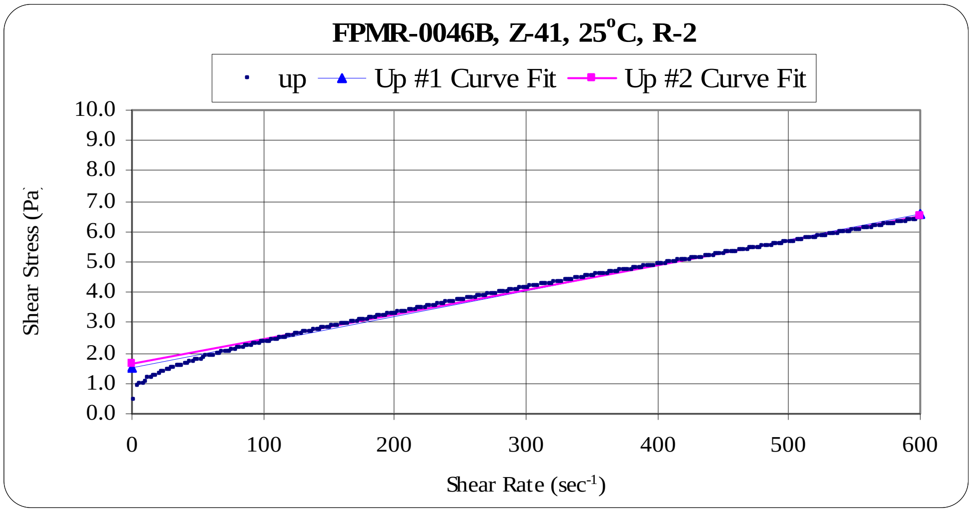


Figure 8.1c – Rheology Profile, 0% Na_2O SRAT Product

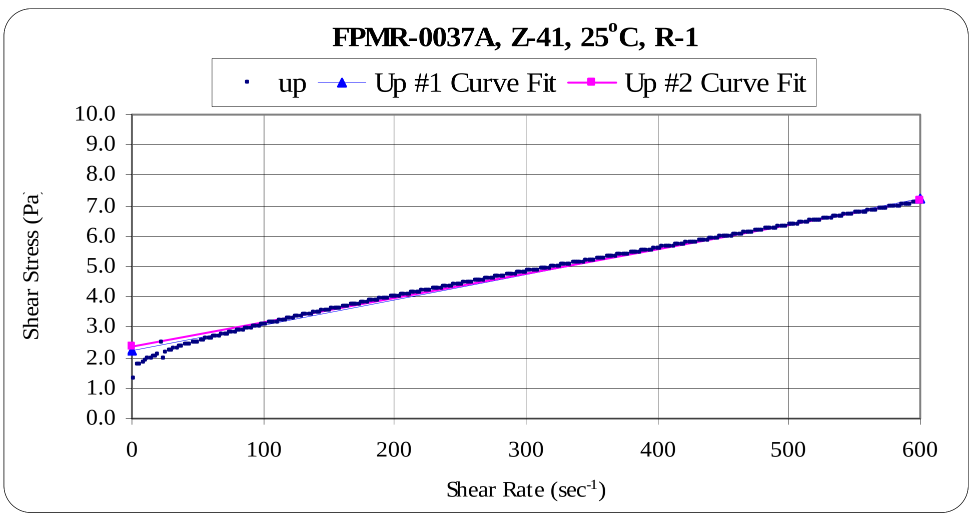


Figure 8.2a – Rheology Profile, 4% Na_2O Sludge

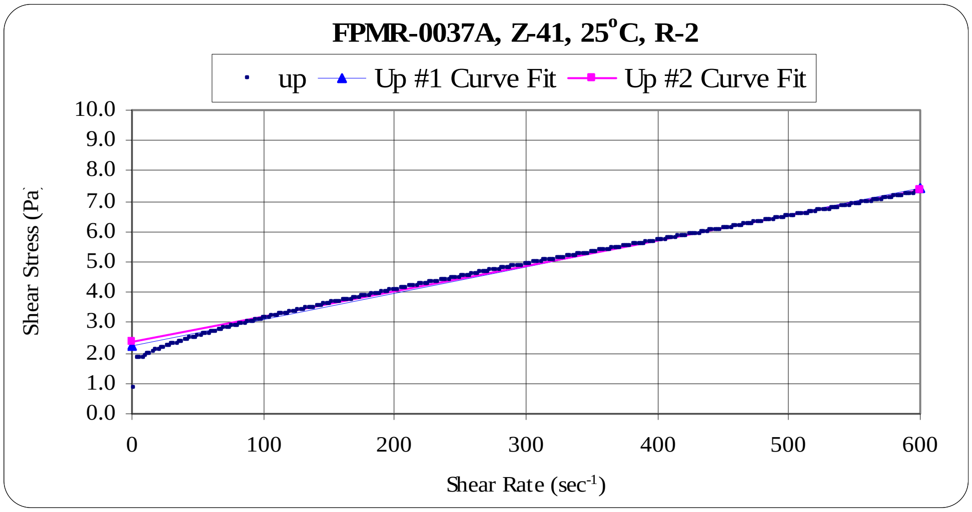


Figure 8.2b – Rheology Profile, 4% Na_2O Sludge

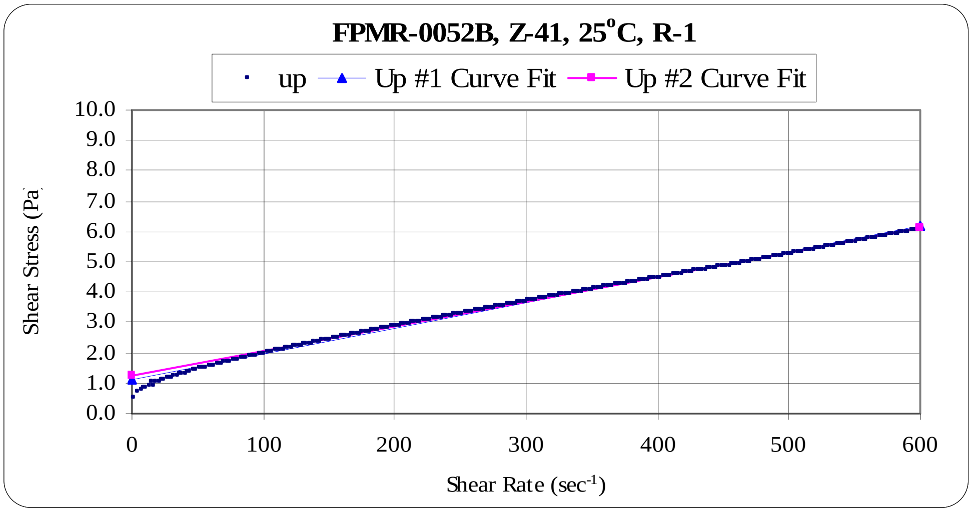


Figure 8.2c – Rheology Profile, 4% Na_2O SRAT Product

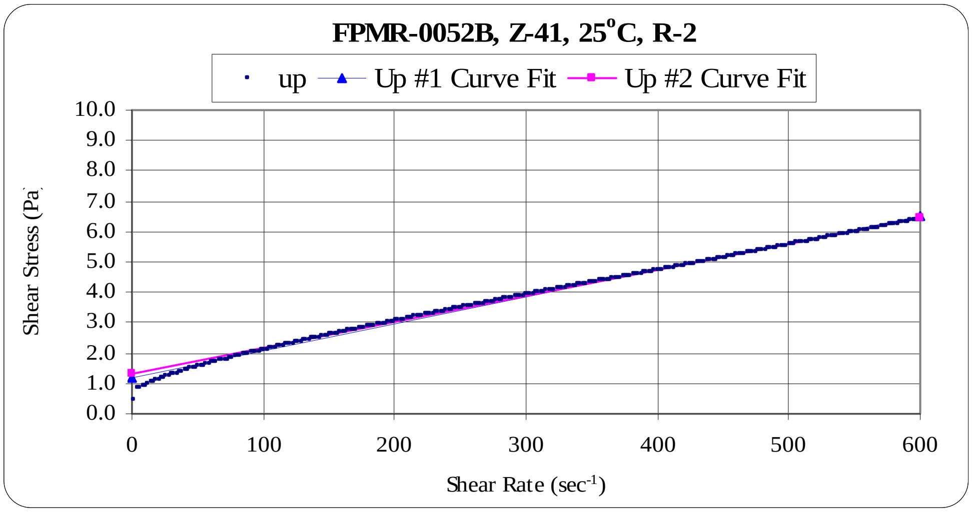


Figure 8.2d – Rheology Profile, 4% Na_2O SRAT Product

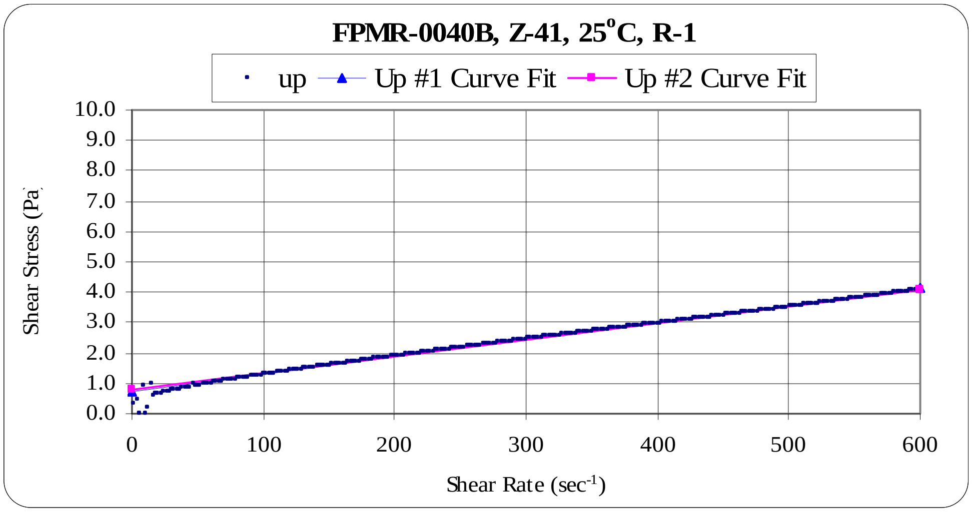


Figure 8.3a – Rheology Profile, 8% Na_2O SRAT Product

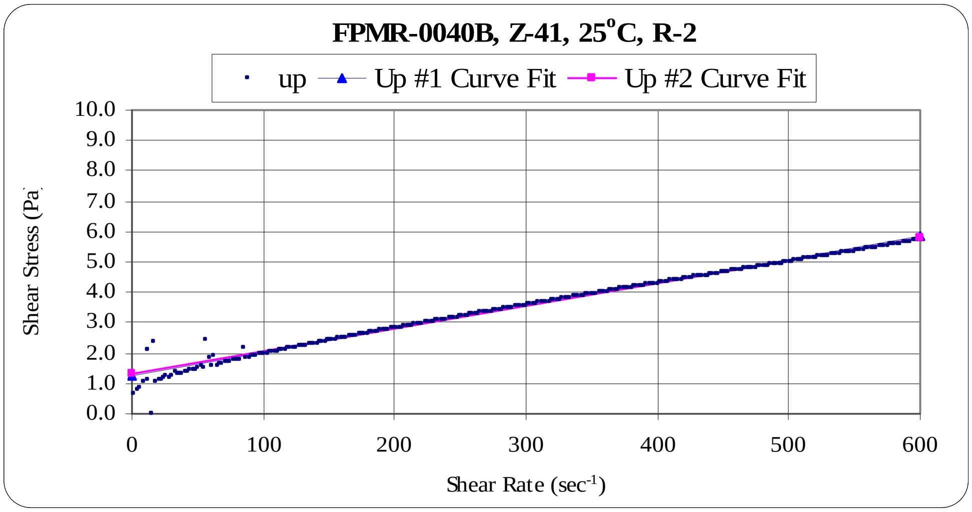


Figure 8.3b – Rheology Profile, 8% Na_2O SRAT Product

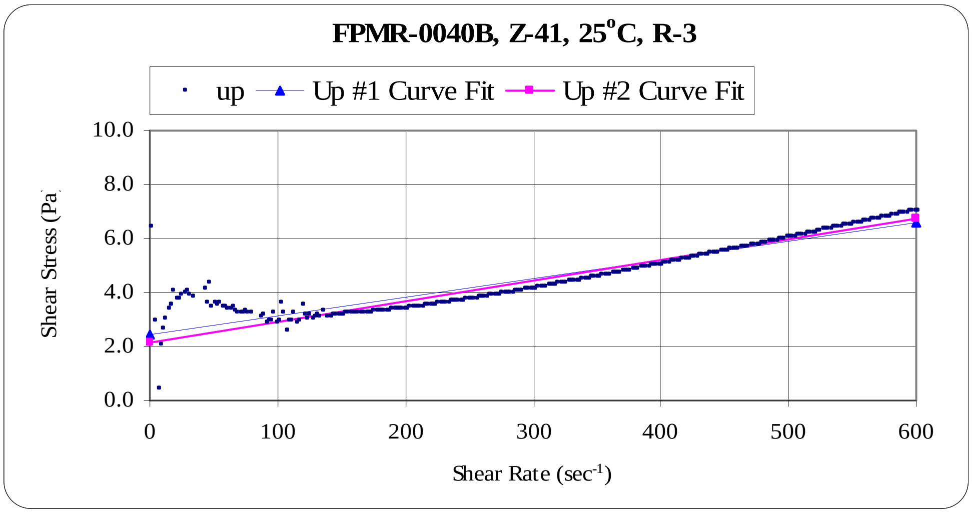


Figure 8.3c – Rheology Profile, 8% Na_2O SRAT Product

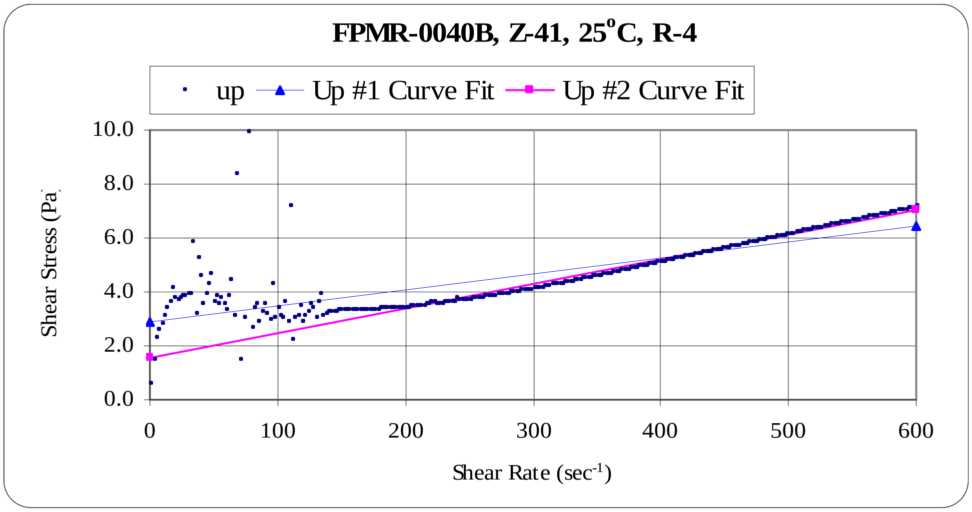


Figure 8.3d – Rheology Profile, 8% Na_2O SRAT Product

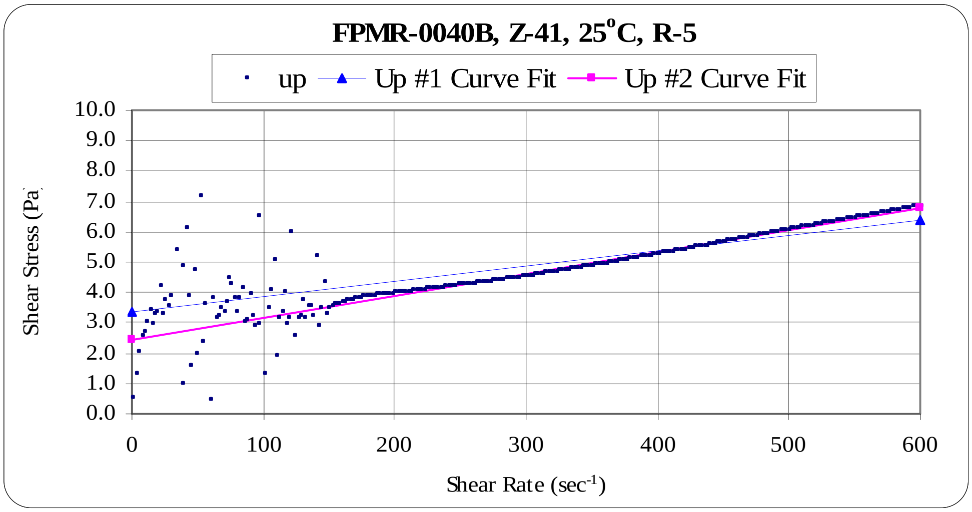
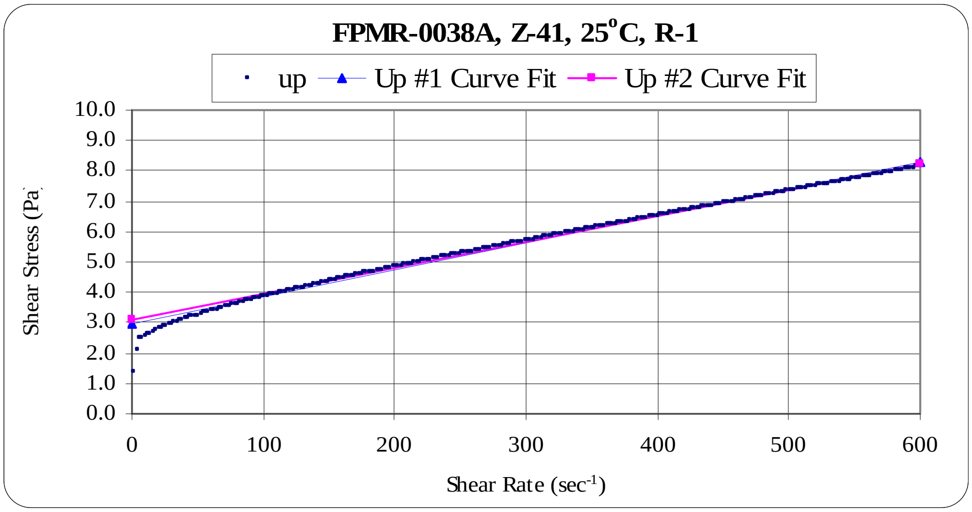


Figure 8.3e – Rheology Profile, 8% Na_2O SRAT Product



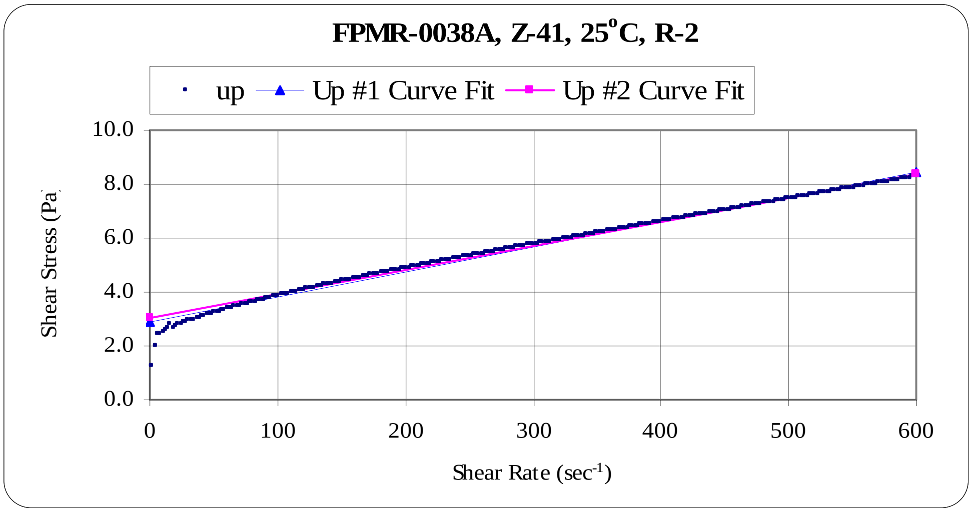


Figure 8.4b – Rheology Profile, 12% Na_2O Sludge

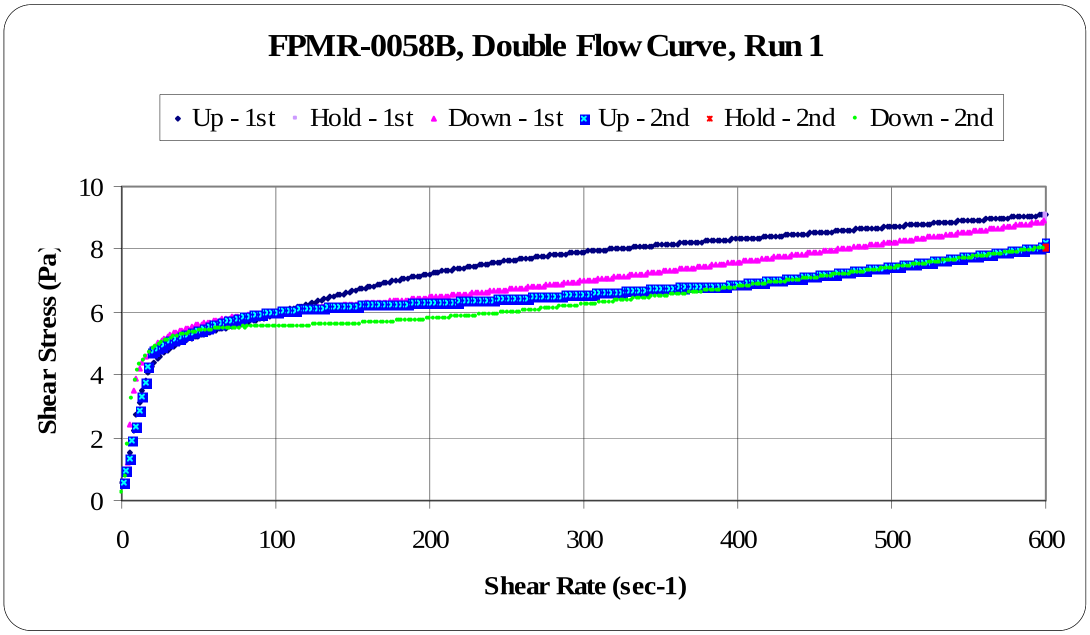


Figure 8.4c – Rheology Profile, 12% Na_2O SRAT Product

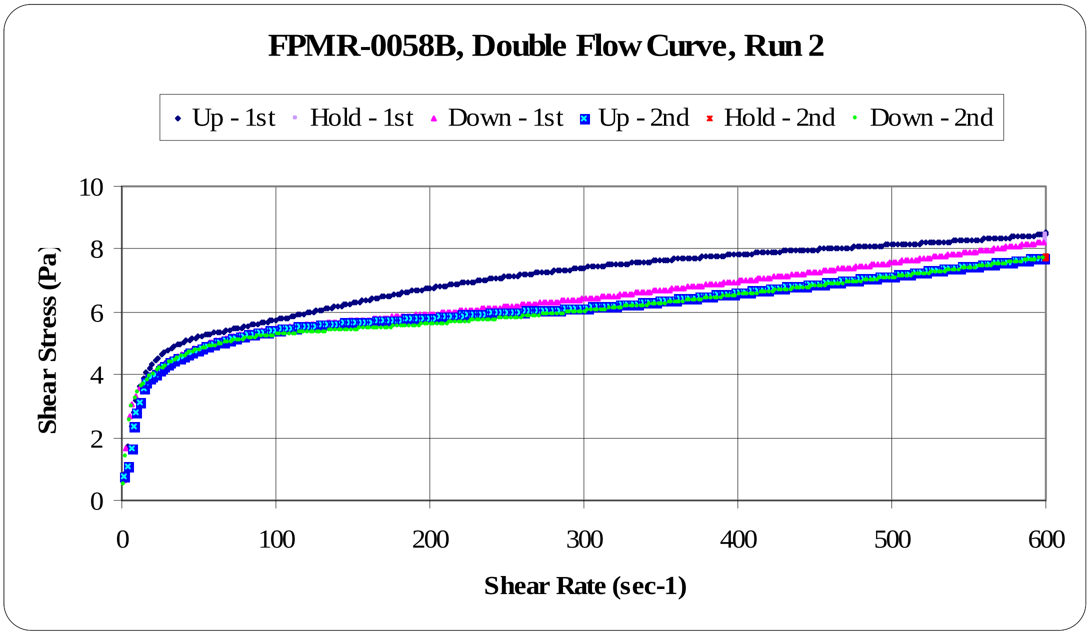


Figure 8.4d – Rheology Profile, 12% Na_2O SRAT Product

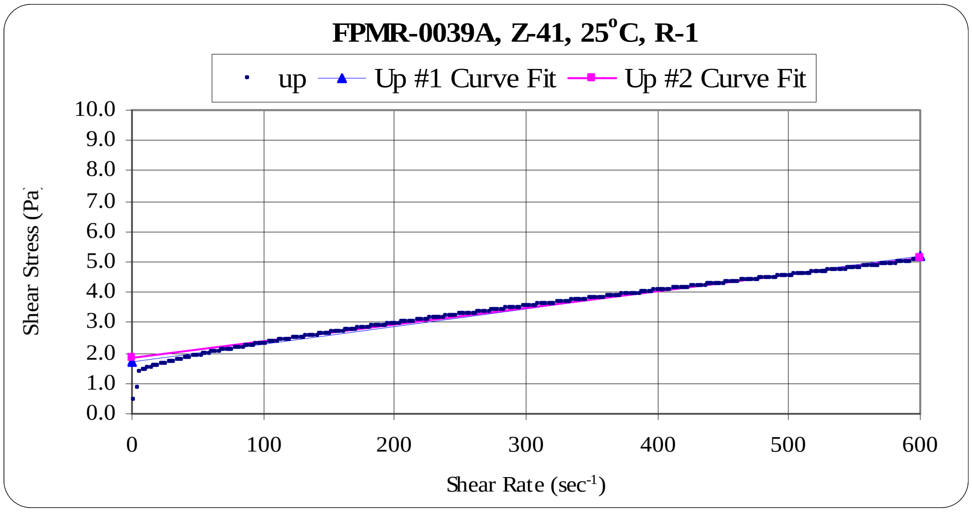


Figure 8.5a – Rheology Profile, 16% Na_2O Sludge

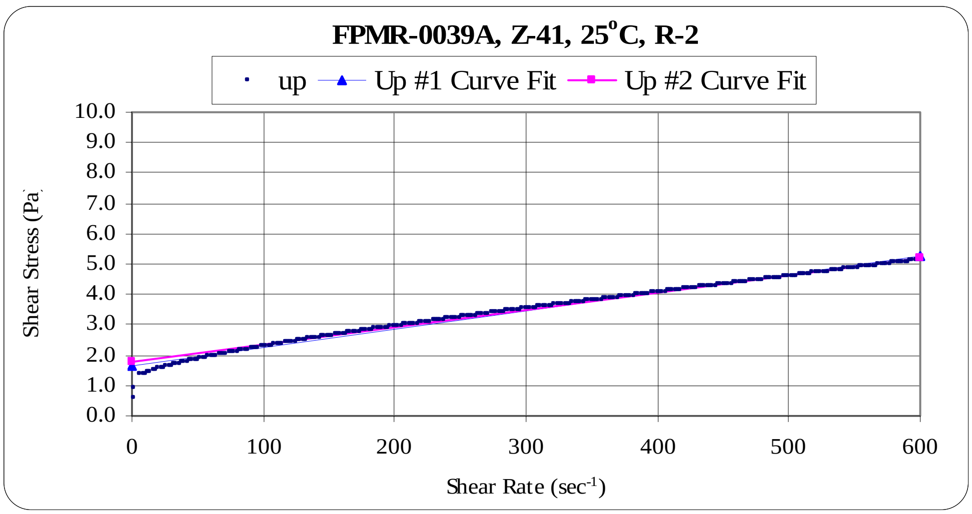


Figure 8.5b – Rheology Profile, 16% Na_2O Sludge

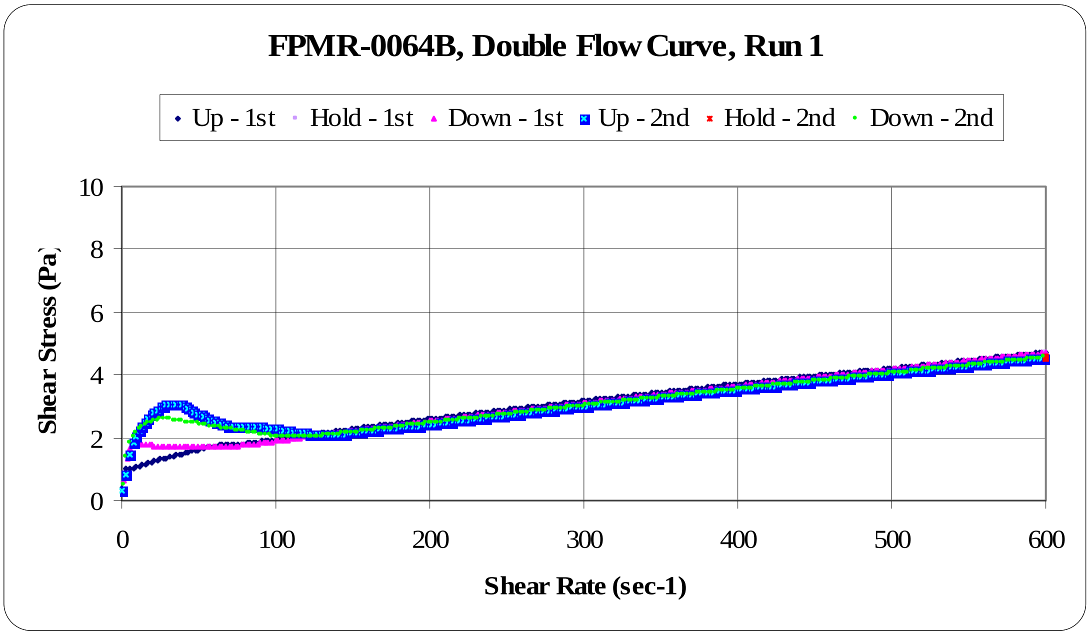


Figure 8.5a – Rheology Profile, 16% Na_2O SRAT Product

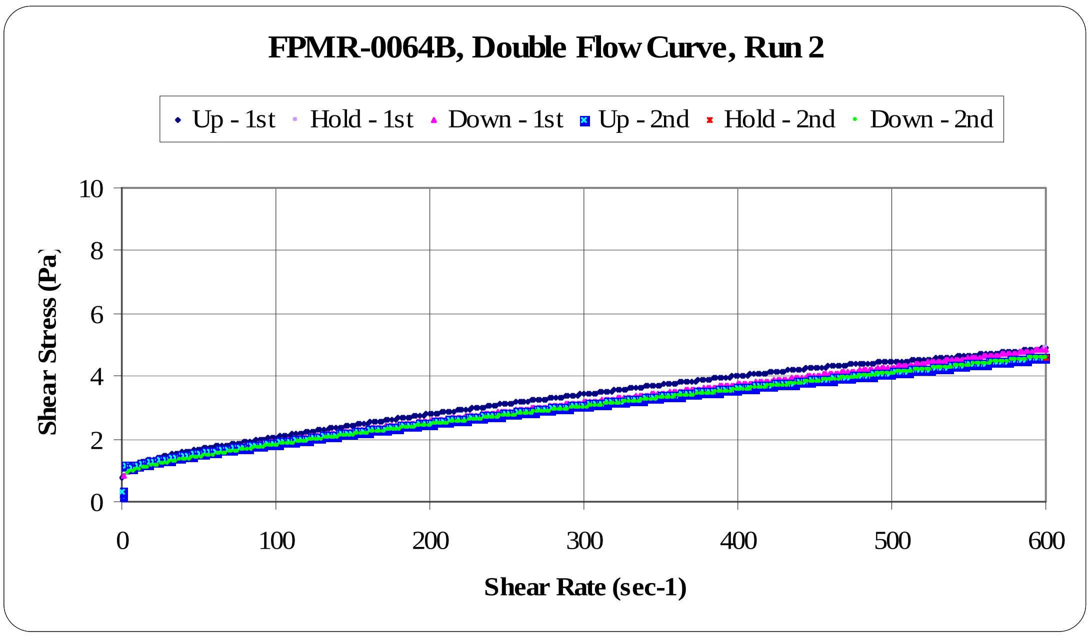


Figure 8.5b – Rheology Profile, 16% Na_2O SRAT Product

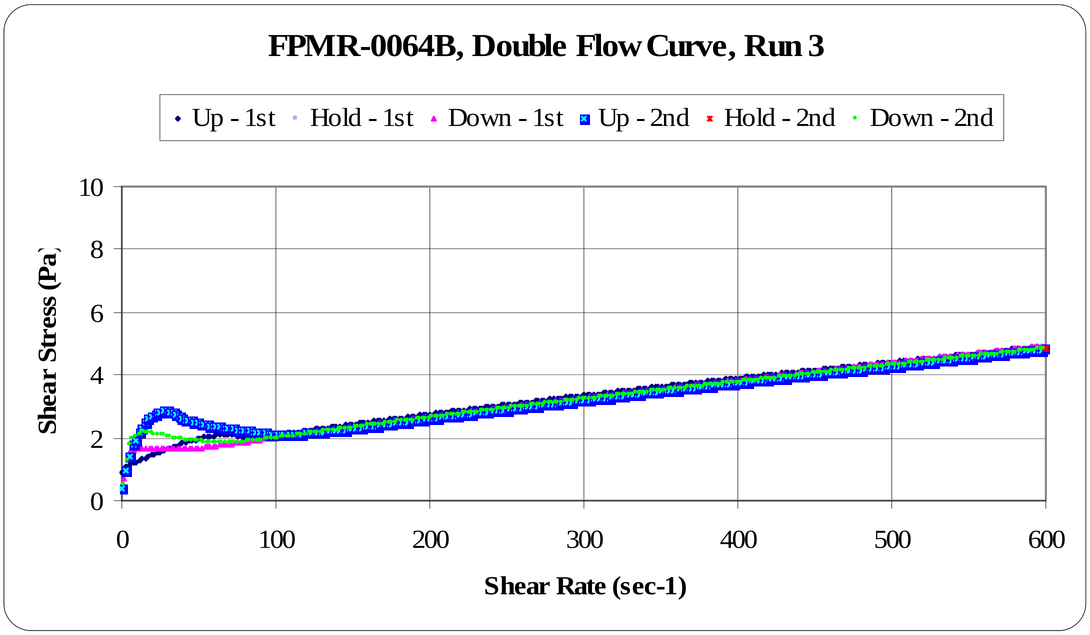


Figure 8.5c – Rheology Profile, 16% Na_2O SRAT Product

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