

DEMONSTRATION OF THE GLYCOLIC- FORMIC FLOWSHEET IN THE SRNL SHIELDED CELLS USING ACTUAL WASTE

D. P. Lambert
J. M. Pareizs
D. R. Click

November 2011

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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Printed in the United States of America

**Prepared For
U.S. Department of Energy**

SRNL-STI-2011-00622

Revision 0

**Key Words: DWPF, Sludge,
Glycolic Acid, Formic Acid,
Flowsheet**

Retention: Permanent

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REVIEWS AND APPROVALS

AUTHORS:

D. P. Lambert, Process Technology Programs Date

J. M. Pareizs, Process Technology Programs Date

D. R. Click, Analytical Development Date

TECHNICAL REVIEWER:

M. E. Stone, Process Technology Programs Date

J. D. Newell, Process Technology Programs Date

APPROVERS:

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

S. L. Marra, Manager, Environmental & Chemical Process Technology Research Programs Date

J. E. Occhipinti, Manager, Date
Waste Solidification Engineering

EXECUTIVE SUMMARY

The first run utilizing the Glycolic-Formic-Nitric Acid Flowsheet (referred to as the Glycolic-Formic Flowsheet throughout this report) with actual waste was demonstrated in Shielded Cells Run 13 (SC-13). The Glycolic-Formic Flowsheet utilizes 4 moles of glycolic acid per mole of formic acid mixture (the reducing acid) to replace the formic acid used in the DWPF Sludge Receipt & Adjustment Tank (SRAT) processing. Nitric acid, an oxidizing acid, is still also used. Other than the change in acids, there are no differences in the flowsheets. The testing was completed with a Tank 51 Sludge Batch 5 (SB5) slurry sample that was available. Some conclusions include:

- No significant issues with processing were noted. SC-13 SRAT cycle processing of Tank 51 –SB5 sludge met DWPF qualification criteria (nitrite was destroyed, mercury was removed, and hydrogen generation was below DWPF limits) with 12 hours of reflux.
- SSC-13 processing was comparable to the respective simulant work. The comparable simulant run (GF6 – 100% Koopman acid stoichiometry and 1.45 mol/L of acid added⁸ to a SB6 simulant) produced a peak hydrogen of 0.013 lb/hr DWPF-Scale, higher than observed in the SC-13 SRAT cycle.

The Glycolic-Formic Flowsheet has the following advantages over the current DWPF flowsheet:

- The hydrogen generation is greatly reduced as free formic acid concentration is much lower in the Glycolic-Formic Flowsheet. No catalytic hydrogen was detected throughout processing. The hydrogen detection limit of the gas chromatograph (GC) is 0.001 volume %.
- The generation volume of carbon dioxide and nitrous oxide was approximately one-third what is typical in a baseline flowsheet run. This will reduce emissions and has the potential to lower foaming in the SRAT.
- Each mole of glycolic acid has two moles of carbon. To produce a melter feed with a balanced REDOX (as measured by $\text{Fe}^{2+}/\Sigma\text{Fe}$), this allows the use of more nitric acid and less reducing acid.
- Since little formic acid is used, there is little acid consumed during processing. This keeps the slurry at approximately the same pH throughout processing and avoids the high pH melter feeds that can be very thick and tacky.
- The Glycolic-Formic Flowsheet is able to dissolve a large fraction of the insoluble metals in the sludge. This is a combination of the chelating ability of glycolic acid and the use of more nitric acid. This makes the resulting slurry more fluid and allows more concentration (i.e., higher total solids) of the SRAT product without exceeding DWPF rheological limits.
- With lower hydrogen generation levels, the flowsheet offers a much larger acid window for processing than the current flowsheet.
- The glassware, pH probe, agitator and thermocouple post-testing were very clean compared to baseline flowsheet runs. The Glycolic-Formic Flowsheet's ability to dissolve metals is likely leading to fewer deposits and less scale build-up during processing.

In addition to being the first demonstration of the Glycolic-Formic Flowsheet with radioactive sludge, a prime focus was the determination of solubility of iron and fissile isotopes during processing. The use of iron as a neutron poison relies on its insolubility to prevent a criticality if mixing is lost. No Slurry Mix Evaporator (SME) cycle was performed because the preparation of the pH 3, pH 2, and pH 1 SRAT products consumed virtually the entire SRAT product. During

SRAT processing, the iron was very insoluble, with a peak solubility of 2.2% at a pH of 1. It should be noted that a pH of less than 4 is not expected during normal processing. The data also indicated that a fraction of the fissile components in the slurry became soluble.

- The soluble Pu-239 in solution had a concentration of 18.5 mg/kg (0.0427 g $^{239}\text{Pu}(\text{NO}_3)_4/\text{L}$, 0.58% of the 7.3 g/L limit (ANS Standard for nuclear criticality safety).
- The Fe/Pu-239 ratio in the slurry was 383:1 at pH 1, greater than the 160:1 Fe:Pu-239 safe weight ratio. The Mn/Pu-239 ratio in the slurry was 90.3:1 at pH 1, greater than the 29:1 Mn:Pu-239 safe weight ratio.

Although the run was a successful demonstration utilizing the Glycolic-Formic Acid Flowsheet for DWPF SRAT processing of actual waste, no SME cycle was completed. As a result, it is recommended that:

- SRNL complete a SRAT/SME cycle using actual waste to demonstrate the entire flowsheet. Utilize the SME product to produce glass and measure resulting glass Reduction/Oxidation Potential (REDOX) potential.
- Continue developing and testing of the Glycolic-Formic Flowsheet to eliminate formic acid. This testing should be completed before the testing of the full Chemical Process Cycle (CPC) with radioactive slurry so the flowsheet to be used is demonstrated, which would reduce the number of radioactive demonstrations required. The use of a single acid would simplify acid procurement and processing in DWPF by reducing the hazards associated with formic acid.

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LIST OF ACRONYMS

ACTL	Aiken County Technologies Laboratory
AD	Analytical Development
ASP	Analytical Study Plan
CPC	Chemical Process Cell
DSA)	Documented Safety Analysis
DWPF	Defense Waste Processing Facility
FAVC	Formic Acid Vent Condenser
GC	Gas Chromatograph
HLW	High Level Waste
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
LDL	Less than Detectable Limit
LIMS	Laboratory Information Management System
MWWT	Mercury Water Wash Tank
NIST	National Institute of Standards and Testing
NM	Not Measured
PSAL	Process Science Analytical Laboratory
PSE	Process Science and Engineering Section
QA	Quality Assurance
REDOX	REDuction / OXidation potential
SB5	Sludge Batch 5
SB6	Sludge Batch 6
SC-7	Shielded Cells Run 7
SC-13	Shielded Cells Run 13
SEE	Systems Engineering Evaluation
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRS	Savanna River Site
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TS	Total Solids
TT&QAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
WAC	Waste Acceptance Criteria
WAPS	Waste Acceptance Product Specification

1.0 INTRODUCTION AND BACKGROUND

Glycolic acid was effective at dissolving many metals, including iron, during processing with simulants. Criticality constraints take credit for the insolubility of iron during processing to prevent criticality of fissile materials. Testing with actual waste was needed to determine the extent of iron and fissile isotope dissolution during Chemical Process Cell (CPC) processing.

The Alternate Reductant Project was initiated by the Savannah River Remediation (SRR) Company to explore options for the replacement of the nitric-formic flowsheet used for the CPC at the Defense Waste Processing Facility (DWPF). The goals of the Alternate Reductant Project are to reduce CPC cycle time, increase mass throughput of the facility, and reduce operational hazards. In order to achieve these goals, several different reductants were considered during initial evaluations conducted by Savannah River National Laboratory (SRNL). After review of the reductants by SRR, SRNL, and Energy Solutions (ES) Vitreous State Laboratory (VSL), two flowsheets were further developed in parallel. The two flowsheet options included a nitric-formic-glycolic flowsheet, and a nitric-formic-sugar flowsheet.

As of July 2011, SRNL and ES/VSL have completed the initial flowsheet development work for the nitric-formic-glycolic flowsheet and nitric-formic-sugar flowsheet, respectively. On July 12th and July 13th, SRR conducted a Systems Engineering Evaluation (SEE)¹ to down select the alternate reductant flowsheet. The SEE team selected the Formic-Glycolic Flowsheet for further development.²

Two risks were identified in SEE¹ for expedited research. The first risk is related to iron and plutonium solubility during the CPC process with respect to criticality. Currently, DWPF credits iron as a poison for the fissile components of the sludge. Due to the high iron solubility observed during the flowsheet demonstrations with simulants, it was necessary to determine if the plutonium in the radioactive sludge slurry demonstrated the same behavior. The second risk is related to potential downstream impacts of glycolate on Tank Farm processes. The downstream impacts will be evaluated by a separate research team.

Waste Solidification Engineering (WSE) has requested a radioactive demonstration of the Glycolic-Formic Flowsheet with radioactive sludge slurry be completed in the Shielded Cells Facility of the SRNL³. The Shielded Cells demonstration only included a Sludge Receipt and Adjustment Tank (SRAT) cycle, and not a Slurry Mix Evaporator (SME) cycle or the co-processing of salt products. Sludge Batch 5 (SB5) slurry was used for the demonstration since it was readily available, had been previously characterized, and was generally representative of sludges being processed in DWPF. This sample was never used in the planned Shielded Cells Run 7 (SC-7).

This report documents:

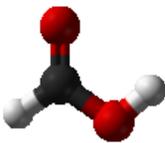
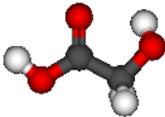
- General processing parameters collected during a SRAT cycle,
- The solubility of iron and fissile isotopes for SRAT products with added nitric and glycolic acid to produce solutions at pH 3, 2, and 1 as worst case SRAT products, and
- Rheology of the SRAT product

This work is controlled by a Task Technical and Quality Assurance Plan (TTQAP)⁴, and analyses are guided by an Analytical Study Plan⁵.

1.1 Development of the Glycolic-Formic Flowsheet

At the request of SRR, a study was completed evaluating 19 reductants to replace formic acid in CPC Processing⁶. In the DWPF CPC, formic acid is both an acid and a reductant. Six reductants that are not acids were evaluated in addition to thirteen reducing acids. Glycolic acid was chosen based on this study. Since this initial study, all testing has been completed with glycolic acid, which like formic acid, is also both an acid and a reductant. Note that for every mole of glycolic acid added, two moles of carbon are added as glycolic acid is a two carbon organic acid. Glycolic acid mixed with formic acid was recommended to ensure mercury would be reduced even if glycolic acid was not effective in reducing mercury.⁶ Acid properties⁷ for glycolic and formic acid are listed in Table 1-1.

Table 1-1. Acid Properties

Reductant	Formula	Acid pKa	Solubility 25 °C g/100 ml	Molarity	Typical Acid Concentration	Carbon Oxidation State	Structure
Formic Acid	CH ₂ O ₂	3.751	Miscible	23.6	90 wt %	2	
Glycolic Acid	C ₂ H ₄ O ₃	3.831	80	11.83	71 wt%	1	

Due to the significant maintenance required for the DWPF Gas Chromatographs (GC) and the potential for production of flammable quantities of hydrogen, reducing the amount of formic acid used in the CPC is one of the options being considered. Earlier work at Savannah River National Laboratory has shown that replacing formic acid in the existing nitric/formic acid flowsheet with an 80:20 molar blend of glycolic and formic acids has the potential to remove mercury in the SRAT without any significant catalytic hydrogen generation.⁶

1.2 Glycolic-Formic Flowsheet Testing with Simulants

A total of twenty-two CPC simulations including SRAT and some SME cycles were performed to develop the flowsheet.⁸ The first four tests were a baseline nitric-formic flowsheet, a baseline Glycolic-Formic Flowsheet, a run without mercury and a run with Actinide Removal Process (ARP) and Modular Caustic Side Solvent Extraction (MCU) streams added. The second set of four simulations included tests at varying acid stoichiometries to define the acid processing window and one test without any formic acid (nitric/glycolic acid only) to determine the effectiveness of glycolic acid as a reductant. No SME cycle was performed on the glycolic acid only flowsheet simulation⁹. Four tests were completed to produce products with a REDOX of 0-0.3 to demonstrate REDOX control for this flowsheet. Four tests were completed at glycolic-formic acid blends of 40-70% glycolic acid to determine the iron solubility at varying glycolic acid blends. Lastly, two runs were completed without mercury and noble metals to determine the conditions for producing melter feed with the baseline and Glycolic-Formic Flowsheets.

A total of eight shortened SRAT cycles were performed to produce sufficient melter feed to test the Glycolic-Formic Flowsheets in the VSL DM10 melter. Approximately 100 kg (25 gallons) of SRAT product was produced for each flowsheet. No noble metals or mercury were added to the sludge in these experiments. The same simulant was used for each of these runs.¹⁰

In addition a total of ten SRAT cycles were performed with a sludge matrix to test the Glycolic-Formic Flowsheet¹¹ over a wide sludge compositional range. Testing was done at both 100% and 150% stoichiometry. This was the first testing without the SB6H sludge simulant.

2.0 APPROACH

2.1 General Description of Analytical Methods

Analyses for this task used guidance of an Analytical Study Plan (ASP).⁵ Sample request forms were used for samples to be analyzed, and analyses followed the guidelines and means of sample control stated in the ASP for the task. A unique laboratory identification management system (LIMS) number was assigned to each sample for tracking purposes. Analyses were performed using approved analytical and Quality Assurance (QA) procedures.

Procedures for analysis of the simulant material can be found in Reference 12 and 13. For the radioactive materials, procedures and work instructions for density, percent solids, and supernate and slurry dilutions are also given in Reference. For the radioactive materials, procedures and work instructions for density, percent solids, and supernate and slurry dilutions are also given in Reference 3. Procedures for digestions and sample analyses are given in Reference 4. It should be noted that the anion AD method was modified to add the analysis of glycolate. This method could not distinguish between the acetate and glycolate peaks. The anion results report a concentration of glycolate but this could also be acetate as these peaks overlap. The anion tables contain a note to explain the results. The glycolate anion result in the SRAT product sample is about 70 times higher than the glycolate concentration in the SRAT receipt sample as expected with the addition of glycolic acid.

Total base for the radioactive slurry was determined by a direct in-cell titration of slurry and a titration by SRNL Analytical Development (SRNL-AD) using diluted slurry.

2.2 Chemical Process Cell (CPC) Processing (SRAT Cycle, SME Cycle)

The SRAT cycle was conducted following procedures in the L29 procedure manual.¹⁴ After the SRAT cycle was complete and samples taken, the pH was adjusted to 3, 2, and 1 and samples were taken after each pH adjustment. A summary of the SRAT experimental work is presented in Table 2-1 below.

Table 2-1. Summary of SRAT Processing

SRAT Cycle
<ul style="list-style-type: none">• Acid Calculation• Heating of SRAT Receipt to 93 °C• Addition of nitric, formic, and glycolic acids• Heat to boiling• Concentration (water removal) to a target wt% total solids• Reflux for 12 hours• Cooldown• Sample• Add glycolic/formic acid to pH 3• Sample• Add nitric acid to pH 2• Sample• Add nitric acid to pH 1• Sample

Processing was performed using a vessel designed to process one liter of radioactive sludge. The SRAT rig was assembled and tested in the SRNL Shielded Cells Mockup area and placed into the Shielded Cells fully assembled. A detailed description of the SRAT rig and testing of the rigs can be found in Reference 15. The intent of the equipment is to functionally replicate the DWPF processing vessels. The glass kettle is used to replicate both the SRAT and the SME, and it is connected to the SRAT Condenser and the Mercury Water Wash Tank (MWWT). Because the DWPF Formic Acid Vent Condenser (FAVC) does not directly impact SRAT and SME chemistry, it is not included in SRNL Shielded Cells CPC processing. Instead, an electronic ice cube is used to cool offgas to approximately 20 °C below ambient to remove excess water before the gas reaches the gas chromatograph for characterization. The Slurry Mix Evaporator Condensate Tank (SMECT) is represented by a sampling bottle that is used to remove condensate through the MWWT. For the purposes of this report, the condensers and wash tank are referred to as the offgas components. A sketch of the experimental setup is given as Figure 2-1.

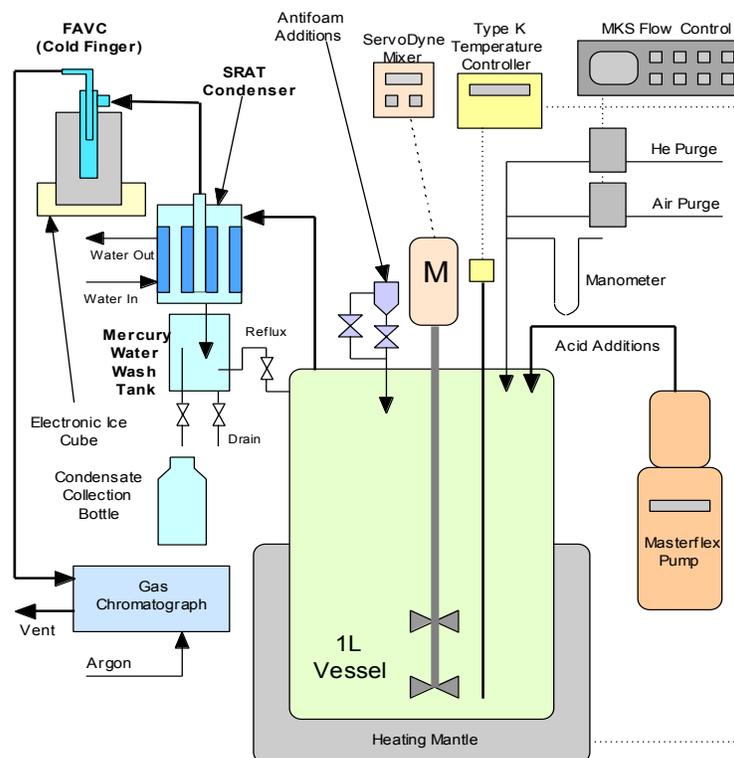


Figure 2-1. Schematic of SRAT Equipment Set-Up

SRAT processing parameters are summarized in Appendix A. In-line instrumentation was used to measure concentrations in the offgas system of the SRAT vessel and included helium, hydrogen, oxygen, nitrogen, nitrous oxide, and carbon dioxide. Helium was introduced at a concentration of 0.5% of the total air purge as an inert tracer gas, so that total amounts of generated gas and peak generation rates could be calculated. During the runs, the kettle was monitored to observe reactions that were occurring to include foaming, air entrainment, rheology changes, loss of heat transfer capabilities, and offgas carryover. Observations were recorded in a laboratory notebook¹⁶ and are discussed in Section 3.0.

Concentrated nitric acid (50-wt%), and a blend of one mole of formic acid (90 wt%) to four moles of glycolic acid (71 wt.%) were used to acidify the sludge and perform neutralization and reduction reactions during processing. The amounts of acid to add for each run were determined using the existing DWPF acid addition equation¹⁷. The split of the acid was determined using the REDOX equation currently being used in DWPF processing¹⁸ with the addition of a glycolic acid term. To account for the reactions and anion destructions that occur during processing, assumptions about nitrite destruction, nitrite to nitrate conversion, and formate and glycolate destruction were made for each run. The values used for each run are provided in Section 3.0.

SRAT processing included the dewater time in boiling plus an additional time of reflux to simulate DWPF processing conditions. The SRAT condenser was maintained at 25 °C during the run, while the electronic ice cube condenser remained below 10 °C.

2.3 Rheology

Rheological properties were determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. Heating/cooling of the cup/sample/bob is through the cup holder. The shear stress is determined from the torque measurement and is independent of rheological properties. Conditions that impact the measured torque are; slip (material does not properly adhere to the rotor or cup), phase separation (buildup of liquid layer on rotor), sedimentation (particles settling out of the shearing zone), homogeneous sample (void of air), lack of sample (gap not filled), excess sample (primarily impacts rheologically thin fluids), completely filling up the void below the bob (air buffer that is now filled with fluid) and Taylor vortices. The first five items yield lower stresses and the last three add additional stresses.

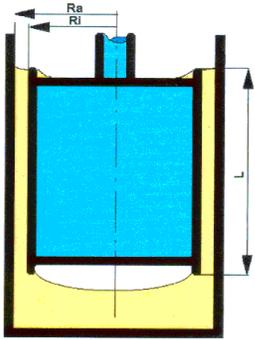
The shear rate is geometrically determined using the equations of change (continuity & motion) and is that for a Newtonian fluid. This assumption also assumes that the flow field is fully developed and the flow is laminar. The shear rate can be calculated for non-Newtonian fluid using the measured data and fitting this data to the rheological model or corrected as recommended by Darby¹². In either case, for shear thinning non-Newtonian fluids, typical of Savannah River Site (SRS) sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a thinner fluid. The bob typically used for measuring tank sludge or SRAT product is the MV I rotor. The shape, dimensions, and geometric constants for the MV I rotor are provided in Table 2-2.

Prior to performing the measurements, the rotors and cups are inspected for physical damage. The torque/speed sensors and temperature bath are verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard, using the MV I rotor. The resulting flow curves are then fitted as a Newtonian fluid and this calculated viscosity must be within $\pm 10\%$ of the reported NIST viscosity at a given temperature for the system to be considered functionally operable. A N10 oil standard was used to verify system operability prior to the sludge measurements. The flow curves for the sludge are fitted to the down curves using the Bingham Plastic rheological model, equation¹⁹, where τ is the measured stress (Pa), τ_0 is the Bingham Plastic yield stress (Pa), μ_p is the plastic viscosity (Pa·sec), and $\dot{\gamma}$ is the measured shear rate (sec^{-1}).

During all these measurements, typically the sample remained in the cup for the 2nd measurement, due to the sample available. If thixotropic properties or unique flow behavior were obvious on the first sample, efforts we made to perform additional measurements by reloading the sample.

$$\tau = \tau_0 + \mu_p \dot{\gamma} \quad [1]$$

Table 2-2. MV I Rotor Specifications and Flow Curve Program

Rotor Design	Dimensions and Flow Curve Program	
	Rotor Type	MV I
	Rotor radius - R_i (mm)	20.04
	Cup Radius - R_a (mm)	21.0
	Height of rotor - L (mm)	60
	Sample Volume (cm^3) minimum	40
	A factor (Pa/%torque)	3.22
	M factor ($\text{s}^{-1}/\% \text{RPM}$)	11.7
	Shear rate range (s^{-1})	0 – 600
	Ramp up time (min)	5
	Hold time (min)	1
	Ramp down time (min)	5

3.0 RESULTS AND DISCUSSION

3.1 SRAT Receipt Characterization and Acid Addition Calculations

The DWPF SRAT process relies upon use of the acid calculation to estimate the required acid necessary to complete reactions. This calculation relies upon measured analytical inputs. Errors in these measurements can result in too little acid being added resulting in incomplete reactions or too much acid being added resulting in excess hydrogen being generated. Therefore, the SB5 Tank 51 sample had been extensively characterized in 2008. The results of the Shielded Cells Run 13 (SC-13) SRAT receipt characterization are provided in Table 3-1. The sample was also reanalyzed after the run and the results are summarized in the "Post Run Analyses" column in Table 3-1. Table 3-2 gives the additional acid calculation inputs (e.g., formate destruction).

Table 3-1. Characterization Results and Acid Calculation Inputs of the SB5 Tank 51 (SC-13 SRAT Receipt) Sample with Added Neptunium

Measurement	2008 Analyses	Post Run Analyses	Units
Fresh Sludge Mass without trim chemicals	1,140.0	1,127.36	g slurry
Fresh Sludge Weight % Total Solids	17.10	16.49	wt%
Fresh Sludge Weight % Calcined Solids	13.40	12.70	wt%
Fresh Sludge Weight % Insoluble Solids	10.97	10.72	wt%
Fresh Sludge Density	1.140	1.125	kg / L slurry
Fresh Sludge Supernate density	1.059	1.056	kg / L supernate
Fresh Sludge Nitrite	7,460	11,200	mg/kg slurry
Fresh Sludge Nitrate	14,500	8,760	mg/kg slurry
Fresh Sludge Sulfate (mg/kg)	N/A	453	mg/kg slurry
Fresh Sludge Chloride (mg/kg)	N/A	<161	mg/kg slurry
Fresh Sludge Phosphate (mg/kg)	N/A	<161	mg/kg slurry
Fresh Sludge Oxalate	N/A	444	mg/kg slurry
Fresh Sludge Glycolate/Acetate#	N/A	<161	mg/kg slurry
Fresh Sludge Slurry TIC (treated as carbonate)	1,011	1,400	mg/kg slurry
Fresh Supernate TIC (treated as carbonate)	1,202	1,230	mg/L supernate
Fresh Sludge Hydroxide (Base Equivalents) pH = 7	0.5548	0.5680	Eq Moles Base/L slurry
Fresh Sludge Manganese (% of Calcined Solids)	4.844	4.33	wt % calcined basis
Fresh Sludge Mercury (% of Total Solids)	1.94	NM	wt% dry basis
Fresh Sludge Magnesium (% of Calcined Solids)	0.727	0.709	wt % calcined basis
Fresh Sludge Sodium (% of Calcined Solids)	19.2	18.8	wt % calcined basis
Fresh Sludge Potassium (% of Calcined Solids)	0.079	<0.065	wt % calcined basis
Fresh Sludge Calcium (% of Calcined Solids)	1.53	1.56	wt % calcined basis
Fresh Sludge Strontium (% of Calcined Solids)	N/A	<0.064	wt % calcined basis
Fresh Sludge Nickel (% of Calcined Solids)	2.71	2.95	wt % calcined basis
Fresh Sludge Supernate Manganese	N/A	<1.021	mg/L supernate

The method used did not separate the glycolate and acetate peaks. <161 mg/kg glycolate-acetate in the SRAT receipt sample may have been <161 mg/kg glycolate or <155 mg/kg acetate or any combination of the two that would give a peak of <161 mg/kg glycolate).

The SRAT cycle acid calculation utilizes the amount of nitrite, mercury, manganese, carbonate, and base equivalents to calculate the stoichiometric amount of acid to be added. Nitric acid, glycolic acid and formic acid amounts are calculated based on the applied stoichiometric factor and the ratio needed to achieve the target glass REDOX ($\text{Fe}^{+2}/\Sigma\text{Fe}$). The equation for prediction of glass REDOX utilizes estimates of the amount of glycolate, formate, oxalate, nitrate, nitrite, manganese, and total solids in the SME product. A modified REDOX equation was used in these calculations with the addition of the glycolate term with a factor of 6.

The REDOX prediction equation used in this study with an added term for glycolate is¹⁸:

$$\text{Fe}^{2+}/\text{Fe} = 0.2358 + 0.1999 * (2[\text{F}] + 4[\text{C}] + 6[\text{G}] + 4[\text{OT}] + 5[\text{N}] - 5[\text{Mn}])/45/\text{T}$$

Where

- [F] = formate (mol/kg feed)
- [C] = coal (carbon) (mol/kg feed)
- [G] = glycolate (mol/kg feed)
- [OT] = oxalate Total (soluble and insoluble) (mol/kg feed)
- [N] = nitrate + nitrite (mol/kg feed)
- [Mn] = manganese (mol/kg feed)
- T = Total Solids, wt %

The estimation of the final concentration for the anions requires assumptions to be made concerning how these species will react during the SRAT and SME cycles. Glycolate, formate and oxalate are destroyed by reactions with oxidizing species, and formate is destroyed by noble metal-catalyzed reactions. Nitrite is typically consumed during acid additions, but can react to form different species including nitrate. Inputs for the SC-13 run are based on simulant studies.⁸ The acid calculation inputs and assumptions are shown in Table 3-1 and Table 3-2.

Table 3-2. Acid Calculation Inputs of the of the SB5 Tank 51 (SC-13 SRAT Receipt) Sample with Added Neptunium

Processing Assumptions	Predicted	Units
Conversion of Nitrite to Nitrate in SRAT Cycle	0.00	gmol NO_3^- /100 gmol NO_2^-
Destruction of Nitrite in SRAT and SME cycle	100	% of starting nitrite destroyed
Destruction of Formic acid charged in SRAT	90	% formate converted to CO_2 etc.
Destruction of Glycolic acid charged in SRAT	0.00	% glycolate converted to CO_2 etc.
Conversion of Glycolic acid to Oxalate	3.00	% glycolate converted to C_2O_4
Destruction of Oxalate charged	0.00	% of total oxalate destroyed
Percent Acid in Excess Stoichiometric Ratio	110	%
SRAT Product Target Solids	25.	%
Nitric Acid Molarity	10.200	Molar
Formic Acid Molarity	23.537	Molar
Glycolic Acid Molarity	11.826	Molar
DWPF Nitric Acid addition Rate	2.0	gallons per minute
DWPF Formic Acid addition Rate	2.0	gallons per minute
REDOX Target	0.100	$\text{Fe}^{+2} / \Sigma\text{Fe}$

The acid calculation determines the values for a large number of processing parameters as well as the amount of acid to be used. Acid requirements (per liter of SRAT receipt) are shown in Table 3-3 with all values tabulated in Appendix A.

Table 3-3. SRAT Cycle Acid Requirements

Run ID	SC-13
Koopman Stoichiometry. Acid, moles/L	1.21
Hsu Stoichiometry. Acid, moles/L	1.07
Actual Acid Added, moles/L	1.334
Ratio of Reducing Acid to Total Acid	0.600
Moles glycolic/moles total acid in glycolic/formic acid Blend	0.800

3.2 CPC Processing Results

The run was completed as planned, with the exception that the pH probe had a loose connection in the cells leading to erroneous readings throughout the SRAT cycle, and the acid pump did not work and had to be replaced. The pH probe was replaced, and a different electrical connection was used after the SRAT cycle to acidify the SRAT product to a pH of 3, 2 and 1. The acid pump was replaced with a single speed pump so the addition rate could not be controlled. The acid pump added acid at 0.20 ml/min, instead of the 0.34 ml/min planned for nitric acid and the 0.69 ml/min planned for the glycolic-formic acid blend. DWPF acid adds both nitric and formic acid at 2 gallons/min, which is scaled to 0.34 mL/min for Shielded Cell's experiments. This extended the time of the acid addition and especially the time for the water flush of the acid line extended the time at 93°C. DWPF typically adds formic acid at 1 gallon per minute (0.17 ml/min scaled), so the addition rate of the glycolic-formic acid blend was higher than the formic acid is typically added in DWPF. The higher addition rates were planned to add the glycolic-formic acid blend at the same molar flowrate as the design basis for formic acid, which would cut the glycolic-formic acid blend addition time in half. The slower addition of acid mainly lowers the offgas peaks and increases the time when the offgas is being produced. The main impact of this strategy is to minimize foam generation rate, which may minimize foam volume during acid addition.

One interesting note is the glassware was much cleaner in the Glycolic-Formic Flowsheet demonstration. For example, the original pH probe was very clean when removed (it was expected to be sludge coated and broken) as were the agitator and thermocouple. In addition, the glassware was clean enough that the cell wall could be seen through the vessel back "window".

3.2.1 SRAT Cycle Processing Observations

No significant processing problems occurred during the SC-13 SRAT cycle other than minimal foaming. No difficulties in mixing or heating the sludge slurry were experienced, but the agitation was increased from 400 rpm to 600 rpm compared to earlier shielded cells runs to maintain adequate mixing. The newest batch of antifoam (Lot# 111128-0613), based on the revised antifoam purchase specification was used. The following was the antifoam addition strategy:

- 200 ppm addition prior to starting the cycle
- 100 ppm addition between nitric and formic /glycolic acid additions
- 500 ppm addition after acid addition, prior to boiling
- 100 ppm addition every 8 hours thereafter

No apparent mercury collected in the MWWT. Black deposits were evident in the tubing between the condenser and MWWT. In simulant runs with higher chloride or iodine, there are more black deposits and less elemental mercury collected. No chloride was detected in the SRAT receipt sample. The

measured mercury in the SRAT receipt sample was 1.94 wt % (0.018 g-moles). No chloride was detected in any of the SRAT product supernate and slurry samples.

3.2.2 SRAT Cycle Sample Results

A sample was pulled at the conclusion of the SRAT cycle. The total solids, anions, and mercury analysis were performed. These results are presented in Table 3-4. As shown in the table, nitrite was adequately destroyed, and mercury was removed below 0.8 wt.% of total solids.

Table 3-4. SC-13 SRAT Product Characterization Results

	SRAT Product	Adjusted to pH 3	Adjusted to pH 2	Adjusted to pH 1
Wt % Total Solids (slurry basis)	24.30%	25.70%	25.70%	24.66%
Wt % Insoluble Solids (slurry basis)	13.07%	12.56%	12.71%	11.86%
Wt % Soluble Solids (slurry basis)	11.23%	13.14%	12.99%	12.80%
Wt % Supernate Solids (supernate basis)	14.84%	17.68%	17.48%	16.99%
Wt % Calcined Solids (slurry basis)	14.81%	14.70%	14.26%	13.55%
Slurry Density (g/mL)	1.205	1.215	1.218	1.221
Supernate Density (g/mL)	1.119	1.132	1.140	1.148
Fluoride	<328	<325	<321	<309
Glycolate (Acetate)#	42,600	48,300	46,600	45,500
Formate	3,540	4,620	4,120	3,730
Chloride	<164	<325	<321	<309
Nitrite	426	<325	<321	<309
Bromide	<164	<325	<321	<309
Nitrate	52,890	68,230	82,330	98,200
Phosphate	<164	<325	<321	<309
Sulfate	426	397	393	407
Oxalate	434	1,190	1,190	1,810
Mercury (wt % of total solids)	0.681	NM	NM	NM

The method used did not separate the glycolate and acetate peaks.

3.2.3 SRAT Cycle Anion Destruction and Conversion

Inputs to the acid calculation include formate, glycolate and nitrite destruction and conversion of nitrite to nitrate. Presented in Table 3-5 is a comparison between these assumed values (based on simulant runs) and measured results. As can be seen in the table, glycolate destruction and nitrite to nitrate conversion were higher than predicted. This means that more nitrate and less glycolate were present in the SRAT product. The predicted REDOX of the SRAT product, based on anion measurements, was $0.05 \text{ Fe}^{+2}/\sum\text{Fe}$ instead of the targeted $0.10 \text{ Fe}^{+2}/\sum\text{Fe}$. The agreement between assumed and measured destruction is adequate as no simulant runs had been completed at the same acid stoichiometry, mercury or noble metals targets as is generally completed before a shielded cells demonstration.

Table 3-5. Assumed and Measured Anion Destruction and Conversion in the SC-13 SRAT Cycle

Anion Conversion	Assumed	Measured
Nitrite Destruction (%)	100	94.9
Formate Destruction (%)	90.0	90.2
Glycolate Destruction (%)	3.0	17.7
Nitrite to Nitrate Conversion (%)	0.0	35.2

3.2.4 SRAT Cycle Offgas Analysis

Offgas data for the SC-13 SRAT cycle are presented in the following figures. The peak generation rates (lb/hr DWPF-Scale) are summarized in Table 3-6. No catalytic hydrogen was detected throughout the SRAT cycle. The detection limit for hydrogen is 0.001 volume %. The N₂O and CO₂ generation rates are significantly lower than have been seen in nitric-formic flowsheet runs.

Table 3-6. Peak Offgas Generation in the SRAT Cycle, DWPF Scale

Gas	lb/hr
N₂O	3.63
CO₂	115
H₂*	<0.001

* Hydrogen detection limit approximately 0.001 volume %

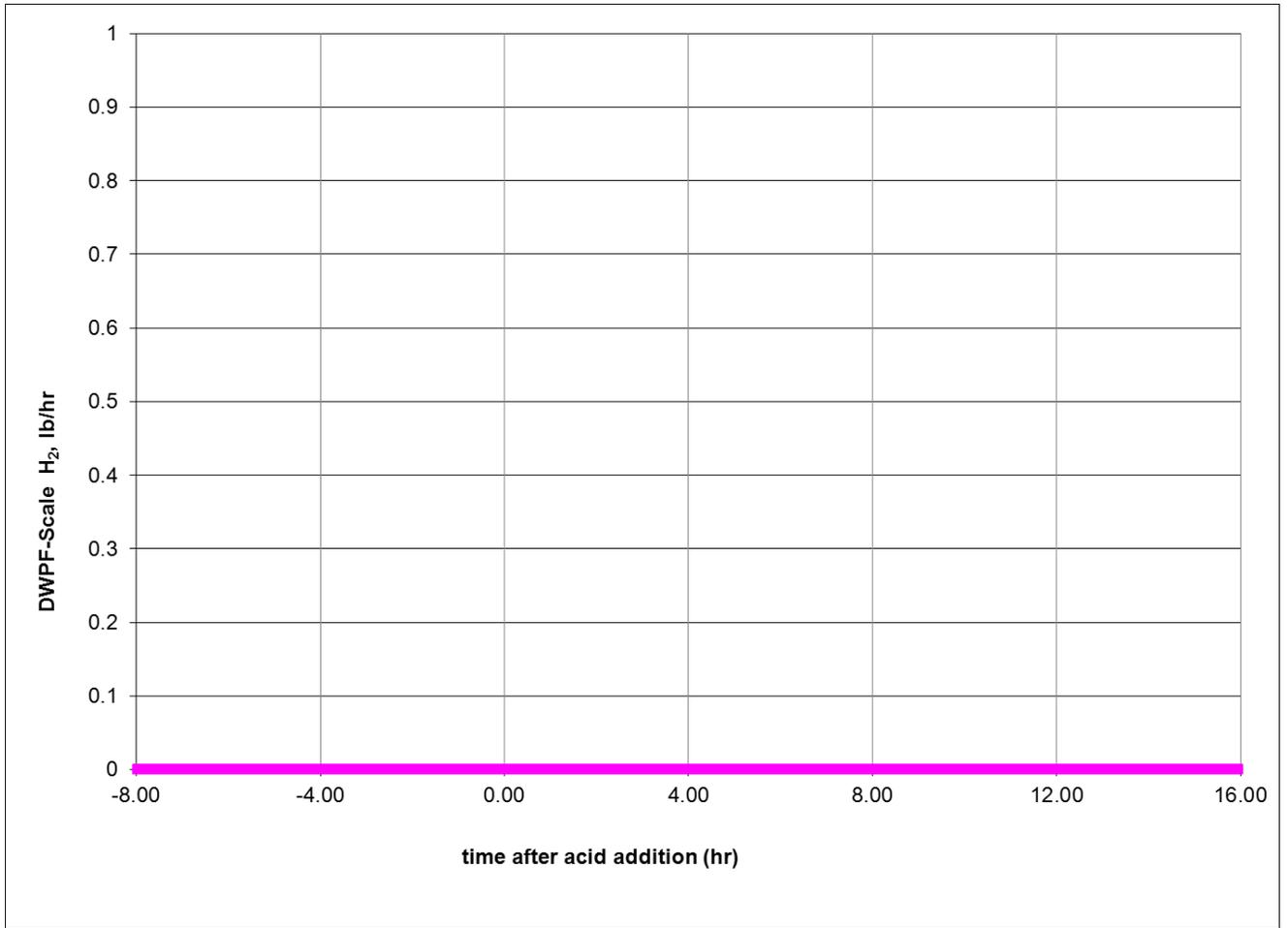


Figure 3-1. Hydrogen Generation during SC-13 SRAT Cycle

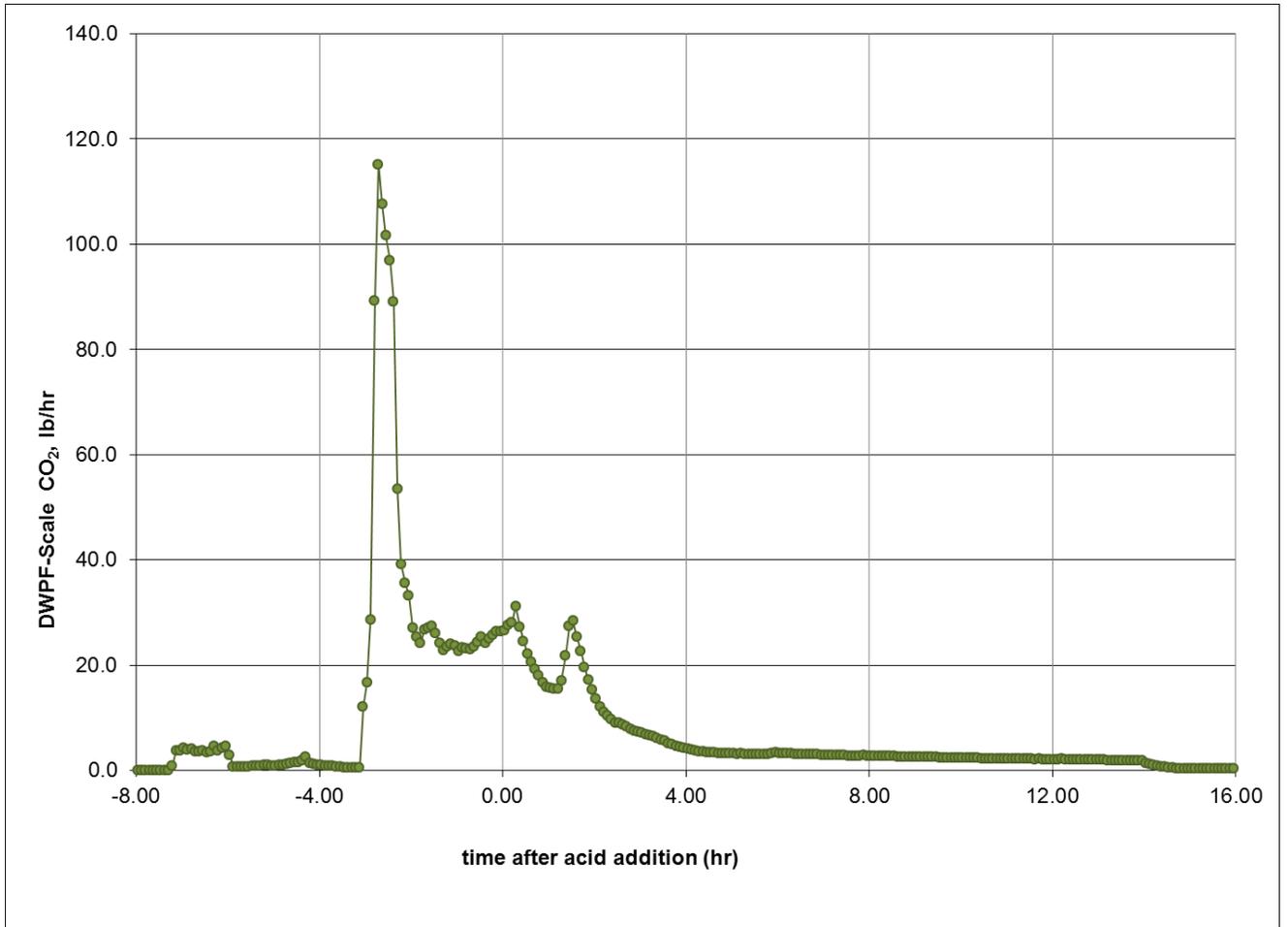


Figure 3-2. Carbon Dioxide Generation during SC-13 SRAT Cycle

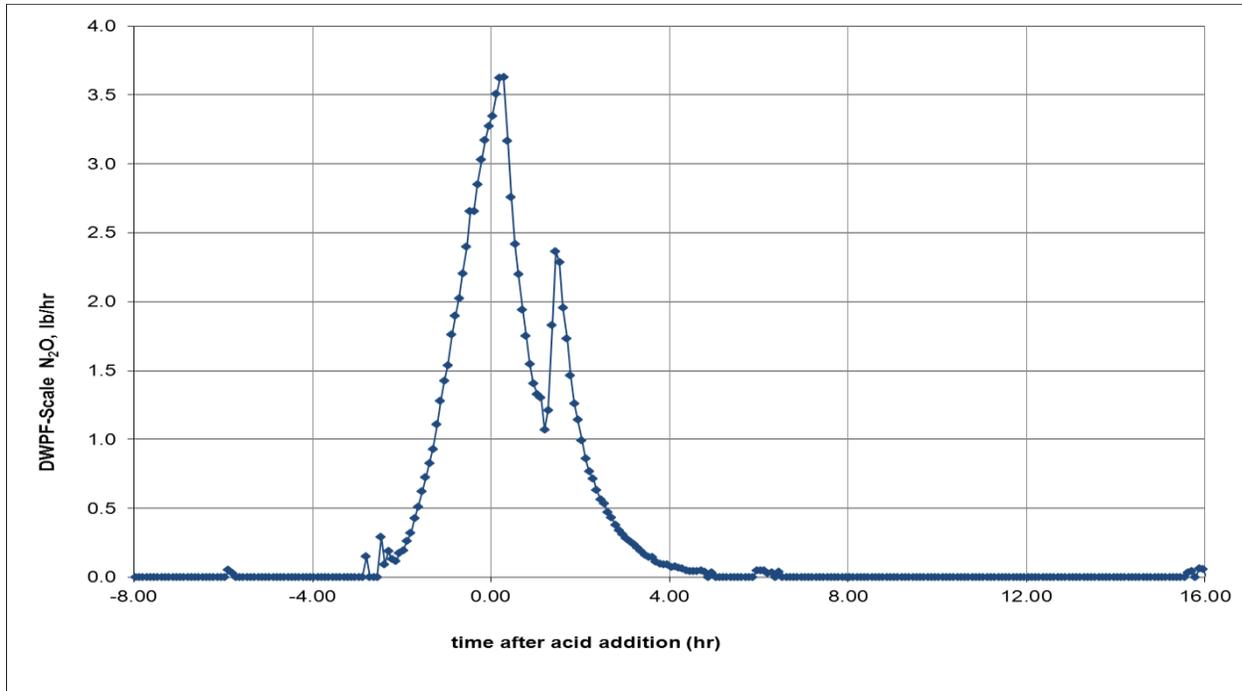


Figure 3-3. Nitrous Oxide Generation during SC-13 SRAT Cycle

3.3 Iron and Fissile Solubility

DWPF credits iron as a poison for the fissile components of the sludge. Iron and the fissile isotopes such as Pu-239 are typically both fairly insoluble throughout CPC processing. However, in testing with simulated sludge and the formic-glycolic flowsheet, a significant fraction of the iron became soluble in CPC processing, increasing from 3% at an acid stoichiometry of 100% (Simulant Run GF6) to 65% at an acid stoichiometry of 200% (Simulant Run GF7) as summarized in Figure 3-4. Due to the iron solubility observed during the flowsheet demonstrations with sludge simulant, it was necessary to determine if the Fe and fissile isotopes in the actual waste demonstrated the same solubility behavior.

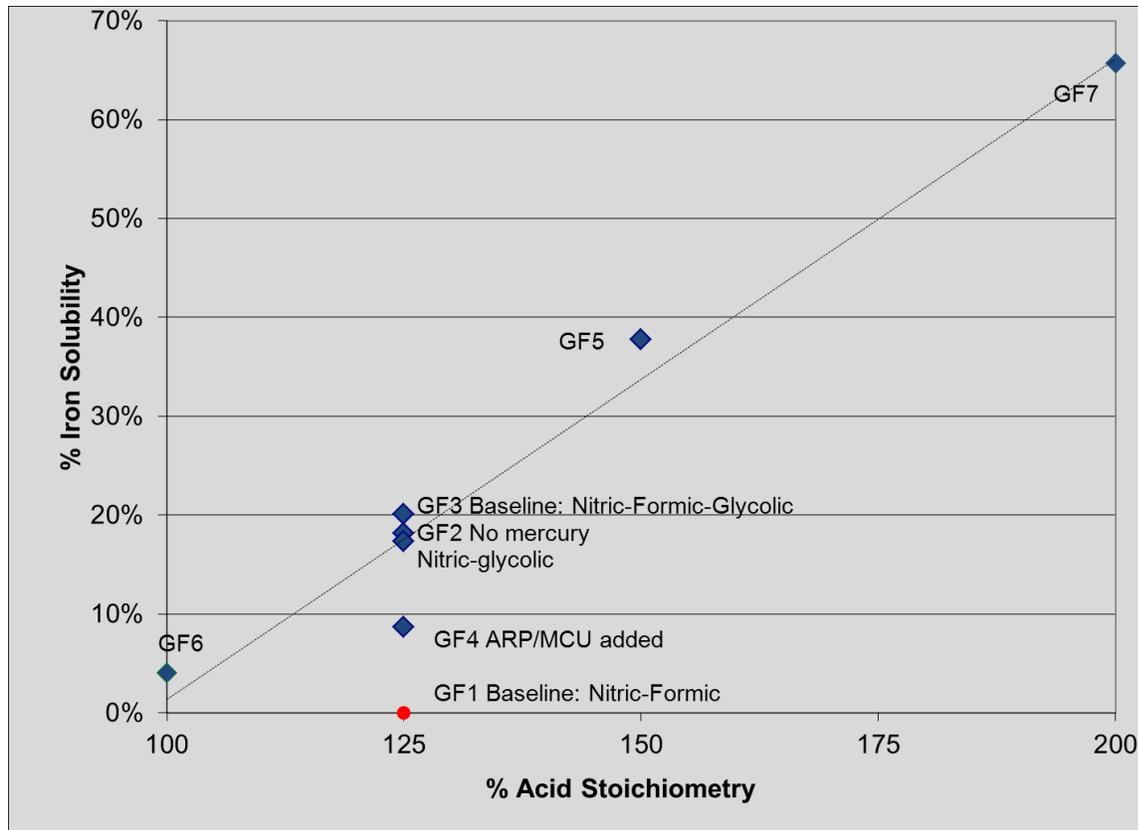


Figure 3-4. Iron Solubility as a Function of Acid Stoichiometry in Simulant Runs

Four SRAT product samples were prepared by the addition of both nitric and glycolic-formic acid. The SRAT product had a pH of 6. After all SRAT product samples were collected, the glycolic/formic acid mixture was added to the remaining SRAT product until the pH remained steady (both acids act as buffers, typically at a pH of 3.75 for formic acid and 3.83 for glycolic acid). The addition of 50 wt% nitric was used to bring the pH down to the final pH targets of 3, 2 and 1. Note that the SRAT product pH stayed at pH 4.2 until nitric acid was added. Therefore during normal processing, the pH will not drop below 4 so these solutions are very conservative from a pH standpoint.

The solubility of a number of elements in the SRAT products at pH 6, 3, 2 and 1 are summarized in Table 3-7. Iron has a very low solubility in the SRAT receipt sample (2.2% in the SRAT product at pH 1) in the Glycolic-Formic Flowsheet as is the case with the nitric-formic-glycolic flowsheet. The fissile masses had significantly higher solubilities than iron in all testing. For example, the mass 239 solubility peak was 27.6%, higher than iron. The solubility of the masses from ICP-MS data are summarized in Table 3-8.

Table 3-7. Solubility of Elements Measured by ICP-AES in SRAT Product

Element	SRAT Receipt	% Solubility of SRAT Product			
		pH 6	pH 3	pH 2	pH 1
Al	10.1	3.36	7.89	10.2	10.3
Fe	0.0024	0.600	1.56	2.20	2.22
Gd	2.38	32.3	66.6	67.5	67.8
Mg	0.0287	55.8	63.6	65.6	65.1
Mn	0.0166	46.3	55.7	56.9	57.1
Na	87.1	92.9	93.6	96.1	95.2
Ni	0.209	7.56	10.70	12.1	12.0
S	69.5	78.6	LDL*	50.5	54.5
U	0.706	30.5	71.9	71.6	78.1

*S was less than the detectable limits in slurry samples

Table 3-8. Solubility of Mass as Measured by ICP-MS in SRAT Product

Element or Isotope	SRAT Receipt	% Solubility of SRAT Product			
		pH 6	pH 3	pH 2	pH 1
La (139)	LDL	14.6	30.5	35.0	33.9
Nd (143-46, 148,150)	LDL	16.9	34.5	36.1	32.8
Gd (155-58, 160)	LDL	30.7	65.8	63.7	51.4
U (235,238)	5.73E-3	30.3	81.2	67.3	51.5
Pu (239)	LDL	7.65	27.6	27.4	18.9
Np (237)	LDL	40.8	62.5	61.2	69.1

The mass ratio of iron to Pu-239 is summarized in Table 3-9. A number of other ratios important to criticality are also summarized in Table 3-9. For example, the minimum Fe:Pu-239 ratio was 383, approximately 2.4 times the current DWPF limit. The minimum Mn:Pu-239 ratio was 90.3, greater than the 29:1 limit.²⁰ Also, the concentration of ²³⁹Pu(NO₃)₄ in the supernate was far below the 7.3 g/L limit specified by the ANS regulation for nuclear criticality safety in operations with fissionable material outside reactors²¹.

In the future, sludge batches enriched in uranium (i.e., greater than 0.93 wt% in U-235) may be processed at DWPF. Currently, sludge batches are blended less than or equal to 0.93 wt% U-235 to ensure the uranium stays subcritical during DWPF processing. If a sludge batch were prepared that exceeded the current 0.93 wt% Documented Safety Analysis (DSA) / Waste Acceptance Criteria (WAC) requirement, then crediting another neutron poison may be desired. Manganese could serve as a neutron poison for the U-235 due to their similar chemistries under caustic and acidic conditions. It should be noted in Table 3-9 that all SRAT Products (pH of 6 to 1) exceeded the Mn:Pu-239 safe weight ratio of 29:1.²⁰

Table 3-9. Iron, Manganese and Pu-239 Concentration and Ratio (Slurry Basis unless noted)

Element or Isotope or Ratio	Limit	SRAT Receipt	SRAT Product			
			pH 6	pH 3	pH 2	pH 1
Slurry Iron, mg/kg	NA	23,800	28,100	26,600	25,200	25,000
Filtrate Iron, mg/kg	NA	0.581	169	415	551	555
Slurry Manganese, mg/kg	NA	5,505	6,643	6,360	6,140	5,893
Slurry Pu-239, mg/kg	NA	55.1	60.9	59.3	62.6	65.3
Filtrate Pu-239, mg/kg	NA	0.0265	15.1	18.5	16.4	6.79
Fe:Pu-239 ratio in slurry	≥ 160	432	462	449	402	383
Fe:Pu-239 ratio in filtrate	NA	21.9	11.2	22.4	33.6	81.8
Mn:Pu-239 ratio in slurry	≥ 29	100	109	107	98.0	90.3
²³⁹ Pu(NO ₃) ₄ g /L filtrate basis	≤ 7.3	$<6.40E-5$	0.0122	0.0432	0.0493	0.0327

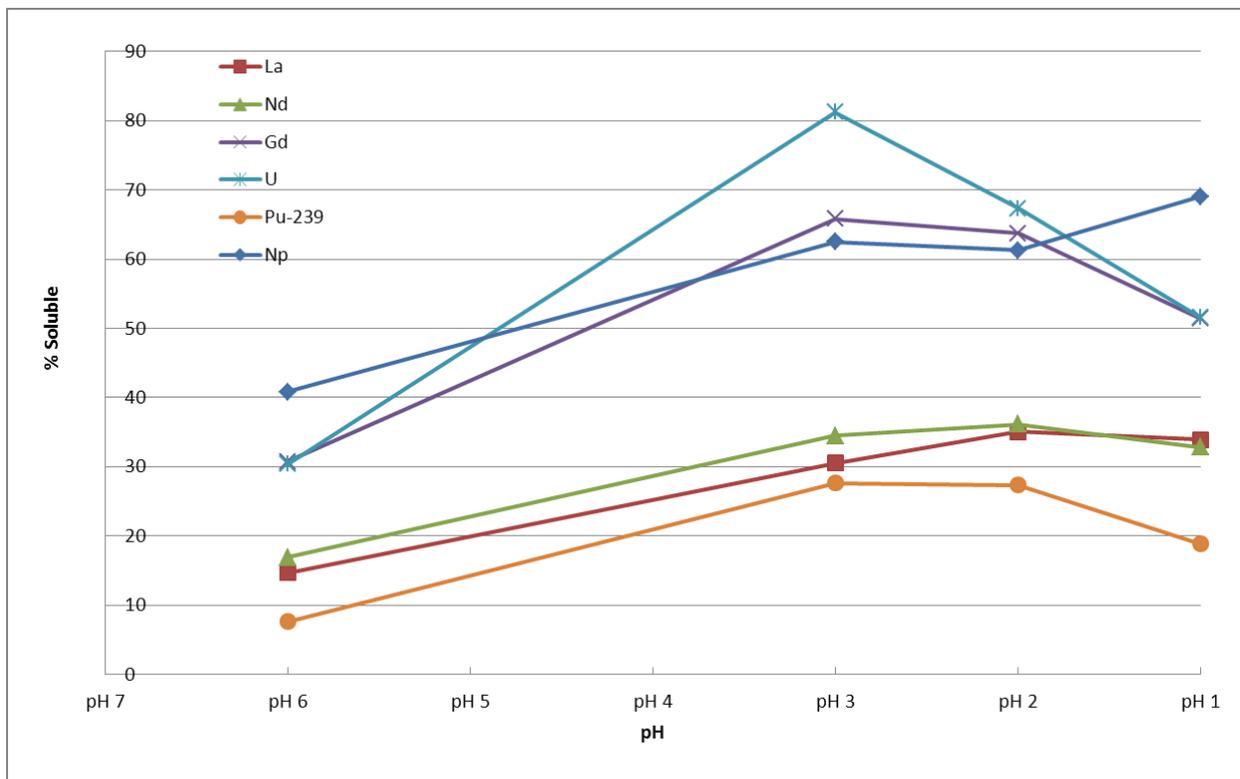


Figure 3-5. Element Solubility from ICP-MS as a Function of pH

3.4 Rheology

The rheological properties, solids and density data are summarized in Table 3-10. Rheology was measured on two of the SRAT products. There was no yield stress so the slurry was Newtonian.

Table 3-10. Rheological Properties of As-Received and SRAT Product

Sample	Yield Stress (Pa)	Plastic Viscosity (CP)	Wt% Total Solids	Wt% Insoluble Solids	Slurry Density (g/ml)
SC-13 SRAT Product	0.20	6.70	24.3	13.07	1.205
SC-13 SRAT Product pH 2	0	6.13	25.7	12.71	1.218

3.5 REDOX

No SME cycle was completed during this testing. Insufficient SRAT product was available to measure REDOX. It is recommended that a SRAT/SME cycle demonstration should be completed using actual waste to demonstrate the entire flowsheet. Utilize the SME product to produce glass and measure resulting glass REDOX potential.

4.0 CONCLUSIONS

The first run utilizing the Glycolic-Formic-Nitric Acid Flowsheet (referred to as the Glycolic-Formic Flowsheet throughout this report) with actual waste was demonstrated in Shielded Cells Run 13 (SC-13). The Glycolic-Formic Flowsheet utilizes 4 moles of glycolic acid per mole of formic acid mixture (the reducing acid) to replace the formic acid used in the DWPF Sludge Receipt & Adjustment Tank (SRAT) processing. Nitric acid, an oxidizing acid, is still also used. Other than the change in acids, there are no differences in the flowsheets. The testing was completed with a Tank 51 Sludge Batch 5 (SB5) slurry sample that was available. Some conclusions include:

- No significant issues with processing were noted. SC-13 SRAT cycle processing of Tank 51 –SB5 sludge met DWPF qualification criteria (nitrite was destroyed, mercury was removed, and hydrogen generation was below DWPF limits) with 12 hours of reflux.
- SC-13 processing was comparable to the respective simulant work. The comparable simulant run (GF6 – 100% Koopman acid stoichiometry and 1.45 mol/L of acid added to a SB6 simulant) produced a peak hydrogen of 0.013 lb/hr DWPF-Scale, higher than observed in the SC-13 SRAT cycle.

The Glycolic-Formic Flowsheet has the following advantages over the current DWPF flowsheet:

- The hydrogen generation is greatly reduced as free formic acid concentration is much lower in the Glycolic-Formic Flowsheet. No catalytic hydrogen was detected throughout processing. The GC hydrogen detection limit is >0.001 volume %.
- The generation volume of carbon dioxide and nitrous oxide was approximately one-third what is typical in a baseline flowsheet run. This will reduce emissions and has the potential to lower foaming in the SRAT.
- Each mole of glycolic acid has two moles of carbon. To produce a melter feed with a balanced REDOX (as measured by $\text{Fe}^{2+}/\Sigma\text{Fe}$), this allows the use of more nitric acid and less reducing acid.
- Since little formic acid is used, there is little acid consumed during processing. This keeps the slurry at approximately the same pH throughout processing and avoids the high pH melter feeds that can be very thick and tacky.
- The Glycolic-Formic Flowsheet is able to dissolve a large fraction of the insoluble metals in the sludge. This is a combination of the chelating ability of glycolic acid and the use of more nitric acid. This makes the resulting slurry more fluid and allows more concentration (i.e., higher total solids) of the SRAT product without exceeding DWPF rheological limits.
- With lower hydrogen generation levels, the flowsheet offers a much larger acid window for processing than the current flowsheet.
- The glassware, pH probe, agitator and thermocouple post-testing were very clean compared to baseline flowsheet runs. The Glycolic-Formic Flowsheet's ability to dissolve metals is likely leading to fewer deposits and less scale build-up during processing.

In addition to being the first demonstration of the Glycolic-Formic Flowsheet with radioactive sludge, a prime focus was the determination of solubility of iron and fissile isotopes during processing. The use of iron as a neutron poison relies on its insolubility to prevent a criticality if mixing is lost. No Slurry Mix Evaporator (SME) cycle was performed because the preparation of the pH 3, pH 2, and pH 1 SRAT products consumed virtually the entire SRAT product. During SRAT processing, the iron was very insoluble, with a peak solubility of 2.2% at a pH of 1. It should be noted that a pH of less than 4 is not expected during normal processing. The data also indicated that a fraction of the fissile components in the sludge slurry became soluble.

- The soluble Pu-239 in solution had a concentration of 18.5 mg/kg (0.0427 g $^{239}\text{Pu}(\text{NO}_3)_4/\text{L}$, 0.58% of the 7.3 g/L limit (ANS Standard for nuclear criticality safety).
- The Fe/Pu-239 ratio in the slurry was 383:1 at pH 1, greater than the 160:1 Fe:Pu-239 safe weight ratio.
- The Mn/Pu-239 ratio in the slurry was 90.3:1 at pH 1, greater than the 29:1 Mn:Pu-239 safe weight ratio.

5.0 RECOMMENDATIONS

Although the run was a successful demonstration utilizing the Glycolic-Formic Acid Flowsheet for DWPF SRAT processing of actual waste, no SME cycle was completed. As a result, it is recommended that:

- SRNL complete a SRAT/SME cycle using actual waste to demonstrate the entire flowsheet. Utilize the SME product to produce glass and measure resulting glass Reduction/Oxidation Potential (REDOX) potential.
- Continue developing and testing of the Glycolic-Formic Flowsheet to eliminate formic acid. This testing should be completed before the testing of the full Chemical Process Cycle (CPC) with radioactive slurry so the flowsheet to be used is demonstrated, which would reduce the number of radioactive demonstrations required. The use of a single acid would simplify acid procurement and processing in DWPF by reducing the hazards associated with formic acid.

6.0 ACKNOWLEDGEMENTS

This run was planned quickly in order to expedite the testing needed to answer a potential Achilles' heel for this flowsheet, namely the solubility of iron during processing. A number of people did extensive work in preparing for the testing and in analyzing the samples. This run generated four times the samples compared to a typical SRAT cycle so the work by the shielded cells technicians and AD are truly appreciated. Some of those deserving special acknowledgement include:

Rita Sullivan and Dee Wheeler were the shielded cells technicians that led these experiments. Their expertise and experience was invaluable in making these experiments successful. In addition, Carl Black and Linda Bush did an excellent job in supporting this experiment.

David Healy prepared most of the bottles that were used for samples during this run and his help is much appreciated.

Monica Jenkins, Jane Howard and Rita Sullivan prepared the samples in the cells and transferred them to AD. In addition, they completed digestions as needed for these samples, along with density and solids measurements. This work took them several weeks to complete after the run was finished.

Curtis Johnson (now retired), Mark Jones, Boyd Wiedenman, and Tom White, together with their technicians were responsible for analyzing the anion, ICP-AES and ICP-MS samples. Their help was much appreciated, especially their effort to get it completed in FY11. This was the first time that AD has analyzed samples for glycolate using the IC.

John Pareizs took the night shift for this run and was responsible for the majority of the processing, as the day shift spent their time fixing problems and did little actual processing. His oversight of this run is appreciated.

Curt Sexton assisted the shielded cells technicians by providing a tool to allow the installation of an O-ring on the pH probe in cell. If I hadn't seen this, I wouldn't have believed it could be done. Curt's experience and "let's figure out how do this" attitude at the end of the day was much appreciated.

7.0 REFERENCES

- ¹ Wagon, T.J., *Defense Waste Processing Facility Alternate Reductant, Systems Engineering Study*, Technical Report G-AES-S-00003, Revision 0, Savannah River Site: Aiken, SC 29808 (2011).
- ² Fellingner, T. L.,; Occhipinti, J. E., *Follow-up Action for Senior Review Group (SRG) – Alternative Reductant Flowsheet Selection*, Interoffice Memorandum SRR-WSE-2011-00184, Rev. 0, Savannah River Site: Aiken, SC 29808 (2011).
- ³ Fellingner, T. L., *Nitric/Formic/Glycolic Flowsheet Tasks – Determination of Fe/Pu Solubility and Impact to Solvent Extraction*, Technical Task Request HLW-DWPF-TTR-2011-0025, Rev. 1, Savannah River Site: Aiken, SC 29808 (2011).
- ⁴ Lambert, D. P.; Pareizs, J. M.; *Task Technical and Quality Assurance Plan for Demonstration of the Glycolic-Formic Flowsheet in the Shielded Cells*, Technical Report WSRC-RP-2011-01281, Rev. 0, Savannah River Site: Aiken, SC 29808 (2011).
- ⁵ Lambert, D. P.; Pareizs, J. M.; Click, D. R. *Analytical Study Plan for Demonstration of the Glycolic-Formic Flowsheet in the Shielded Cells*, Technical Report WSRC-RP-2011-01282, Rev. 0, Savannah River Site: Aiken, SC 29808 (2011).
- ⁶ Pickenheim, B.R., Stone, M.E., Peeler, D.K., *Selection and Preliminary Evaluation of Alternative Reductants for SRAT Processing*, SRNL-STI-2009-00120, Savannah River National Laboratory, Aiken, SC, June 29808 (2009).
- ⁷ Pickenheim, B.R., Bibler, N.E., Lambert, D.P., Hay, M.S., *Glycolic-Formic Acid Flowsheet Development*, SRNL-STI-2010-00523, Rev 0, Savannah River National Laboratory, Aiken, SC, 29808 (2010).
- ⁸ Lambert, D.P., Pickenheim, B.R., Stone, M.E., Newell, J.D., Best, D.R., *Glycolic - Formic Acid Flowsheet Final Report for Downselection Decision*, Technical Report SRNL-STI-2010-00523, Rev 1, Savannah River Site: Aiken, SC 29808 (2011).
- ⁹ Pickenheim, B.R., M.E. Stone, J.D.M.E., Newell, *Glycolic-Formic Acid Flowsheet Development, Glycolic Acid Physical Properties, Impurities, and Radiation Effects Assessment*, SRNL-STI-2010-0052300314, Rev 01, Savannah River National Laboratory, Aiken, SC, November 29808 (2010).
- ¹⁰ Choi, A.S., *Melter Off-Gas Flammability Assessment for DWPF Alternate Reductant Flowsheet Options*, Technical Report SRNL-STI-2011-00321, Revision 0, Savannah River Site: Aiken, SC 29808 (2011).
- ¹¹ Lambert, D.P., Koopman, D.C., *Glycolic-Formic Acid Flowsheet Sludge Matrix Study*, Technical Report SRNL-STI-2011-00275, Revision 0, Savannah River National Laboratory, Aiken, SC, 29808 (2011).
- ¹² *Analytical Development Procedures Manual*; Manual L16; Savannah River National Laboratory: Aiken, SC 29808.

- ¹³ *Analytical Development Procedures Manual*; Manual L16; Savannah River National Laboratory: Aiken, SC 29808.
- ¹⁴ *Process Science and Engineering Section Procedure Manual*; Manual L29; Savannah River National Lab: Aiken, SC 29808.
- ¹⁵ Stone, M. E. *Lab-Scale CPC Equipment Set-up*; Interoffice Memorandum SRNL-PSE-2006-00074; Rev, 2, Savannah River National Laboratory: Aiken, SC 29808. (2010).
- ¹⁶ *SC13 Shielded Cells Demo of Glycolic-Formic Flowsheet*; Notebook WSRC-NB-2011-00118; Savannah River National Laboratory: Aiken, SC, 29808 (2011.).
- ¹⁷ Lambert, D. P. *Acid Calculation Spreadsheet for DWPF Simulations, Revision 1*; Inter-Office Memorandum SRNL-PSE-2006-00173; Savannah River National Laboratory: Aiken, SC, 29808 (2006.).
- ¹⁸ Jantzen, C.M.; Zamecnik, J. R.; Koopman, D. C.; Herman, C. C.; Pickett, J. B. *Electron Equivalents Model for Controlling Reduction-Oxidation (REDOX) Equilibrium during High Level Waste (HLW) Vitrification*, Technical Report WSRC-TR-2003-00126, Savannah River Site, Aiken, SC 29808 (2003).
- ¹⁹ Darby, R., *Chemical Engineering Fluid Mechanics, 2nd edition*. Marcel Dekker: 2001.
- ²⁰ Murray, M. D., *Nuclear Criticality Safety Evaluation: H-Canyon Waste Stream Poisoning with Fe and Mn (U)*, N-NCS-H-00152, Revision 0, February 2003.
- ²¹ “*American National Standard for Nuclear Criticality Safety in Operations with Fissionable Material Outside Reactors*”, ANSI-ANS-8.1-1998, September 1998.

APPENDIX A. MASS BALANCES

Table A - 1. Acid Calculation

<i>SRNL SRAT Acid, Trim Chemical, Dewater and Redox Calc Revised:</i>		<i>3/12/2009</i>	
<i>Run Description:</i>		SC-13 110% 80:20 glycolic:formic ratio	
Sludge Feed Batch #		SB5 Qual SRNL washed Tk51	
SRAT Vessel Volume, L	4		
REDOX Equation (7 for Mn ⁺⁷ , otherwise assumes Mn ⁺⁴)	7		Enter 7 for newest redox equation
Acid Equation	2		Enter 1 for Hsu, 2 for Koopman, 3 for cation
Will ARP be added?	No		Yes
Will MCU be added?	No		No
Will there be a SME cycle?	No		
Fresh Sludge Slurry TIC (treated as carbonate)	1,011	1,401	mg/kg slurry
Fresh Supernate TIC (treated as carbonate)	1,202	1,228	mg/L supernate
Fresh Sludge Hydroxide (Base Equivalents) pH = 7	0.5548	0.5680	Equiv Moles Base/L slurry
Fresh Sludge Manganese (% of Calcined Solids)	4.844		wt % calcined basis
Fresh Sludge Mercury (% of Total Solids in untrimmed sludge)	1.9400	NM	wt% dry basis
Fresh Sludge Magnesium (% of Calcined Solids)	0.727		wt % calcined basis
Fresh Sludge Sodium (% of Calcined Solids)	19.200		wt % calcined basis
Fresh Sludge Potassium (% of Calcined Solids)	0.079		wt % calcined basis
Fresh Sludge Calcium (% of Calcined Solids)	1.530		wt % calcined basis
Fresh Sludge Nickel (% of Calcined Solids)	2.710		wt % calcined basis
Fresh Sludge Supernate manganese		0.053	mg/L supernate
Run #	SC-13	Units	
Table 2 -- SRAT Processing Assumptions, Run #	SC-13		
Conversion of Nitrite to Nitrate in SRAT Cycle	0.00	gmol NO ₃ ⁻ /100 gmol NO ₂ ⁻	

Run #	SC-13	Units
Destruction of Nitrite in SRAT and SME cycle	100.00	% of starting nitrite destroyed
Destruction of Formic acid charged in SRAT	90.00	% formate converted to CO ₂ etc.
Destruction of Glycolic acid charged in SRAT	0.00	% glycolate converted to CO ₂ etc.
Conversion of Glycolic acid to Oxalate	3.00	% glycolate converted to C2O4
Destruction of Oxalate charged	0.00	% of total oxalate destroyed
Percent Acid in Excess Stoichiometric Ratio	110.00	%
SRAT Product Target Solids	25.00	%
Nitric Acid Molarity	10.200	Molar
Formic Acid Molarity	23.537	Molar
Glycolic Acid Molarity	11.826	Molar
DWPF Nitric Acid addition Rate	2.0	gallons per minute
DWPF Formic Acid addition Rate	4.080	gallons per minute
REDOX Target	0.100	Fe ⁺² / ∑Fe
Total nitrite	0.185	gmol
Total Mn minus soluble Mn	0.135	gmol
Total carbonate	0.096	gmol
Total hydroxide	0.555	gmol
Total mercury	0.038	gmol
Total oxalate	0.000	gmol
Total grams of calcined oxides	152.760	g
Trim Chemicals Calculations	0.0000	
Fresh Sludge Calcine Factor (1100°C), g oxide/g dry solids (calculated)	0.7836	g/g
ARP calcine factor	0.0000	g/g
Total solids before trim addition	194.936 8	g
Total solids before trim less HgO, NaOxalate, coal)	190.85	g
Predicted total solids at target levels	194.936 8	g
Predicted total mass at target levels	1,270.00 00	g
Target Ag metal content in trimmed sludge	0.00000 0	total wt% dry basis
AgNO₃ to add (CF=0.682)	0.00000	g
Ag₂O calcined solids	0.00000	g
Water added with Ag	0.00000	g
Target wt% Hg dry basis	1.940	total wt% dry basis
Total HgO in fresh Sludge	4.083	g
Total HgO in trimmed Sludge	4.08341	g
HgO to add	0.00000	g
HgO calcined solids	0.00000	g
Water added with Hg	0.00000	g
Calculated total wt% Hg dry basis	1.9400	wt% dry basis
Target Pd metal content in trimmed sludge	0.0000	total wt% dry basis

Run #	SC-13	Units
Wt % Pd in reagent solution	15.2700	wt% in solution
Pd(NO ₃) ₂ *H ₂ O solution to add (CF=1.150 g metal oxide/g metal)	0.00000	g of solution
Pd(NO ₃) ₂ to add	0.00000	g
PdO calcined solids	0.00000	g
Water added with Pd	0.000	g
Target Rh metal content in trimmed sludge	0.0000	total wt% dry basis
Wt% Rh in reagent solution	4.93	wt% in solution
Rh(NO ₃) ₃ *2H ₂ O (CF=1.311g metal oxide/g metal)	0.0000	g of solution
Rh(NO ₃) ₃ to add	0.00000	g
Rh ₂ O ₃ calcined solids	0.00000	g
Water added with Rh	0.000	g
Target Ru metal content in trimmed sludge	0.0000	total wt% dry basis
Wt% Ru in RuCl ₃ reagent solids	41.74	wt% in solids
RuCl ₃ to add (CF=1.0)	0.0000	g solid
Target Cr metal content in trimmed sludge	0.0000	total wt% dry basis
Cr ₂ O ₃ to add	0.0000	g
Target Ba metal content in trimmed sludge	0.0000	total wt% dry basis
BaO to add	0.0000	g
Target Cd metal content in trimmed sludge	0.0000	total wt% dry basis
CdO to add	0.0000	g
Target Gd metal content in trimmed sludge	0.0000	total wt% dry basis
wt% Gd in reagent	34.8392	wt% in solution
Gd(NO ₃) ₃ *6H ₂ O to add	0.0000	g of reagent
Gd(NO ₃) ₂ to add	0.0000	g
Gd ₂ O ₃ calcined solids	0.0000	g
water added with Gd	0.0000	g
Target wt% Coal/carbon source in trimmed sludge, dry basis	0.00	total wt% dry basis
Total Coal in fresh Sludge	0.000	g
Total Coal in trimmed Sludge	0.000	g
Mass of Coal to add (CF =.08)	0.00	g
Calculated wt% coal after trim additions	0.00	wt%
Total solids after trim addition	194.94	g
Match of actual to predicted total solids mass	100.00 %	
Total Calcined solids after trim	152.76	g
Water added to dilute and/or rinse trim chemicals	130.0	g
Mass of trimmed sludge	1,270.00	g
Calculated wt% total solids in trimmed sludge	15.3	wt%
Sample mass of trimmed sludge	0.00	g
Mass of trimmed feeds reacted	1,270.00	g
Mass of equivalent sludge w/o ARP	1,140.00	g, used to calculate scaling factors, etc.

Run #	SC-13	Units
Sample removal ratio at start of ARP boil	1.000	
Sample removal ratio at start of SRAT	1.000	
Calcined solids at start of SRAT	152.8	g
Hsu Total Stoichiometric Acid required	1.0658	gmol
Koopman Minimum Stoichiometric Acid required	1.2125	gmol
Koopman Minimum Stoichiometric Acid required	1.2125	gmol
Cation Nominal Stoichiometric Acid required	1.6432	gmol
Cation Minimum Stoichiometric Acid required	1.4412	gmol
Percent Acid in Excess Stoichiometric Ratio	110.000	%
Actual acid to add to SRAT	1.3338	gmol
Acid required in moles per liter of starting sludge (less receipt samples)	1.3337	gmol/L
REDOX CALCULATION (SME PRODUCT REDOX PREDICTION)		
REDOX Target	0.100	Fe+2 / Fe
Predicted REDOX	0.100	
Ratio of glycolic/formic acid to total acid	0.6003	moles formic + glycolic acid blend / mole total acid
Delta between predicted REDOX and target REDOX	- 0.00003 1	
Acid blend ratio	0.79996 0383	moles glycolic / mole blend
Activation of SME cycle corrections? (1=SME corrections performed):	0	
Nitric acid density, 20 °C	1.30502	g/mL
Formic acid density, 20 °C	1.2043	g/mL
Glycolic/Formic blend density, 20°C	1.2644	g/mL
Nitric acid, wt %	49.25	wt %
Formic acid, wt %	89.95	wt %
Glycolic acid, wt %	71.00	wt %
Glycolic acid in blend, wt%	68.66	wt%
Formic acid in blend, wt%	10.39	wt%
Formic acid amount	0.160	gmol
Nitric acid amount	0.533	gmol
Glycolic acid amount	0.641	gmol
Total Manganese in fresh feed	0.135	gmol
Manganese removed with SRAT product sample	0.033	gmol
Projected Melter Feed Manganese, total moles	0.101	gmol
Formate moles with fresh sludge	0.000	gmol

Run #	SC-13	Units
Formate moles added with formic acid	0.160	gmol
Formate moles destroyed in SRAT (% of acid charged)	0.144	gmol
Formate moles removed with SRAT product sample	0.004	gmol
Formate moles reacted in SME (% of acid charged)	0.000	gmol
Formate Moles after SME	0.012	gmol
Frit slurry formate (when SME cycle frit additions are made with formic acid)	0.000	gmol
Projected Melter Feed Formate, total moles	0.012	gmol
Nitrate moles from fresh sludge, ARP, and MCU	0.267	gmol
Nitrate moles from nitric acid	0.533	gmol
Nitrate from conversion of nitrite to nitrate in SRAT and SME	0.000	gmol
Nitrate from minor trim chemicals	0.00000	gmol
Nitrate removed with SRAT product sample	0.19787	gmol
Nitrate destroyed in the SME	0.00000	gmol
Projected Melter Feed Nitrate, total moles (Sum of inputs - destroyed)	0.602	gmol
Glycolate added with acid	0.641	gmol
Glycolate moles destroyed in SRAT	0.019	gmol
Glycolate moles removed with SRAT product sample	0.154	gmol
Glycolate moles reacted in SME	0.000	gmol
Projected Melter Feed Glycolate, total moles	0.468	gmol
Oxalate in fresh feed	0.000	gmol
Oxalate from trim	0.000	gmol
Oxalate destroyed during reaction	0.000	gmol
Oxalate created from glycolate	0.019	gmol
Oxalate removed with SRAT product sample	0.000	gmol
Projected Melter Feed Oxalate, total moles	0.019	gmol
Carbon from Coal in fresh feed	0.000	gmol
Carbon from trim coal	0.000	gmol
Carbon removed in SRAT product Sample	0.000	gmol
Projected Melter Feed Carbon from coal, total moles	0.000	gmol
	- 335544. 320	
Projected Melter Feed Nitrite, total moles	0.0000	gmol
Assumed SME density	1.400	g/ml
Projected final SME mass	0.897	kg
Manganese concentration in final melter feed	0.113	gmol/kg melter feed slurry
Formate concentration in final melter feed	0.013	gmol/kg melter feed slurry

Run #	SC-13	Units
Glycolate concentration in final melter feed	0.521	gmol/kg melter feed slurry
Oxalate concentration in final melter feed	0.021	gmol/kg melter feed slurry
Carbon from coal concentration in final melter feed	0.000	gmol/kg melter feed slurry
Nitrate concentration in final melter feed	0.671	gmol/kg melter feed slurry
Nitrite concentration in final melter feed	0.000	gmol/kg melter feed slurry
Projected final SME volume	0.641	liters
BENCH SCALE CALCULATIONS		
Bench Scale Operational Setting		
Scaled formic/glycolic acid feed rate based on nominal 23.551 M	0.6890	ml/min
Scaled nitric acid feed rate based on nominal 10.395 M	0.3440	ml/min
Prototypical formic/glycolic acid feed time	88.5	min
Prototypical nitric acid feed time	151.9	min
Formic/glycolic acid volume required	60.968	ml
Nitric acid volume required	52.265	ml
Wt% active agent in antifoam solution	10	%
Target concentration for overall SRAT cycle	800	ppm
Total SRAT antifoam charge for 1:10 dilution	9.12	g
100 ppm SRAT antifoam charge at 1:10	1.14	g
Dewatering Calc for Target Wt. % Total Solids in SRAT Product		
Final SRAT Product Total Solids (UNDER TOOLS USE SOLVER)	25.00	%
Water in Trimmed (and sampled) Sludge	1,075.06	g
Water added with antifoam	17.33	g
Water added with formic/glycolic acid blend	17.34	g
Water added with nitric acid	34.61	g
Water added in acid flushing	20.00	g
Water made during base equiv neutralization	9.99	g
Water made in TIC destruction	1.73	g
Water made in SRAT nitrite destruction	1.11	g
Water made in Mercury Reduction	0.34	
Revised water mass in slurry	1,177.52	g
Solids in Trimmed (and sampled) Sludge	194.94	g
Mass 1:20 antifoam added	0.91	g
Mass of pure formic acid (HCOOH) added	7.37	g
Mass of pure nitric acid (HNO3) added	33.59	g
Mass of pure glycolic acid (HOCH₂COOH)	48.71	g
Solids lost during base equiv neutralization	9.99	g
Solids lost in TIC destruction	5.95	g
Solids lost in SRAT nitrite destruction	4.81	g

Run #	SC-13	Units
Solids lost in SRAT nitrite destruction	8.50	g
Solids lost in SRAT formate destruction (formic acid)	6.63	g
Solids lost in SRAT glycolate destruction (glycolic acid)	1.46	g
Solids lost in Mercury Stripping	4.08	
Revised solids mass in slurry	252.59	g
Target final water mass in slurry to hit total solids target	757.77	g
Total water to remove (ARP and in SRAT)	419.75	g
Total water to remove in ARP step	0.00	g to return to wt% TS of starting sludge
Calculated total water to remove to return to starting volume	293.39	g
net (used in Macro iteration)	-126.36	g
Time to dewater ARP	0.00	minutes
Mass of carbonate lost as CO ₂	4.22	g
Mass of nitrite lost as NO	3.70	g
Formate converted to CO ₂	6.63	g
Formate converted to CO ₂ in SRAT	6.63	g
Glycolate converted to CO ₂ in SRAT	1.69	
Final sludge mass in SRAT after acid addition and dewater (neglecting samples)	1010.36	g
Mass of SRAT cycle samples (excluding SRAT Receipt)	250.00	g
Mass of treated sludge going into SME cycle	760.36	g
SME sample ratio	0.7526	
Calcined Solids going to SME	114.96	g
DWPF SCALE TO BENCH SCALE		
DWPF Scale SRAT cycle		
density estimate =	1.126	
Volume based scale factor 6000 gal starting SRAT	22426.1	
Minimum SRAT conflux time	720.0	min
Bench Scale SRAT cycle		
99.5% of scaled air purge	289.0	sccm
Helium purge rate at 0.5 vol%	1.4	sccm
Scaled boil-up rate	1.69	g/min
Required dewatering time at above rate (ARP and after acid)	249.0	min
DWPF Scale SME cycle		
Bench Scale SME cycle		

Run #	SC-13	Units
SME scale factor (ADJUSTED FOR SRAT SAMPLES)	29799.6	
99.5% scaled SME air purge	70.0	sccm
Helium purge rate at 0.5 vol%	0.35	sccm
Solids remaining at start of SME	190.1	g
SRAT product Calcine Factor (calculated)	0.605	g oxide/g dry SRAT Product
Sludge calcined solids - based on SRAT product	114.96	g
Sludge oxide contribution in SME	35.00	%
Frit oxide contribution	65.00	%
Frit slurry wt % solids	50.00	wt%
Frit slurry formic acid ratio	0.00	g 90 wt% FA/100 g Frit
Added water simulating decontamination of canisters	0.0	g
SME cycle antifoam addition at 1:10	0.76	g
Frit solids (total)	213.5	g
90 wt% formic acid (corrections necessary for other concentrations)	0.00	g
Water in frit slurry	213.5	g
Scaled transfer water	0.00	g
Total frit slurry water	213.5	g
Total mass of frit slurry	427.0	g
Number of equal SME frit slurry additions	2	
Each SME frit addition	106.8	g
Each SME 90-wt% formic acid addition	0.00	g
Each SME water addition	106.8	g
Scaled SME boil-up rate	1.27	g/min
Approximate time to remove water:	84.2	min
Final solids content in SME	403.7	g
Target SME solids total wt%	45.0	%
Mass of water to boil off for final SME concentration	78.3	g
Scaled boil-up rate	1.27	g/min
Approximate time to reach solids target concentration.	61.8	min

Table A - 2. Mass Balance

SRAT Mass Balance SC-13	Planned VERSUS Actual Additions			
	Planned, g	Actual, g	Delta, g	% Delta
Sludge Simulant (grams)	1,140.00	1,127.36	-12.64	-1.11%
Water to dilute/rinse trim chemicals (grams)	130.00	130.00	0.00	0.00%

Total Slurry (grams)	1,270.00	1,257.36	-12.64	-1.00%
Slurry Mass after sample (grams)	1,270.00	1,257.36	-12.64	-1.00%
SRAT Antifoam (and water) (grams)	18.24	18.24	0.00	0.00%
Nitric Acid solution (grams)	68.21	68.21	0.00	0.00%
Glycolic/Formic Acid solution (grams)	77.08	77.08	0.00	0.00%
Water added to flush Nitric and Formic Acid Lines	20.00	20.00	0.00	0.00%
Total Dewater (grams)	419.75	415.65	25.90	6.17%
MCU Simulant	0.00	0.00	0.00	NA
MCU Dewater		0.00	0.00	NA
MWWT Dewater mass (grams)		30.00		
SRAT FAVC Dewater mass (grams)		0.00		
SRAT Sample (grams)	250.00	251.05	1.05	0.4%
SRAT Product Mass after sampling (grams)	760.36	759.31	-1.05	-0.14%

Table A - 3. Formate and Nitrate Balance

Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta	% Delta
SRAT Product Analysis:				
SRAT Product Total Solids, wt %	25.00	24.30	-0.7	-2.80%
SRAT Calcined Solids, wt %	15.12	14.81	-0.3	-2.05%
SRAT Mn, wt. % calcined element	4.84	4.485	-0.4	-7.41%
SRAT Formate, mg/kg	758	759	0.5	0.07%
SRAT Glycolate, mg/kg	49,476	42,561	-6,915	-13.98%
SRAT Oxalate, mg/kg	2,411	434	-1,977	-82.00%
SRAT Nitrite, mg/kg	0	426	426.0	NA
SRAT Nitrate, mg/kg	50,136	52,893	2,757.3	5.50%
SRAT Formate Added as acid (best basis, grams)	7.834	7.834	0	0.00%
SRAT Glycolate Added as acid (best basis, grams)	52.222	52.222	0	0.00%
Nitrate Added as acid (best basis, grams)	33.054	33.054	0	0.00%
Nitrite in Feed (grams)	8.50	8.41	0	-1.11%
Nitrate in Feed & trim chemicals (grams)	16.53	16.35	0	-1.11%
Nitrite in SRAT product (grams)	0.00	0.43	0	NA
Nitrate in SRAT product (grams)	50.655	53.44	3	5.50%
Formate in SRAT product (grams)	0.78	0.77	0	-2.11%
Glycolate in SRAT product (grams)	50.66	43.00	-8	-15.11%
SRAT Formate Destruction (grams)	7.050	7.067	0.0	0.23%
SRAT Formate Destruction (%)	90.0	90.2	0.2	0.23%
SRAT Glycolate Destruction (grams)	1.6	9.2	7.7	488.51%
SRAT Glycolate Destruction (%)	3.0	17.7	14.7	488.51%
SRAT Nitrite Destruction (grams)	8.5	8.0	-0.5	-6.17%
SRAT Nitrite Destruction (%)	100.0	94.9	-5.1	-5.12%

Anion Conversion Balance (SRAT Cycle)	Planned, g	Actual, g	Delta	% Delta
Nitrite to Nitrate Conversion (grams)	1.07	4.04	3.0	277.28%
Nitrate from nitrite in SRAT product, mol	0.017	0.065	0.05	277.28%
Moles of nitrite reacted	0.185	0.185	0.000	0.00%
% nitrite conversion to nitrate	0.0	35.2	35.2	NA
Predicted SME product mass from forwarded SRAT mass	896.9	874.1		
Predicted SME Product Formate, gmol/kg SME slurry	0.013	0.015		
Predicted SME Product Oxalate, gmol/kg SME slurry	0.021	0.022		
Predicted SME Product Glycolate, gmol/kg SME slurry	0.521	0.493		
Predicted SME Product Coal, gmol/kg SME slurry	0.000	0.000		
Predicted SME Product Nitrate, gmol/kg SME slurry	0.671	0.686		
Predicted SME Product Nitrite, gmol/kg SME slurry	0.000	0.008		
Predicted SME Product Mn, gmol/kg SME slurry	0.113	0.105		
Predicted Fe⁺²/Fe total in glass (no SME cycle)	0.100	0.052		

APPENDIX B. OFFGAS COMPOSITION DATA

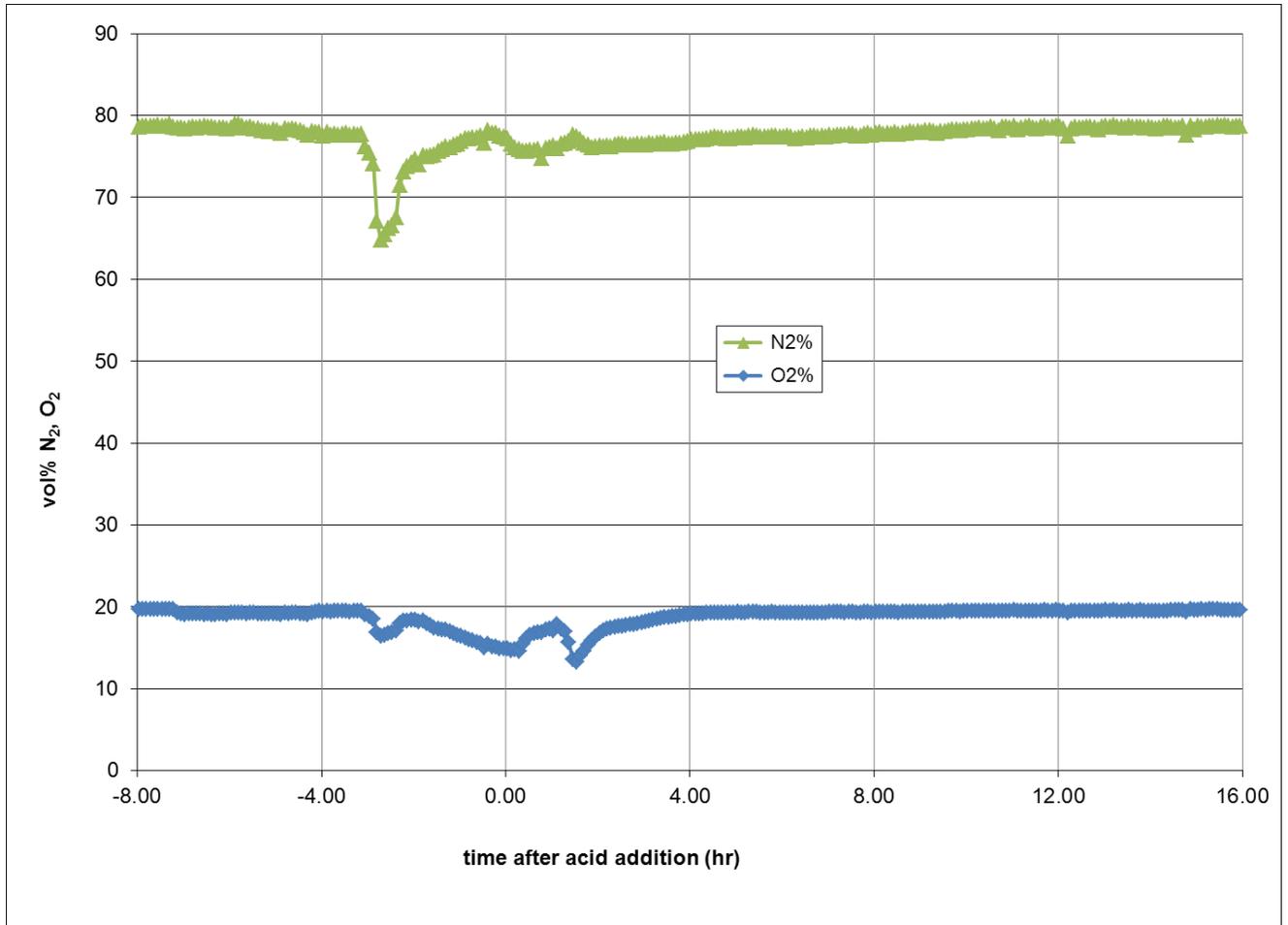
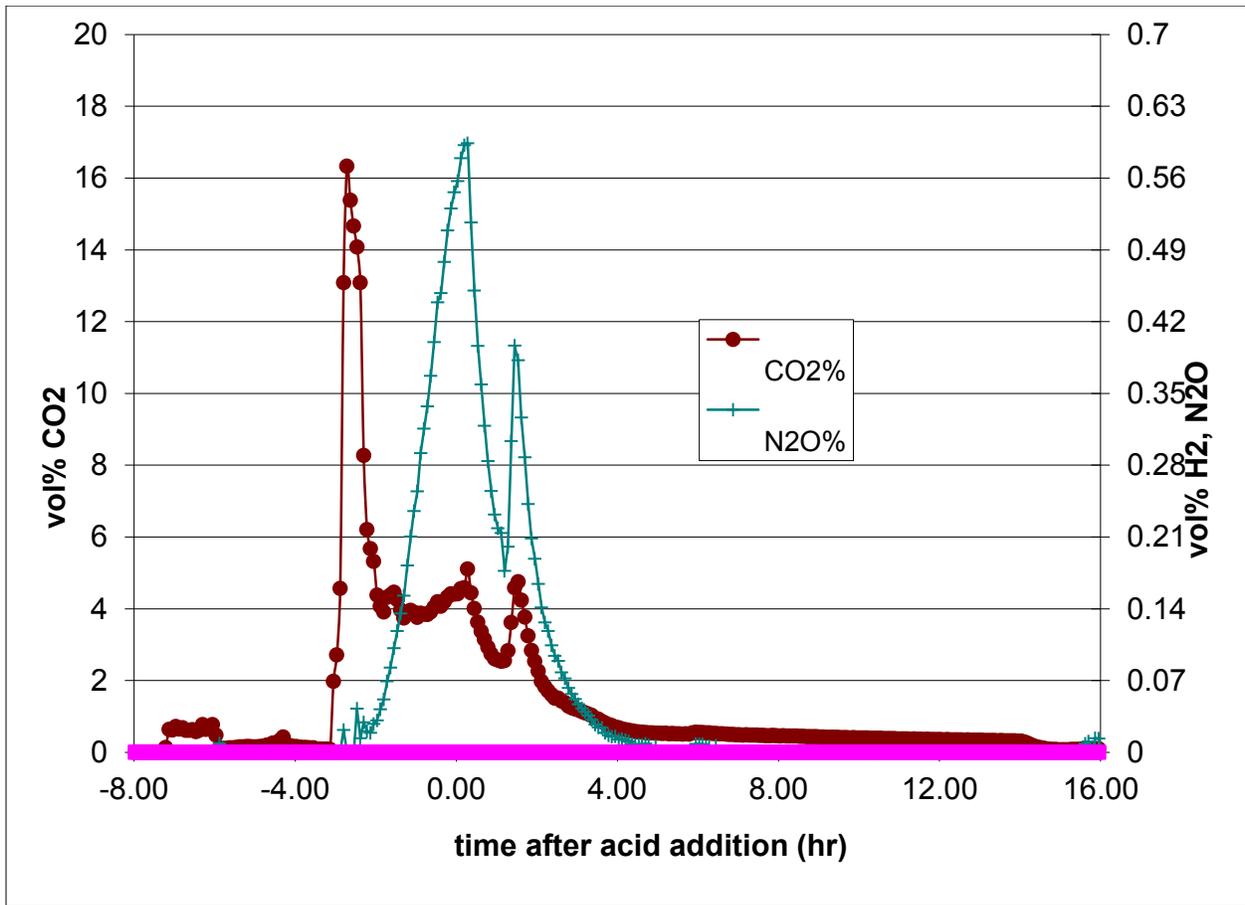
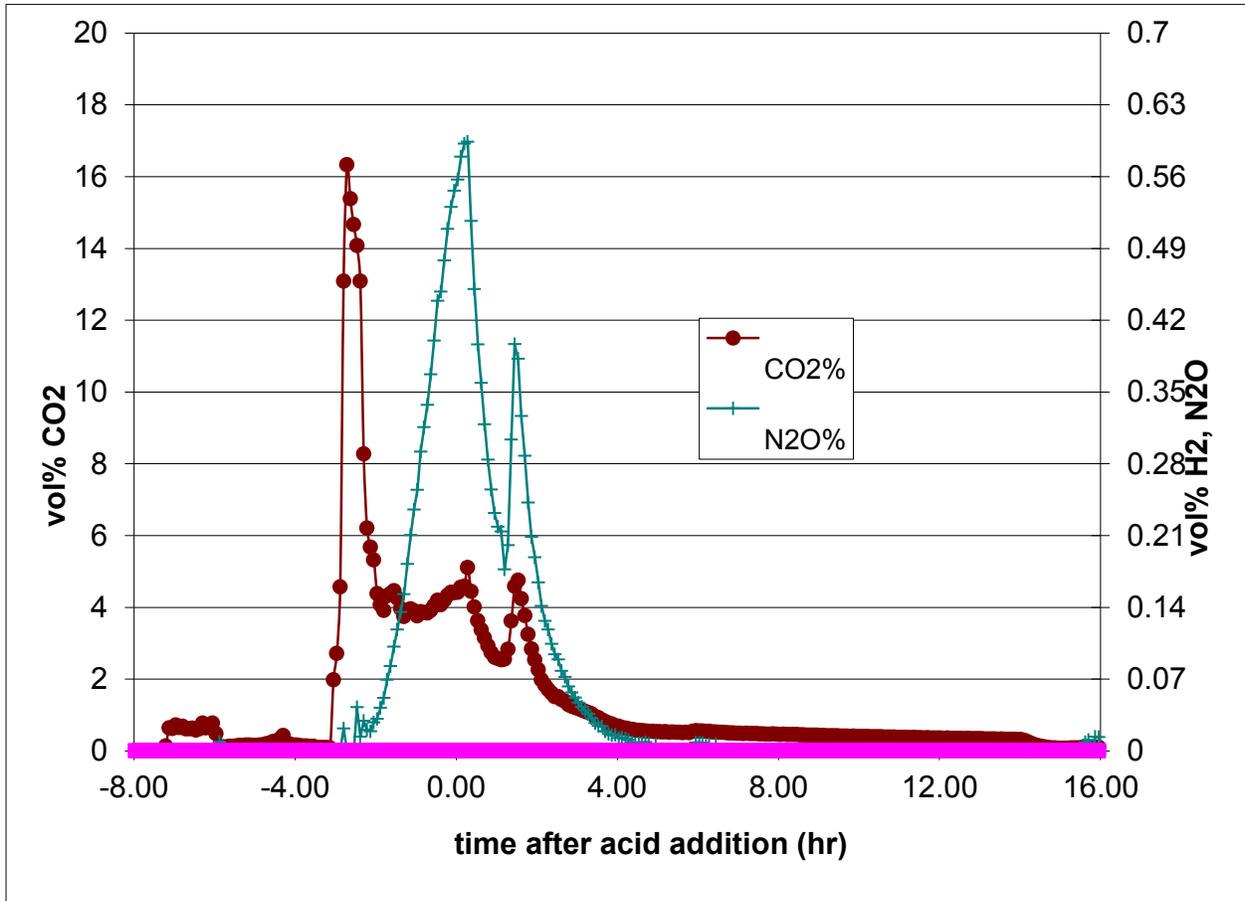


Figure B - 1. SC-13 SRAT Cycle O₂, N₂ Offgas Data





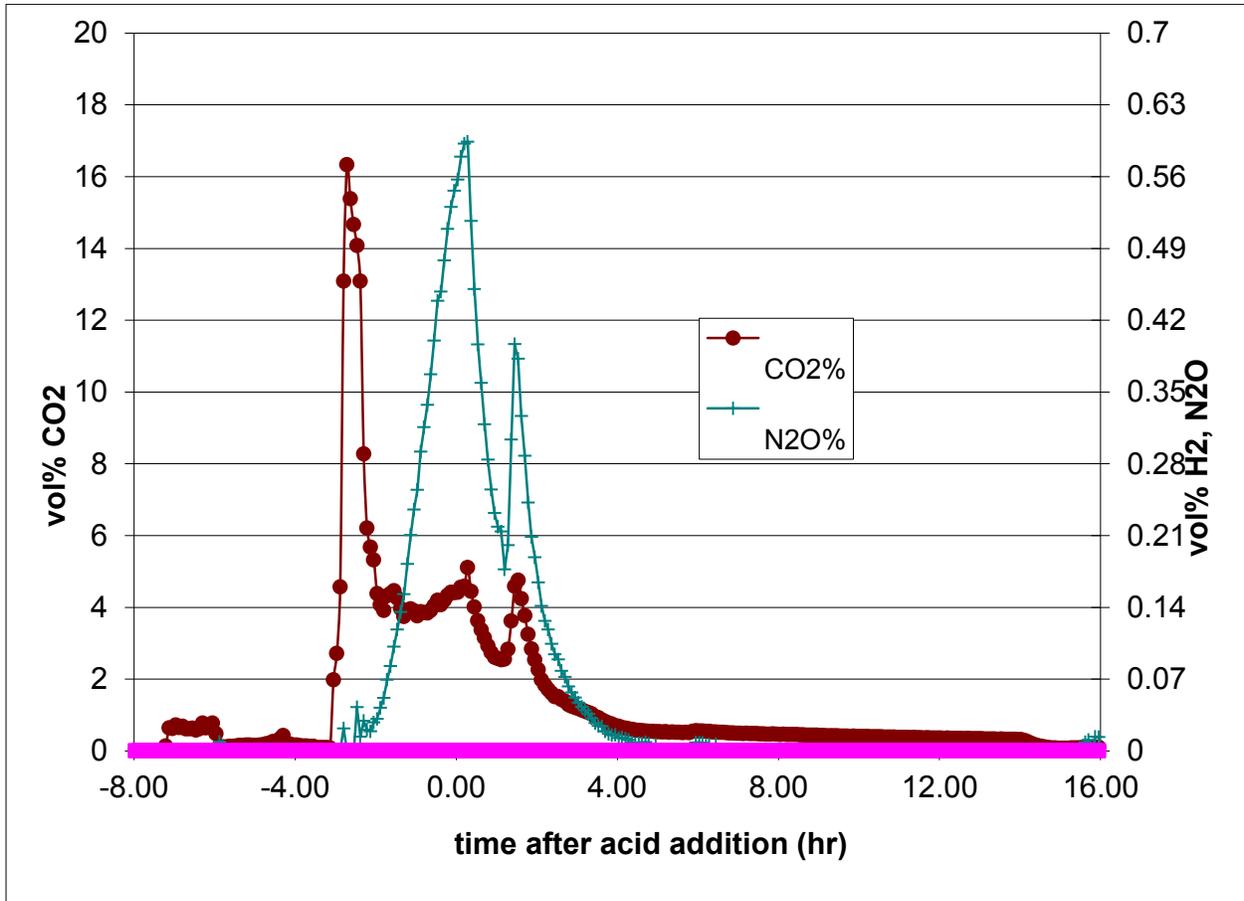


Figure B - 2. SC-13 SRAT Cycle N₂O, CO₂, H₂ Offgas Data

APPENDIX C. RHEOLOGICAL RESULTS CHARTS AND FLOW CURVES

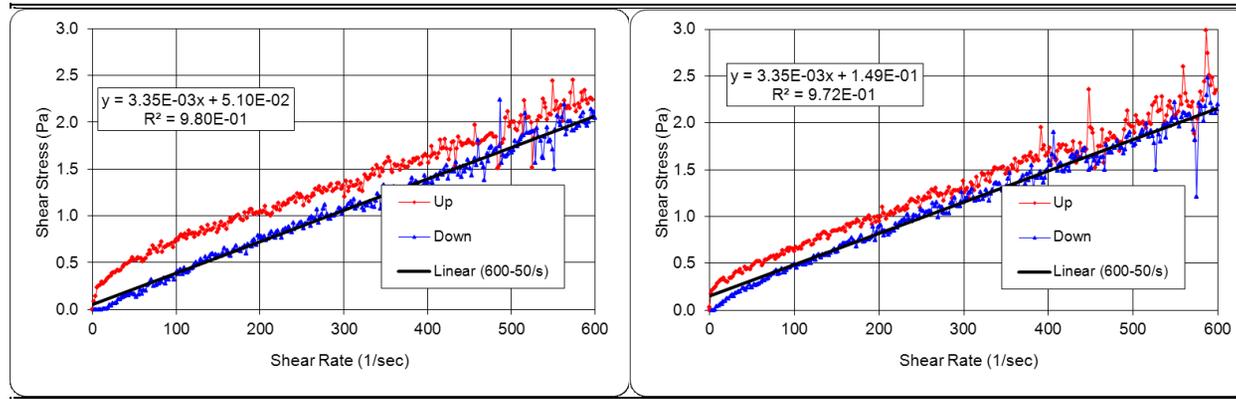


Figure C - 1 SRAT Product, Flow Curve

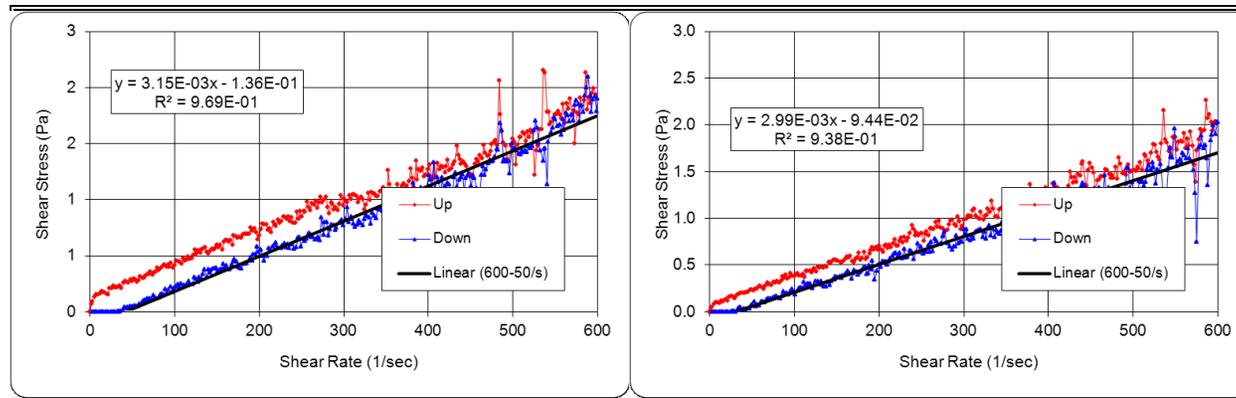


Figure C - 2 SRAT Product pH 2, Flow Curve

APPENDIX D. ICP-AES and ICP-MS Data Compilation

Table D - 4. SRAT Receipt ICP-AES

3002 Units	Aqua regia ICP-AES Slurry				Peroxide Fusion ICP-AES Slurry				ICP-AES Supernate			
	Aqua regia ICP-AES Slurry, mg/kg slurry				Peroxide Fusion ICP-AES Slurry, mg/kg slurry				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
	92299	92300	92301	92302	92307	92308	92309	92310	92239	92240	92241	92242
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	mg/L	mg/L	mg/L	mg/L
Ag	<18	<18	<18	<18	179	181	179	180	<28.54	<28.13	<28.51	<25.77
Al	11,300	12,100	11,900	10,600	13,500	12,800	13,700	12,800	1502.18	1494.14	1497.03	1503.22
B	<24.8	<25.0	<25.1	<25.2	<25.1	<25.4	<25.1	<25.3	4.56	4.71	4.69	4.29
Ba	162	161	162	160	162	157	169	156	<1.25	<1.23	<1.25	<1.13
Be	1.43	1.42	1.42	1.43	1.24	1.26	1.49	1.38	<0.12	<0.12	<0.12	<0.11
Ca	2,000	1,990	1,980	1,970	2,210	2,300	2,300	2,140	3.77	4.32	4.32	4.03
Cd	73.0	71.8	71.8	71.2	71.1	70.3	76.5	70.2	<0.61	<0.61	<0.61	<0.55
Ce	194	195	191	193	183	167	210	179	<11.95	<11.78	<11.94	<10.79
Co	18.9	19.2	19.1	18.8	19.2	19.5	16.5	15.3	<1.68	<1.66	<1.68	<1.52
Cr	69	69	69	69	74	72	76	75	15.40	15.12	15.39	15.48
Cu	88.1	87.6	87.5	87.7	64.3	61.1	65.1	60.3	<1.25	<1.23	<1.25	<1.13
Fe	23,800	23,900	23,900	23,700	24,500	23,600	25,100	23,400	0.65	0.70	0.65	0.59
Gd	156	155	156	154	118	112	123	118	<4.26	<4.20	<4.26	<3.85
K	<79	<84	<77	<92	<319	<322	<319	<321	57.27	64.65	57.43	61.38
La	133	134	133	133	115	115	123	115	<1.07	<1.05	<1.07	<0.97
Li	52.9	51.6	52.2	55.4	82.8	75.2	89.4	68.7	<2.89	<2.85	<2.89	<2.61
Mg	903	902	901	893	940	918	973	902	<0.30	<0.29	<0.30	<0.27
Mn	5,530	5,510	5,510	5,470	5,760	5,530	5,900	5,490	<1.05	<1.04	<1.05	<0.95
Mo	<9.7	<10.5	<9.9	<9.5	<33.3	<33.7	<33.4	<33.5	<5.31	<5.23	<5.31	<4.80
Na	23,900	23,900	24,000	23,800	NM	NM	NM	NM	23,385	23,438	23,366	23,085
Ni	<3,750	<3,750	<3,740	<3,730	<3,840	<3,730	<3,960	<3,700	<9.02	<8.89	<9.01	<8.14
P	<166	<180	<160	<146	<226	<302	<251	<109	<32.50	<32.03	<32.48	<29.35
Pb	<48	<50	<53	<51	<89	<90	<89	<90	<14.19	<13.98	<14.18	<12.81

3002 Units	Aqua regia ICP-AES Slurry				Peroxide Fusion ICP-AES Slurry				ICP-AES Supernate			
	Aqua regia ICP-AES Slurry, mg/kg slurry				Peroxide Fusion ICP-AES Slurry, mg/kg slurry				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
S	92299	92300	92301	92302	92307	92308	92309	92310	92239	92240	92241	92242
Sb	157	158	159	159	132	134	133	145	<21.20	<20.90	<21.19	<19.15
Sn	NM	NM	NM	NM	1460	1440	1480	1320	12.37	10.45	10.18	10.93
Sn	<62	<62	<63	<63	<70	<71	<70	<70	<11.12	<10.96	<11.11	<10.04
Sr	81.3	81.1	81.5	80.4	79.5	77.3	83.7	77.4	<0.10	<0.10	<0.10	<0.09
Th	271	270	272	268	218	207	224	205	<5.31	<5.23	<5.31	<4.80
Ti	27.2	27.3	27.2	28.3	34.9	32.9	34.1	32.0	<0.75	<0.74	<0.75	<0.68
U	8,010	7,970	7,960	7,910	7,970	7,700	8,250	7,530	<64.80	<63.87	<64.75	<58.52
V	<5.78	<5.81	<5.85	<5.86	<5.83	<5.90	<5.85	<5.88	<0.93	<0.92	<0.93	<0.84
Zn	77.5	77.5	77.9	76.9	82.4	79.0	87.4	80.8	<1.82	<1.80	<1.82	<1.65

Table D - 5. SRAT Product ICP-AES

3002 Units	Aqua regia ICP-AES Slurry, mg/kg slurry				Peroxide Fusion ICP-AES Slurry, mg/kg slurry				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	mg/L	mg/L	mg/L	mg/L
Ag	92314	92315	92316	92317	92322	92323	92324	92325	92441	92442	92443	92444
Al	<28	<28	<28	<28	287	287	287	279	<28.54	<28.13	<28.51	<25.77
B	13,300	14,300	13,200	14,100	15,400	15,400	15,600	15,200	590.57	628.91	603.96	520.76
Ba	<39.3	<39.1	<39.2	<39.8	<40.2	<40.3	<40.2	<39.1	5.55	6.31	5.82	4.58
Be	177	189	167	181	193	198	195	191	5.35	5.59	5.39	4.56
Ca	1.60	1.67	1.47	1.60	1.59	1.60	1.59	1.55	<0.12	<0.12	<0.12	<0.11
Cd	2,330	2,500	2,250	2,400	2,750	2,670	2,780	2,650	1,278.24	1,371.09	1,318.81	1,139.94
Ce	88.9	95.9	85.3	92.6	84.7	88.3	90.7	86.2	20.21	21.68	20.99	18.07
Co	215	231	206	226	249	265	262	219	<11.95	<11.78	<11.94	<10.79
Cr	23.6	25.6	22.2	24.3	24.8	23.9	25.3	25.7	3.15	3.63	3.35	2.83
	161	173	153	168	217	250	277	260	24.38	26.37	25.15	21.83

3002 Units	Aqua regia ICP-AES Slurry, mg/kg slurry				Peroxide Fusion ICP-AES Slurry, mg/kg slurry				ICP-AES Supernate, mg/L			
	1 92314 ug/g	2 92315 ug/g	3 92316 ug/g	4 92317 ug/g	1 92322 ug/g	2 92323 ug/g	3 92324 ug/g	4 92325 ug/g	1 92441 mg/L	2 92442 mg/L	3 92443 mg/L	4 92444 mg/L
Cu	97.1	106.0	92.8	101.0	69.3	66.8	68.5	69.0	<1.25	<1.23	<1.25	<1.13
Fe	27,700	29,600	26,700	28,500	28,200	28,600	29,200	28,100	191.84	205.08	197.43	170.90
Gd	165	177	157	169	127	113	86	97	61.43	65.63	63.37	54.04
K	<59	<81	<67	<73	<511	<512	<511	<497	83.23	88.67	87.33	82.68
La	128	133	125	130	137	132	139	129	19.76	20.90	20.00	17.09
Li	59.4	64.3	56.2	60.9	82.1	83.5	83.6	76.2	5.65	7.05	5.27	4.97
Mg	1,130	1,220	1,080	1,170	1,200	1,190	1,220	1,180	729.29	781.25	750.50	647.82
Mn	6,530	7,040	6,260	6,740	6,750	6,830	6,900	6,680	3,507.73	3,750.00	3,603.96	3,095.92
Mo	<15.8	<16.9	<14.4	<14.6	<53.4	<53.4	<53.3	<51.9	<12.23	<12.05	<12.22	<11.04
Na	27,600	29,300	26,400	27,900	NM	NM	NM	NM	29,330	31,445	30,297	26,127
Ni	<4,230	<4,570	<4,100	<4,370	<4,400	<4,410	<4,530	<4,360	370.59	398.44	382.18	329.28
P	<203	<180	<275	<232	<173	<213	<260	<168	<32.50	<32.03	<32.48	<29.35
Pb	<65	<71	<63	<68	<1,050	<1,050	<1,050	<1,020	<14.19	<13.98	<14.18	<12.81
S	<259	<250	<145	<167	<1,490	<1,500	<1,490	<1,450	229.89	146.48	217.82	141.55
Sb	249	247	248	252	212	213	212	207	<25.37	<25.00	<25.35	<22.91
Sn	NM	NM	NM	NM	1660	1600	1600	1520	59.85	66.80	62.38	50.64
Sn	<98	<98	<98	<99	<112	<112	<112	<109	<11.12	<10.96	<11.11	<10.04
Sr	89.8	95.1	84.4	91.4	94.9	97.7	96.6	94.1	50.54	54.10	52.08	44.92
Th	202	214	193	209	240	216	235	217	<10.60	<10.45	<10.59	<9.57
Ti	31.4	32.5	29.1	32.2	41.6	49.7	46.0	45.2	2.28	2.34	2.36	1.97
U	8,770	9,760	8,660	9,180	8,990	8,640	9,050	8,660	3,151.01	3,378.91	3,227.72	2,809.59
V	<9.15	<9.11	<9.12	<9.26	<9.36	<9.37	<9.35	<9.11	<0.93	<0.92	<0.93	<0.84
Zn	97.5	107.0	93.9	103.0	99.8	103.0	99.1	93.8	2.66	2.75	2.65	2.25

Table D - 6. SRAT Product pH 3 ICP-AES

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1 92330 ug/g	2 92331 ug/g	3 92332 ug/g	4 92333 ug/g	1 92345 ug/g	2 92346 ug/g	3 92347 ug/g	4 92348 ug/g	1 92265 mg/L	2 92266 mg/L	3 92267 mg/L	4 92268 mg/L
Ag	<29	<28	<29	<27	285	282	281	287	<27.06	<27.16	<27.46	<28.07
Al	6,060	5,680	5,410	6,060	15,500	14,300	14,700	16,300	1,436	1,283	1,346	1,423
B	<40.2	<39.7	<40.0	<38.2	<39.9	<39.6	<39.4	<40.3	6.20	5.04	5.28	5.83
Ba	176	176	174	175	193	177	183	204	5.41	4.55	4.86	5.13
Be	1.45	1.46	1.46	1.49	1.58	1.57	1.56	1.60	<0.11	<0.11	<0.11	<0.12
Ca	2,270	2,290	2,270	2,280	2,720	2,440	2,460	2,930	2,161	1,924	2,021	2,125
Cd	83.2	82.9	82.7	83.3	89.3	81.8	84.5	95.4	36.45	32.44	33.75	35.87
Ce	213	219	212	216	184	184	163	222	13.28	13.33	13.48	13.78
Co	21.3	22.3	21.8	22.3	25.8	24.8	25.9	27.7	6.37	5.55	5.68	5.93
Cr	159	156	154	155	172	159	164	183	36.45	32.44	33.94	35.87
Cu	97.9	97.9	96.8	98.2	70.7	70.8	68.5	76.6	<11.84	<11.88	<12.01	<12.28
Fe	26,600	26,700	26,600	26,500	28,600	26,400	27,400	30,200	499.81	443.23	465.29	491.23
Gd	166	165	165	167	125	115	108	133	132.66	118.63	123.00	130.41
K	<90	<103	<95	<92	<507	<503	<501	<512	104.66	104.49	106.60	107.02
La	127	121	123	123	128	118	122	145	53.55	47.72	49.58	52.63
Li	60.9	60.3	59.7	59.8	76.7	64.9	74.1	86.4	11.46	8.85	10.18	11.03
Mg	1,090	1,110	1,100	1,110	1,180	1,080	1,120	1,260	839.91	748.77	785.66	832.36
Mn	6,310	6,380	6,350	6,400	6,840	6,300	6,520	7,180	4,247	3,791	3,966	4,191
Mo	<16.1	<14.0	<15.3	<15.7	<53.0	<52.5	<52.3	<53.5	7.25	5.83	7.25	6.67
Na	27,500	27,400	27,400	27,400	NM	NM	NM	NM	30,064	31,120	28,223	28,070
Ni	<4,100	<4,140	<4,080	<4,120	<4,590	<4,200	<4,350	<4,840	528.00	469.63	493.90	520.47
P	<275	<274	<207	<250	<283	<170	<169	<255	30.82	30.93	31.27	31.97
Pb	<55	<60	<58	<60	<1,040	<1,040	<1,030	<1,050	<13.45	<13.50	<13.65	<13.96
S	<217	<147	<154	<191	<1,480	<1,470	<1,460	<1,500	255.54	141.46	244.09	214.42
Sb	254	251	253	242	211	209	208	213	<24.05	<24.14	<24.41	<24.95
Sn					1650	1520	1560	1740	306.28	267.82	272.69	290.45
Sn	<100	<99	<100	<95	<111	<110	<109	<112	<10.54	<10.58	<10.70	<10.94

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
	92330	92331	92332	92333	92345	92346	92347	92348	92265	92266	92267	92268
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	mg/L	mg/L	mg/L	mg/L
Sr	88.2	88.5	90.8	88.1	87.5	80.4	82.4	98.0	66.89	59.79	62.55	66.08
Th	206	206	201	204	231	206	209	253	<102.97	<103.36	<104.50	<106.82
Ti	29.2	28.3	33.1	28.3	38.7	35.9	37.1	40.5	<0.71	<0.72	<0.72	<0.74
U	8,940	9,340	9,190	9,160	8,360	8,000	7,950	9,160	7,704	7,865	7,265	7,290
V	<9.36	<9.24	<9.30	<8.89	<9.29	<9.22	<9.17	<9.38	<0.88	<0.89	<0.90	<0.92
Zn	90.5	91.7	91.3	92.1	98.2	86.9	94.0	107.0	5.20	4.45	5.09	5.07

Table D - 7. SRAT Product pH 2 ICP-AES

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
	92359	92360	92361	92362	92352	92353	92354	92355	92485	92486	92487	92488
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	mg/L	mg/L	mg/L	mg/L
Ag	<28	<28	<28	<28	282	285	277	278	<2.79	<2.80	<2.86	<2.84
Al	13,000	14,000	13,800	14,100	13000	14000	13800	14100	1,634	1,620	1,671	1,515
B	<39.3	<39.9	<39.6	<39.3	39.5	40	38.9	39	4.74	4.50	4.75	4.19
Ba	170	172	155	167	161	170	166	173	6.13	6.09	6.30	5.71
Be	1.56	1.58	1.57	1.56	1.57	1.78	1.54	1.74	<0.12	<0.12	<0.12	<0.12
Ca	2,190	2,250	2,000	2,150	2360	2420	2380	2320	2,186	2,181	2,247	2,035
Cd	79.8	81.7	74.6	78.6	71.8	76.6	79.3	78.3	35.8	35.8	37.0	33.4
Ce	215	189	198	211	224	215	223	213	<20.5	<20.6	<21.1	<20.9
Co	23.8	20.6	21.6	22.8	20.5	20.6	20	23.5	7.56	7.83	7.68	7.25
Cr	156	159	147	154	148	151	152	154	36.4	36.2	37.0	34.0
Cu	65.4	67.7	60.8	65.2	61.6	62.8	62.4	65	<1.22	<1.23	<1.25	<1.24
Fe	25,800	26,200	23,600	25,200	24000	25500	25000	25700	638	637	654	595
Gd	175	152	161	168	120	130	122	114	128	127	130	119
K	<119	<105	<107	<113	502	508	494	495	87.2	104.4	96.9	92.3
La	132	116	122	129	112	122	118	120	47.2	47.9	49.1	44.6

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1 ug/g	2 ug/g	3 ug/g	4 ug/g	1 ug/g	2 ug/g	3 ug/g	4 ug/g	1 mg/L	2 mg/L	3 mg/L	4 mg/L
Li	59.6	52.1	55.7	58.3	63.8	68.5	67	80.4	9.63	9.56	10.24	8.22
Mg	1,080	1,110	989	1,060	984	1050	1030	1040	805	802	827	753
Mn	6,260	6,440	5,720	6,140	5720	6090	6000	6130	4,043	4,030	4,157	3,773
Mo	<24.0	<24.4	<24.2	<24.0	52.4	53.1	51.6	51.7	<11.9	<12.0	<12.3	<12.2
Na	26,700	27,500	24,400	26,100	NM	NM	NM	NM	29,207	29,011	29,833	27,262
Ni	<4,070	<4,160	<3,710	<3,980	3910	4160	4100	4210	557	557	573	522
P	<138	<238	<165	<203	169	171	167	167	<16.8	<16.9	<17.2	<17.1
Pb	<51	<47	<48	<52	140	142	138	138	<13.8	<13.9	<14.2	<14.1
S	<280	<263	<278	<236	1470	1490	1470	1450	145	146	172	148
Sb	104	105	105	104	209	211	205	208	<24.8	<24.9	<25.5	<25.3
Sn	<467	<423	<438	<454	1370	1470	1380	1440	111	108	109	89
Sn	109.0	111.0	110.0	109.0	110	111	108	108	<10.9	<10.9	<11.2	<11.1
Sr	91	81	85	89	78.8	81.9	90.5	91.8	71.0	70.5	72.6	65.8
Th	198.0	176.0	203.0	212.0	214	219	205	223	<41.4	<41.7	<42.6	<42.3
Ti	32	32	28	31	33.7	33.7	44.9	45.3	<7.35	<7.40	<7.56	<7.51
U	<9,040.00	<9,300.00	<8,000.00	<8,510.00	7530	8280	7920	7870	7,253	7,204	7,399	6,717
V	9.2	9.3	9.2	9.1	9.2	9.31	9.05	9.06	<1.82	<1.83	<1.87	<1.86
Zn	95.2	84.0	88.9	93.6	86.5	88.5	90.3	94.4	7.10	6.78	6.78	6.34

Table D - 8. SRAT Product pH 1 ICP-AES

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1 ug/g	2 ug/g	3 ug/g	4 ug/g	1 ug/g	2 ug/g	3 ug/g	4 ug/g	1 mg/L	2 mg/L	3 mg/L	4 mg/L
Ag	<28	<29	<28	<28	283	288	282	280	<2.86	<2.84	<2.82	<2.85
Al	9,410	7,940	7,520	12,000	13,700	14,500	13,700	14,400	1,593	1,756	1,690	1,560
B	<39.6	<40.4	<39.6	<39.6	<39.7	<40.4	<39.5	<39.3	5.54	5.14	4.86	5.05
Ba	170	167	168	169	170	177	164	175	<10.4	<11.4	<11.0	<10.2
Be	1.51	1.50	1.49	1.51	1.57	1.60	1.56	1.56	0.12	0.12	0.12	0.12
Ca	2,120	2,090	2,120	2,130	2,380	2,520	2,430	2,380	1,984	2,193	2,098	1,943
Cd	79.2	76.9	78.0	78.2	77.8	82.7	79.0	79.5	34.7	38.3	37.1	33.9
Ce	205	200	205	208	172	184	182	189	<21.0	<20.9	<20.8	<21.0
Co	21.1	20.4	21.0	20.8	26.3	29.0	28.5	24.5	7.26	8.26	7.41	6.89
Cr	152	149	152	150	162	171	164	169	34.52	38.13	36.47	33.86
Cu	94.1	92.1	93.5	92.8	66.2	66.7	66.9	68.5	<1.25	<1.24	<1.24	<1.25
Fe	25,300	24,700	25,000	25,100	25,500	26,800	25,300	26,600	607	670	647	594
Gd	163	158	164	165	113	127	124	119	121	134	128	118
K	<107	<102	<108	<107	<504	<513	<502	<500	91.9	94.0	87.6	90.3
La	140	136	140	140	117	127	121	124	51.6	57.1	54.1	50.5
Li	54.5	53.0	54.3	54.6	68.0	106.0	87.6	91.1	11.1	12.4	12.3	11.0
Mg	1,040	1,020	1,040	1,040	1,020	1,080	1,030	1,060	738	814	782	723
Mn	5,930	5,770	5,930	5,940	5,950	6,290	5,950	6,240	3,690	4,070	3,902	3,604
Mo	<16.5	<12.2	<16.6	<16.1	<52.7	<53.5	<52.4	<52.2	<12.2	<12.2	<12.1	<12.2
Na	25,300	24,600	25,100	25,200	NM	NM	NM	NM	26,190	28,842	27,647	25,545
Ni	<4,070	<3,970	<4,010	<4,060	<4,120	<4,390	<4,120	<4,330	528	587	559	517
P	<186	<120	<184	<180	<170	<173	<169	<169	74.4	71.5	63.1	60.8
Pb	<52	<50	<49	<52	<141	<143	<140	<139	<14.2	<14.1	<14.0	<14.2
S	<195	<309	<246	<277	<1,470	<1,500	<1,470	<1,460	176	148	158	152
Sb	251	255	250	250	209	213	208	208	<25.4	<25.3	<25.1	<25.3
Sn	NM	NM	NM	NM	1470	1530	1400	1530	69.6	81.8	71.8	61.6
Sn	<99	<101	<99	<99	<110	<112	<110	<109	<11.1	<11.1	<11.0	<11.1

3002 Units	Aqua regia ICP-AES Slurry, mg/kg				Peroxide Fusion ICP-AES Slurry, mg/kg				ICP-AES Supernate, mg/L			
	1	2	3	4	1	2	3	4	1	2	3	4
	92366	92367	92368	92369	92373	92374	92375	92376	92556	92557	92558	92559
	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	mg/L	mg/L	mg/L	mg/L
Sr	86.2	83.4	85.2	85.8	78.4	84.5	86.7	80.4	64.7	71.5	68.6	63.6
Th	287	281	288	288	206	217	203	200	<42.5	<42.3	<42.0	<42.4
Ti	28.1	27.5	27.5	27.8	36.5	38.4	42.0	36.8	<7.54	<7.51	<7.45	<7.52
U	8,380	8,210	8,470	8,340	7,820	8,220	7,880	8,180	7,143	7,882	7,569	7,010
V	<9.22	<9.40	<9.21	<9.21	<9.23	<9.39	<9.19	<9.15	<1.87	<1.86	<1.84	<1.86
Zn	83.6	81.9	82.5	83.2	96.9	102.0	91.9	104.0	6.69	7.53	7.06	6.30

Table D - 9. SRAT Receipt ICP-MS

3002 m/z	Aqua regia ICP-MS, mg/kg slurry basis					Supernatant Basis, mg/L			
	1 92299 μg/g	1 92300 μg/g	3 92301 μg/g	4 92302 μg/g	1 92247 mg/L	2 92248 mg/L	3 92249 mg/L	4 92250 mg/L	
85	9.23E+00	9.27E+00	9.33E+00	9.35E+00	8.88E-02	9.82E-02	8.89E-02	8.73E-02	
92	4.84E+01	1.93E+01	3.79E+01	3.73E+01	5.23E-02	5.71E-02	5.38E-02	6.99E-02	
94	4.89E+01	1.52E+01	4.16E+01	4.78E+01	<4.19E-02	5.90E-02	4.67E-02	4.39E-02	
95	2.35E+00	2.32E+00	3.11E+00	2.34E+00	3.49E-01	3.74E-01	3.62E-01	4.24E-01	
96	5.98E+01	2.09E+01	4.32E+01	7.95E+01	7.23E-02	7.08E-02	6.90E-02	8.85E-02	
97	1.54E+00	1.55E+00	1.62E+00	1.56E+00	2.83E-01	2.96E-01	3.20E-01	3.50E-01	
98	2.31E+00	2.32E+00	2.33E+00	2.34E+00	3.71E-01	3.68E-01	3.50E-01	4.02E-01	
99	3.84E+00	3.86E+00	3.89E+00	3.89E+00	2.75E-01	2.50E-01	2.66E-01	3.05E-01	
100	6.92E+00	6.95E+00	7.00E+00	7.01E+00	3.25E-01	3.10E-01	3.13E-01	3.59E-01	
101	6.90E+01	6.49E+01	6.70E+01	6.21E+01	3.19E-02	3.16E-02	3.30E-02	3.91E-02	
103	3.56E+01	3.48E+01	3.44E+01	3.12E+01	3.84E-02	2.87E-02	2.56E-02	2.45E-02	
104	3.37E+01	3.52E+01	3.70E+01	3.50E+01	7.10E-02	5.83E-02	4.76E-02	6.92E-02	
105	2.39E+00	2.98E+00	1.98E+00	3.18E+00	4.04E-01	3.85E-01	3.97E-01	4.37E-01	
106	4.88E+00	5.87E+00	4.29E+00	6.09E+00	5.32E-01	4.80E-01	5.14E-01	5.48E-01	
107	1.16E+01	1.56E+01	1.10E+01	9.69E+00	1.62E-01	1.53E-01	1.39E-01	1.78E-01	
108	1.54E+00	1.55E+00	1.56E+00	1.56E+00	1.04E-01	1.05E-01	1.00E-01	1.15E-01	
133	2.61E+01	2.63E+01	2.64E+01	2.65E+01	3.75E-01	3.61E-01	3.41E-01	3.72E-01	
137	2.17E+01	2.03E+01	2.18E+01	1.96E+01	1.06E-01	1.04E-01	9.84E-02	1.08E-01	
138	1.30E+02	1.22E+02	1.27E+02	1.25E+02	3.72E-02	4.85E-02	3.32E-02	9.69E-02	
182	1.54E+00	1.55E+00	1.56E+00	1.56E+00	4.59E-02	6.23E-02	6.68E-02	4.79E-02	
183	3.08E+00	3.09E+00	3.11E+00	3.12E+00	2.27E-02	3.47E-02	3.86E-02	4.24E-02	
184	2.31E+00	2.32E+00	2.33E+00	2.34E+00	6.90E-02	5.23E-02	7.56E-02	5.34E-02	
196	2.74E+00	2.32E+00	2.82E+00	3.18E+00	6.24E-02	8.67E-02	6.05E-02	6.52E-02	
198	1.53E+02	1.47E+02	1.57E+02	1.57E+02	2.60E+00	2.73E+00	2.72E+00	3.10E+00	
204	7.77E+01	7.75E+01	8.18E+01	7.95E+01	1.38E+00	1.44E+00	1.42E+00	1.54E+00	
237	1.22E+01	1.11E+01	1.10E+01	1.14E+01	1.05E-01	7.55E-02	1.03E-01	1.10E-01	
238	8.81E+03	8.35E+03	8.57E+03	7.98E+03	6.28E-01	5.30E-01	5.93E-01	6.21E-01	
239	5.51E+01	5.41E+01	6.16E+01	5.64E+01	3.14E-02	3.14E-02	3.14E-02	3.14E-02	

Table D - 10. SRAT Product ICP-MS

3002 m/z	Aqua regia ICP-MS Slurry, mg/kg				Supernate, mg/L			
	1 92314 µg/g	2 92315 µg/g	3 92316 µg/g	4 92317 µg/g	1 92446 mg/L	2 92447 mg/L	3 92448 mg/L	4 92449 mg/L
59	1.98E+01	1.99E+01	1.82E+01	2.88E+01	2.68E+00	2.32E+00	2.66E+00	2.44E+00
88	4.83E+01	4.82E+01	5.04E+01	5.44E+01	3.73E+01	3.53E+01	3.66E+01	3.60E+01
89	5.37E+01	5.36E+01	5.18E+01	5.74E+01	1.63E+01	1.75E+01	1.57E+01	1.52E+01
90	4.87E+01	3.66E+01	5.65E+01	5.31E+01	3.34E+01	3.53E+01	3.40E+01	3.46E+01
91	2.71E+01	1.71E+01	4.42E+01	3.56E+01	6.89E+00	6.87E+00	6.01E+00	7.47E+00
92	3.25E+01	2.46E+01	5.35E+01	4.32E+01	6.60E+00	7.17E+00	7.37E+00	6.92E+00
93	3.57E+01	2.70E+01	6.42E+01	4.68E+01	8.05E+00	8.11E+00	6.29E+00	7.69E+00
94	3.13E+01	2.25E+01	5.23E+01	5.29E+01	7.60E+00	7.52E+00	6.98E+00	8.24E+00
96	3.46E+01	3.00E+01	6.38E+01	6.41E+01	7.52E+00	8.06E+00	8.84E+00	8.02E+00
101	7.28E+01	7.31E+01	6.95E+01	7.07E+01	2.26E+01	2.37E+01	2.27E+01	2.40E+01
102	6.99E+01	6.93E+01	6.73E+01	7.20E+01	2.27E+01	2.25E+01	2.14E+01	2.15E+01
103	3.37E+01	3.69E+01	2.94E+01	3.70E+01	5.45E+00	5.69E+00	5.32E+00	4.99E+00
104	3.50E+01	3.99E+01	3.62E+01	4.12E+01	1.21E+01	1.19E+01	1.22E+01	1.27E+01
110	8.13E+00	1.01E+01	1.38E+01	9.55E+00	2.93E+00	2.55E+00	3.16E+00	2.68E+00
111	1.06E+01	1.05E+01	1.15E+01	1.39E+01	3.41E+00	4.24E+00	3.99E+00	2.84E+00
112	1.70E+01	1.76E+01	1.70E+01	1.72E+01	4.22E+00	5.75E+00	5.21E+00	5.60E+00
114	2.14E+01	2.26E+01	2.15E+01	2.27E+01	6.39E+00	8.30E+00	6.68E+00	6.13E+00
116	8.33E+00	8.40E+00	7.91E+00	6.42E+00	<2.69E+00	<2.73E+00	<2.74E+00	<2.74E+00
119	8.98E+01	8.78E+01	8.78E+01	9.54E+01	<3.40E+01	<3.64E+01	<3.68E+01	<3.61E+01
125	3.41E+01	3.39E+01	3.39E+01	3.45E+01	<8.62E+00	<8.74E+00	<8.77E+00	<8.76E+00
133	1.22E+01	1.21E+01	1.21E+01	1.23E+01	<2.02E+00	<2.99E+00	<2.18E+00	<1.64E+00
138	1.36E+02	1.33E+02	1.36E+02	1.37E+02	7.54E+00	1.20E+01	5.43E+00	1.19E+01
139	1.36E+02	1.31E+02	1.30E+02	1.39E+02	2.56E+01	2.58E+01	2.56E+01	2.35E+01
140	1.28E+02	1.37E+02	1.31E+02	1.32E+02	6.86E+00	4.81E+00	4.76E+00	4.16E+00
141	1.25E+02	1.14E+02	1.22E+02	1.20E+02	2.36E+01	2.28E+01	2.33E+01	2.22E+01
142	1.28E+02	1.22E+02	1.27E+02	1.31E+02	3.61E+00	3.72E+00	3.62E+00	3.08E+00
143	1.12E+02	1.12E+02	1.14E+02	1.16E+02	2.70E+01	2.51E+01	2.53E+01	2.73E+01
144	1.25E+02	1.30E+02	1.24E+02	1.36E+02	2.46E+01	2.51E+01	2.62E+01	2.50E+01
145	8.19E+01	8.31E+01	8.16E+01	8.72E+01	1.80E+01	1.85E+01	1.88E+01	1.91E+01

3002 m/z	Aqua regia ICP-MS Slurry, mg/kg				Supernate, mg/L			
	1 92314 μg/g	2 92315 μg/g	3 92316 μg/g	4 92317 μg/g	1 92446 mg/L	2 92447 mg/L	3 92448 mg/L	4 92449 mg/L
146	6.73E+01	6.69E+01	6.50E+01	7.04E+01	1.58E+01	1.47E+01	1.47E+01	1.49E+01
147	4.02E+01	4.38E+01	4.31E+01	4.23E+01	9.08E+00	1.01E+01	8.46E+00	1.06E+01
148	4.14E+01	4.26E+01	4.02E+01	4.95E+01	9.50E+00	1.04E+01	8.42E+00	9.90E+00
150	3.56E+01	3.82E+01	3.98E+01	4.44E+01	8.85E+00	8.57E+00	8.54E+00	8.62E+00
152	1.22E+01	1.42E+01	1.35E+01	1.08E+01	3.23E+00	3.12E+00	2.93E+00	2.47E+00
153	7.30E+00	7.27E+00	7.27E+00	7.39E+00	1.36E+00	1.75E+00	1.26E+00	1.13E+00
154	6.01E+00	5.41E+00	6.51E+00	7.25E+00	2.17E+00	2.28E+00	1.98E+00	2.35E+00
155	2.24E+01	2.21E+01	2.36E+01	2.31E+01	1.04E+01	7.83E+00	8.05E+00	8.03E+00
156	3.23E+01	3.00E+01	3.20E+01	3.17E+01	1.33E+01	1.31E+01	1.18E+01	1.24E+01
157	2.24E+01	2.10E+01	2.26E+01	2.92E+01	9.22E+00	9.52E+00	8.92E+00	9.61E+00
158	3.28E+01	3.55E+01	3.73E+01	3.59E+01	1.54E+01	1.63E+01	1.52E+01	1.46E+01
160	3.41E+01	3.46E+01	3.08E+01	3.79E+01	1.27E+01	1.21E+01	1.18E+01	1.26E+01
235	6.38E+01	6.44E+01	6.27E+01	6.52E+01	2.54E+01	2.19E+01	2.18E+01	2.18E+01
236	<4.94E+00	<6.13E+00	<6.75E+00	<5.48E+00	<4.31E+00	<4.37E+00	<4.39E+00	<4.38E+00
237	1.36E+01	2.16E+01	1.77E+01	1.37E+01	9.22E+00	7.64E+00	8.00E+00	9.97E+00
238	8.87E+03	8.63E+03	8.68E+03	9.24E+03	3.47E+03	3.39E+03	3.41E+03	3.52E+03
239	6.38E+01	6.41E+01	5.74E+01	5.82E+01	5.60E+00	5.65E+00	5.70E+00	6.93E+00

Table D - 11. SRAT Product pH 3 ICP-MS

3002 m/z	Aqua regia ICP-MS, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92366 μg/g	2 92367 μg/g	3 92368 μg/g	4 92369 μg/g	1 92468 mg/L	2 92469 mg/L	3 92470 mg/L	92471 mg/L
59	<1.74E+01	<1.72E+01	<2.07E+01	<1.90E+01	<6.38E+00	<6.41E+00	<6.48E+00	<6.62E+00
88	4.62E+01	5.04E+01	5.04E+01	5.36E+01	4.22E+01	4.08E+01	4.60E+01	4.63E+01
89	5.13E+01	5.17E+01	4.96E+01	5.11E+01	3.14E+01	3.54E+01	2.89E+01	3.10E+01
90	4.92E+01	4.03E+01	5.00E+01	4.97E+01	3.85E+01	4.78E+01	4.73E+01	4.66E+01
91	<3.54E+01	<2.17E+01	<3.99E+01	<3.55E+01	<1.06E+01	<1.07E+01	<1.08E+01	<1.10E+01
92	3.87E+01	<2.32E+01	3.99E+01	4.28E+01	1.19E+01	6.94E+00	<6.48E+00	6.67E+00
93	4.13E+01	3.10E+01	4.86E+01	4.46E+01	9.47E+00	1.02E+01	8.55E+00	1.13E+01
94	4.42E+01	2.88E+01	3.93E+01	4.64E+01	7.44E+00	8.34E+00	7.46E+00	1.06E+01
96	4.25E+01	2.63E+01	4.90E+01	4.16E+01	8.78E+00	7.56E+00	6.91E+00	1.10E+01
101	6.57E+01	6.64E+01	6.98E+01	6.26E+01	2.46E+01	2.54E+01	2.64E+01	2.94E+01
102	6.24E+01	6.33E+01	6.23E+01	5.71E+01	2.41E+01	2.33E+01	2.47E+01	2.69E+01
103	<3.33E+01	<3.07E+01	3.32E+01	<3.31E+01	<8.51E+00	<8.54E+00	9.90E+00	<8.83E+00
104	3.47E+01	3.35E+01	3.46E+01	3.61E+01	1.71E+01	1.52E+01	1.43E+01	1.54E+01
110	8.00E+00	7.49E+00	9.64E+00	8.88E+00	5.13E+00	5.03E+00	5.53E+00	4.88E+00
111	<1.06E+01	<9.19E+00	<8.19E+00	<1.05E+01	<1.49E+01	<1.49E+01	<1.51E+01	<1.54E+01
112	1.74E+01	1.54E+01	1.65E+01	1.88E+01	7.32E+00	1.13E+01	7.70E+00	1.01E+01
114	2.17E+01	1.89E+01	2.06E+01	2.14E+01	1.12E+01	1.73E+01	1.66E+01	1.18E+01
116	<7.76E+00	8.40E+00	<6.83E+00	<6.31E+00	<4.25E+00	4.93E+00	<4.32E+00	<4.41E+00
119	9.76E+01	9.74E+01	1.03E+02	1.04E+02	8.97E+01	9.76E+01	9.06E+01	1.00E+02
125	<1.10E+01	<1.13E+01	<1.10E+01	<1.10E+01	<1.91E+01	<1.92E+01	<1.94E+01	<1.99E+01
133	<1.10E+01	1.13E+01	<1.10E+01	<1.10E+01	<8.51E+00	8.65E+00	<8.63E+00	<8.83E+00
138	1.30E+02	1.29E+02	<1.24E+02	<1.30E+02	1.11E+01	1.29E+01	<1.08E+01	<1.10E+01
139	1.20E+02	1.27E+02	1.27E+02	1.22E+02	4.91E+01	4.85E+01	4.92E+01	4.96E+01
140	<1.24E+02	<1.25E+02	<1.24E+02	<1.22E+02	<1.06E+01	<1.07E+01	<1.08E+01	<1.10E+01
141	1.08E+02	1.06E+02	1.11E+02	1.08E+02	4.48E+01	4.26E+01	4.72E+01	5.43E+01
142	1.13E+02	1.09E+02	1.13E+02	1.12E+02	6.87E+00	8.87E+00	5.91E+00	6.57E+00
143	1.04E+02	1.08E+02	1.11E+02	1.05E+02	4.78E+01	5.79E+01	4.71E+01	4.87E+01
144	1.17E+02	1.12E+02	1.14E+02	1.15E+02	4.42E+01	4.18E+01	4.24E+01	4.52E+01
145	7.22E+01	7.74E+01	7.31E+01	7.42E+01	3.25E+01	3.55E+01	3.32E+01	4.09E+01

3002 m/z	Aqua regia ICP-MS, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92366 μg/g	2 92367 μg/g	3 92368 μg/g	4 92369 μg/g	1 92468 mg/L	2 92469 mg/L	3 92470 mg/L	92471 mg/L
146	6.65E+01	6.34E+01	6.26E+01	6.39E+01	2.78E+01	2.95E+01	3.35E+01	2.89E+01
147	4.10E+01	3.74E+01	3.91E+01	4.14E+01	1.74E+01	1.63E+01	1.77E+01	2.33E+01
148	3.53E+01	3.82E+01	4.15E+01	3.67E+01	1.70E+01	1.98E+01	1.65E+01	2.01E+01
150	3.60E+01	3.39E+01	3.72E+01	3.63E+01	1.60E+01	1.62E+01	1.58E+01	1.56E+01
152	1.39E+01	1.15E+01	1.19E+01	1.16E+01	7.64E+00	6.51E+00	7.03E+00	5.80E+00
153	<7.36E+00	<7.50E+00	<7.35E+00	<7.35E+00	<4.25E+00	<4.27E+00	<4.32E+00	<4.41E+00
154	5.66E+00	<5.95E+00	<6.65E+00	<6.48E+00	4.32E+00	<4.27E+00	<4.32E+00	<4.41E+00
155	2.11E+01	2.01E+01	2.02E+01	2.26E+01	1.70E+01	1.76E+01	1.64E+01	1.61E+01
156	3.00E+01	2.89E+01	2.93E+01	2.98E+01	2.35E+01	2.61E+01	2.37E+01	2.61E+01
157	2.08E+01	1.99E+01	1.91E+01	2.07E+01	1.85E+01	2.05E+01	2.21E+01	2.32E+01
158	3.56E+01	3.15E+01	3.53E+01	3.45E+01	2.84E+01	2.37E+01	2.58E+01	2.79E+01
160	3.16E+01	3.00E+01	3.19E+01	3.02E+01	2.49E+01	2.68E+01	2.77E+01	2.66E+01
235	6.13E+01	6.04E+01	6.25E+01	6.23E+01	6.51E+01	6.87E+01	6.48E+01	6.64E+01
236	4.50E+00	<3.75E+00	<5.70E+00	<4.30E+00	6.41E+00	<4.27E+00	<4.32E+00	<4.41E+00
237	1.68E+01	1.36E+01	1.73E+01	1.37E+01	1.12E+01	1.36E+01	1.12E+01	1.37E+01
238	8.97E+03	8.60E+03	8.86E+03	8.58E+03	9.38E+03	8.82E+03	9.21E+03	9.39E+03
239	6.30E+01	5.57E+01	6.04E+01	5.81E+01	1.92E+01	1.93E+01	2.61E+01	2.03E+01

Table D - 12. SRAT Product pH 2 ICP-MS

3002 m/z	Aqua regia ICP-MS Slurry, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92359 μg/g	2 92360 μg/g	3 92361 μg/g	4 92362 μg/g	1 92352 mg/L	2 92353 mg/L	3 92354 mg/L	4 92355 mg/L
59	1.87E+01	2.12E+01	1.74E+01	2.10E+01	7.79E+00	<6.66E+00	1.41E+01	<6.75E+00
88	4.89E+01	4.86E+01	4.91E+01	4.75E+01	5.01E+01	4.86E+01	4.39E+01	4.37E+01
89	5.18E+01	5.38E+01	5.05E+01	5.25E+01	3.36E+01	3.15E+01	3.02E+01	3.06E+01
90	6.57E+01	6.72E+01	6.62E+01	6.57E+01	5.29E+01	5.44E+01	5.13E+01	5.08E+01
91	2.47E+01	5.16E+01	2.74E+01	3.63E+01	1.03E+01	9.08E+00	1.12E+01	8.28E+00
92	<2.68E+01	5.16E+01	<3.13E+01	4.78E+01	<1.10E+01	<1.11E+01	1.15E+01	<1.13E+01
93	<2.38E+01	6.48E+01	<3.35E+01	4.97E+01	1.03E+01	1.12E+01	1.18E+01	1.44E+01
94	2.63E+01	6.25E+01	2.97E+01	4.59E+01	9.86E+00	7.89E+00	9.94E+00	1.07E+01
96	2.45E+01	5.69E+01	3.70E+01	4.66E+01	1.20E+01	1.24E+01	1.21E+01	<1.13E+01
101	7.19E+01	7.15E+01	6.94E+01	7.28E+01	3.26E+01	2.55E+01	2.80E+01	3.00E+01
102	6.38E+01	6.22E+01	6.08E+01	6.29E+01	3.33E+01	2.77E+01	2.52E+01	2.45E+01
103	3.13E+01	3.03E+01	3.17E+01	3.42E+01	1.30E+01	9.64E+00	1.28E+01	1.04E+01
104	3.19E+01	3.40E+01	3.46E+01	3.46E+01	1.84E+01	1.40E+01	1.36E+01	1.46E+01
110	7.90E+00	7.93E+00	7.10E+00	8.81E+00	7.17E+00	4.44E+00	<6.83E+00	5.70E+00
111	1.17E+01	<8.53E+00	1.04E+01	<7.71E+00	<1.32E+01	<1.33E+01	<1.36E+01	<1.35E+01
112	1.55E+01	1.76E+01	1.80E+01	1.48E+01	8.33E+00	7.25E+00	7.63E+00	8.21E+00
114	2.26E+01	2.17E+01	2.38E+01	2.48E+01	1.20E+01	1.52E+01	1.28E+01	1.47E+01
116	<7.76E+00	<6.53E+00	<7.88E+00	<6.29E+00	<6.61E+00	<6.66E+00	<6.80E+00	<6.75E+00
119	8.80E+01	9.06E+01	8.26E+01	8.63E+01	1.05E+02	8.07E+01	8.75E+01	8.53E+01
125	<2.19E+01	<2.23E+01	<2.21E+01	<2.19E+01	<1.32E+01	<1.33E+01	<1.36E+01	<1.35E+01
133	<5.10E+00	<5.07E+00	<4.91E+00	<4.86E+00	8.02E+00	6.76E+00	6.85E+00	6.75E+00
138	1.40E+02	1.32E+02	1.33E+02	1.36E+02	8.26E+00	5.75E+00	5.37E+00	<4.50E+00
139	1.38E+02	1.30E+02	1.28E+02	1.30E+02	6.17E+01	6.11E+01	6.14E+01	5.62E+01
140	1.35E+02	1.31E+02	1.28E+02	1.33E+02	8.85E+00	9.46E+00	7.93E+00	7.09E+00
141	1.14E+02	1.14E+02	1.16E+02	1.18E+02	5.74E+01	5.02E+01	5.05E+01	5.18E+01
142	<1.16E+02	<1.17E+02	<1.18E+02	<1.24E+02	8.82E+00	8.88E+00	9.07E+00	9.01E+00
143	1.12E+02	1.16E+02	1.10E+02	1.19E+02	5.52E+01	5.65E+01	5.52E+01	5.82E+01
144	1.26E+02	1.24E+02	1.23E+02	1.26E+02	5.61E+01	5.03E+01	5.06E+01	4.72E+01
145	7.96E+01	7.99E+01	7.65E+01	7.84E+01	3.87E+01	4.27E+01	3.75E+01	3.52E+01

3002 m/z	Aqua regia ICP-MS Slurry, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92359 μg/g	2 92360 μg/g	3 92361 μg/g	4 92362 μg/g	1 92352 mg/L	2 92353 mg/L	3 92354 mg/L	4 92355 mg/L
146	5.86E+01	6.95E+01	6.68E+01	6.10E+01	3.68E+01	3.09E+01	3.09E+01	3.20E+01
147	4.08E+01	4.40E+01	3.72E+01	4.10E+01	2.29E+01	1.70E+01	2.28E+01	1.84E+01
148	4.63E+01	4.39E+01	3.84E+01	4.14E+01	2.27E+01	2.16E+01	1.89E+01	1.91E+01
150	4.21E+01	4.13E+01	3.87E+01	4.09E+01	2.05E+01	1.92E+01	1.97E+01	2.13E+01
152	1.28E+01	1.34E+01	1.08E+01	1.30E+01	7.37E+00	5.79E+00	6.82E+00	8.31E+00
153	<5.30E+00	<5.86E+00	7.85E+00	6.44E+00	<4.41E+00	<4.44E+00	<5.98E+00	<4.50E+00
154	<5.63E+00	<6.64E+00	8.25E+00	9.06E+00	5.06E+00	5.77E+00	5.76E+00	6.20E+00
155	2.26E+01	2.35E+01	2.19E+01	2.26E+01	2.01E+01	2.22E+01	1.95E+01	1.73E+01
156	3.54E+01	3.49E+01	3.17E+01	2.92E+01	2.90E+01	2.58E+01	2.63E+01	2.54E+01
157	2.42E+01	2.31E+01	2.40E+01	2.07E+01	1.93E+01	2.05E+01	1.72E+01	2.07E+01
158	3.39E+01	3.74E+01	3.49E+01	3.52E+01	3.24E+01	2.96E+01	2.69E+01	2.76E+01
160	3.35E+01	3.30E+01	3.17E+01	3.41E+01	2.67E+01	2.47E+01	2.85E+01	2.94E+01
235	6.43E+01	6.81E+01	6.35E+01	5.88E+01	5.25E+01	5.87E+01	5.71E+01	5.77E+01
236	<9.74E+00	<9.93E+00	<9.81E+00	<9.73E+00	<1.54E+01	<1.55E+01	<1.59E+01	<1.58E+01
237	1.27E+01	1.44E+01	1.99E+01	1.58E+01	1.03E+01	1.09E+01	1.47E+01	1.43E+01
238	8.90E+03	9.02E+03	8.83E+03	8.84E+03	8.21E+03	7.78E+03	7.54E+03	7.76E+03
239	6.57E+01	7.49E+01	7.18E+01	5.84E+01	2.78E+01	2.66E+01	2.11E+01	2.12E+01

Table D - 13. SRAT Product pH 1 ICP-MS

3002 m/z	Aqua regia ICP-MS, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92329 μg/g	2 92330 μg/g	3 92331 μg/g	4 92332 μg/g	1 92563 mg/L	2 92564 mg/L	3 92565 mg/L	4 92566 mg/L
59	2.09E+01	1.72E+01	1.88E+01	1.80E+01	5.60E+00	8.48E+00	6.48E+00	1.02E+01
88	5.28E+01	5.07E+01	5.56E+01	4.90E+01	4.63E+01	4.92E+01	5.12E+01	5.82E+01
89	5.70E+01	5.50E+01	5.39E+01	5.12E+01	3.60E+01	3.52E+01	3.55E+01	4.03E+01
90	6.14E+01	6.10E+01	4.88E+01	5.09E+01	3.89E+01	4.13E+01	4.46E+01	4.36E+01
91	5.03E+01	5.40E+01	3.42E+01	3.94E+01	6.12E+00	5.98E+00	6.58E+00	5.49E+00
92	<5.06E+01	<6.06E+01	<4.90E+01	<4.52E+01	<1.14E+01	<1.13E+01	<1.13E+01	<1.14E+01
93	<6.89E+01	<6.29E+01	<4.90E+01	<5.33E+01	<1.14E+01	<1.13E+01	<1.13E+01	<1.14E+01
94	6.11E+01	8.17E+01	4.16E+01	5.13E+01	7.17E+00	8.26E+00	5.70E+00	5.98E+00
96	6.54E+01	6.17E+01	4.61E+01	5.35E+01	6.64E+00	7.98E+00	9.92E+00	7.59E+00
101	7.22E+01	6.82E+01	7.15E+01	6.90E+01	3.01E+01	2.39E+01	2.70E+01	2.45E+01
102	6.28E+01	6.50E+01	5.98E+01	6.47E+01	2.57E+01	2.24E+01	2.29E+01	2.67E+01
103	3.91E+01	3.66E+01	5.12E+01	2.97E+01	9.10E+00	8.29E+00	6.77E+00	8.51E+00
104	3.72E+01	3.58E+01	3.50E+01	3.42E+01	1.38E+01	1.54E+01	1.48E+01	1.33E+01
110	7.31E+00	<7.42E+00	<1.13E+01	7.96E+00	7.19E+00	<4.53E+00	<4.50E+00	6.77E+00
111	<9.25E+00	<8.79E+00	9.63E+00	8.37E+00	<6.83E+00	<6.80E+00	1.10E+01	7.16E+00
112	1.81E+01	2.12E+01	1.76E+01	1.80E+01	1.55E+01	9.18E+00	1.04E+01	9.12E+00
114	2.12E+01	2.27E+01	2.12E+01	1.88E+01	1.31E+01	1.27E+01	1.57E+01	1.38E+01
116	<7.05E+00	<7.02E+00	<6.26E+00	<8.55E+00	<4.55E+00	<4.53E+00	<4.50E+00	<4.54E+00
119	7.95E+01	8.37E+01	8.10E+01	8.12E+01	8.59E+01	8.51E+01	7.84E+01	8.43E+01
125	<9.96E+00	<9.83E+00	9.89E+00	<9.46E+00	<4.55E+00	4.53E+00	<5.64E+00	<4.54E+00
133	<4.76E+00	<4.75E+00	<4.30E+01	<3.55E+00	<4.55E+00	<4.53E+00	<4.50E+00	<4.54E+00
138	1.38E+02	1.35E+02	1.33E+02	1.28E+02	1.27E+01	1.01E+01	9.18E+00	4.03E+01
139	1.36E+02	1.36E+02	1.31E+02	1.30E+02	6.46E+01	6.59E+01	6.09E+01	6.96E+01
140	1.34E+02	1.32E+02	1.26E+02	1.27E+02	8.17E+00	9.73E+00	8.52E+00	1.62E+01
141	1.15E+02	1.16E+02	1.12E+02	1.13E+02	6.27E+01	5.76E+01	5.88E+01	5.74E+01
142	<1.21E+02	<1.22E+02	<1.20E+02	<1.20E+02	<9.11E+00	<9.07E+00	<9.00E+00	1.02E+01
143	1.15E+02	1.15E+02	1.17E+02	1.17E+02	5.96E+01	6.09E+01	6.20E+01	6.00E+01
144	1.23E+02	1.21E+02	1.18E+02	1.19E+02	5.79E+01	5.54E+01	5.20E+01	5.69E+01
145	8.04E+01	8.20E+01	8.06E+01	8.09E+01	4.51E+01	4.27E+01	4.58E+01	4.40E+01

3002 m/z	Aqua regia ICP-MS, mg/kg Slurry Basis				Supernate Basis, mg/L			
	1 92329 μg/g	2 92330 μg/g	3 92331 μg/g	4 92332 μg/g	1 92563 mg/L	2 92564 mg/L	3 92565 mg/L	4 92566 mg/L
146	6.52E+01	6.33E+01	6.39E+01	6.53E+01	3.48E+01	3.89E+01	3.55E+01	3.63E+01
147	4.06E+01	4.32E+01	4.27E+01	4.45E+01	2.74E+01	2.68E+01	2.40E+01	2.17E+01
148	4.17E+01	4.26E+01	3.82E+01	3.83E+01	2.46E+01	2.28E+01	2.28E+01	2.41E+01
150	3.67E+01	3.62E+01	3.77E+01	3.66E+01	2.22E+01	2.26E+01	2.12E+01	2.04E+01
152	1.20E+01	1.28E+01	1.21E+01	1.26E+01	7.14E+00	6.94E+00	7.72E+00	8.08E+00
153	6.60E+00	<5.02E+00	<5.03E+00	6.93E+00	4.67E+00	<4.53E+00	<4.50E+00	5.67E+00
154	5.92E+00	4.79E+00	<5.66E+00	<5.66E+00	5.64E+00	5.70E+00	<4.50E+00	<4.54E+00
155	2.34E+01	1.93E+01	2.05E+01	2.10E+01	1.78E+01	2.15E+01	1.80E+01	1.80E+01
156	3.54E+01	3.36E+01	3.16E+01	3.34E+01	2.50E+01	2.65E+01	2.65E+01	2.36E+01
157	2.36E+01	2.21E+01	2.05E+01	2.12E+01	1.68E+01	2.09E+01	1.98E+01	1.82E+01
158	3.66E+01	3.59E+01	3.63E+01	3.56E+01	2.96E+01	3.34E+01	3.04E+01	2.67E+01
160	3.30E+01	3.17E+01	3.31E+01	3.14E+01	2.23E+01	2.56E+01	2.86E+01	2.33E+01
235	6.27E+01	6.49E+01	6.33E+01	6.15E+01	6.17E+01	6.06E+01	5.26E+01	5.84E+01
236	<4.40E+00	<5.29E+00	<5.17E+00	<5.64E+00	<9.11E+00	<9.07E+00	<9.00E+00	<9.09E+00
237	1.16E+01	1.37E+01	1.55E+01	1.44E+01	1.32E+01	1.60E+01	1.31E+01	1.38E+01
238	9.42E+03	9.17E+03	9.48E+03	9.32E+03	8.41E+03	8.47E+03	8.40E+03	8.16E+03
239	6.57E+01	6.14E+01	6.78E+01	6.62E+01	1.94E+01	1.98E+01	<1.58E+01	<1.59E+01

Distribution:

A. B. Barnes, 999-W
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
J. M. Gillam, 766-H
J. F. Iaukea, 704-30S
J. E. Occhipinti, 704-S
W. O Pepper, 704-S
D. K. Peeler, 999-W
J. W. Ray, 704-S
H. B. Shah, 766-H
D. C. Sherburne, 704-S
M. E. Stone, 999-W
B. R. Pickenheim, 704-28S
J. R. Zamecnik, 999-W
M. A. Broome, 704-29S
R. N. Hinds, 704-S
J. P. Vaughan, 773-41A
J. M. Bricker, 704-27S
T. L. Fellingner, 704-26S
E. W. Holtzscheiter, 704-15S
A. V. Staub, 704-27S
K. R. Shah, 704-S
M. E. Smith, 704-30S
M. T. Keefer, 766-H
D. P. Lambert, 999-W
D. C. Koopman, 999-W
J. D. Newell, 999-W
D. R. Best, 999-W
R. E. Eibling, 999-W
W. T. Riley, 999-W
P. R. Jackson, 703-46A