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Radioactive Waste Sludge Washing and Demonstration of the Nitric-Glycolic Acid Flowsheet for Sludge Batch 10 Qualification

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EXECUTIVE SUMMARY

For each sludge batch that is processed in the Defense Waste Processing Facility (DWPF), the Savannah River National Laboratory (SRNL) performs qualification testing to demonstrate that the sludge batch (SB) is processible. During processing of SB9, DWPF will be transitioning from the Nitric-Formic Acid (NFA) flowsheet to the Nitric-Glycolic Acid (NGA) flowsheet. Thus, the qualification of SB10 was requested to only be performed using the NGA flowsheet.

In order to qualify the batch for the NGA flowsheet, Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) cycles, designated SC-19, were performed using SB10 Tank 51 sample material. SRNL received Tank 51 material in the midst of Tank Farm washing. SRNL continued the washing in the SRNL Shielded Cells. The SRNL process included the addition of Sodium Reactor Experiment (SRE) material from H Canyon, simulating the transfer of SRE from H Canyon to Tank 51 during Tank Farm washing. The washed SB10 Tank 51 material, with SRE, was characterized prior to flowsheet qualification testing.

The Chemical Process Cell demonstration with the NGA flowsheet utilized an acid stoichiometry of 107% Koopman Minimum Acid basis (102% Hsu basis). The qualification was performed on the "batch", which is representative of the Tank 51 material that will be transferred to Tank 40, rather than the "blend", which is representative of the Tank 40 material that will be fed to DWPF. Thus, specifics for the DWPF processing of the Tank 40 blend may differ in varying degrees from this qualification test. Additionally, the qualification did not include Salt Waste Processing Facility (SWPF) feeds (Monosodium Titanate (MST)/Sludge Solids and Strip Effluent), which would contribute soluble salts and the MST solids, which could influence the acid addition and the product rheology, respectively. For these reasons, the results of this qualification testing demonstration are used in conjunction with simulant testing to inform the processing of the Tank 40 blend with inclusion of SWPF feeds.

Highlights of the testing results are summarized in the paragraphs below.

Prior to the addition of antifoam, foaming was encountered in the SRAT cycle during the addition of nitric acid to the extent that sludge foamed over into the off-gas system. Based on observations from simulant testing, during which foaming was not noted during nitric acid addition, the plan involved addition of Momentive^{TM} Y-17112 antifoam after the completion of nitric acid addition and prior to glycolic acid addition. The foaming during nitric acid addition was likely due to the release of carbon dioxide from the acid reacting with carbonate. After a time out (3 weeks), the SRAT cycle was resumed with an antifoam addition prior to resumption of nitric acid addition. Foaming was successfully mitigated during the remainder of the SRAT and SME cycles.

Total dried solids measurements of the SRAT and SME Products were 20.1 and 48.1 weight percent (wt%), respectively. Calcined solids measured 11.3 wt% in the SRAT Receipt and SRAT Product, and 37.7 wt% in the SME Product. Waste loading in the SME Product was 33.9% based on iron concentration in the SRAT and SME Products, versus the target of 36% waste loading. When other elements were considered, the waste loading averaged 34.4%.

The pH stayed below 4.0 during qualification test SRAT and SME processing. Nitrite was destroyed to below the detection limit (<130 mg/L) during the SRAT cycle. Glycolate destruction was 9.0% during the SRAT cycle and 5.5% during the SME cycle.

The SRAT Receipt Bingham-Plastic yield stress averaged 8.3 Pa and viscosity averaged 8.0 cP. The SRAT Product yield stress averaged 0.83 Pa with a viscosity of 3.9 cP. This is below the lower range of the SRAT

Product design basis of 1.5 Pa and 5 cP, which would be expected in this case where the total dried solids content of the SRAT Product was below the target. The SME Product yield stress averaged 2.3 Pa with a viscosity of 10.0 cP. This was very close to the lower limit of the SME Product design basis of 2.5 Pa and 10 cP. SB10 qualification SME Product was significantly closer to the DWPF design basis rheology range than the similar NGA qualification for SB9.

The off-gas peak observed hydrogen generation rate scaled to 6,000 gallons of SRAT and SME feed was 2.7×10^{-4} lb/h in both the SRAT and SME cycles, which is approximately 90-times less than the DWPF NGA flowsheet limit of 2.4×10^{-2} lb/h. Methane was sporadically detected at low concentrations in both the SRAT and SME cycles.

A series of glasses were prepared from dried SB10 SME Product in sealed crucibles in order to determine the glass REDOX ratio (Fe(II)/ΣFe). Ten total analyses of three separate sealed crucible glasses yielded an average iron REDOX of 0.062 versus the predicted REDOX from the SME Product slurry analysis of 0.10 and the target REDOX of 0.1.

Additional glass was produced from the SME Product, digested and chemically analyzed. All Product Composition Control System (PCCS) criteria were met.

Both SRAT and SME condensate were generally dilute, but both contained measurable quantities of nitrate, glycolate, total mercury, methylmercury, and elemental mercury, with the SME condensate being generally more concentrated than the SRAT condensate. Formate was additionally present in the SME condensate. Glycolate concentrations in the SRAT and SME condensates averaged 32.2 mg/L and 60.5 mg/L, respectively. Ammonium was less than 100 mg/L in the SRAT concentrate and was 30 mg/L in the SME condensate. No antifoam degradation products seen with the previous antifoam (i.e., hexamethyldisiloxane, trimethylsilanol, and propanal) were noted in either condensate.

Final mercury concentration in the SRAT Product was 0.77 wt% of the total dried solids, which met the current target of <0.8 wt% mercury in the SRAT Product total dried solids. Mercury was further removed to 0.16 wt% of the SME Product total dried solids. Mercury speciation in the SRAT Receipt, SRAT Product, and SME Product identified soluble methylmercury, ionic mercury, and elemental mercury in the supernatant associated with each slurry.

Based on the results of this radioactive-waste testing, SRNL has qualified SB10 with the NGA flowsheet. SRNL demonstrated acceptable mercury stripping, slurry rheology, off-gas composition, and glass REDOX at a single acid addition amount to a Tank 51 material. Specific recommendations for processing SB10 are based on a series of simulant tests and are published in a separate document.

Due to the foaming encountered during nitric acid addition (which was prior to the addition of antifoam for this test), it is recommended that antifoam be added prior to nitric acid addition.

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LIST OF ABBREVIATIONS

ACSM	Analytical Characterization and Sample Management
AF	alkali fusion
AR	aqua regia
ARG-1	Analytical Reference Glass-1
CQ	caustic quench preparation
CPC	Chemical Process Cell
CS	calcined solids
CVAFS	Cold Vapor Atomic Fluorescence Spectroscopy
DAC	Data Acquisition and Control
DMA	Direct Mercury Analysis
DS	dissolved solids in the supernate
DWPF	Defense Waste Processing Facility
EA	Environmental Assessment
ES	shorthand for ICP-ES
FAVC	Formic Acid Vent Condenser
GC	micro gas chromatograph
GC-MS	Gas Chromatography with Mass Spectrometry
HDPE	high-density polyethylene
HMDSO	hexamethyldisiloxane
IC	ion chromatography
ICA	Ion Chromatography for Anions
ICA-G	Ion Chromatography for Anions, Glycolate measurement
ICC	Ion Chromatography for Cations
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
IS	Insoluble Solids in the slurry
LOQ	limit of quantification
LSC	Liquid Scintillation Counting
MDL	method detection limit
MS	shorthand for ICP-MS
MST	Monosodium Titanate
multi	Multiple methods used
MWWT	Mercury Water Wash Tank
n	number of replicates
N/A	Not Applicable
n.d.	Not Determined
ND	Not Detected

NFA	Nitric-Formic Acid
NGA	Nitric-Glycolic Acid
NIST	National Institute of Standards and Technology
NM	Nuclear Measurements
n.m.	not measured
P&T-TD-CVAFS	Purge & Trap, Thermal Desorption, CVAFS
PCCS	Product Composition Control System
PCT	Product Consistency Test
ppm_v	parts per million volume
REDOX	reduction/oxidation
RSD	Relative Standard Deviation
SB	Sludge Batch
sccm	standard cubic centimeters per minute
SCFM	standard cubic feet per minute
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	Sludge Receipt and Adjustment Tank
SRE	Sodium Reactor Experiment
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SS	soluble solids in the slurry
SVOA	Semivolatile Organics Analysis
TIC	total inorganic carbon
TMSOH	trimethylsilanol
TOC	total organic carbon
TS	total dried solids in the slurry
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UV/Vis	ultraviolet/visible
VOA	Volatile Organic Analysis
vol. %	volume percent
WAPS	Waste Acceptance Product Specifications
wt%	weight percent

1.0 Introduction

For each sludge batch that is processed in the Defense Waste Processing Facility (DWPF), the Savannah River National Laboratory (SRNL) performs qualification testing to demonstrate that the sludge batch (SB) is processible. During processing of SB9, DWPF will be transitioning from the Nitric-Formic Acid (NFA) flowsheet to the Nitric-Glycolic Acid (NGA) flowsheet. Thus, this qualification of SB10 was requested to only be performed using the NGA flowsheet.

SRMC Engineering requested that the qualification be performed on the "batch," which is representative of the Tank 51 material that will be transferred to Tank 40, rather than the "blend," which is representative of the Tank 40 material that will be fed to DWPF. Thus, specifics for the DWPF processing of the Tank 40 blend may differ in varying degrees from this qualification test. Additionally, SRMC Engineering requested that the qualification not include any Salt Waste Processing Facility (SWPF) feeds (Monosodium Titanate (MST)/Sludge Solids or Strip Effluent). The lack of the MST/Sludge Solids stream excluded the additional soluble salts and the MST solids, which could influence the acid addition and the product rheology, respectively. The tasks covered by this report were requested by the Technical Task Request (TTR) and are governed by the Task Technical and Quality Assurance Plan (TTQAP).[1,2]

SRNL personnel have been requested to qualify the next sludge batch (SB10) for processing at DWPF.[1] To accomplish this task, Savannah River Mission Completion (SRMC)[a] sent SRNL two 3-L samples of Tank 51 slurry to be characterized, washed, and then used in a lab-scale demonstration of the DWPF flowsheet. Sample HTF-51-19-114 was received on January 28, 2020, and HTF-51-20-15 was received on February 4, 2020. SRNL has washed the combined Tank 51 samples per the Tank Farm washing strategy as of August 2020, and revised February 2021. During washing, material from H Canyon Tanks 16.3 and 16.4 was also added. A part of the qualification process is extensive radionuclide and chemical characterization of the SRNL-washed Tank 51 slurry, which has previously been reported.[3-4] Using the washed slurry, SRNL completed a small-scale demonstration of the DWPF Chemical Process Cell (CPC) utilizing the NGA flowsheet to support SB10 qualification.[1-2, 5-6] A sample of glass was fabricated from the Slurry Mix Evaporator (SME) Product for chemical characterization and evaluation against DWPF process control models. Samples for reduction/oxidation (REDOX) determination were also prepared and analyzed.

This report documents sludge washing, Sludge Receipt and Adjustment Tank (SRAT) and SME processing, characterization of the SRAT and SME Products, glass fabrication and analysis of glass.

2.0 Experimental

2.1 Analytical Techniques

2.1.1 Sampling

The first Tank 51 sample, nominally 3-L with a mass of 3,660 g, was received and placed into a 4-L high-density polyethylene (HDPE) bottle. The sample was mixed and a ~100 mL subsample was taken while maintaining mixing. The same subsampling process was used here as was developed and used for the Sludge Batch 9 waste acceptance product specification sample.[7] That subsample was used for corrosion control and evaporator feed qualification analyses.[8] The remainder of the first 3-L sample was transferred to an 8-L glass beaker. The second sample, also nominally 3-L and mass of 3,690 g, was removed from the Tank Farm sampler into a 4-L HDPE bottle to obtain a weight and approximate volume, then transferred into the 8-L beaker with the first sample. The contents of the 8-L beaker (nearly 6-L of Tank 51) were

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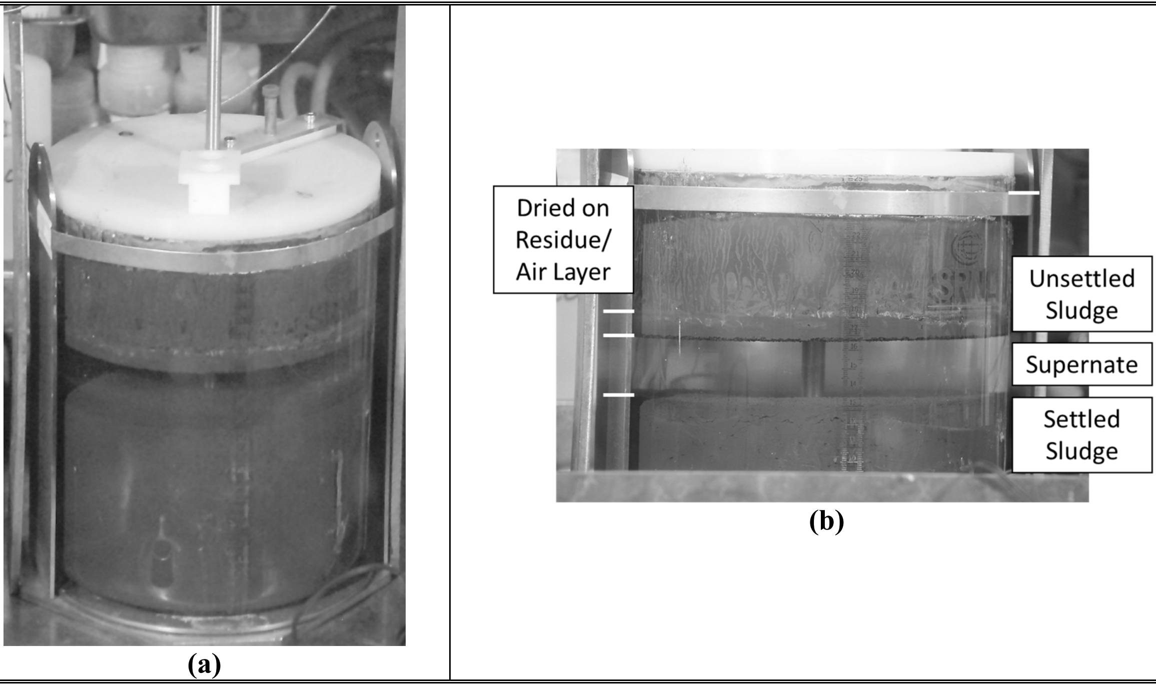
[a] The liquid waste contractor at the Savannah River Site transitioned from Savannah River Remediation to Savannah River Mission Completion on February 27, 2022. For simplicity, the liquid waste contractor is referred to as SRMC in this document.

subsampled as described above (mixing maintained during sampling) and a 500 mL subsample was taken. This 500 mL subsample was then used for characterization.

See Figure 2-1 for a photograph of the washing vessel (image a) and qualification sample HTF-51-19-114/HTF-51-20-15 (image b) at an intermediate state of sludge settling. Note the ports on the vessel (image a) that allow for sampling while mixing without sample tubing interfering with agitation. From top-to-bottom (image b), there was a thin coating of sludge solids on the vessel wall in the headspace, an approximately 0.5 to 1 cm region of sludge at the interface of the headspace, supernate, and vessel wall, an approximately 3.5 cm layer of supernate, and an approximately 13 inch layer of settled sludge.



(a) **(b)**

Figure 2-1. Photos of the Washing Vessel with Qualification Sample HTF-51-19-114/HTF-51-20-15, a) washing vessel and lid, b) side-on photo showing layers in the settled sample.

During the washing process, inhibited water was added to the slurry, the slurry was mixed for 30 minutes, and the agitation was stopped to allow the insoluble solids to settle. After a time of settling, the sample was decanted by pumping supernatant from the vessel. Several slurry samples were taken while agitating during washing. When pulling aliquots from the slurry samples, the subsamples were mixed by shaking these relatively small bottles. These slurry samples were pumped while the content of the larger vessel was being mixed. The final washed analytical slurry sample was also obtained in this way. At the conclusion of the SRAT and SME cycles, analytical samples were taken immediately after mixing was stopped.

During washing, decanted supernatant was utilized for aqueous phase characterization, including the washed sample; the final decant was used in this case. For the as-received analyses, SRAT Product and SME Product, slurry was filtered to obtain filtrate, which was used for aqueous phase characterization.

Details of the washing process and the SRAT and SME cycles are included in the Results and Discussion (Sections 3.1 and 3.2).

2.1.2 Density and Solids Distribution

Slurry and supernatant densities were determined gravimetrically from sample weights in vessels of known volume (plastic test tubes of nominally 8 mL capacity).

Aliquots of slurry and supernatant were dried to a constant weight (change between subsequent weights of <0.005 g or weight increase) at 110 °C for total dried solids (TS) and dissolved solids (DS), respectively, in weight percent (wt%). TS (in wt%) is the measured weight of dried slurry divided by the measured weight of wet slurry, multiplied by 100%. DS (in wt%) is the measured weight of dried supernate or filtrate divided by the measured wet weight of supernate or filtrate, multiplied by 100%. Insoluble solids (IS) and soluble solids (SS) were calculated in wt% from TS and DS measurements (see Eq. 2-1 and Eq. 2-2 and Marek[9] for a derivation of these equations).

$$IS(wt\%) = \frac{TS\ (wt\%) - DS(wt\%)}{100\% - DS(wt\%)} \times 100\% \qquad \text{Eq. 2-1}$$

$$SS(wt\%) = TS(wt\%) - IS(wt\%) \qquad \text{Eq. 2-2}$$

Dried slurry samples were heated to 1100 °C, held at that temperature for two hours, and then cooled and weighed to determine calcined solids (CS) in wt%.

2.1.3 Rheology Measurements

The rheological properties (shear stress as a function of shear rate) were measured on the following materials:

- SRNL-washed SB10 qualification sample
- SRAT Product
- SME Product

Rheological properties of radioactive samples were determined using a Haake VT550 rotoviscometer.[10] The VT550 is a Searle sensor system, where the bob rotates and the cup is fixed. The bob is rotated to vary the shear rate and the shear stress is measured. The instrument is equipped with a water jacket to facilitate temperature control. The plot of shear stress as a function of shear rate is then used to determine the rheological properties. For Savannah River Site (SRS) samples, the results are modeled as a Bingham plastic. The SRAT Receipt and Product samples are fit to a line from shear rates of 600 s^{-1} to 50 s^{-1}. The SME Product sample is fit to a line from shear rate of 300 s^{-1} to 50 s^{-1}. The slope of the line fit is reported as the viscosity (μ_∞) and the intercept as the Bingham plastic yield stress (τ_o). See Eq. 2-3:

$$\tau = \tau_o + \mu_\infty \dot{\gamma} \qquad \text{Eq. 2-3}$$

where:

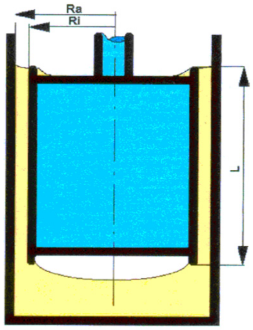
τ is the measured shear stress (Pa)
τ_o is the Bingham Plastic yield stress (Pa)
μ_∞ is the plastic viscosity (Pa·s)
$\dot{\gamma}$ is the shear rate (s^{-1})

Each sample was run twice, remaining in the rheometer cup for both runs.

The bob used for measuring the SRAT Receipt and SRAT Products is the MV I rotor. For SME Product, the slightly smaller MV II rotor is used to perform the measurements. The smaller rotor is needed so there is a larger gap between the rotor and cup, due to the larger frit particles that are present in the SME Product.

The shape, dimensions, and geometric constants for the MV I and MV II rotors, and other data are provided in Table 2-1.

Table 2-1. MV I and MV II Rotor Specifications and Flow Curve Program

Rotor Design	Dimensions and Flow Curve Program		
	Rotor Type	MV I	MV II
	Rotor radius - R_i (mm)	20.04	18.40
	Cup Radius - R_a (mm)	21.0	21.0
	Height of rotor -L (mm)	60	60
	Sample Volume (cm^3) minimum	40	55
	A factor (Pa/%torque)	3.22	3.76
	M factor (s^{-1}/%RPM)	11.7	4.51
	Shear rate range (s^{-1})	0 – 600	0 – 300
	Ramp up time (min)	5	5
	Hold time (min)	1	1
	Ramp down time (min)	5	5

2.1.4 General Sample Preparation Descriptions

The following are general descriptions of sample preparations and analytical methods. Specific preparations and analytical methods are presented with the results.

2.1.4.1 Elemental and Radiochemical Analysis of Slurry Solids

To characterize slurry, aliquots of slurry were digested and resulting liquids analyzed. Slurry samples were digested by two methods, aqua regia (AR) and alkali fusion (AF). For the AR digestions, aliquots of slurry were mixed with AR and heated in closed vessels for several hours at ~110 °C. The resulting liquids were diluted to 100 mL with water. For the AF digestions, aliquots of slurry were dried (~110 °C) and fused at 675 °C with sodium peroxide. The flux was then dissolved with nitric acid and diluted to 100 mL with water. Slurry aliquot amounts were chosen to target ~0.25 g of solids in each digestion. Details of the digestion methods can be found in Procedure L16.1-ADS-2226[11] for AR and Procedure L16.1-ADS-2502[12] for AF. The digestates were then submitted to Analytical Characterization and Sample Management (ACSM) for analyses. The results were then converted from a slurry basis to a wt% of TS basis using the measured TS. Reagent blanks and digested glass of known composition were processed and analyzed at the same time as process samples. Results of these samples were used in evaluating AR and AF slurry digestion effectiveness and evaluation of contamination in the samples from the Shielded Cells. Wherever possible, results from both methods are utilized, except for the following cases:

- The element Hg (total Hg) is determined from AR digestions by Direct Mercury Analysis (DMA). A portion of Hg would be volatilized in the AF digestion, therefore, AF digestions are not submitted for Hg analysis.
- For Al, AF and AR results are compared. If the two digestion results are within 10%, all digestion results are used. If they are not within 10%,the AF result was used.
- For Ca, results from the AR digestion are used. Ca is often detected in the AF blank, likely due to a Ca impurity in the reagent chemicals.
- For Na, S, and Zr, the AR digestion results are used. AFs utilize Na as a reagent and they are performed in Zr crucibles, thus these elements cannot be determined from AF. Some S may volatilize in the AF process, therefore, only the AR digestion is used for this element.
- For Si, results from the AF digestion are used. AR does not completely digest Si as shown in reference glass digestion results.

- Several elements were determined from Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS) results from AR digestions.
- When analytes are not detected in either digestion, the lower of the two methods is reported.

Table 2-2. Interpretation and assumptions for converting ICP-MS data to element concentrations.

Cobalt is mass 59. This ignores the relatively smaller concentrations of radioactive cobalt isotopes.

Mass 88 is attributable to non-radioactive strontium but does not account for the entire strontium element concentration.

Technetium is mass 99.

Ruthenium is the sum of masses 101, 102, and 104.

Rhodium is mass 103.

Palladium is mass 105 multiplied by 1.663 to compensate for isotopes of other masses that cannot be disambiguated from other components in the mixture. Palladium isotopic distribution assumed as that of uranium-235 fission products.

Silver is taken as the sum of masses 107 and 109 and potentially includes a minor interference from palladium.

Antimony is the sum of masses 121 and 123.

Tin is the sum of masses 117 through 120, 122, and 124.

Mass 133 is attributable to non-radioactive cesium but does not account for the entire cesium element concentration due to the presence of cesium-137. Cesium-135 and 137 cannot be disambiguated from barium-135 and 137 in the slurry.

Lanthanum is mass 139.

Cerium is the sum of masses 140 and 142.

Praseodymium is mass 141.

Neodymium is the sum of masses 143 through 146, 148, and 150. Mass 142 is not included due to the potential interference from cerium-142.

Europium is taken as the sum of masses 151 and 153.

Gadolinium is the sum of masses 155 through 158 and 160.

Terbium is mass 159.

Tantalum is mass 181.

Lead is the sum of masses 206, 207, and 208.

Thorium is mass 232. Contributions from other Th isotopes are insignificant for this evaluation.

Uranium is the sum of masses 233, 234, 235, 236 and 238. This ignores the very small fraction of mass 238 attributable to Plutonium-238.

Neptunium is mass 237.

Masses 239 and 240 are assumed to be due to plutonium but do not account for the entire plutonium element concentration.

Masses 241 and 242 are a combination of plutonium and americium and were not disambiguated.

Table 2-2 contains a description of how ICP-MS data was interpreted for elemental analysis. In converting ICP-MS data to elemental data, most of the elements could be slightly underestimated or overestimated based on the assumptions made and interferences from isotopes of another element. Ion Chromatography

(IC) analyses of slurry dilutions were used for Cl and F. It is assumed that Cl and F are soluble in the slurry (see Section 2.1.4.3).

Table 2-3 gives the 36 radionuclides to be reported for the SRNL-washed SB10 qualification sample.[1,2] Also needed are total alpha, total beta, total gamma, and total beta-gamma. These analyses are needed for DWPF Radiological Evaluation Program, DWPF Technical Safety Requirements/Waste Acceptance Criteria Evaluation, and the DWPF Solid Waste Characterization Program. Additional details on preparation and radiochemical methods used for SB10 qualification sample analysis is provided in the results report.

Table 2-3. SRNL-Washed SB10 Tank 51 Qualification Sample Requested Radionuclides to Report

Radionuclides			
H-3	Te-125m	Sm-151	Np-237
Co-60	I-129	Eu-152	Pu-238
Sr-90	Cs-134	Eu-154	Pu-239
Y-90	Cs-137	Eu-155	Pu-240
Tc-99	Ba-137m	U-233	Pu-241
Ru-106	Ce-144	U-234	Am-241
Rh-106	Pr-144	U-235	Am-242m
Ag-110m	Pr-144m	U-236	Cm-244
Sb-125	Pm-147	U-238	Cm-245

2.1.4.2 Mercury Analyses

Supernatant and condensate were analyzed for mercury. Supernatant was collected by decanting from a settled slurry. Condensate was taken during the SRAT and SME cycle dewatering periods. In addition to total mercury analysis by DMA,[13] other mercury species were analyzed from diluted decants of the supernates in the SRAT Receipt, SRAT Product and SME Product. Methylmercury and ethyl mercury were measured by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS).[14] Soluble elemental mercury (Hg^0) was measured by ACSM using a Purge & Trap, Thermal Desorption, CVAFS (P&T-TD-CVAFS) method.[15] A variation of this method was also developed by ACSM to analyze for inorganic mercury (Hg(I/II)).[16] Dimethyl mercury was analyzed using a semi-quantitative Volatile Organic Analysis (VOA) method that involves extraction followed by Gas Chromatography with Mass Spectrometry (GC-MS).[17]

Samples were prepared in the Shielded Cells by ACSM personnel using a nominal 1 mL sample:100 mL dilution of supernate decant aliquots into Teflon bottles containing high purity water with approximately 0.5% ultrapure HCl to preserve the various mercury species in the solution.[18] Unlike all the other SRAT Product supernate and SME Product supernate analyses that derived from filtration of slurry, filtration was not used for the mercury analyses to prevent any potential interaction with total mercury and mercury species with the filter media. Each quadruplicate analytical sample for each matrix was obtained by pouring off approximately 30 mL of the 100 mL diluted sample into a Teflon bottle to near zero-headspace. These unshielded 30-mL Teflon bottles were removed from the Shielded Cells and were immediately refrigerated until the various mercury analyses. A single blank was prepared for each matrix. It consisted of 1 mL high purity water added in the Shielded Cells to approximately 99 mL of a 0.5% ultrapure HCl/high purity de-ionized water mixture that had been previously prepared outside of the Shielded Cells.

2.1.4.3 Anions in Slurry

SRAT Receipt, SRAT Product, and SME Product slurry samples were diluted nominally 30× with de-ionized water. Unfiltered aliquots were submitted to ACSM for TIC/TOC, VOA, and (for the SRAT

Receipt) total base (base to reach pH of 7). After pulling unfiltered aliquots, the dilutions were allowed to settle and the aqueous portions were sampled and submitted for anions by IC.

The caustic quench (CQ) method was used for glycolate measurement in the SRAT and SME Product slurry samples.[19] In the CQ method, approximately 2 g of 50 wt% NaOH solution was mixed with approximately 10 g of slurry followed by dilution of a 1 mL aliquot of the CQ mixture to 100 mL with de-ionized water and filtration of the diluted mixtures. These preparations were performed in quadruplicate. The diluted solutions were analyzed by IC for bromide, chloride, fluoride, formate, glycolate, nitrate, nitrite, oxalate, phosphate, and sulfate. Blank samples of de-ionized water were run on the IC between each analytical sample.

2.1.4.4 Anions in Supernatant

During washing, supernatant samples were diluted with water and submitted to ACSM for anions. It was determined that the washed SB10 material had low enough radioactivity (estimated beta/gamma did not exceed SRNL hood limits) and was not diluted. Supernatant samples were obtained from SRAT and SME Products by vacuum filtration through a 0.45 μm membrane. Filtrate samples were diluted with water and submitted to ACSM for anion analysis by IC and TIC/TOC.

2.1.4.5 Elemental and Radiochemical Analysis of Supernatant

Diluted supernatant throughout washing and straight (undiluted) SRAT Receipt decanted supernatant, were submitted to ACSM for Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES) analysis. Additionally, undiluted SRAT Receipt supernate was submitted to ACSM for analysis by ICP-MS and to the Nuclear Measurements (NM) group for various radiochemical analyses.

SRAT and SME Product filtrates were diluted nominally 50× with 2M nitric acid and submitted to ACSM for elemental analysis by ICP-ES and ICP-MS, and submitted to NM for analysis by the "PU238_PU241" and "AM_CM" methods.[2]

2.1.5 Off-Gas Measurements

Off-gas was quantified using an Agilent model 3000 micro gas chromatograph (GC). Channel A of the instrument consists of a molecular sieve column with thermal conductivity detector. Gases quantified on channel A are hydrogen, krypton (used as a tracer), and methane. The method was optimized for quantification of small concentrations of hydrogen and methane. Oxygen and nitrogen were also detected on channel A but could not be accurately quantified due to saturation of the detector. Qualitative oxygen results are presented for a portion of the testing. Channel B consists of a PoraPLOT Q column with thermal conductivity detector. Gases quantified on this channel are carbon dioxide and nitrous oxide. Argon is used as the carrier gas on both channels. Note that nitric oxide does elute on a molecular sieve column, but nitric oxide readily reacts with oxygen, so any detection would be qualitative at best.

Concentrations were measured in volume percent (vol. %) of the gas or parts per million by volume (ppm_v). The GC was calibrated and checked using a calibration gas containing, nominally, 50 ppm_v hydrogen, 0.5 vol. % krypton, 100 ppm_v methane, 1 vol. % carbon dioxide, 0.5 vol. % nitrous oxide, 20 vol. % oxygen, and the balance nitrogen. This gas was chosen as it represents, approximately, the gas composition when hydrogen and methane are at their projected peaks. Ideally, calibration gas should represent the gas to be analyzed as closely as possible, thus the calibration gas has low carbon dioxide. As a result, the peak carbon dioxide concentration encountered in the SB10 qualification test is a significant extrapolation of the calibration and may have a low bias (based on a scoping test with several different carbon dioxide concentrations).

The experimental apparatus was purged with a gas containing 0.5 vol. % krypton, 20 vol. % oxygen, and the balance nitrogen. The krypton was added as an internal standard to assist in calculating the flow out of the vessel during times of high off-gas generation, may aid in identifying major off-gas in-leakage, and gives an indication that off-gas from the vessel is indeed being sampled and analyzed by the GC.

Vessel purge rate during the demonstration was determined by scaling the applicable DWPF purge rate (94 standard cubic feet per minute (SCFM) in the SRAT and 72 SCFM in the SME) with a 6,000 gallon sludge volume to the SRNL volume.

2.1.5.1 Off-Gas Generation Rate Calculations

Off-gas generation rates (lb/h) for nitrous oxide, carbon dioxide, hydrogen, and methane were calculated and scaled to a DWPF 6,000 gallon SRAT or SME cycle using the following equation:

$$Gen\ Rate_i = F_{out} \times C_i \times \frac{mol}{24.1\ L} \times MW_i \frac{g}{mol} \times \frac{lb}{454\ g} \times \frac{60\ min}{h} \times DWPF\ Scale\ Factor \qquad \text{Eq. 2-4}$$

where:

F_{out} = flow out of the vessel, L/min. See Equation 2-5 and following text for explanation.

C_i = concentration of component *i* (hydrogen, methane, nitrous oxide, or carbon dioxide) as mol fraction in the off-gas (vol. %/100 or $ppm_v/10^6$). The ambient temperature and pressure offgas (after the condenser) is assumed to be an ideal gas.

24.1 L/mol is the molar volume for ideal gas at 70 °F and 1 atm, the reference conditions for the gas flow controller and DWPF purge flowrate.

MW_i = molecular weight of i (hydrogen, methane, nitrous oxide, or carbon dioxide), g/mol

DWPF Scale Factor = ratio of a 6,000 gal SRAT or SME divided by the SRNL SRAT or SME volume, as calculated and published in the respective demonstration run plan.[5-6]

For the SRAT cycle, the flow out of the vessel was calculated as follows:

$$F_{out} = F_{in} \times \frac{Kr_{ave}}{Kr_t} \qquad \text{Eq. 2-5}$$

where:

F_{in} = the flow into the vessel, L/min

Kr_{ave} = the average of multiple Kr concentrations during times of low gas generation

Kr_t = the Kr concentration at time *t*

For the SME cycle, the gas generation was minimal. Therefore, the flow out of the vessel was taken as equal to the flow into the vessel without impact, i.e. $F_{out} = F_{in}$.

The molecular weights and scale factors used in the generation rate calculations are given in Table 2-4.

Table 2-4. Molecular Weights and Scaling Factors for Off-gas Calculations

Parameter	Units	Value
Nitrous oxide molecular weight	g/mol	44.01
Hydrogen molecular weight	g/mol	2.016
Methane molecular weight	g/mol	16.04
Carbon dioxide molecular weight	g/mol	44.01
SRAT Cycle Scale Factor	6,000 gal/SRNL SRAT cycle starting volume (gal)	7,214
SME Cycle Scale Factor	6,000 gal/SRNL SME cycle starting volume (gal)	8,538

2.1.5.2 Retained hydrogen measurements

Prior to the SRAT cycle and SME cycle and after the SME cycle, the vessel was periodically mixed and purged to quantify retained hydrogen.

2.1.5.3 Estimation of Limits of Quantification

The limits of quantification (LOQs) for hydrogen and methane were previously estimated to be 3 and 14 ppm_v, respectively.[20] The LOQs for carbon dioxide and nitrous oxide are estimated to be 0.05 vol% and 0.03 vol%, respectively. These LOQs were determined using a method where the t-statistic is applied to the standard deviation of the results of a known concentration, the calibration gas in this case.[21] Equation 2-5, with $F_{out} = F_{in}$, is used to calculate the limit of quantification (in lb/h on DWPF scale) for hydrogen, nitrous oxide, and methane for the SRAT and SME cycles. LOQs are presented in Table 2-5.

Table 2-5. Limits of Quantification for Off-gas Measurements

Gas	Unit	SRAT Cycle	SME Cycle
Carbon Dioxide	volume %	0.05	0.05
	lb/h	0.3	0.2
Nitrous Oxide	volume %	0.03	0.03
	lb/h	0.2	0.2
Hydrogen (DWPF limit: 2.4×10^{-2} lb/h)	ppm_v	3	3
	lb/h	9×10^{-5}	7×10^{-5}
Methane	ppm_v	14	14
	lb/h	4×10^{-3}	3×10^{-3}

2.1.6 Format of the Reported Results

Mean results, based on the average of all applicable analytical determinations, are reported in this document, along with the percent relative standard deviation (RSD) and the number of determinations (n) feeding each mean. RSD provides an indication of the measurement variation between replicate determinations but is typically not an indicator of the overall analytical uncertainty. When an RSD or other value in a table is not applicable, either two dashes (--) or the abbreviation "N/A" is used. If an analyte was not measured, the abbreviation "n.m." is used. In some cases, analytes that are not detected are reported as the abbreviation "n.d."

In general, the one sigma analytical uncertainty (σ) reported by SRNL Analytical was 10%, although it was sometimes lower or higher. Specifically, the one sigma analytical uncertainties reported by AD were: a) ~10% for base titration, IC, ICP-ES, and TIC/TOC analyses; b) ~20% for ICP-MS, DMA, CVAFS, and VOA analyses; and c) directly based on radiometric (counting) methods, with 5% on the low end, but often in the 20-30% range. As such, only the two leading digits reported for the SRNL Analytical results should be considered significant.

RSD is not reported when the reported result average is below the detection limit. Individual results and averages are preceded by "<" when reporting below detection limit. Percent soluble results are preceded by "<" when the slurry concentration is a measured value and the supernate concentration is below detection limit. Percent soluble results are preceded by ">" when the slurry concentration is below detection limit and the supernate concentration is a measured value. Average values are preceded by "≤" when results are a combination of above and below detection limit values. The ≤ values typically include the below detection limit values when calculating the average and RSD. In cases where the below detection limit values are outliers, the ≤ values exclude the below detection limit values when calculating the average and RSD. In either case, the ≤ values should be considered qualitative.

2.2 SRAT Receipt Preparation

Following is a narrative of SRNL's Tank 51 washing/SB10 SRAT Receipt preparation. Amounts of additions and decants can be found in the Results and Discussion section, Section 3.1.2, Table 3-8. After the initial sample analysis of the Tank 51 combined samples, the slurry was washed utilizing the washing plans of 9/24/19 (see Appendix A). While implementing the plan, and following discussions between SRNL and SRMC, washing plans were modified to change the endpoint, which is shown in Appendix A. While SRNL targeted the same amount of H Canyon addition and the Tank Farm wash endpoint, SRNL accomplished this through fewer wash cycles (larger washes and decants). Thus, SRNL wash and decant amounts and nomenclature do not align directly with SRMC labeling. When an SRNL wash does not align directly with an SRMC wash, a prime (′) is used in the nomenclature of the SRNL wash. An outline of the SRNL washing follows:

> The as-received, combined Tank 51 material was allowed to settle, and a decant was completed (designated as Decant K). Washing was paused from March 2020 to September 2020 as plans for the addition of Sodium Reactor Experiment (SRE) solutions from H Canyon Tanks 16.3 and 16.4[22] were finalized.
>
> In September 2020, washing resumed with the addition of wash water, settling and a decant (designated as Decant L). Separately, Mn (as $MnNO_3$) and NaOH (50 wt%) were added to H Canyon samples from Tanks 16.3 and 16.4 per H Canyon plans.[22] $MnNO_3$ was added to Tank 16.3 and 16.4 material to attain a target of 64:1 Mn: $^{235}U(eq_{SLU})$[b] mass ratio. $^{235}U(eq_{SLU})$ is defined in the DWPF Waste Acceptance Criteria document.[23] NaOH was added to attain 1.2 M excess free hydroxide. The H Canyon/Mn/NaOH material was then added to the post Decant L Tank 51 material. The amount of material added was determined by ratioing SRNL volumes to Tank Farm volumes. Following the H Canyon/Mn/NaOH additions, an analysis of the supernatant showed a higher than projected concentration of sodium, indicating loss of water due to evaporation since the washing vessel is not leak tight. The subsequent wash water addition was adjusted to account for this loss.
>
> Wash water was added and the sludge allowed to settle for Decant N′. Wash water was added and the sludge allowed to settle for Decant Q′. Wash water and 40 wt% $NaNO_2$ to target a NO_2^-/NO_3^- ratio of

---

[b] Equivalent uranium-235 for use in sludge is defined as $^{235}U(eq_{SLU}) = {}^{235}U + 1.4({}^{233}U)$, where all species are on a mass basis

1.8 was added, followed by settling and a decant (Decant R′). Wash water was added and the sludge allowed to settle for Decant S′.

Decant S′ was analyzed and the NO_2^-/NO_3^- was 2.3, significantly higher than the target of 1.8. Therefore, a portion of Decant N was added with the final wash to lower the ratio. The sludge was allowed to settle and then decanted. The resulting slurry, the SRNL washed Tank 51 SB10 material, was sampled and characterized. This slurry was then used in the DWPF CPC demonstration.

2.3 Process Equipment

SRAT and SME cycles were performed in a 4-L glass vessel with a stainless steel lid. Heating was provided using two 0.5 inch diameter stainless steel heating rods powered by an automated direct current power supply (TDK Lambda Genesys, GEN150-10). Slurry mixing was controlled using a mixer system consisting of a ServoDyne mixing head coupled to an agitator shaft utilizing two 3 inch 45° pitched turbine impeller blades. The process air purge was provided by the building air supply and controlled using a MKS Flow Controller. Values for pH were acquired using a Mettler-Toledo probe coupled to a Thermo Scientific Orion Star™ pH meter. Diagrams and photographs of the setup are shown in Figure 2-2, Figure 2-3, and Figure 2-4.

The SRAT condenser was cooled to 25 °C using a 12,000 BTU/hr water/glycol chiller manufactured by Dimplex Thermal Solutions. A bypass line with a needle valve was installed to reduce the coolant flowrate from 20 gpm to 0.8 gpm. The condensate dropped into the Mercury Water Wash Tank (MWWT) at a point below the gas-liquid interface inside the MWWT. The MWWT is configured to allow reflux of condensate back to the SRAT vessel or to drain the condensate to the collection bottle. To replicate the function of the Formic Acid Vent Condenser (FAVC), the off-gas exiting the condenser is passed through a vapor trap (i.e. cold finger, typically less than 10 °C, but there were periods with temperatures up to 14 °C) cooled by an aluminum block mounted on a Torrey Pines Scientific electronic ice cube before continuing to the GC. No ammonia scrubber was used in this testing.

Acids were metered into the 4-L vessel through an injection tube below the surface of the slurry. Acid was added using a Fluid Metering, Inc., QV drive with RHO piston pump head and V300 controller. Antifoam additions were added to the surface using a valved injection port and preloaded syringes.

A Data Acquisition and Control (DAC) application was programmed using National Instruments LabVIEW software. The DAC logged process data and controlled mixing speed, purge gas flow, and heating rod temperature.

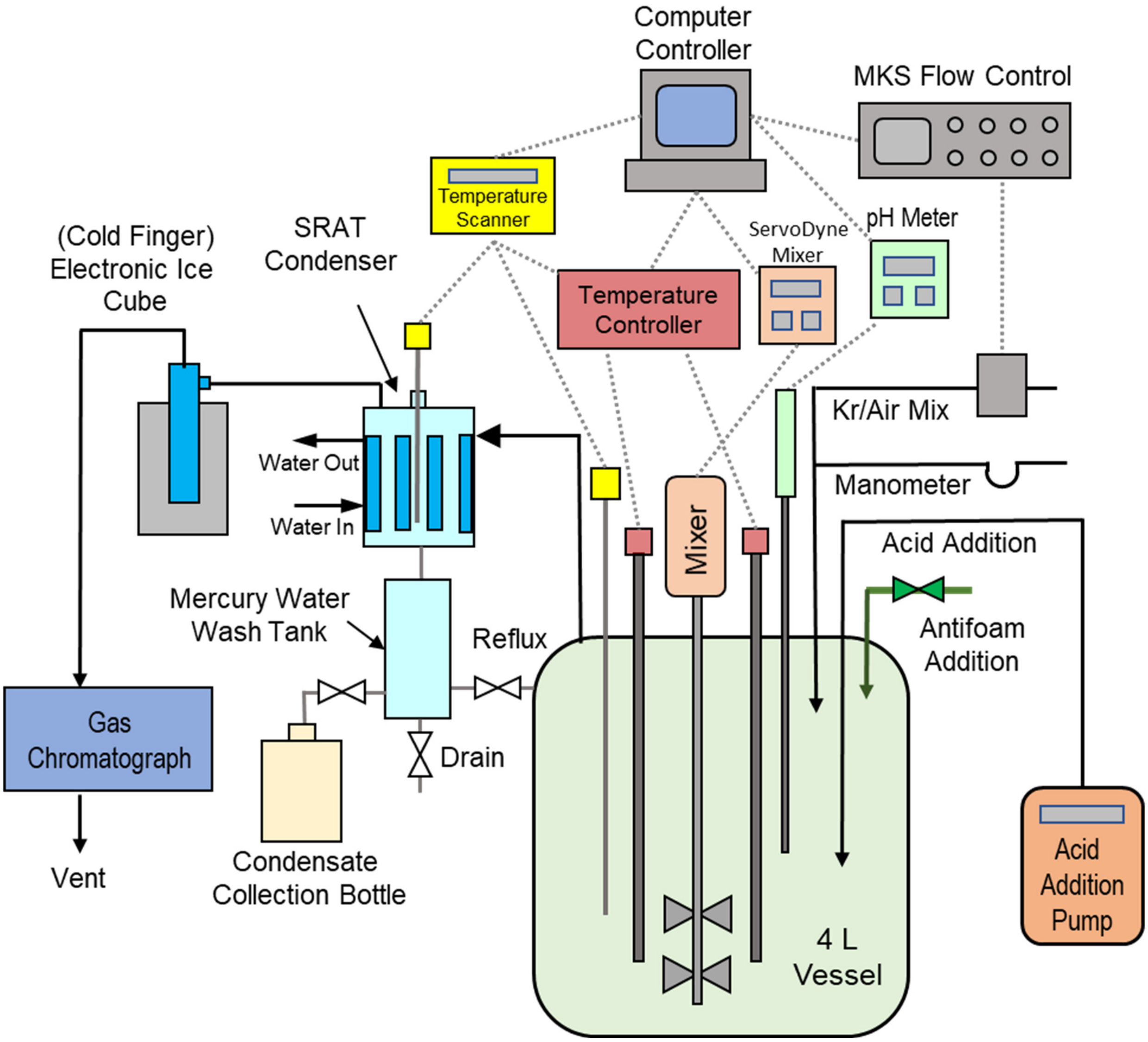


Figure 2-2. Schematic of Shielded Cells Process Equipment

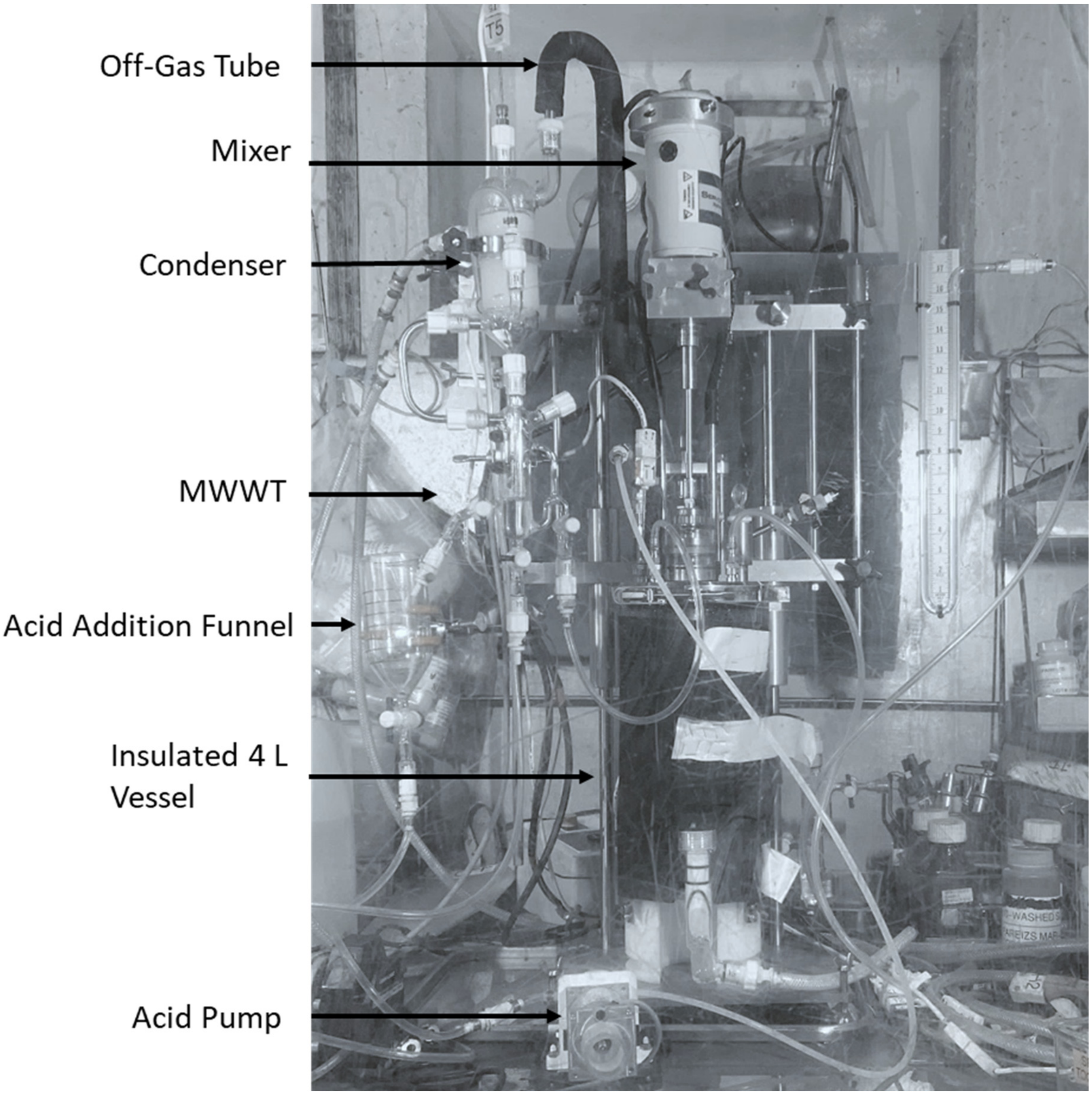


Figure 2-3. Photograph of 4 L Equipment in A Block Shielded Cell 2

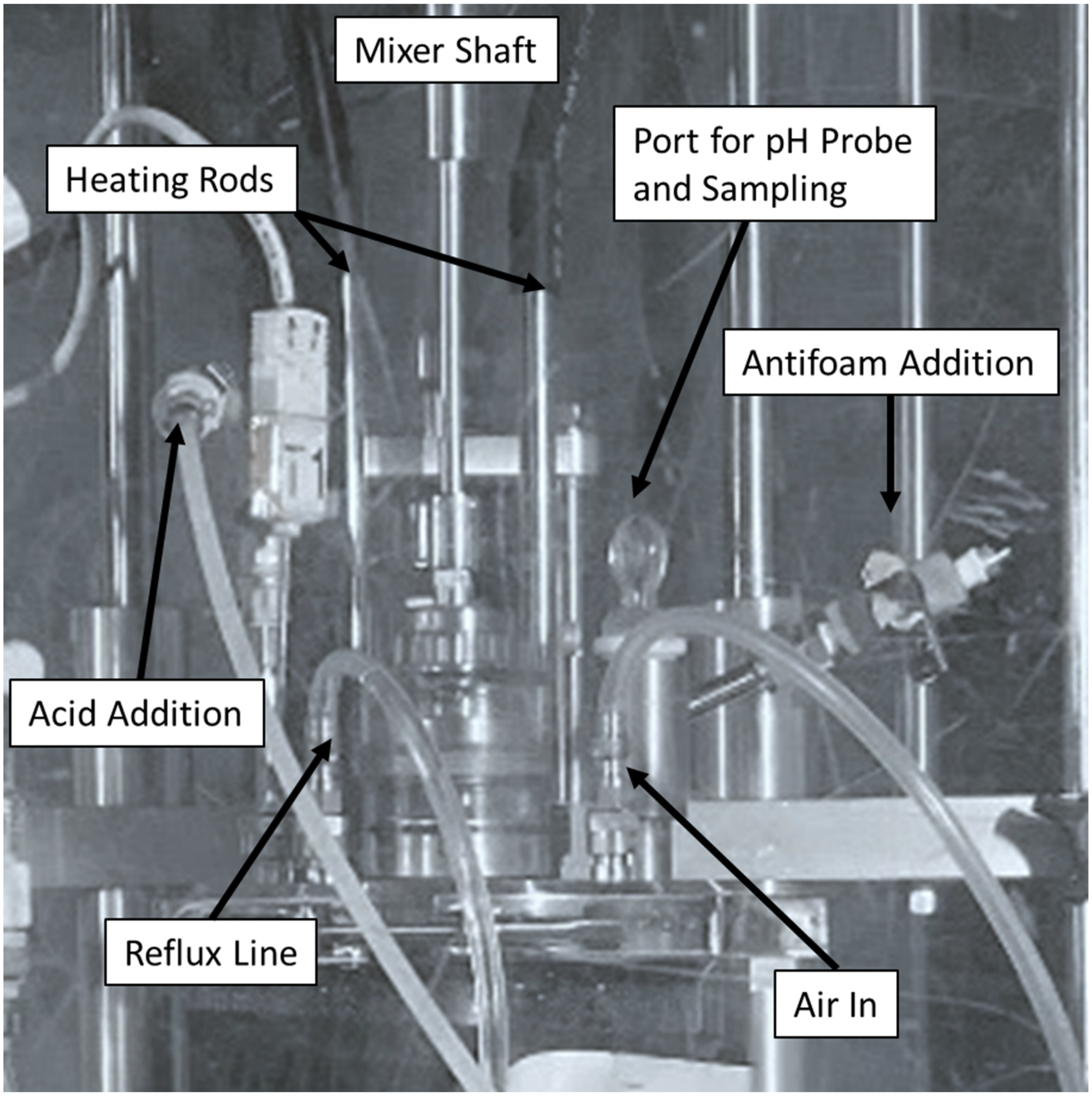


Figure 2-4. Annotated photograph of 4 L vessel lid

2.4 SRAT and SME Cycle Overview

The following is a summary of the planned SRAT and SME cycles. Details are presented in the Results and Discussion section.

For the SRNL demonstration of the first stage of the CPC process, the SRAT cycle begins with mixing, purging, and heating the vessel to 93 °C. Nitric acid addition began as the vessel was heating. Glycolic acid was to be added after nitric acid (with vessel at 93 °C). The acids lower pH and destroy nitrite and carbonate. The glycolic acid reduces mercury to its elemental form so it can be steam stripped. Following acid addition, contents are taken to boiling and water is removed to obtain a target final wt% TS. Boiling is then continued under reflux to steam strip mercury. Following the reflux, the vessel is allowed to cool and an analytical sample is taken.

For the SME cycle, water is added and then boiled off five times to simulate the addition and removal of canister blast water (water used in the decontamination of filled canisters of glass). Frit is then added, followed by water (to simulate a 50 wt% frit and 50 wt% water mixture). Frit is added in two separate, equal batches. The added water is boiled off and additional water is removed to attain a predetermined target wt% TS.

For the SRNL real waste SRAT and SME demonstrations, SRNL planned on using the same antifoam (Momentive™ Y-17112) and antifoam addition strategy as simulant testing. Antifoam was to be added as a 5 wt% solution in de-ionized water to ensure addition to the vessel. The planned SRAT addition strategy was: 25 mg/kg prior to glycolic acid addition, 50 mg/kg prior to boiling, and 25 mg/kg every 12 hours during boiling. The planned SME addition strategy was: 25 mg/kg prior to the first canister blast addition and 25 mg/kg prior to the first frit/water addition. This strategy was recommended by Lambert et al.[24] The SRAT antifoam addition strategy was modified due to a foamover and is discussed in the Results and Discussion below.

2.5 Glass Fabrication and Compositional Analysis

Due to the high level of radioactivity, most of this work was performed remotely in the SRNL Shielded Cells except when sub-samples were removed for further compositional analysis.

2.5.1 Glass Fabrication

Approximately 50 g of the SB10 SC-19 SME Product was divided into two nearly equal portions, placed into quartz crucibles, and dried overnight at 110 °C. Subsequently, both portions were transferred to an open Pt/Au crucible and heated to 1150 °C at approximately 10 °C/min in an electrically heated furnace.[25] Upon reaching the target furnace temperature of 1150 °C, the sample was held at temperature for approximately three hours. The crucible was removed from the furnace at temperature and bottom quenched in a shallow pan of water. The surfaces of the glasses were then visually examined to confirm that they were homogeneous (dark and shiny appearance). The final mass of the glass was approximately 19 g.

2.5.2 Compositional Analysis

Approximately 2.5 g of the glass was ground in an agate mechanical pulverizing mixer mill. The glass was sieved and the portion that passed through a 200 mesh stainless steel sieve (<75 μm) was used for the dissolutions. Four replicate samples (nominally 0.25 g each) of the sieved glass were dissolved for analysis by two different methods: AF dissolution and AR dissolution.[11-12] Reruns were performed using new AF dissolutions due to an initial low recovery of Si. Concurrent with the dissolutions, two samples of the Analytical Reference Glass-1 (ARG-1) were dissolved by AF and one sample by AR to confirm the performance of the glass dissolution methods.[26]

Aliquots from both dissolution methods were analyzed by ICP-ES.[27] A multi-element standard solution containing known concentrations of Al, B, Fe, Li, and Si and one containing known concentrations of Al, Fe, Mn, Ni, and S were also analyzed with the AF samples to confirm the accuracy of the ICP-ES measurements.[28-29] Aliquots of the AR dissolutions were also analyzed by ICP-MS to analyze for certain elements in addition to those from ICP-ES in the glasses.[30] Blanks were processed through the applicable dissolutions for ICP-ES and ICP-MS and were submitted for analysis to check for any gross contamination.

2.6 Glass Fabrication and REDOX Analysis

2.6.1 Closed Crucible Hot Method for Glass Fabrication

Approximately 30 g of glass per sample was fabricated at 1150 °C from the SB10 SC-19 SME Product following the closed sealed crucible methodology within the SRNL Shielded Cells.[31] Utilizing the measured

SME chemical composition, the theoretical glass viscosity at 1150 °C was predicted as 115 Poise. An amount (0.75 g) of lithium metaborate ($LiBO_2$) was added to the SME Product before vitrification to aid in lowering viscosity, to approximately 45 Poise, and improving thermal convective mixing. Five samples of the SME Product were dried to approximately 95% solids and sealed in crucibles using nepheline gel; three crucibles remained sealed during the heat treatment process.[c]

2.6.2 REDOX Preparation and Measurement

Samples from the three crucibles that remained sealed during vitrification were selected, ground using an agate mortar and balls, and digested with sulfuric and hydrofluoric acids. The dissolved iron in the samples is then complexed with ammonium metavanadate and analyzed using the ultraviolet/visible (UV/Vis) spectrometer.[32] The spectrometer measures the color produced by the complexed iron(II) (Fe(II)). The first measurement results in the initial Fe(II) content; the second measurement is performed after all of the iron in the sample has been reduced to Fe(II) and results in the total iron (ΣFe) value for the sample. The ratio of Fe(II) to total iron (Fe(II)/ΣFe) is the reported REDOX value.

2.7 Quality Assurance

Data used to evaluate DWPF Technical Safety Requirements Safety Administrative Controls 5.8.2.11 (H_2 Generation, Radiolytic Hydrogen Generation, and Inhalation Dose Potential), 5.8.2.25 (Ammonium Concentration of SRAT/SME Condensates), 5.8.2.36 (Retained Hydrogen), and 5.8.2.38 (Flammability Assumptions, Inhalation Dose Potential, Canister Heat Generation, Salt Solubility in Glass) are as follows:[1]

- Density and weight percent solids (TS, DS, IS, SS, and CS) measurements
- Hydrogen generation rate for SRAT and SME cycles on a 6,000 gallon basis
- Ammonium concentration of the SRAT and SME condensates
- Rheology data
- SRAT Receipt characterization

All other items have the functional classification of Production Support. With the exception of the SRAT Receipt characterization, these tasks are not waste-form affecting and do not need to follow the quality assurance requirements of DOE/RW-0333P. Microsoft Excel and JMP® 16.0.0 were used to support this work.[33-34] Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.[35] Density and solids results in Table 3-11, hydrogen results in Section 3.2.4.1, ammonium results in Table 3-42, rheology results in Section 3.2.9, and SRAT Receipt characterization of Section 3.1.3, including calculations, with functional classification of Safety Class, were subjected to a Design Verification by Document Review. All other sections in this report, including calculations, with a functional classification of Production Support were reviewed by a Design Check. SRNL documents the Design Verification and Design Check(s) using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[36] Experimental details and the Design Checklists for this report are stored in the PerkinElmer E-Notebook system as experiments L3293-00022-41, A6583-00142-31, K9691-00478-11, B9108-00327-18, I7770-00338-17, and related experiments.

3.0 Results and Discussion

3.1 Results from Addition of SRE Material and Tank 51 Sample Washing

In this section, the chemical and radionuclide compositions of the previously published as-received composition, analyses of supernate and slurry samples during washing, and the washed sample chemical and radionuclide compositions are presented.[37]

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[c] The risk of the sealed lids popping off the crucible during vitrification is an inherent risk in the procedure. The lids must be sealed in such a way as to allow slow venting of gases to prevent pressure build-up within the crucible during heat-up but completely seal to the outside atmosphere once off-gassing has completed and the dried material is vitrifying.

3.1.1 Tank 51 Sample HTF-51-19-114/HTF-51-20-15

Table 3-1 shows the density and solids results for HTF-51-19-114/HTF-51-20-15, which is the combination of the two nominally 3-L Tank 51 SB10 qualification sample at the start of washing.

Table 3-1. Sample HTF-51-19-114/HTF-51-20-15 Densities and Weight Percent Solids

Property	Result	RSD, n
Slurry Density (g/mL)	1.19	0.6%, 4
Supernatant Density (g/mL)	1.17	0.3%, 5
Total Dried Solids (wt%, Slurry Basis)	23.1	0.4%, 5
Dissolved Solids (wt%, Supernatant Basis)	18.8	0.5%, 5
Insoluble Solids (wt%, Slurry Basis)	5.3	NA
Soluble Solids (wt%, Slurry Basis)	17.8	NA

Filtrate was used for the supernatant characterization. Aliquots were diluted before removal from the cells. Water was the diluent for anions, OH and carbon analyses. Nitric acid (2 M) was used as the diluent for Cs-137 and the elementals. Results are presented in Table 3-2 and Table 3-3, along with the analytical technique.

Table 3-2. Sample HTF-51-19-114/HTF-51-20-15 Supernatant Anions and ^{137}Cs

Analyte	Method	Units	Result	RSD, n	σ (%)
Bromide	IC	M	<3E-02	N/A	N/A
Chloride	IC	M	<1E-02	N/A	N/A
Fluoride	IC	M	<3E-02	N/A	N/A
Formate	IC	M	<1E-02	N/A	N/A
Nitrate	IC	M	5.89E-01	0.9%, 4	10
Nitrite	IC	M	3.58E-01	1.2%, 4	10
Oxalate	IC	M	1.25E-02	1.1%, 4	10
Phosphate	IC	M	<5E-03	N/A	N/A
Sulfate	IC	M	9.55E-02	1.1%, 4	10
Carbonate	TIC	M	2.31E-01	1.6%, 4	10
Free OH	Titration	M	1.53E+00	6.0%, 4	10
TOC	TOC	mg C/L	5.78E+02	3.7%, 4	10
^{137}Cs	γ-scan	dpm/mL	2.19E+08	3.9%, 4	5

Table 3-3. Sample HTF-51-19-114/HTF-51-20-15 Elemental Composition of Supernatant

Element	Result (mg/L)	Result (M)	RSD, n	Element	Result (mg/L)	Result (M)	RSD, n
Ag	<8E+0	<8E-5	N/A	Mn	<1E+0	<2E-5	N/A
Al	5.98E+3	2.22E-1	3.5%, 4	Mo	<2E+1	<2E-4	N/A
B	<1E+2	<1E-2	N/A	Na	8.30E+4	3.61E+0	3.1 %, 4
Ba	<3E-1	<2E-6	N/A	Ni	<4E+1	<6E-4	N/A
Be	<3E-1	<3E-5	N/A	P	<1E+2	<3E-3	N/A
Ca	1.84E+1	4.58E-4	N/A, 1	Pb	<4E+1	<2E-4	N/A
Cd	<3E+0	<2E-5	N/A	S	3.01E+03	9.40E-02	4.6%, 4
Ce	<3E+1	<2E-4	N/A	Sb	<8E+1	<7E-4	N/A
Co	<7E+0	<1E-4	N/A	Si	<2E+1	<7E-4	N/A
Cr	1.25E+2	2.41E-3	4.3%, 4	Sn	<5E+1	<4E-4	N/A
Cu	<1E+1	<2E-4	N/A	Sr	<9E-2	<1E-6	N/A
Fe	<2E+0	<4E-5	N/A	Th	<5E+1	<2E-4	N/A
Gd	<8E+0	<5E-5	N/A	Ti	<2E+0	<4E-5	N/A
Hg	2.22E+01	1.11E-04	4.1%, 4	U	<1E+2	<4E-4	N/A
K	3.34E+2	8.54E-3	3.4%, 4	V	<4E+0	<7E-5	N/A
La	<4E+0	<3E-5	N/A	Zn	<3E+0	<4E-5	N/A
Li	<2E+1	<2E-3	N/A	Zr	<3E+0	<3E-5	N/A
Mg	7.72E-1	3.18E-5	27.7%, 3				

Slurry samples were digested and analyzed for elementals. Results for the HTF-51-19-114/HTF-51-20-15 samples were converted from slurry basis to a total dried solids basis using the wt% TS result in Table 3-1. Specific digestion and analytical techniques for each element are given with the results in Table 3-4. Isotopes of uranium and plutonium are given in Table 3-5.

For the elements Ag, B, Be, Cd, Ce, Co, Cu, Li, Mo, P, Pb, S, Sb, Sn, Sr, Ti, V, and Zn results from ICP-ES of the AR digestions were used either because the detection limit was lower in the AR results or the element was not detected in the AF digestion. For K and Na, results from the ICP-ES of the AR digestions were used because these elements were detected in significant quantities in the AF reagent blank. For Th, ICP-ES from the AR digestion was used because the result agreed well with ICP-MS results (mass 232). For Zr, the AR digestion was used because the AF digestion is done in a Zr crucible that adds Zr to the digestate.

Table 3-4. Sample HTF-51-19-114/HTF-51-20-15 Elemental Composition of Total (Dried) Solids

Element	Digestion And Analytical Method	Average (wt% of TS)	RSD, n	Element	Digestion And Analytical Method	Average (wt% of TS)	RSD, n
Ag	AR, ES	<3E-03	NA	Na	AR, ES	3.12E+01	0.9%, 4
Al	AR/AF, ES	7.29E+00	2.7%, 8	Nd	AR, MS	7.13E-02	0.8%, 4
B	AR, ES	<4E-02	NA	Ni	AR/AF, ES	1.29E-01	3.2%, 8
Ba	AR/AF, ES	2.36E-02	2.4%, 8	P	AR, ES	<4E-02	NA
Be	AR, ES	<1E-04	NA	Pb	AR, ES	<2E-02	NA
Ca	AR, ES	2.60E-01	1.0%, 4	Pd	AR, MS	8.32E-04	0.9%, 4
Cd	AR, ES	<2E-03	NA	Rh	AR, MS	3.85E-03	1.4%, 4
Ce	AR, ES	5.18E-02	0.8%, 4	Ru	AR, MS	1.93E-02	1.2%, 4
Co	AR, ES	<2E-03	NA	S	AR, ES	1.18E+00	1.2%, 4
Cr	AR/AF, ES	1.39E-01	1.7%, 8	Sb	AR, ES	<3E-02	NA
Cu	AR, ES	1.46E-02	0.4%, 4	Si	AF, ES	1.84E-01	2.9%, 4
Fe	AR/AF, ES	3.98E+00	1.1%, 8	Sn	AR, ES	<2E-02	NA
Gd	AR/AF, ES	1.44E-02	5.1%, 8	Sr	AR/AF, ES	1.08E-02	2.7%, 8
Hg	AR, DMA	1.73E+00	3.3%, 4	Th	AR/ES	7.25E-01	2.2%, 4
K	AR, ES	1.26E-01	1.3%, 4	Ti	AR, ES	6.10E-03	3.4%, 4
La	AR/AF, ES	1.34E-02	0.6%, 4	U	AR/AF, ES	1.06E+00	3.0%, 8
Li	AR, ES	<6E-03	NA	V	AR, ES	<1E-03	NA
Mg	AR/AF, ES	9.60E-02	3.2%, 8	Zn	AR, ES	5.95E-03	7.8%, 4
Mn	AR/AF, ES	7.81E-01	1.2%, 8	Zr	AR, ES	7.63E-02	1.0%, 4
Mo	AR, ES	<1E-02	NA				

Table 3-5. Sample HTF-51-19-114/HTF-51-20-15 Isotopes of Uranium and Plutonium

Isotope	Average (wt% of TS)	RSD (%), n=4	σ (%)
U-233	2.25E-04†	0.7	20
U-234	1.78E-04	2.0	20
U-235	6.13E-03	0.7	20
U-236	6.70E-04	1.7	20
U-238	1.06E+00	0.4	20
Pu-238	1.50E-04	3.2	9.5
Pu-239	7.56E-03†	1.8	20
Pu-240	5.90E-04	1.5	20
Pu-241	8.08E-06	5.3	16

† U-233 and Pu-239 may be biased high by hydrides of Th-232 and U-238, respectively.

3.1.1.1 Calculation of Calcined Solids

Based on previous experience, a direct measurement of CS as wt% may not be possible with a high sodium sample such as the Tank 51 material. In the calcining process, compounds are converted to their oxides. Upon cooling, the oxides, mainly sodium oxide, may react with water in the air to form NaOH before a weight can be completed. Therefore, the CS for this material was calculated in the following way. First, the elements detected at greater than 0.1 wt% from Table 3-4, with the exception of Hg, were converted to oxides and summed (see Table 3-6). In this calculation, it is assumed that these elements are converted to oxides and all Hg and anions such as nitrite, nitrate, and hydroxide are driven off in the calcining process. Therefore, this sum represents the mass of oxides (calcined solids) in 100 g of total dried solids. The sum was then used to calculate the CS (wt%, slurry basis):

$$\frac{69.7\ g\ oxides}{100\ g\ total\ solids} \times \frac{23.1\ g\ total\ solids}{100\ g\ slurry} \times 100 = 16.1\ wt\%$$

Table 3-6. Sample HTF-51-19-114/HTF-51-20-15 Elements to Oxides Conversion

Element	wt% of TS	Oxide	El. to Oxide Conv.*	Oxide (wt% of TS)
Al	7.29E+00	Al_2O_3	1.8895	1.38E+01
Ca	2.60E-01	CaO	1.3992	3.64E-01
Cr	1.39E-01	Cr_2O_3	1.4616	2.04E-01
Fe	3.98E+00	Fe_2O_3	1.4297	5.69E+00
K	1.26E-01	K_2O	1.2046	1.52E-01
Mn	7.81E-01	MnO_2†	1.5825	1.24E+00
Na	3.12E+01	Na_2O	1.3480	4.21E+01
Ni	1.29E-01	NiO	1.2726	1.64E-01
S	1.18E+00	SO_4	2.9958	3.54E+00
Si	1.84E-01	SiO_2	2.1393	3.93E-01
Th	7.25E-01	ThO_2	1.1379	8.25E-01
U	1.06E+00	U_3O_8	1.1792	1.24E+00
			Total	6.97E+01

* The Element to Oxide Conversion factor (El. to Oxide Conv), also known as the gravimetric factor, is the ratio of the mass of the oxide to the mass of the element in that oxide.

† Mn is tracked by the Tank Farm as MnO_2, but tracked as MnO when projecting glass composition. In the calculation of wt% calcined solids (the purpose of this table) either form of Mn oxide yields the same calculated wt% solids.

3.1.1.2 Soluble Sodium, Sulfur, and Oxalate

A significant amount of sulfur was found to be in the insoluble solids portion of a 2019 Tank 26 sample; ~60% of the sulfur was determined to be soluble.[38] Because Tank 26 was added to Tank 51, a calculation was performed to determine if a significant amount of insoluble sulfur was present in Tank 51. Results of that calculation are shown in Table 3-7. Also included are results for sodium and oxalate.

Total sulfur and sodium were obtained from aqua regia digestions of the slurry (see discussion on analysis of TS above). Total oxalate and sulfate were determined by IC analysis of an approximately 50× water dilution of slurry.

Soluble sodium and sulfur were calculated from the supernatant elemental results in Table 3-3. Soluble sulfate and oxalate were calculated from the supernatant elemental results in Table 3-2. The supernatant basis was corrected to a slurry basis with supernatant density and IS (wt%).

As can be seen from the table, sulfur is mostly soluble based on sulfur measurements and all the sulfur is soluble based on sulfate measurements. There is significant insoluble oxalate, which is consistent with expectations. Sodium oxalate solubility decreases as sodium concentration increases.[39]

Table 3-7. Sample HTF-51-19-114/HTF-51-20-15 Soluble Sodium, Sulfur, Sulfate, and Oxalate in the Tank 51 Sample

Element /Anion	Total (mg/kg slurry)	Soluble (mg/kg slurry)	% soluble
Na	72,200	67,100	93
S	2,730	2,440	89
Sulfate	7,370	7,430	101
Oxalate	1,940	890	46

3.1.2 Tank 51 Washing

Following the initial sample analysis, the slurry was washed. A description of washing, including Table 3-8. has been published in the SB10 Chemical Composition report[4]. Initially, SRNL followed the washing plans of September 24, 2019 (see Appendix A). During washing and following discussions between SRNL and SRMC, washing plans changed, specifically the endpoint. The changed endpoint is shown in Appendix A. While SRNL targeted the same amount of H Canyon addition and the Tank Farm wash endpoint, SRNL accomplished this through fewer wash cycles (larger washes and decants). Thus, after the addition of SRE material, SRNL decant designations are not comparable to SRMC's and are denoted with a prime (′) after the decant letter. An outline of SRNL's washing follows.

The as-received, combined Tank 51 material was allowed to settle, and a decant was completed (designated as Decant K). Decant included supernate material, but did not include any of the sludge containing phases noted in Figure 2-1 (solids coating vessel wall in headspace, sludge at the headspace/supernate/wall interface, and settled sludge). Washing was paused from March 2020 to September 2020 as plans for the addition of high aluminum-low uranium and SRE solutions from H Canyon Tanks 16.3 and 16.4[22] were finalized.

In September 2020, washing was resumed with the addition of wash water, settling and a decant (designated as Decant L). $MnNO_3$ and 50 wt% NaOH were added to H Canyon samples from Tanks 16.3 and 16.4 per the plans of H Canyon.[22] $MnNO_3$ was added to Tank 16.3 and 16.4 material to attain a target of 64:1 $Mn:^{235}U(eq_{SLU})$ mass ratio. NaOH was added to attain 1.2 M excess free hydroxide. Actual amounts added to the SRNL qualification sample are given in Table 3-8. The resulting mixtures were added to the Tank 51 sample. The amount of material added was determined by ratioing SRNL volumes to Tank Farm volumes. Following the H Canyon additions, an analysis of the supernatant showed a higher than projected concentration of sodium, indicating loss of water due to evaporation since the washing vessel was not leak tight. The subsequent wash water addition was adjusted to account for this loss. See Table 3-8. for addition amount. At this point, SRNL washing deviated from Tank Farm washing in that SRNL did fewer washes (larger additions and decants) relative to the Tank Farm. SRNL washes are designated with a prime symbol to distinguish from Tank farm wash/decant designations.

Wash water was added and the sludge allowed to settle for Decant N′. Wash water was added and the sludge allowed to settle for Decant Q′. Wash water and 40 wt% $NaNO_2$ was added to target a NO_2^-/NO_3^- ratio of 1.8, followed by settling and a decant (Decant R′). Wash water was added and the sludge allowed to settle for Decant S′.

Decant S′ was analyzed and the NO_2^-/NO_3^- ratio was 2.3, significantly higher than the target of 1.8. Therefore, a portion of Decant N′ was added with the final wash to lower the ratio. The sludge was allowed to settle and then decanted. The resulting slurry, the SRNL washed Tank 51 SB10 material, was sampled and characterized.

Table 3-8. Summary of SRNL SB10-Tank 51 Washing

Initial Mass of SB10-Tank 51 Qualification Sample in Washing Vessel	6,540 g slurry
Decant K	–1,028 g supernatant
Wash Water Addition	+564 g wash water
Decant L	–362 g supernatant
Tank 16.4 Addition	+23.66 g Tank 16.4 +19.59 g 50% $MnNO_3$ +22.12 g 50% NaOH +48.8 g water
Tank 16.3 Addition	+52.81 g Tank 16.3 +32.60 g 50% $MnNO_3$ +45.61 g 50% NaOH +56.94 g water
An analysis after the 16.4 and 16.3 additions showed higher than expected Na and anion concentrations. This was attributed to evaporation. Wash water and NaOH was added as a wash and to account for the evaporation and readjust the Na to anion ratios.	+391 g 50 wt% NaOH +2,444 g water
Decant N′	–2,656 g supernatant
Wash Water addition	+2,040 g wash water
Decant Q′ (small slurry sample taken after Decant Q′)	–2,078 g supernatant
Wash water and $NaNO_2$ addition, targeting a NO_2/NO_3 ratio of 1.8	+152 g $NaNO_2$ +1,745 g wash water
Decant R′	–1232 g supernatant
Wash water addition	+2,050 Wash Water
Decant S′ (small slurry sample taken after Decant S′)	–1,918 g
Wash water and material from N′ addition to adjust NO_2/NO_3 ratio	+1,200 g of Decant N′ +1,000 g wash water
Decant T′	–1,826 g supernatant
Wash Water Addition	+1,147 g wash water
Decant U′ (final)	–1,552 g

The results for supernatant and slurry characterization for several samples taken during SRNL washing of the qualification sample are given in Table 3-9 (Decants N′ through S′) and Table 3-10 (post Decant Q′ and post Decant S′ slurries). Note that the projections are not SRMC projections. They are SRNL projections based on targeting lower and lower Na concentrations at each wash to attain the final wash endpoint. Generally, SRNL can do larger (and therefore fewer) washes than the Tank Farm. These results were used in subsequent washing decisions. For example, the Decant S′ results were used in the decision to use a portion of Decant N′ to lower the nitrite/nitrate ratio.

With respect to the solubility calculations, the calculations have several inputs and likely significant uncertainty around these results. For example, soluble sodium and sulfur on a slurry basis is calculated from ICP-ES of diluted supernate and then put on a slurry basis using supernate density and wt% insoluble solids (which are a function of wt% total and dissolved solids). It is not possible to differentiate uncertainty and an increase in sodium and sulfur solubility based on the magnitude and degree of fluctuation in the measurements, and thus there may not be a difference in solubility between the two slurries shown in Table 3-10.

Table 3-9. Analytical Results of Decants During Washing (mol/L)

	Decant N′		Decant Q′		Decant R′		Decant S′	
	Projected	Measured	Projected	Measured	Projected	Measured	Projected	Measured
Na	3.06	2.99	2.00	1.56	1.80	1.53	1.30	1.17
NO_2^-	0.238	0.234	0.159	0.128	0.447	0.431	0.324	0.339
NO_3^-	0.519	0.460	0.338	0.239	0.248	0.186	0.178	0.145
SO_4^{2-}	0.064	0.0572	0.041	0.0293	0.030	0.0225	0.021	0.0188
S	0.064	0.0617	0.041	0.0298	0.030	0.0233	0.021	0.0193
Al	0.159	0.142	0.101	0.0704	0.075	0.0549	0.052	0.0447
$C_2O_4^{2-}$	0.015	0.0189	0.015	0.0102	0.011	8.10E–03	0.008	6.81E–03

Table 3-10. Density, Solids, and Solubility Results During Washing

	Post Decant Q′ Slurry	Post Decant S′ Slurry
Total Dried Solids (wt% of slurry)	16.4	15.0
Insoluble Solids (wt% of slurry)	8.01	8.44
Supernatant Density (g/mL supernate)	1.08	1.06
Total Na (mg/kg slurry)	41,800	25,800
Soluble Na (mg/kg slurry)	30,600	23,200
Soluble Na (%)	73%	90%
Total S (mg/kg slurry)	1,010	528
Soluble S (mg/kg slurry)	813	535
Soluble S (%)	80%	101%

3.1.3 SRAT Receipt Characterization

3.1.3.1 Physical and Chemical Characterization

A sample of slurry from the SRNL washing apparatus was taken in April 2021 and characterized for anions and sodium in the supernatant and elements in the slurry. After discussions with SRMC Engineering personnel, it was determined that the sludge had been adequately washed and could make an acceptable glass. The slurry sample was then extensively characterized as the washed SB10 qualification sample. The washed SB10 sample is also referred to as the SRAT Receipt. Results for this material have been previously reported[4] and are exhibited again in the following tables.

Table 3-11 shows the density and wt.% solids of the washed Tank 51 sample.

Table 3-11. Density and Solids of the SRNL-Washed SB10 Tank 51 Qualification Sample (SRAT Receipt)

Property	Result	RSD, n
Slurry Density (g/mL) T = 18 °C	1.10	1.0%, 4
Supernatant Density (g/mL) T = 22 °C	1.06	0.4%, 4
Total Dried Solids (wt%, Slurry Basis)	15.0	0.8%, 4
Dissolved Solids (wt%, Supernatant Basis)	7.4	3%, 3
Insoluble Solids (wt%, Slurry Basis)	8.2	N/A
Soluble Solids (wt%, Slurry Basis)	6.8	N/A
Calcined Solids (wt%, Slurry Basis)	11.3	0.1%, 3

Presented in Table 3-12 are anion, carbonate, and free OH supernatant results, along with the analytical technique used. These results were determined from analysis of decanted (unfiltered) supernatant – the final decant.

Table 3-12. SRNL-Washed SB10 Tank 51 Qualification Sample Supernatant Anion Results

Analyte	Analytical Method	Units	Result	RSD, n=4
Bromide	IC	M	<6.26E-04	N/A
Chloride	IC	M	1.06E-03	0.3%
Fluoride	IC	M	<5.26E-03	N/A
Formate	IC	M	<2.22E-04	N/A
Nitrate	IC	M	1.46E-01	1%
Nitrite	IC	M	2.34E-01	1%
Oxalate	IC	M	7.11E-03	0.1%
Phosphate	IC	M	2.37E-04	3%
Sulfate	IC	M	2.12E-02	0.4%
Carbonate	TIC	M	2.24E-01	1%
Free OH	Titr.	M	2.40E-01	5%

Elements quantified by ICP-ES and DMA in the supernatant are reported in Table 3-13, and only Al, Cr, Hg, Na, and S were detected.

Table 3-13. SRNL-Washed SB10 Tank 51 Qualification Sample Supernatant Elemental Results

Element	mg/L	M	RSD, n=4	Element	mg/L	M	RSD, n=4
Ag	<2.4E-01	<2.2E-06	N/A	Mn	<8.1E-01	<1.5E-05	N/A
Al	1.21E+03	4.48E-02	3%	Mo	<4.1E+00	<4.3E-05	N/A
B	<2.1E+01	<2.0E-03	N/A	Na	2.37E+04	1.03E+00	4%
Ba	<1.5E-01	<1.1E-06	N/A	Ni	<3.8E+00	<6.4E-05	N/A
Be	<1.1E-01	<1.3E-05	N/A	P	<6.9E+00	<2.2E-04	N/A
Ca	<4.3E+00	<1.1E-04	N/A	Pb	<1.5E+00	<7.2E-06	N/A
Cd	<3.0E-01	<2.7E-06	N/A	S	6.38E+02	1.99E-02	3%
Ce	<1.3E+00	<9.4E-06	N/A	Sb	<7.4E+00	<6.0E-05	N/A
Co	<7.1E-01	<1.2E-05	N/A	Si	<1.0E+01	<3.6E-04	N/A
Cr	5.35E+01	1.03E-03	3%	Sn	<6.1E+00	<5.2E-05	N/A
Cu	<1.7E+00	<2.7E-05	N/A	Sr	<6.9E-01	<7.9E-06	N/A
Fe	<1.3E+00	<2.4E-05	N/A	Th	<3.2E+00	<1.4E-05	N/A
Gd	<5.3E-01	<3.4E-06	N/A	Ti	<1.4E+00	<2.8E-05	N/A
Hg[*]	3.42E+02	1.70E-03	1.1%	U	<6.8E+00	<2.9E-05	N/A
K	<5.5E+01	<1.4E-03	N/A	V	<1.4E+00	<2.8E-05	N/A
La	<3.6E-01	<2.6E-06	N/A	Zn	<1.0E+00	<1.5E-05	N/A
Li	<2.0E+00	<2.9E-04	N/A	Zr	<2.1E-01	<2.3E-06	N/A
Mg	<3.2E-01	<1.3E-05	N/A				

[*] The Hg reported in this table is a reanalysis compared to the originally published result. This result has less scatter among replicates.

A comparison between Tank Farm targets from projections from February 16, 2021 and measurements of the SRNL washed Tank 51 sample is presented in Table 3-14. See Appendix A, Exhibit A-2, for a snapshot from the Tank Farm planning spreadsheet. SRNL targeted Na and IS when washing and did not adjust for free OH and carbonate concentrations. The two analytes are significantly different between targets and measured results with lower hydroxide and higher carbonate. This difference was also seen with the SB9 sample.[40]

The SRNL sample is slightly less washed than the tank farm Na target for Tank 51-SB10, resulting in a higher TS and soluble components (with the exception of free OH and carbonate) than the Tank Farm projection. SRNL completed the final decant after the sludge stopped settling, resulting in 8.2 wt% IS. As much supernatant was decanted as possible without disturbing the sludge.

Table 3-14. Comparison Between Tank Farm Projections and Measurements

Analysis	Units	Projection	Measurement
IS	wt%	8.85	8.2[*]
TS	wt%	13.85	15.0[*]
Supernatant Density	g/mL	1.04	1.06[*]
Sodium	M	0.940	1.03[‡]
Nitrite	M	0.209	0.234[†]
Nitrate	M	0.143	0.146[†]
Free OH	M	0.425	0.240[†]
Chloride	M	0.001	0.00106[†]
Sulfur	M	0.0175	0.0199[‡]
Fluoride	M	0.005	<5.3E-03
Carbonate	M	0.036	0.224[†]
Aluminum	M	0.042	0.0448[‡]
Oxalate	M	0.006	0.00711[†]

[*] From Table 3-11. [†] From Table 3-12. [‡]From Table 3-13

Presented in Table 3-15 are the analytical results from slurry dilutions. The analytical methods for each analyte are also presented. Slurry nitrite, nitrate, and total base are significant inputs to the CPC acid calculation.

Table 3-15. SRNL-Washed SB10 Tank 51 Qualification Sample Slurry Dilution Results

Analyte	Analytical Method	Units	Result	RSD, n=4
Bromide	IC	mg/kg	<1.6E+03	NA
Chloride	IC	mg/kg	<3.1E+02	NA
Fluoride	IC	mg/kg	<3.1E+02	NA
Formate	IC	mg/kg	<3.1E+02	NA
Nitrate	IC	mg/kg	7.67E+03	3%
Nitrite	IC	mg/kg	9.27E+03	3%
Oxalate	IC	mg/kg	5.66E+02	5%
Phosphate	IC	mg/kg	<3.1E+02	NA
Sulfate	IC	mg/kg	1.69E+03	5%
Total Base (titration to pH 7)	Titration	M	6.50E-01	3%
Inorganic Carbon	TIC	mg/kg	2.10E+03	7%
Organic Carbon	TOC	mg/kg	<6.3E+02	NA
Volatile organic carbon	VOA	mg/kg	<6.3E+00	NA

Presented in Table 3-16 are elemental analyses of the total dried solids of the SRNL-washed Tank 51 sample. As described above, slurry material was digested by both AR and AF. Both digestions were submitted for ICP-ES; AR digestions were submitted for DMA and ICP-MS. In addition to the slurry samples, reagent blanks and digestions of ARG-1 (a reference glass of known composition)[26] were analyzed by ICP-ES. The results of the blanks and reference glass digestions were used in evaluating the slurry results (also discussed above).

Samples were digested by each method in quadruplicate. Thus, if only one digestion method is reported, number of samples (n) would equal 4. If both digestions are used, n=8. For La, the AR by ES result was used. There was not good agreement between the two digestion results. The result from AR was nearly 50% higher than the result from AF.

Table 3-16. SRNL-Washed SB10 Tank 51 Qualification Slurry Sample Elemental Composition of Total Dried Solids

Element	Dig, Analytical Method	wt% of TS	RSD (%), n	Element	Dig, Analytical Method	wt% of TS	RSD (%), n
Ag	AR, ES	<8.7E-03	NA	Mo	AR, ES	<7.7E-03	NA
Al	AF, ES	1.24E+01	5, 4	Na	AR, ES	1.66E+01	1, 4
As §	AR, MS	3.28E-03	4, 4	Nd	AR, MS	1.27E-01	NA
B	AF, ES	<8.3E-02	NA	Ni	AR/AF, ES	2.74E-01	5, 8
Ba	AR/AF, ES	4.59E-02	5, 8	P	AF, ES	<9.3E-02	NA
Be	AR, ES	<2.4E-03	NA	Pb	AR, MS	9.32E-03	NA
Ca	AR, ES	5.30E-01	3, 4	Pd	AR, MS	1.45E-03	NA
Cd	AF, ES	<4.0E-03	NA	Rh	AR, MS	8.70E-03	NA
Ce	AR/AF, ES	9.72E-02	9, 8	Ru	AR, MS	4.24E-02	NA
Cl	SL, IC	<2.1E-01	NA	S	AR, ES	3.79E-01	2, 4
Co	AF, ES	<4.7E-03	NA	Sb	AF, ES	<1.7E-02	NA
Cr	AR/AF, ES	1.61E-01	3, 8	Se	AR, MS	<1.0E-04	NA
Cu	AR/AF, ES	3.52E-02	4, 8	Si	AF, ES	4.24E-01	2, 4
F	SL, IC	<2.1E-01	NA	Sn	AF, ES	<2.5E-02	NA
Fe	AR/AF, ES	8.51E+00	3, 8	Sr	AR/AF, ES	1.63E-02	5, 8
Gd	AR/AF, ES	4.44E-02	5, 8	Th	AR/AF, ES	1.73E+00	3, 8
Hg	AR, DMA	2.85E+00	6, 4	Ti	AF, ES	<1.4E-02	NA
K	AR, ES	<7.6E-02	NA	U	AR/AF, ES	2.22E+00	5, 8
La	AR, ES	3.38E-02	3, 4	V	AF, ES	<3.4E-03	NA
Li	AF, ES	<6.3E-03	NA	Zn	AR/AF, ES	1.75E-02	5, 8
Mg	AR/AF, ES	2.09E-01	3, 8	Zr	AR, ES	1.13E-01	3, 4
Mn	AR/AF, ES	2.66E+00	3, 8				

§ The As result may be biased high due to possible interferences with double-charged lanthanides. See Jones (2018)[41] for a discussion.

SRNL was requested to compare the elemental composition of the washed sample calcined solids to the Tank 51 projections. The SRMC projected elemental composition is a normalized composition (see Exhibit A-3). Therefore, SRNL normalized the results on a CS basis for comparison. Elements that were detected by SRNL and listed by SRMC were converted to oxides using oxide and element molecular weights. The oxides were then summed. Each oxide was then divided by the sum. Finally, the oxide was converted from oxide to elemental concentrations. SRMC's projected composition is presented in Appendix B. SRNL's calculation is presented in Appendix C. A comparison between SRMC's projection and SRNL results is presented in Table 3-17. As can be seen, with the exception of the low concentration elements, there is good agreement between the projections and results for elements on a calcined solids basis; percent difference is less than 4% for elements greater than 1% in the calcined solids. The agreement between projected Hg and the measured Hg result is greater than 10%. Hg is not reported on a calcined solids basis as it is not present in the final glass waste form.

Table 3-17. Comparison Between the SRMC Projections and SRNL Results (Elements on a wt% of Calcined Solids Basis)

Element	Tank Farm Projection (wt% CS)	SRNL Result† (wt% CS)	% Difference *
Al	17.53	17.7	-1.0
B	0.003	ND	N/A
Ba	0.06	0.0655	-8.8
Ca	0.74	0.756	-2.1
Ce	0.12	0.139	-14.7
Cr	0.23	0.230	0.0
Cu	0.05	0.0502	-0.4
Fe	11.91	12.1	-1.6
K	0.08	ND	N/A
La	0.03	0.0482	-46.5
Li	0.02	ND	NA
Mg	0.29	0.298	-2.7
Mn	3.72	3.79	-1.9
Na	24.44	23.7	3.1
Ni	0.38	0.391	-2.9
Pb	0.02	0.0133	40.2
S	0.54	0.540	0.0
Si	0.59	0.605	-2.5
Th	2.42	2.47	-2.0
Ti	0.02	ND	N/A
U	3.08	3.17	-2.9
Zn	0.02	0.03	-22.2
Zr	0.16	0.161	-0.6
Hg (wt% of TS)§	3.23	2.85	12.4

† ND=not detected in the total dried solids and thus not shown here.

$$^{*}\ \%Difference = \frac{Tank\ Farm\ Projection - SRNL\ Result}{Average(Tank\ Farm\ Projection, SRNL\ Result)} \times 100$$

§ Hg is not reported on a calcined solids basis as it is not present in the final glass waste form.

Presented in Table 3-18 are results of various carbon measurements (TIC, TOC and VOA) for the washed SB10 Tank 51 sample. Of which, only inorganic carbon was detected in the slurry. No volatile organic compounds were detected.

Table 3-18. SRNL-Washed SB10 Tank 51 Qualification Slurry Sample Carbon Analysis

Analysis	**Result (mg/kg slurry)**	**RSD, n**
Total Inorganic Carbon	2.10E+03	7%, 4
Total Organic Carbon	<6.3E+02	NA
Volatile Organics Analysis	<6.3E+00	NA

3.1.3.2 Radiological Characterization

Table 3-19 gives the measured radionuclide values of the washed SB10 qualification sample, as previously reported.[3] The results reported in units of wt% of TS (column two) and microcuries per gram (µCi/g) of TS (column three) were determined using the TS of the slurry, 15.0 wt%. Additionally, the specific activities of each individual radionuclide was used for the conversion between the two.[42] The curies per gallon (Ci/gal) of sludge slurry (column five) is calculated using the slurry density of 1.10 g/mL. Th-232, Am-243, Cm-242, Cm-246, Cm-247, Cm-248, and Cf-250 concentrations were above their respective method detection limits (MDLs) and are reported in addition to the thirty-six radionuclides requested in the TTR. Y-90, Rh-106, Pr-144, and Pr-144m are in secular equilibrium with 100% of their parent radionuclides, and as such, have the same activities. Ba-137m is in secular equilibrium with 94.6% of Cs-137, its parent radionuclide. Te-125m is in secular equilibrium with Sb-125, its parent radionuclide, at 22.9% of its activity.[43] However, it is treated in Table 3-19 as being 100% of the activity, as a worst-case scenario, due to the Sb-125 measurement being below its MDL. [43] Table 3-20 gives the fissile isotope results for each of the four sample aliquots of the SB10 washed qualification sample in µCi/g of TS.

Table 3-19. Tank 51 SB10 Washed Qualification Sample Average Concentrations of Radionuclides

Radionuclide	wt% in TS	µCi/g in TS	RSD (%) (n=4)	Ci/gal in Sludge Slurry	Method
H-3	N/A[*]	N/A[*]	N/A[*]	3.89E-05	Tritium (LSC)
Co-60	4.12E-09	4.66E-02	7.8	2.91E-05	Cs-Removed Gamma Counting
Sr-90	5.27E-03	7.18E+03	15	4.49E+00	SR90 (LSC)
Y-90	1.32E-06	7.18E+03	15	4.49E+00	Calculated (Secular Equilibrium w/Y-90)
Tc-99	1.90E-03	3.22E-01	3.3	2.01E-04	ICP-MS
Ru-106	<4.02E-09	<1.35E-01	N/A	<8.41E-05	Cs-Removed Gamma Counting
Rh-106	<3.78E-15	<1.35E-01	N/A	<8.41E-05	Calculated (Secular Equilibrium w/Ru-106)
Ag-110m	<5.13E-10	<2.44E-02	N/A	<1.52E-05	Cs-Removed Gamma Counting
Sb-125	<8.70E-09	<8.98E-02	N/A	<5.61E-05	Cs-Removed Gamma Counting
Te-125m	<4.98E-10	<8.98E-02	N/A	<5.61E-05	Calculated (Secular Equilibrium w/Sb-125)
I-129	5.10E-05	9.00E-05	28	5.62E-08	I-129 with Separation
Cs-134	<1.53E-07	<1.98E+00	N/A	<1.24E-03	Gamma Counting
Cs-137	2.81E-04	2.44E+02	1.5	1.52E-01	Gamma Counting
Ba-137m	4.29E-11	2.31E+02	1.5	1.44E-01	Calculated using 0.946 x Cs-137 activity
Ce-144	<1.47E-08	<4.68E-01	N/A	<2.92E-04	Cs-Removed Gamma Counting
Pr-144	<6.20E-13	<4.68E-01	N/A	<2.92E-04	Calculated (Secular Equilibrium w/Ce-144)
Pr-144m	<2.58E-13	<4.68E-01	N/A	<2.92E-04	Calculated (Secular Equilibrium w/Ce-144)
Pm-147	<5.18E-06	<4.80E+01	N/A	<3.00E-02	Pm-147/SM-151 (LSC)
Sm-151	2.09E-04	5.50E+01	5.0	3.43E-02	Pm-147/SM-151 (LSC)
Eu-152	<2.53E-08	<4.37E-02	N/A	<2.73E-05	Cs-Removed Gamma Counting
Eu-154	1.76E-06	4.74E+00	10	2.96E-03	Cs-Removed Gamma Counting
Eu-155	<6.85E-08	<3.19E-01	N/A	<1.99E-04	Cs-Removed Gamma Counting
Th-232	1.75E+00	1.92E-03	2.9	1.20E-06	ICP-MS
U-233	5.10E-04	4.94E-02	3.5	3.09E-05	ICP-MS
U-234	5.93E-04	3.70E-02	1.1	2.31E-05	ICP-MS
U-235	2.93E-02	6.34E-04	3.7	3.96E-07	ICP-MS
U-238	2.43E+00	8.16E-03	2.9	5.10E-06	ICP-MS
Np-237	1.11E-03	7.82E-03	4.2	4.89E-06	ICP-MS
Pu-238	3.76E-04	6.43E+01	6.8	4.02E-02	PU238/PU241 (α-PHA)
Pu-239	1.44E-02	8.98E+00	3.8	5.61E-03	ICP-MS
Pu-240	1.15E-03	2.63E+00	3.6	1.64E-03	ICP-MS
Pu-241	3.00E-05	3.09E+01	16	1.93E-02	PU238/PU241 (LSC)
Am-241	2.83E-04	9.72E+00	6.0	6.07E-03	Cs-Removed Gamma Counting
Am-243	9.05E-07	1.80E-03	4.4	1.13E-06	Am/Cm
Am-242m	1.52E-09	1.48E-04	11	9.25E-08	Am/Cm
Cm-242	3.71E-12	1.23E-04	15	7.66E-08	Am/Cm
Cm-243	2.38E-09	1.23E-03	9.4	7.66E-07	Am/Cm
Cm-244	4.47E-08	3.62E-02	27	2.26E-05	Am/Cm
Cm-245	5.27E-09	9.05E-06	16	5.66E-09	Am/Cm
Cm-246	6.83E-09	2.10E-05	16	1.31E-08	Am/Cm
Cm-247	2.25E-09	2.09E-09	31	1.31E-12	Am/Cm
Cm-248	8.11E-08	3.45E-06	74	2.15E-09	Am/Cm
Cf-250	3.45E-12	3.80E-06	25	2.37E-09	Am/Cm
Total alpha	N/A	1.08E+02	21	6.72E-02	LSC
Total beta	N/A	1.35E+04	1.1	8.42E+00	LSC
Total gamma[†]	N/A	2.49E+02	N/A	1.53E-01	Calculated
Total beta-gamma[‡]	N/A	1.37E+04	N/A	8.57E+00	Calculated

N/A = not applicable

*Drying the slurry sample would drive off the Tritium (H-3) due to it being mainly present as HTO. The concentration was measured in the slurry with a value of 9.34E-03 µCi/g and converted to Ci/gal using the slurry density. Only the one measurement of four replicates that was above the MDL for H-3 is reported in the table.

† Total activity of reported gamma emitters: Co-60, Ru-106, Rh-106, Sb-125, Te-125m, Cs-134, Ba-137m, Ce-144, Pr-144, Eu-152, Eu-154, Eu-155, and Am-241. The MDL value was used in the calculations for radionuclides that had concentrations below the MDL.

‡ Total activity from beta and gamma.

Table 3-20. Replicate Activities of Fissile Radionuclides for the SB10 Washed Qualification Sample in µCi/g of TS

Radionuclide	Repl. 1	Repl. 2	Repl. 3	Repl. 4	Reported Average	RSD (%)
^{233}U	5.11E-02	4.89E-02	5.04E-02	4.72E-02	4.94E-02	3.48
^{235}U	6.57E-04	6.19E-04	6.49E-04	6.09E-04	6.34E-04	3.67
^{239}Pu	9.40E+00	8.83E+00	9.06E+00	8.61E+00	8.98E+00	3.76
^{241}Pu	3.72E+01	3.18E+01	2.88E+01	2.59E+01	3.09E+01	15.66

3.2 SRAT and SME Cycles

3.2.1 Acid Calculation Inputs and Outputs

In late August 2021, the material in the washing apparatus was transferred to an HDPE bottle. The washing vessel was not leak tight (see description of evaporation in the washing section) Therefore, the TS of the August sample was measured and found to be 18.0 wt%, which corresponds to a water loss of 577 g of water. Using the mass of material recovered from the washing vessel and measured TS, the mass of water to add to the material was calculated by a TS mass balance:

$$2{,}886\ g\ slurry\ at\ 18\ wt\%\ TS \times 18.0\% = x \times 15.0\%$$

where x = mass of slurry with 15.0 wt% TS. Solving for x, x = 3,463 g. The difference between this result and the slurry at 18.0 wt% TS is 577 g, the amount of water to be added to lower the concentration from 18.0 wt% to 15.0 wt% TS. This amount of water was added to the slurry. The water was added back so the sample would closer match projections and to have similar rheological properties to the planned SRAT receipt.

Given in Table 3-21 are acid calculation inputs. The SRAT Receipt amount (first row) is the actual amount of slurry at 18.0 wt% TS plus the 577 g of water needed to lower the TS to 15.0 wt%. The analytical results are taken from the April 2021 subsample analyses (see above section). Note that SRNL does not have (or simulate) a SRAT heel. Acid stoichiometry and anion conversions are based on simulant testing.[44]

Table 3-21. SB10 Tank 51 Qualification SRAT Receipt Acid Calculation Inputs

SRAT Receipt Mass	3,463	g slurry
SRAT Receipt Volume	3,148	mL slurry
SRAT Receipt Weight % Total Solids	15.0	wt%
SRAT Receipt Weight % Insoluble Solids	8.20	wt%
SRAT Receipt Density	1.10	kg / L slurry
SRAT Receipt Supernatant Density	1.06	kg / L supernate
SRAT Receipt Nitrite	9,270	mg/kg slurry
SRAT Receipt Nitrate	7,670	mg/kg slurry
SRAT Receipt Oxalate	566	mg/kg slurry
SRAT Receipt Slurry TIC (treated as carbonate)	2,330	mg/kg slurry
Fresh Supernatant TIC (treated as carbonate)	2,690	mg/L supernate
SRAT Receipt Hydroxide (Base Equivalents) pH = 7	0.650	mol/L slurry
SRAT Receipt Manganese	2.66	wt% of total solids
SRAT Receipt Mercury	2.85	wt% of total solids
SRAT Receipt Magnesium	0.53	wt% of total solids
SRAT Receipt Calcium	0.209	wt% of total solids
Conversion of Nitrite to Nitrate in SRAT Cycle	17.80	gmol NO_3^-/100 gmol NO_2^-
Destruction of Nitrite in SRAT and SME Cycle	100.00	% of starting nitrite destroyed
Glycolate conversion to formate	1.60	% glycolate converted to formate.
Destruction of Glycolic acid charged in SRAT	24.60	% glycolate converted to CO_2 etc.
Conversion of Glycolic acid to Oxalate	1.50	% glycolate converted to C_2O_{40}
Percent Acid in Excess Stoichiometric Ratio (Koopman Min Acid Eq) – for Total acid to add determination	107	%
Water to rinse sample bottle	0.00	g
Total water added to flush nitric and glycolic acid lines/bottles	20	g
SRAT Product Target Solids	22	wt%
REDOX Target	0.10	Fe^{+2} / ΣFe
SRAT air purge	94	SCFM
DWPF Acid addition Rate	179	mol/min (both acids)
Nitric Acid Molarity	10.20	Molar
Glycolic Acid Molarity	11.84	Molar
SRAT boil-up rate	5,000	lb/h
SRAT Mercury Product Target Concentration	0.80	wt% of total solids
SRAT Steam Stripping Factor	750	g steam/g Hg

Acid calculation outputs are given in Table 3-22. The stoichiometric acid amount using both the Hsu and Koopman acid equations[45] is calculated and reported. The total acid required is based on 107% of the stoichiometric acid amount from the Koopman Minimum Acid (KMA) equation, which is the recommended amount of acid to be added for this demonstration. The excess acid based on the Hsu equation was 102%, as determined by dividing the acid to be added based on 107% of the KMA by the stoichiometric acid amount, as calculated with the Hsu equation.

Table 3-22. Sludge Batch 10 Acid Calculation Outputs

Hsu Total Stoichiometric Acid required (100%)	4.29	gmol
Koopman Minimum Stoichiometric Acid required (100%)	4.08	gmol
Total Acid to Add based on Koopman equation stoichiometric factor (107% of Koopman Min Stoic.)	4.36	gmol
Stoichiometric Acid Hsu Equation (%)	102	%
Fraction of glycolic acid for REDOX Target	0.536	moles glycolic acid / mole total acid
Nitric acid volume required	198	ml
Glycolic acid volume required	198	ml
SRAT Dewater Mass	847	g
Design Basis nitric acid feed time	82	min
Design Basis glycolic acid feed time	94	min
Minimum SRAT conflux time	1,348	min

The SRNL processing parameters for this run were calculated by scaling the SRNL volume to 6,000 gal. Processing parameters are given in Table 3-23 in both DWPF and SRNL scale. After the foamover events, SRNL reduced the targeted acid addition rates by half. Implications of the lower acid addition rates are discussed in the off-gas and anion conversion sections (Sections 3.2.4.1 and 3.2.5, respectively).

Table 3-23. DWPF and SRNL-Scale SRAT Cycle Processing Parameters

	DWPF Scale	SRNL Scale
SRAT Receipt Volume	6,000 gal	3,148 mL
Volume-based scale factor	7,214	
SRAT air purge*	94 SCFM	369 sccm
Nitric acid addition rate†	4.64 gal/min§	2.4 mL/min§
Glycolic acid addition rate†	3.99 gal/min§	2.1 mL/min§
Boil up rate	5,000 lb/h	5.24 g/min
Antifoam addition‡	25 mg/kg	0.087 g
	50 mg/kg	0.173 g

* Standard temperature for DWPF and SRNL flow meters is 70 °F.

† Acid addition rates are based on a recommended flow rate of 179 mol/min for each acid.

‡ For the SRNL demonstration, antifoam will be added in a 5 wt% solution with water.

§ Following the foamover event, the target acid addition rate was reduced by half: 1.2 mL/min for nitric acid and 1.05 mL/min for glycolic acid at SRNL scale.

3.2.2 Description of the SRNL CPC Demonstrations

For the SRNL demonstration of the first stage of the CPC process, the SRAT cycle began with mixing, purging, and heating the vessel to 93 °C per an approved run plan.[6] As nitric acid preparations were being made, a leak in the nitric acid addition funnel was observed and heating to 93 °C was suspended (temperature was held at 40 °C) for troubleshooting. After replacing the leaking parts, the designated amount of nitric acid was added to the new funnel. Heating to 93 °C and acid addition were then initiated. After approximately 60% of the nitric acid was added, a foamover event occurred. An attempt to resume nitric acid addition led to another foamover and the run was suspended.

Per the run plan, antifoam was not to be added until after nitric acid addition was complete. In the following section, this part of the experiment is designated as the SRAT Cycle Part 1. A timeline of events is presented in Table 3-24. SRNL, in consultation with SRMC personnel, evaluated the event and proceeded to resume the run. The evaluation and path forward is documented in a revised run plan.[6] At the resumption of the run, designated as SRAT Cycle Part 2 in this document, antifoam was added, the vessel was heated to 93 °C, nitric acid addition was completed, additional antifoam was added, and glycolic acid addition was completed.

Table 3-24. SRAT Cycle Part 1 Timeline

Elapsed Time (h)	Activity
-3.4	Began heating, loaded acid addition funnel with nitric acid and observed a leak. Heating was suspended. Vessel contents were at 60 °C. Instead of allowing the vessel to cool to room temperature, the heating controller was set to a setpoint of 40 °C while troubleshooting.
--	Acid addition funnel and acid addition line were replaced. Nitric acid was recovered and re-weighted and the acid lost to the leak was replaced.
0.0	Began heating from 40 °C to 93 °C, began nitric acid addition.
0.8	Vessel temperature reached 93 °C.
1.8	Foamover occurred. Acid addition and heating stopped.
--	Called “time out”, discussed event with the SRNL principal investigator, SRNL, management, SRMC engineering manager. Foamover was deemed “minor” and decision was made to proceed.
2.4	Resumed heating and acid addition.
2.5	Foamover occurred, acid addition stopped, and heating stopped. Notifications were made and it was decided to put the experiment in a safe, stable condition and discuss with a broader team.

At the conclusion of glycolic acid addition, another antifoam addition was made, and the vessel was taken to boiling under reflux. The purpose of this added reflux period (planned and documented in the revised run plan[6]) was to remove sludge in the off-gas line and clean up the MWWT. Even after this cleaning, the MWWT was still dirty. The purge rate was lowered during this cleaning reflux period, for a brief six-minute period, to prevent loss of water from the manometer. The experiment continued after the cleaning reflux period, where the SRAT dewater process was initiated to concentrate the SRAT Product to the targeted 20 wt% TS. After water removal, the material was boiled under reflux, as planned, to steam strip mercury. At the conclusion of the cycle a sample was taken and characterized in preparation for the SME cycle. The timeline for the SRAT Cycle Part 2 is presented in Table 3-25.

Table 3-25. SRAT Cycle Part 2 Timeline

Elapsed Time (h)	Activity
0.0	Began heating Added 25 mg/kg Momentive™ Y-17112 antifoam during heating
1.1	Resumed nitric acid addition
4.7	Completed nitric acid addition
5.2	Added 25 mg/kg Momentive™ Y-17112 Began glycolic acid addition
11.6	Completed glycolic acid addition
12.2	Added 50 mg/kg Momentive™ Y-17112 Began boiling under reflux to "steam clean" off-gas line
13.0	Purge reduced, heating paused as vessel pressure increased, sludge visible coming from off-gas tube and into the condenser. See Figure 2-3 for location of off-gas tube and condenser.
13.2	Purge and heating resumed, no additional pressure or visible sludge (continued to "steam clean off-gas line")
16.3	Began dewatering
19.9	Completed Dewatering
23.5	Added 25 mg/kg Momentive™ Y-17112
35.6	Added 25 mg/kg Momentive™ Y-17112
39.7	Completed reflux, thus completing the SRAT cycle

Following the SRAT cycle and analysis of the SRAT cycle analytical sample, a run plan for the SME cycle was generated.[5] The pre-SME calculations and evaluations were published in the run plan and are repeated here.

Frit 473 was used in the SME cycle to target a waste loading of 36%. Frit 473 is the recommended frit for SB10-Tank 40[6] and as such was evaluated as a candidate for SB10 qualification with the SRNL-Tank 51 material. The SRAT Product elemental composition was combined with the Frit 473 composition at 24% through 42% waste loading and evaluated using the DWPF PCCS glass property models and their associated Measurement Acceptance Region constraints.[46] The results indicated acceptable glass compositions for waste loadings of 31 through 40%.

The estimated amount of frit to be added was calculated based on a calcined solids basis.

- 3,458 g SRAT Receipt sample at 11.2 wt% CS in the SRAT Receipt slurry
- 30 g of slurry at 11.2 wt% CS lost in foamover (estimate, assuming same wt% CS concentration as the SRAT Receipt)
- 446 g of SRAT Product sample at 11.3 wt% CS in the SRAT Product slurry

$$(3458g - 30g) \times \frac{11.2\ g\ CS}{100\ g\ SRAT\ Receipt} - 446g \times \frac{11.3\ g\ CS}{100\ g\ SRAT\ Product} = 334g\ CS\ at\ start\ of\ SME$$

With the mass of calcined solids, the amount of frit to be added was calculated.

$$334\ g\ CS \times \frac{64\%\ frit}{36\%\ CS} = 594\ g\ frit$$

Shown in Table 3-26 is the SME final dewater calculation for a final TS of 42 wt%. Canister blast waters and water added with the first frit addition were not included in the calculation since these water amounts were removed shortly after addition and do not contribute to the final mass.

Table 3-26. SME Cycle Dewater Calculation

A	SRAT Product Mass	2,952	g
B	SRAT Product wt% TS	20.0	wt%
C	SRAT Product TS mass (A×B/100)	590	g
D	Added water with antifoam (2 additions at 1.4 g each)	2.8	g
E	Added Frit (solids)	594	g
F	Water with second frit addition	297	g
G	SME Product mass before concentration (A+D+E+F)	3,846	g
H	SME Product TS mass (C+E)	1,184	g
I	SME Product mass at target 42 wt% TS (H/0.42)	2,820	g
J	Amount to dewater (G–I)	1,026	g

Processing parameters were scaled to a 6,000 gal SME feed. The SRNL volume was calculated from the SRAT Product mass and SRAT Product density. DWPF and SRNL-scale processing parameters are shown in Table 3-27.

Table 3-27. SME Cycle DWPF and SRNL Scaled Processing Parameters

	DWPF	SRNL
Volume	6,000 gal	2,660 mL
Volume-based scale factor	8,538	
Purge	72 SCFM (1 atm, 21.11 °C)	239 sccm (1 atm, 21.11 °C)
Boilup Rate	5,000 lb/h	4.43 g/min
Canister Blast Water	1,000 gal	443 mL
Frit 473 Amount (for waste loading of 36 wt%)	N/A	594 g (along with 594 g water)
Final Dewater for 42 wt% TS	N/A	1,026 g

The demonstration included the addition and removal of water representing five canister blast water additions, addition of frit with an equal mass of water, removal of the added water, addition of a second batch of frit with equal mass of water, removal of that water, and finally removal of water to target 42 wt% TS in the SME Product. Momentive™ Y-17112 antifoam was added per the recommendation memo: 25 mg/kg of antifoam (on an undiluted basis) prior to first canister blast water addition, 25 mg/kg of antifoam prior to first frit addition, and 25 mg/kg of antifoam as needed to control foaming throughout the experiment.[44]

A timeline is given in Table 3-28. No significant processing issues such as mixing, fouling, foaming, etc., were encountered during the testing. No additional antifoam was added. Frit addition took longer than planned due to the funnel plugging. A larger opening funnel will be utilized for the next qualification run. This slow addition did not have any impact on results.

Table 3-28. SME Cycle Timeline

Elapsed Time (h)	Activity
Prior to heating	Added canister decontamination water-1 Added Momentive™ Y-17112 antifoam
0.0	Began heating
1.0	Began boiling, dewatered the added water
2.6	Stopped heating
3.2	Added canister decontamination water-2, Began heating
3.7	Began boiling, dewatered the added water
5.3	Stopped heating
5.5	Added canister decontamination water-3, Began heating
5.9	Began boiling, dewatered the added water
7.5	Stopped heating
7.7	Added canister decontamination water-4, Began heating
7.9	Began boiling, dewatered the added water
9.6	Stopped heating
9.8	Added canister decontamination water-5, Began heating
10.1	Began boiling, dewatered the added water
11.8	Stopped heating, allowed vessel to cool below 80 °C, Added Momentive™ Y-17112 antifoam Added frit addition-1 and equal mass of water
13.5	Started heating
14.3	Began boiling, dewatered the added water
15.5	Stopped heating, allowed vessel to cool to below 60 °C, added frit addition-2 and equal mass of water (Note that frit became damp and did not flow well in the first addition, thus the contents were allowed to cool more before the second addition)
17.4	Started heating
18.2	Began boiling. dewatered the added water and the amount of water needed to reach target TS
22.6	Stopped heating, ending the SME cycle

Following the SME cycle, samples were taken for analyses as specified in the TTQAP, which included fabrication of a glass and characterization of that glass.[2]

A summary of antifoam additions is given in Table 3-29.

Table 3-29. Summary of Antifoam Additions

Antifoam added (mg/kg)	Description of Addition
25	Resumption of the SRAT cycle (post foamover, post shutdown)
25	Prior to glycolic acid addition (after nitric acid addition)
50	Prior to boiling (after acid additions)
25	11.3 hours after previous addition
25	12.2 hours after previous addition
25	Start of SME cycle (at initiation of heating)
25	Prior to the first frit addition

3.2.3 Process Measurements

A plot showing the pH, temperature, and mixer torque of the sludge during SRAT Cycle Part 1 (i.e., the run up to and including the foamover events) are provided in Figure 3-1. The first foamover occurred about 110 minutes after the start of acid addition, at the point where the measured pH reached 7.7. Approximately 2 minutes before the foamover event, at pH 7.8, the mixer torque dropped from 11 in-oz to less than 7 in-oz. Additionally, the condenser temperature (vapor space) briefly increased from 25 °C to 29 °C during the foamover event, but then cooled back to 25 °C for the remainder of the cycle.

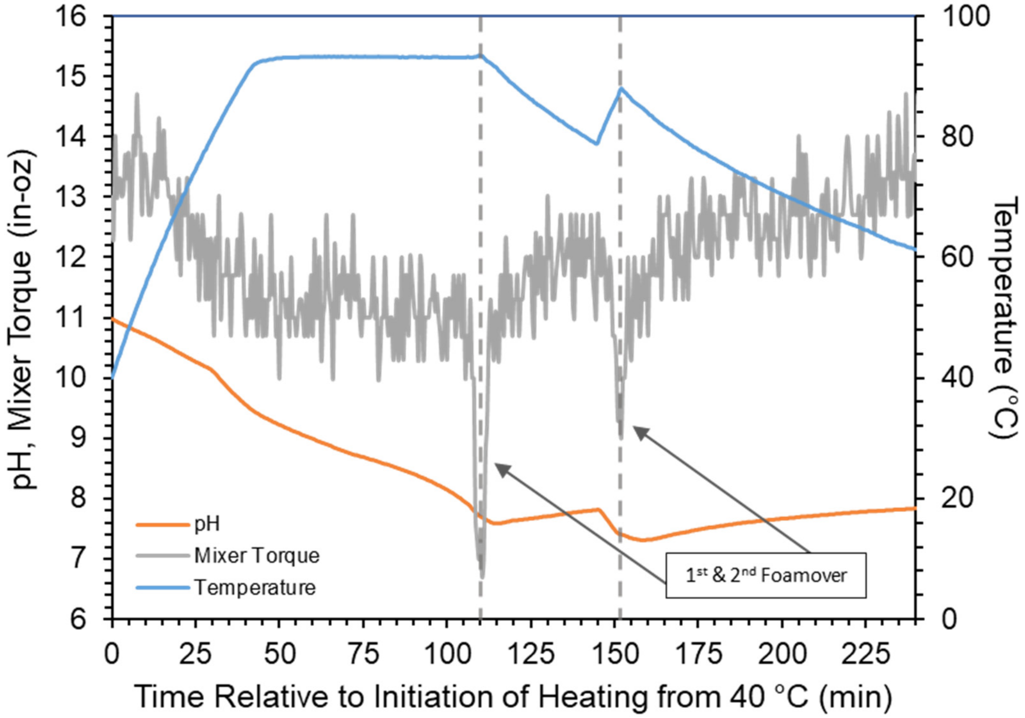

Figure 3-1. Plot of pH, mixer torque, and temperature of SRAT Cycle Part 1 vs time from the start of nitric acid addition through the foamover events

Heating and acid addition were resumed, and a second foamover event occurred at approximately 2.5 hours after the initial start of acid addition. Two minutes before the 2nd event, the mixer torque dropped from 11 in-oz to 9 in-oz. A temperature increase was not observed in the condenser vapor space during the second foamover. Both the lower drop in mixer torque and lack of condenser vapor space temperature increase suggests the second foamover was not as extreme as the first.

Heating and acid addition were suspended immediately following the first foamover event. The pH of the SRAT vessel dropped as low as 7.6, but increased to 7.8 after the system equilibrated (approximately 3 minutes) and the mixer torque returned to 11 in-oz approximately two minutes after foamover. The vessel temperature dropped to 80 °C during the period without heating and acid addition.

Heating and nitric acid addition were resumed, and a second foamover event occurred 151 minutes into the experiment, as shown in Figure 3-1 after the pH had reached 7.5 and the vessel temperature increased to 88 °C. Similar to the first foamover event, the mixer torque decreased from 11.5 to 9 approximately two minutes prior to the foamover and returned to prior levels after nitric acid addition and heating were stopped. The 2.5 in-oz drop in mixer torque was less than the 4 in-oz drop observed during the first foamover, indicating the second event was not as severe. Another indicator the second foamover was less severe is that no temperature increase was observed in the condenser vapor space.

A plot showing the pH, temperature, and mixer torque of the sludge for the second part of the SRAT cycle is shown in Figure 3-2. The SRAT pH dropped to 6.7 at the conclusion of nitric acid addition. The SRAT pH dropped further to 3.4 upon conclusion of glycolic acid addition and remained relatively stable at 3.5 for the remainder of the cycle. During nitric and glycolic acid addition, the mixer torque ranged from 15-17 in-oz. The mixer torque dropped from 17 to 13 in-oz during the dewater step and remained relatively stable at 13 in-oz through reflux. Note the decrease in temperature observed at 13 hours in Figure 3-2 was due to a brief operational pause during the cleaning step and not a function of the process.

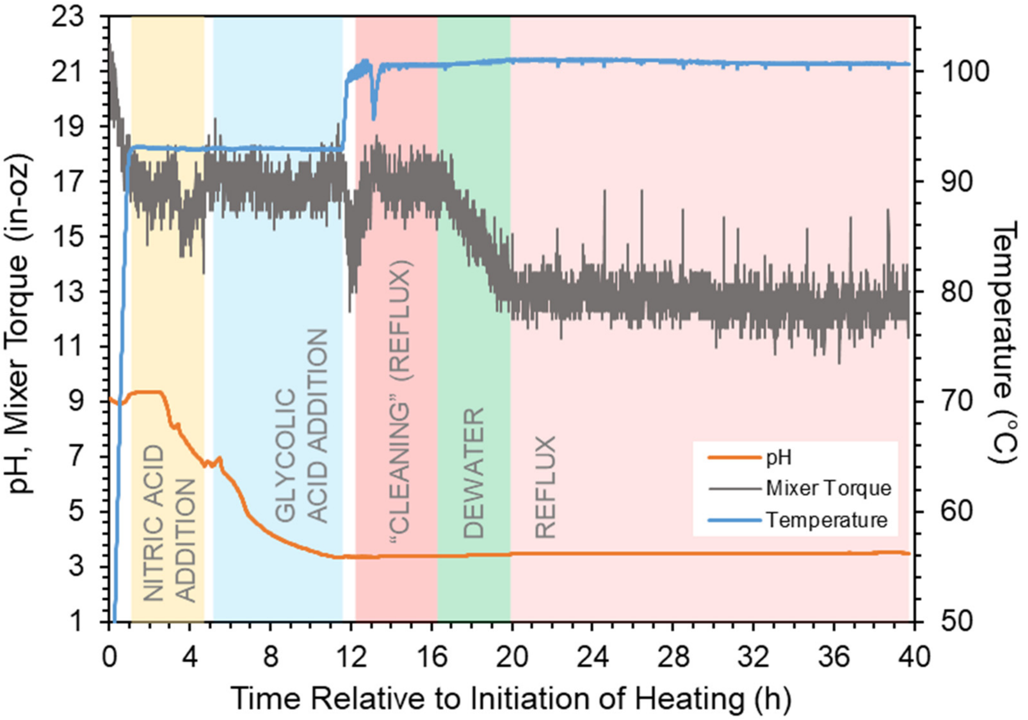

Figure 3-2. Plot of pH, mixer torque, and temperature vs. time for SRAT Cycle Part 2

A plot showing the pH, temperature, and mixer torque of the sludge for the SME cycle is shown in Figure 3-3. In general, rheology improves with increased temperature, which is the case observed here where the mixer torque increased from 11 in-oz when boiling to 17-20 in-oz during cooling and water additions. The pH in the SME cycle remained below 4. Note that water and frit were added via the pH probe port, requiring the pH probe to be briefly removed, resulting in no data ("gaps") in the figure during these times.

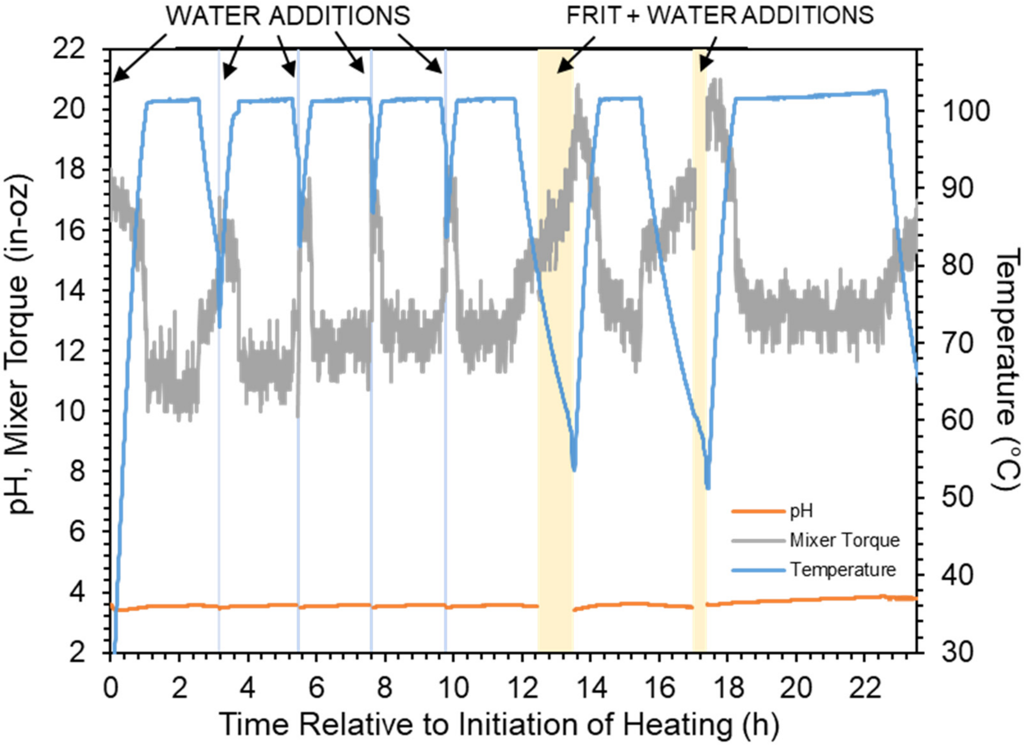

Figure 3-3. Plot of pH, Mixer Torque, and Temperature of the SME Cycle vs Time From Initiation of Heating

3.2.4 Off-gas Results

3.2.4.1 SRAT and SME Off-gas Generation

Presented in Table 3-30 are peak carbon dioxide, nitrous oxide, hydrogen, and methane concentrations and DWPF-scale generation rates for the SRAT and SME cycles, as previously reported.[47] Because SRNL scaled purge rates to DWPF, SRNL-scale and DWPF-scale concentrations are equivalent. The TTR also specifies generation rates for nitrous oxide and hydrogen, which are also included in the table.[1] The DWPF does not have a concentration or generation rate limit for carbon dioxide, nitrous oxide, or methane. With the NGA flowsheet, the DWPF limit for hydrogen generation is 2.4×10^{-2} lb/h in the SRAT and the SME. The maximum SRNL-observed hydrogen generation rates were ~1/90th of this limit. Off-gas plots and commentary follow Table 3-30.

Table 3-30. SB10 SRAT and SME Cycle Peak Off-Gas Concentrations and Generation Rates

Gas	Unit	SRAT Cycle	SME Cycle
Carbon Dioxide	volume %	31#	0.43
	lb/h	320#	2.1
Nitrous Oxide	volume %	0.67	<0.05
	lb/h	4.5	<0.2
Hydrogen (DWPF Limit: 2.4×10^{-2} lb/h)	ppm_v	8.9	12
	lb/h	2.7×10^{-4}	2.7×10^{-4}
Methane	ppm_v	16	110
	lb/h	3.7×10^{-3}	2.0×10^{-2}

Generation rates are scaled to 94 SCFM purge rate for the SRAT cycle and 72 SCFM for the SME cycle.

The peak carbon dioxide measurements in the SRAT cycle has a possible low bias due to a significant extrapolation outside of the calibrated range.

The Table 3-30 and the individual graphs corresponding to the concentration and generation rates have been published before [47] The previously published graphs are also presented in Appendix C.

Off-gas plots for the SRAT Cycle Part 1 (up to the foamover) are shown in Figure 3-4 (concentration) and Figure 3-5 (generation rate). Part 1 was denoted to begin at the time when heating was initiated from 40 °C. The qualitative measurements for oxygen concentration appeared relatively constant and are therefore not plotted. No nitrous oxide or methane was detected during this stage of processing.

During the initial nitric acid addition step of the SB10 SRAT cycle, a foamover resulted in carryover of sludge from the SRAT to the MWWT via the off-gas line. The event occurred after approximately 60% of the nitric acid had been added, with the sludge temperature at 93 °C and agitator set at 600 rpm. Acid addition and heating were immediately stopped. The event can be seen on the SRAT Part 1 off-gas figures at around 1.75 hours (just before the carbon dioxide and hydrogen peaks). Upon observing the liquid level in the manometer continually increasing (approaching approximately 1 inch from the top, or approximately 7 inches of water pressure), the purge rate was set to zero (for approximately two minutes) until the pressure stabilized. At this point, the purge was resumed, and a timeout was called. SRNL management and SRMC personnel were notified and consulted. The decision was made to resume the demonstration (i.e., resume heating and acid addition). Heating and nitric acid addition were resumed concurrently (vessel temperature had dropped to 83 °C). Within seven minutes, a second foamover occurred resulting in additional sludge being transferred to the MWWT via the vessel off-gas line/condenser. Elevated carbon dioxide and hydrogen levels were observed at approximately 2.5 hours. Heating and acid addition were halted, and the purge rate was reduced for (approximately four minutes) to prevent loss of water from the manometer. Another timeout was called, with SRNL management and SRMC personnel notifications.

The experiment was placed in a safe configuration. After internal SRNL discussions and discussions with SRMC Engineering personnel, the run was resumed 22 days later, designated as SRAT Cycle Part 2.

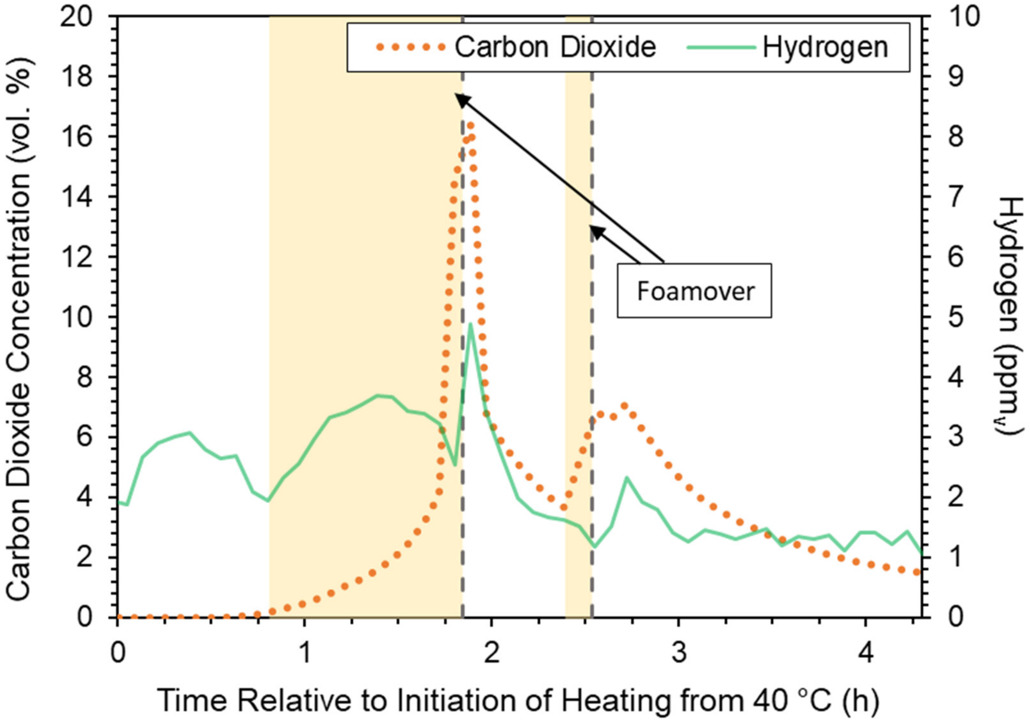

Figure 3-4. SRAT Part 1, Off-gas Concentration (shaded areas show nitric acid addition)

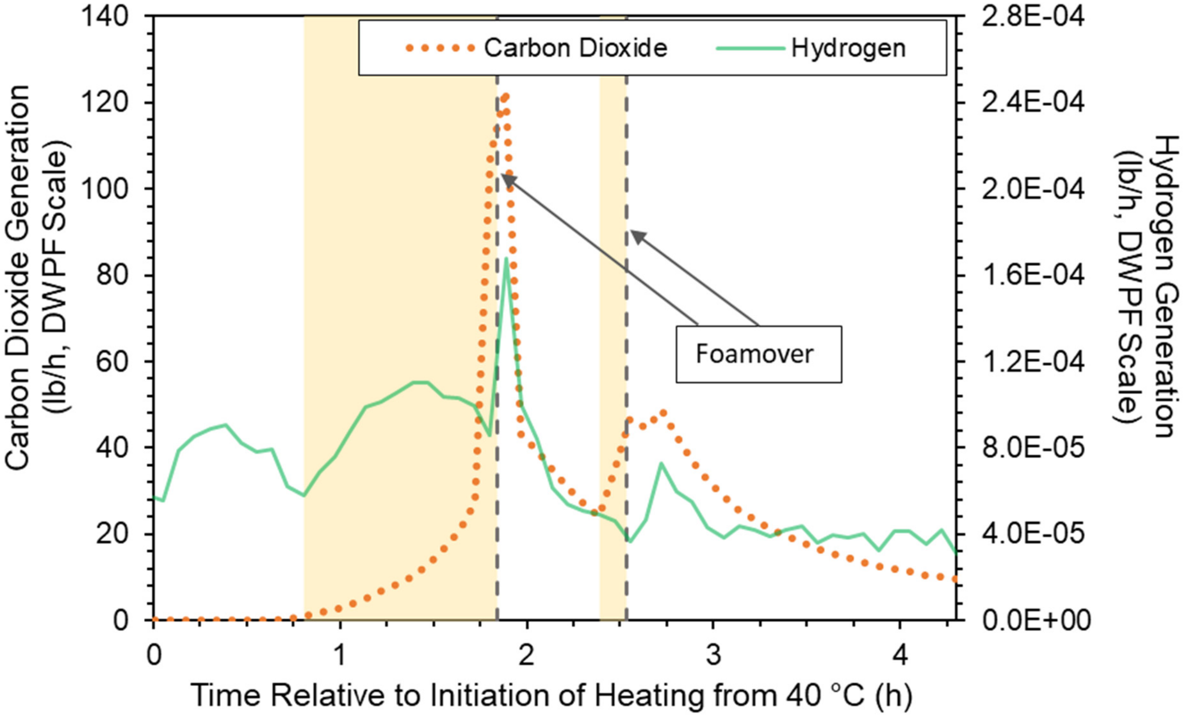

Figure 3-5. SRAT Part 1, Off-gas Generation (shaded areas show nitric acid addition)

Off-gas plots during the completion of the SRAT cycle follow. Carbon dioxide, nitrous oxide, hydrogen, and methane plots are presented in Figure 3-6 (concentration) and Figure 3-7 (DWPF-scale generation rate).

As can be seen in the plots, and as expected, carbon dioxide was produced during acid addition, and nitrous oxide, occurring at the latter part of acid addition and into boiling, shows nitrite was being destroyed.

In comparing the qualification run to typical SRNL SRAT cycles, the carbon dioxide peak was early and low, and generation dropped to baseline by the time of dewater. The early peak shows the carbonate was destroyed earlier than is typical. See the SB10 simulant report, particularly run TK51-4.[48] The time of carbonate destruction and the relatively small peak are likely due to the slow acid addition. Similarly, nitrous oxide peak was lower and broader due to the slower acid addition. The lower acid addition rate may have limited (but certainly affected) nitrogen dioxide production, though this would need more study.

Hydrogen generation peaked after nitrous oxide peaked and then declined. Note that the peak in hydrogen concentration around the 13 hour mark was due to reducing the purge from 368 sccm to 50 sccm for approximately six minutes during initial reflux to clean the off-gas line of sludge from the foamover described above. When the reduced purge is accounted for in the calculation, the hydrogen generation is in line with the calculated generation before and after the high concentration.

Methane was only detected for approximately one hour at near its limit of quantification of 14 ppm during the “cleaning” (reflux) period of the SRAT cycle. Typically, this occurs during the dewater segment of the run.

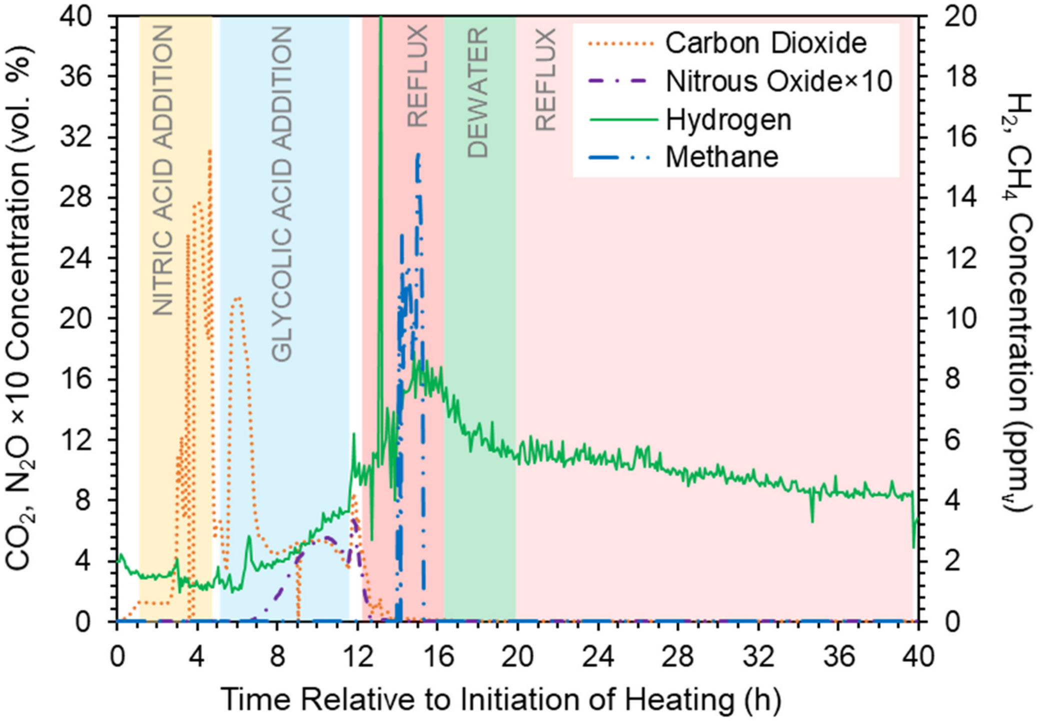

Figure 3-6. SRAT Part 2 Off-gas Concentrations (N_2O concentrations are to be divided by 10)

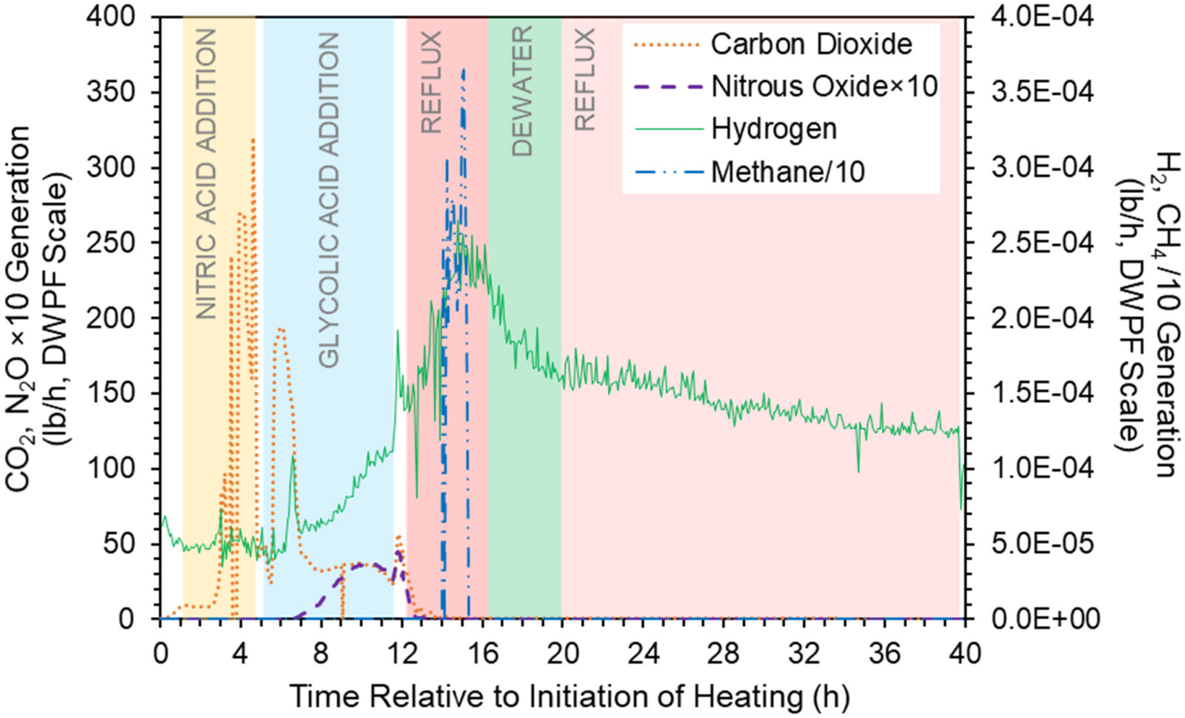

Figure 3-7. SRAT Part 2 Generation Rates(N$_2$O generation rates are to be divided by 10 and CH$_4$ generation rates are to be multiplied by 10)

Qualitative oxygen results are presented in Figure 3-8. As stated in Section 2.1.5, the GC method was optimized to quantify low concentrations of hydrogen and methane. Thus, the detector was saturated with oxygen and nitrogen and could not be reliably calibrated for those gasses. As a result, the oxygen results should be considered qualitative. No detectible drop in oxygen during peak carbon dioxide generation (nitric acid addition) is evidence of detector saturation. A drop in oxygen during glycolic acid addition is discernable, albeit not quantifiable.

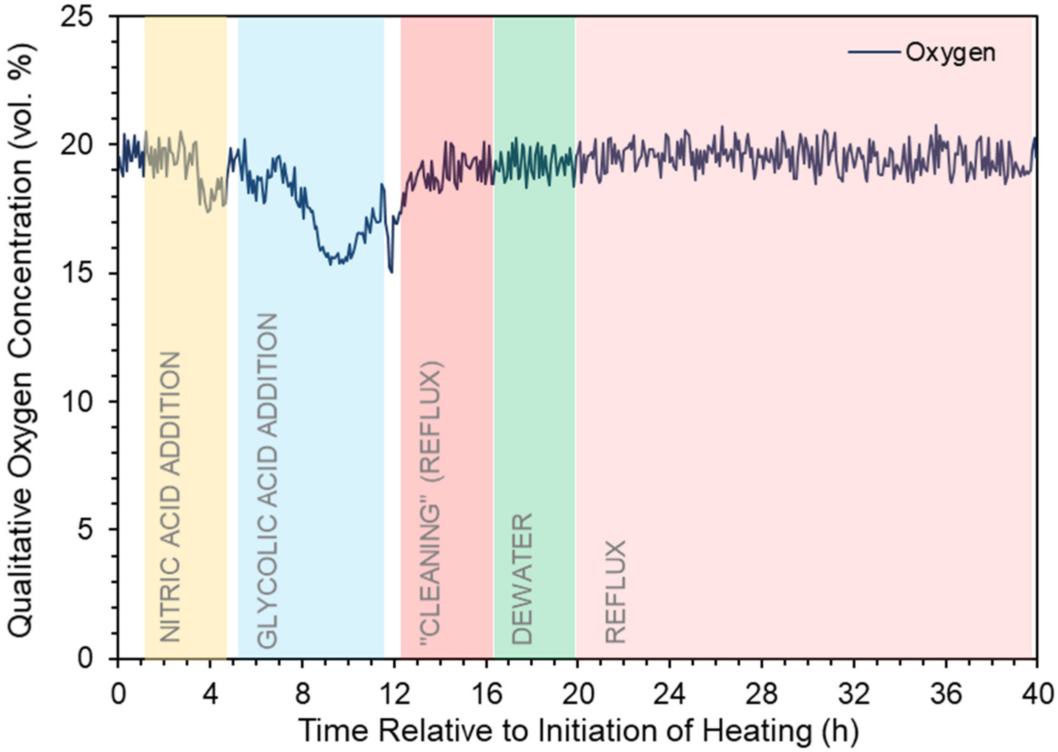

Figure 3-8. SRAT Part 2 Qualitative Oxygen Concentration

One consequence of the reduced rate of acid addition is slower off-gas production – specifically carbon dioxide and nitrous oxide. The peak concentrations and generation rates for these gases would be expected to be higher if the acid were added more quickly. Slow acid addition should not have impacted the hydrogen generation as that typically occurs after nitrite destruction.

SME Cycle off-gas plots for carbon dioxide, hydrogen, and methane (concentration and generation rates) are given in Figure 3-9 (concentration) and Figure 3-10 (generation rate). Nitrous oxide was detected as purging, mixing, and heating began and then the concentration dropped to less than detect. Therefore, a nitrous oxide plot is not shown. The qualitative measurements for oxygen concentration appeared relatively constant and are therefore not plotted.

The relatively high concentrations of carbon dioxide, nitrous oxide, hydrogen, and methane during heat-up are retained gases being released. Thus, the concentrations during heat-up are not considered in determining the maximum concentrations and generation rates under conditions of continuous mixing and gas purge.

All three gases – carbon dioxide, hydrogen, and methane – showed relatively high concentrations as vessel contents were brought back to boiling after canister blast and frit additions. This suggests that when not boiling, generated carbon dioxide, hydrogen, and methane are being generated and retained and they are released when boiling is initiated. See also Section 3.2.4.2 which shows gas release upon mixing. Note that SRNL reaches boiling in the lab scale experiments much faster than DWPF. SRNL peaks are likely conservative as the DWPF slower heat-up and lower boil-up rates would result in lower, broader gas generation peaks.

The peak methane concentration was 110 ppm$_v$, which is 0.22% of the methane lower flammability limit in air of 50,000 ppm$_v$.[49] Each peak is during the cycle is higher than the previous peak.

There is a slight "bump" of carbon dioxide and methane as the vessel began cooling. It is postulated that during cooling, as water vapor condenses, the pressure in the vapor space decreases and the gases are trapped in bubbles. When the pressure decreases, the bubbles grow and break. The cooldown peak is almost as big as the heat-up peak.

Hydrogen had local peaks of similar amount each time boiling began. It is likely hydrogen is being retained to some extent during water and frit additions and is then released with boiling.

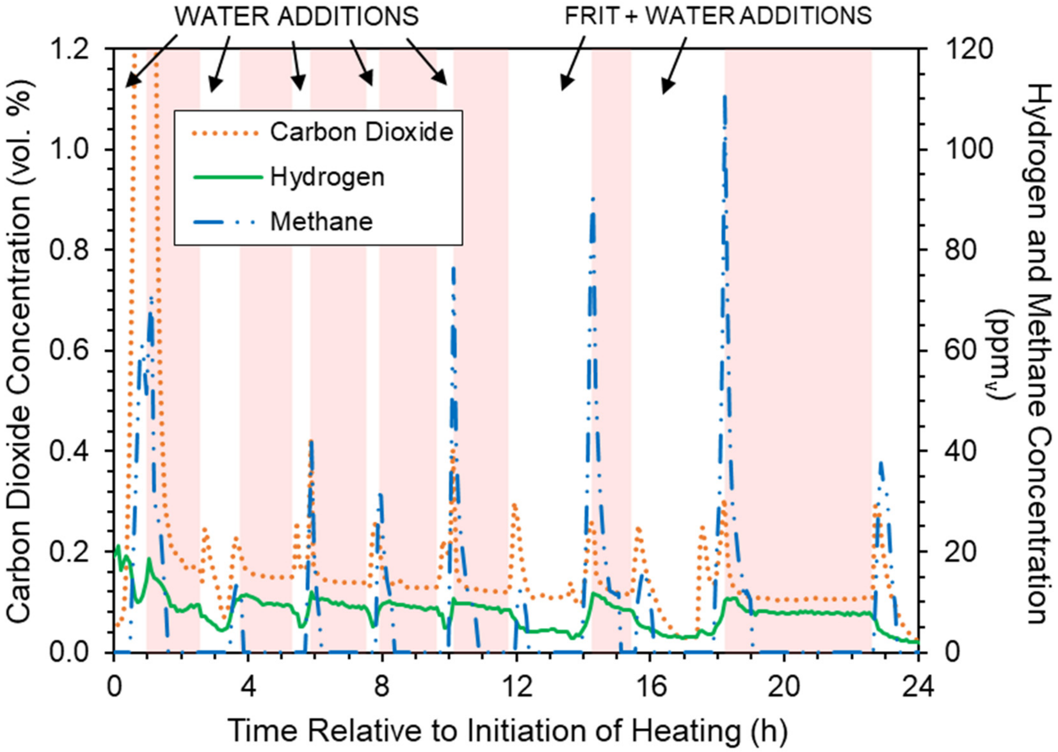

Figure 3-9. SME Cycle Off-gas Concentration (shaded areas represent boiling)

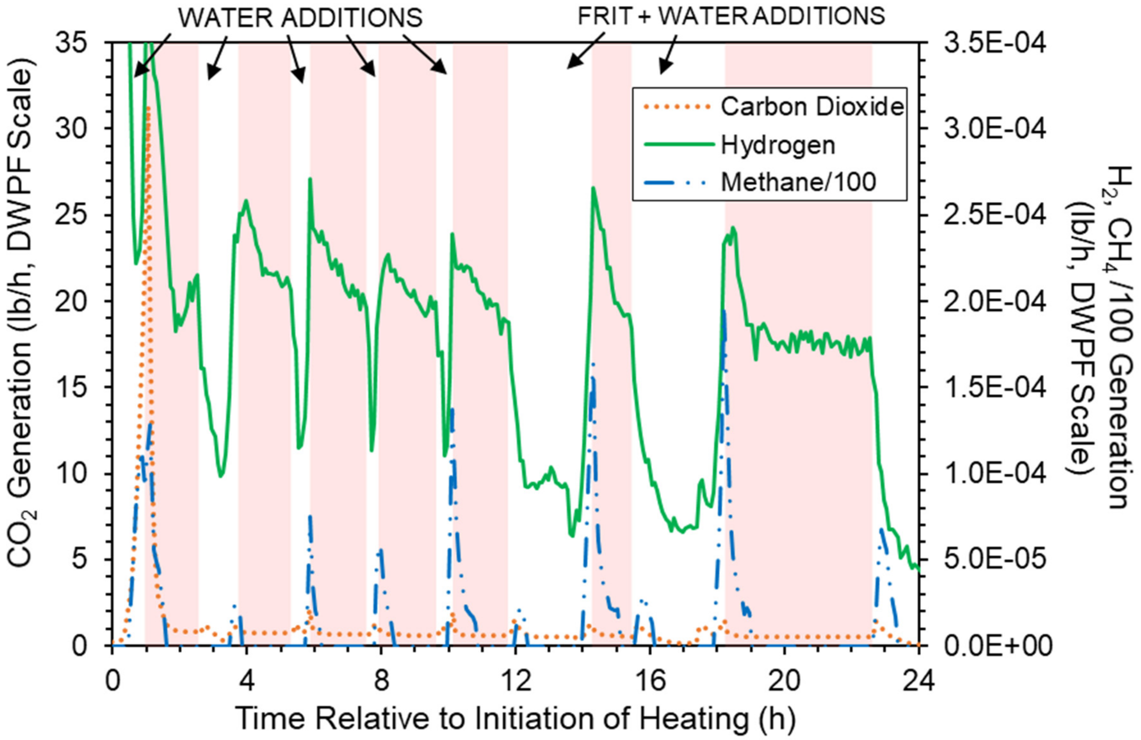

Figure 3-10. SME Cycle Off-gas Generation (shaded areas represent boiling, CH_4 generation rates are to be multiplied by 100)

3.2.4.2 Retained Gas Release

In addition to gas generation during CPC processing, SRMC requested SRNL to monitor the release of retained hydrogen at the start of each cycle. Several other tests were also performed. A list of the tests with estimated quiescent time is presented in Table 3-31.

Table 3-31. Summary of Retained Gas Release Tests

Test and Corresponding Figure	Estimated Quiescent Time (d)
Start of SRAT Cycle (Figure 3-11)	4
Post SRAT Test 1 (Figure 3-12)	13
Post SRAT Test 2 (Figure 3-13)	21
Start of SME Cycle (Figure 3-14)	12
Post SME Test 1 (Figure 3-15)	47
Post SME Test 2 (Figure 3-16)	2

Figure 3-11 shows the retained hydrogen release at the start of the SRAT cycle (SRAT Cycle Part 1). Only hydrogen was detected, which was expected; hydrogen is the main gas, produced from radiolysis, in SRS Tank Farm sludge and supernatant.

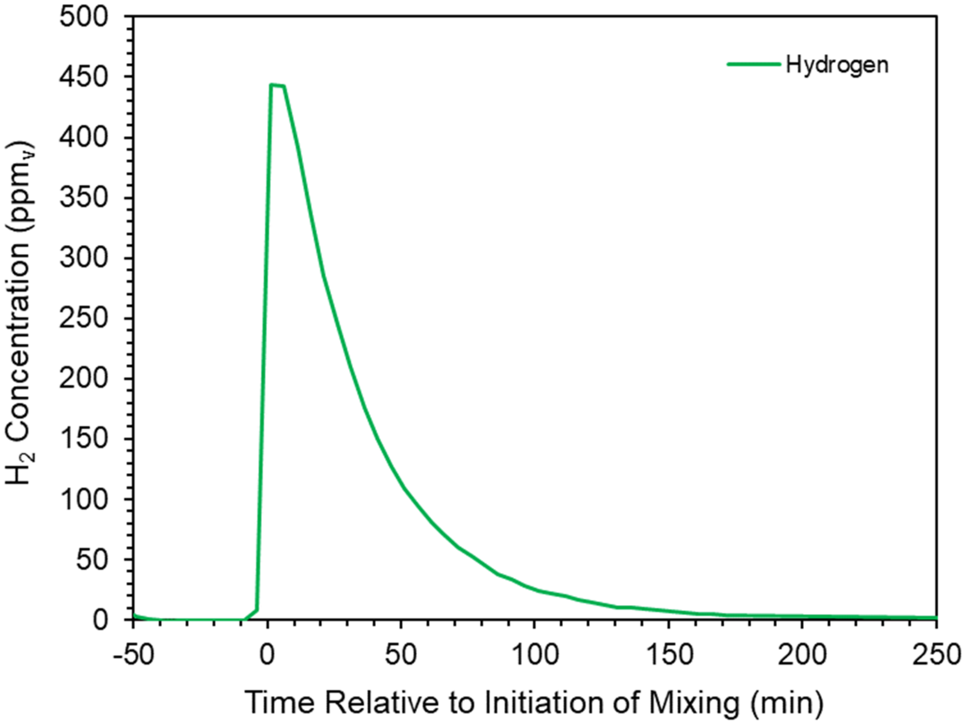

Figure 3-11. Gas Release at Initiation of SRAT Cycle, Agitator 600 RPM, Purge 368 sccm

Figure 3-12 and Figure 3-13 show gas release from tests after the SRAT cycle was completed (prior to the SME cycle). Due to operational issues at the resumption of the SRAT cycle (SRAT Part 2), retained gas release from that phase of testing is not available. As can be seen, all the quantifiable gases were detectible, suggesting that even after the SRAT cycle was completed and cooled, reactions did continue.

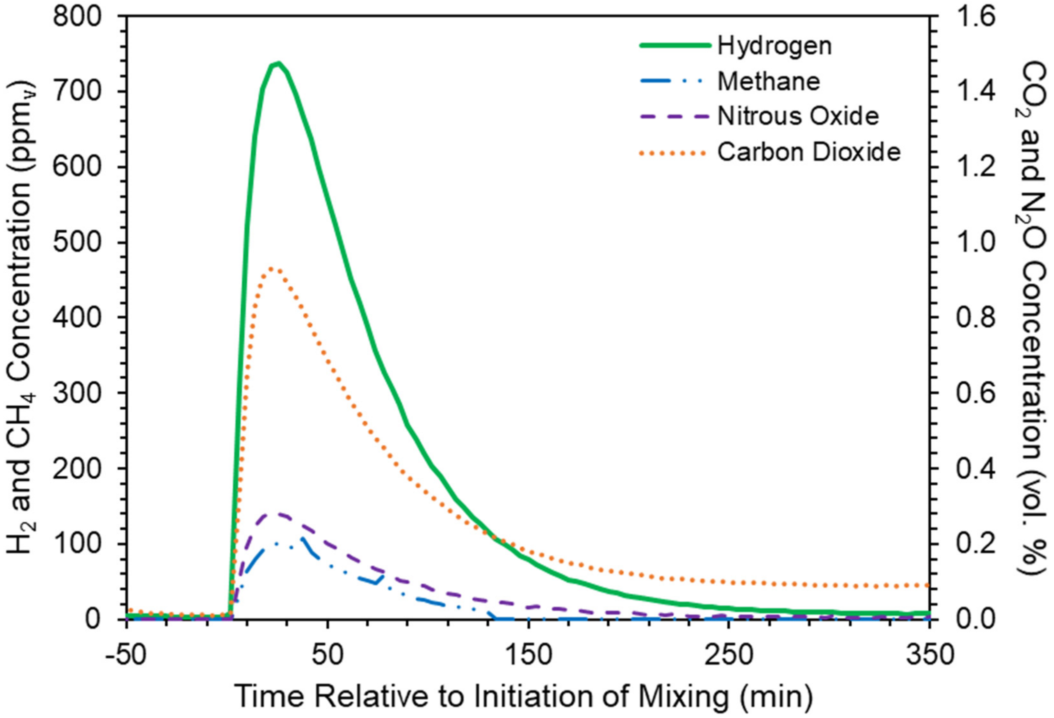

Figure 3-12. Gas Release Post SRAT Agitator 600 RPM, Purge 100 sccm

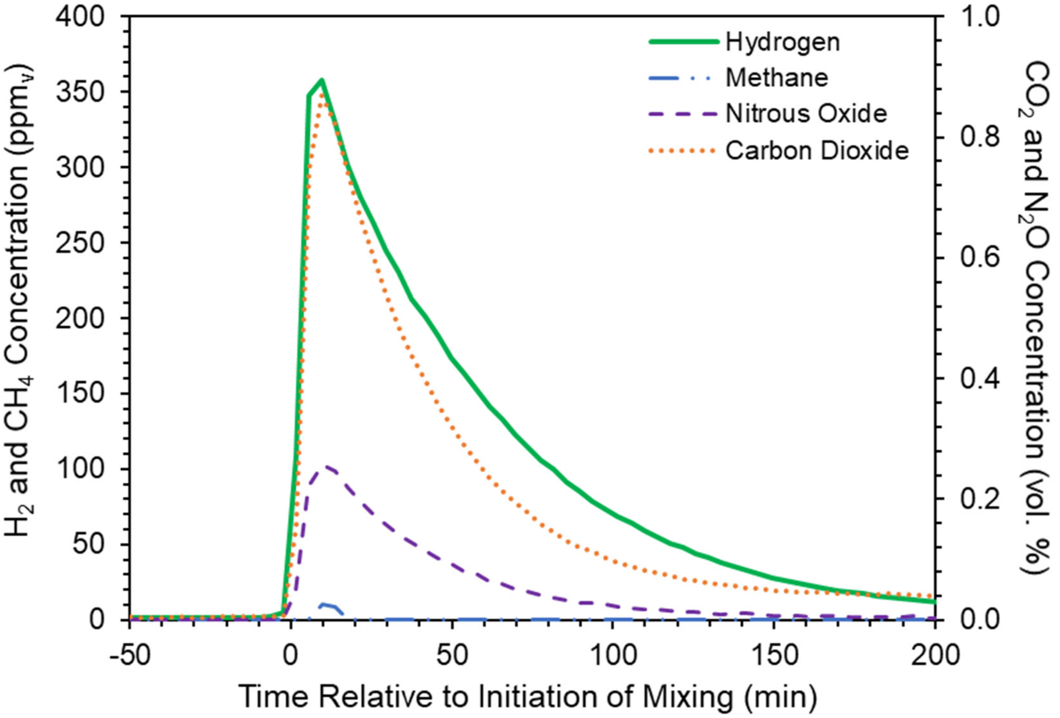

Figure 3-13. Gas Release Post SRAT Agitator 750 RPM, Purge 300 sccm

Figure 3-14 shows the off-gas at the start of the SME cycle and beyond the initiation of boiling to remove the first water canister blast. The methane and carbon dioxide detected as the vessel was heated suggest that a portion of these gases is dissolved in the sludge-slurry.

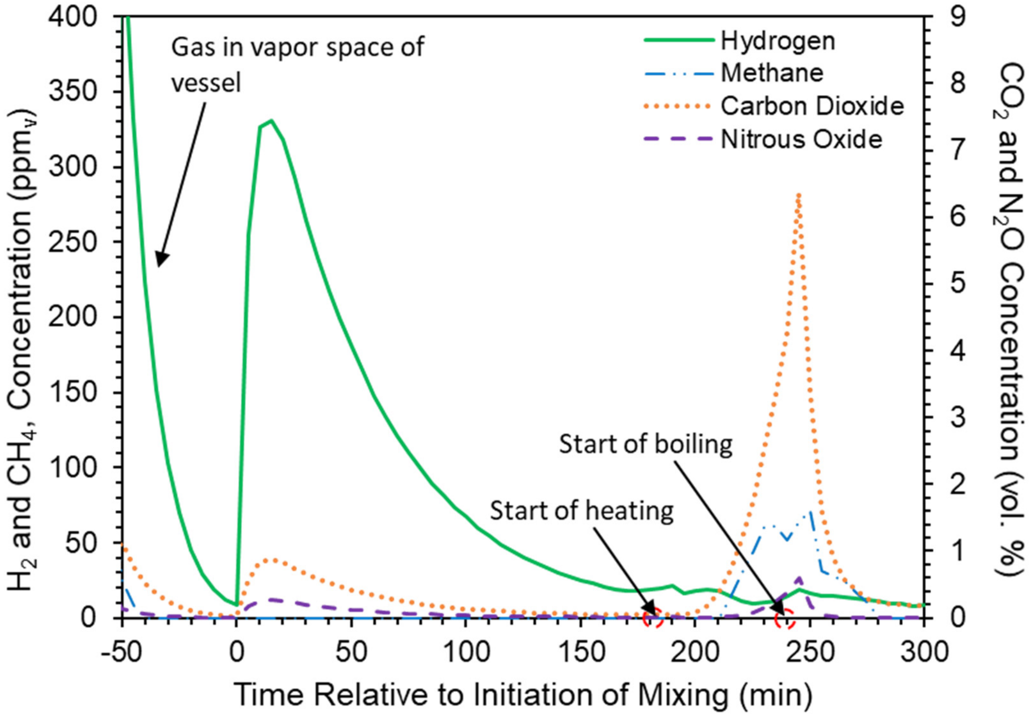

Figure 3-14. Gas Release at the Start of the SME Cycle Agitator 600 RPM, Purge 239 sccm

Figure 3-15 and Figure 3-16 show gas release with SME Product. The first test was completed forty-five days after SME cycle completion, thus the high hydrogen peak. The second test was completed 3 days after the first test. As can be seen in the first test, nitrous oxide, methane, and carbon dioxide were present in the slurry. In both cases, most of the retained gas was removed and purged within 100 minutes of agitation.

For Figure 3-14 through Figure 3-16, the measurements prior initiation of mixing are reflecting gasses that were built-up in the vessel headspace during the quiescent period with no agitation or purge gas flow. When purge gas flow was reestablished in preparation for the gas release test, the gas concentrations were allowed to return to just above detectable levels prior to initiating mixing.

As a separate effort, M-Star® computational fluid dynamics software is being used to simulate the release rate of hydrogen from the mixing the SRAT and SME after quiescent periods.[50-51] That effort will involve a comparison of the data in this section to the computational fluid dynamics simulations. Additional discussion and calculations related to this gas release data will be included in the report resulting from the modeling comparison.

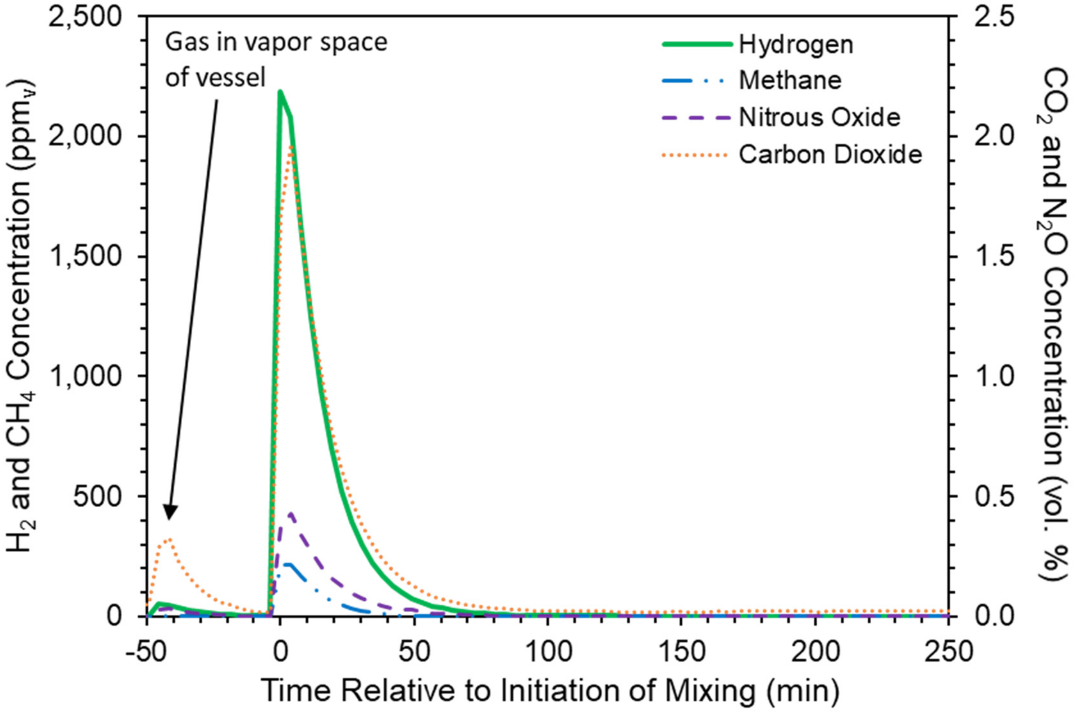

Figure 3-15. Gas Release Post SME Agitator 600 RPM, Purge 300 sccm

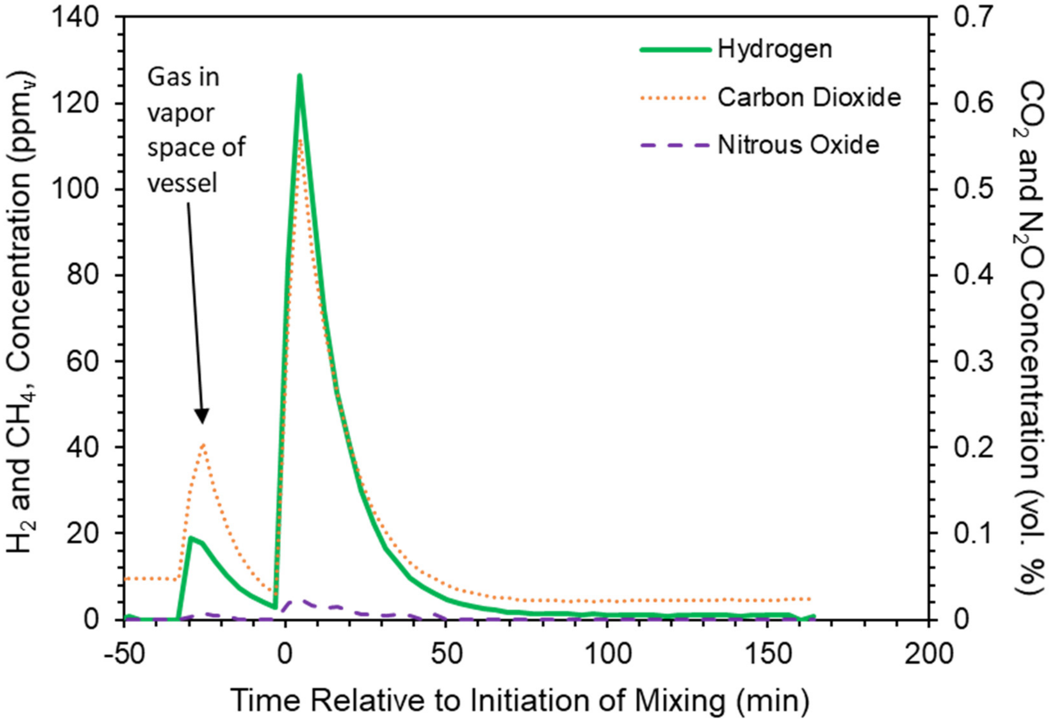

Figure 3-16. Gas Release Post SME Agitator 750 RPM, Purge 300 sccm Note that methane was not detected in this test.

3.2.5 Product Analytical Results

Physical data related to the density and the solids content for the SRAT Receipt and SRAT and SME Products are shown in Table 3-32. As expected, slurry densities are directly proportional (though not linearly) to the TS. The SRAT Product slurry density of 1.14 g/mL reflects the relatively low TS of 20.1 wt%. After frit addition and net water evaporation during the SME cycle, the SME Product had a density of 1.42 g/mL and TS of 48.1 wt% (versus a targeted TS of 42 wt%).

Likewise, supernate densities are roughly directly proportional to DS measurements and SS calculations. SRAT and SME Products had supernate densities of 1.08 and 1.17 g/mL, respectively. DS content increasing from 7.4 wt% in the SRAT Receipt supernate to 14.6 wt% in the SRAT Product filtrate and 23.8 wt% in the SME Product supernate reflects the impact of acid addition and the dissolution of a portion of the metals with the NGA flowsheet.

IS decreased from 8.2 wt% in the SRAT Receipt to 6.4 wt% in the SRAT Product, reflecting the partial dissolution of solids with the NGA flowsheet. Insoluble solids in the SME Product increased to 31.9 wt% due to frit addition. CS in the SRAT Receipt and SRAT Product were consistent at 11.3 wt%, while CS in the SME Product increased to 37.7 wt% due to the addition of frit.

Table 3-32. Densities and Solids Contents of the SRAT Receipt, SRAT Product, and SME Product, *n* = 4

Sludge Batch 10	SRAT Receipt		SRAT Product		SME Product	
	Average	RSD	Average	RSD	Average	RSD
Slurry Density (g/mL slurry)	1.10	1%	1.14	0.3%	1.42	0.6%
Supernate Density (g/mL supernate)	1.06	0.4%	1.08	0.6%	1.17	1.4%
Total Dried Solids (wt% slurry)	15.0	0.8%	20.1	0.4%	48.1	0.8%
Dissolved Solids (wt% supernate)	7.4	3%	14.6	1.1%	23.8	0.3%
Insoluble Solids (wt% slurry)	8.2	--	6.4	--	31.9	--
Soluble Solids (wt% slurry)	6.8	--	13.7	--	16.2	--
Calcined Solids (wt% slurry)	11.3	0.1%	11.3	0.9%	37.7	1.5%

Figure 3-17. SME Product during vacuum filtration

Figure 3-17 contains a photograph taken through the Shielded Cell window of the SME Product during vacuum filtration using 0.45-micron cellulose nitrate filter. This is to show that the SME Product filtrate (at the at the bottom of the image) is clear but relatively dark when compared to the remaining SME Product slurry in the top section of the filter cup.

Table 3-33 contains results for the elemental components of the SRAT and SME Products. The table also includes the SRAT Receipt composition for comparison, which was reported earlier in Table 3-16, but with some additional ICP-MS results for certain elements. In examining the SME Product results, the addition of frit during the SME caused a significant increase in the components present in Frit 473 (B, Li, Na, and Si)[52] and diluted the other components from the slurry on a TS basis. Nitrogen, carbon, and halides are not summarized in this table (they are represented in the anion, TOC, and ammonium analysis table). Arsenic and selenium were not analyzed for in the SRAT and SME Products because they require special analysis and were not seen above the detection limit in the SRAT Receipt.

Final mercury concentration in the SRAT Product was 0.77 wt% of the TS, which met the current target of <0.8 wt% mercury in the SRAT Product TS. Mercury was further removed to 0.16 wt% of the SME Product TS.

Table 3-34 addresses a requirement of the TTQAP that all measurement replicates be reported for iron, gadolinium, manganese, and lead for the SRAT Receipt, SRAT Product, and SME Product. Iron and manganese each had four measurement replicates for ICP-ES of both AR and AF preparations, for a total of eight measurement replicates. Gadolinium and thorium each had an additional four measurement replicates for ICP-MS of the AR preparations, for a total of twelve replicate measurements. For gadolinium, however, the four measurement replicates for ICP-ES of the AF preparations of the SME Product were below the detection limit. Thus, gadolinium had only eight measurement replicates above the detection limit for the SME product.

Table 3-33. Elemental Analysis of the SRAT Receipt and SRAT and SME Product Slurries

Analyte	SRAT Receipt Slurry			SRAT Product Slurry			SME Product Slurry		
	Digestion, Analytical Method	Average (wt% of TS)	RSD, n	Digestion, Analytical Method	Average (wt% of TS)	RSD, n	Digestion, Analytical Method	Average (wt% of TS)	RSD, n
Ag	AR, MS	7.02E-03	2%, 4	AR, MS	4.78E-03	1.9%, 4	AR, MS	1.92E-03	4.9%, 4
Al	AF, ES	1.24E+01	5%, 4	AF, ES	8.96E+00	5.2%, 4	AF, ES	4.69E+00	4.1%, 4
B	AF, ES	<8.3E-02	--	AR, ES	<5.6E-03	--	AF, ES	1.18E+00	2.8%, 4
Ba	AR/AF, ES	4.59E-02	5%, 8	AR/AF, ES	3.58E-02	3.1%, 8	AF, ES	1.96E-02	8.3%, 4
Be	AR, ES	<2.4E-03	--	AR, ES	<1.2E-04	--	AR, ES	<4.0E-04	--
Ca	AR, ES	5.30E-01	3%, 4	AF, ES	6.14E-01	7.4%, 4	AF, ES	3.17E-01	16%, 4
Cd	AF, ES	<4.0E-03	--	AR, ES	<3.1E-03	--	AR, ES	<1.6E-03	--
Ce	AR, MS	1.21E-01	4%, 4	AR, MS	9.09E-02	0.9%, 4	AR, MS	4.17E-02	4.3%, 4
Co	AR, MS	2.52E-03	3%, 4	AR, MS	1.93E-03	1.7%, 4	AR, MS	9.12E-04	4.3%, 4
Cr	AR/AF, ES	1.61E-01	3%, 8	AR/AF, ES	1.15E-01	8.2%, 8	AR/AF, ES	5.85E-02	6.8%, 8
Cu	AR/AF, ES	3.52E-02	3%, 8	AR, ES	<3.4E-02	--	AR, ES	<1.6E-02	--
Eu	AR, MS	1.84E-03	5%, 4	AR, MS	1.37E-03	1.2%, 4	AR, MS	6.23E-04	3.3%, 4
Fe	AR/AF, ES	8.51E+00	3%, 8	AR/AF, ES	6.22E+00	6.1%, 8	AR/AF, ES	2.93E+00	7.6%, 8
Gd	AR/AF, ES	4.44E-02	5%, 8	AR/AF, ES	3.67E-02	3.8%, 8	AR, ES	1.80E-02	4.8%, 4
	AR, MS	4.94E-02	5%, 4	AR, MS	3.74E-02	0.8%, 4	AR, MS	1.71E-02	4.1%, 4
Hg	AR, DMA	2.85E+00	6%, 4	AR, DMA	7.68E-01	2.0%, 4	AR, DMA	1.56E-01	5.5%, 4
K	AR, ES	<7.6E-02	--	AR, ES	<4.3E-02	--	AR, ES	<2.1E-02	--
La	AR, ES	3.38E-02	3%, 4	AR/AF, ES	2.64E-02	5.1%, 8	AR/AF, ES	1.25E-02	7.6%, 8
	AR, MS	3.86E-02	4%, 4	AR, MS	3.05E-02	1.1%, 4	AR, MS	1.32E-02	5%, 4
Li	AF, ES	<6.3E-03	--	AF, ES	<2.7E-02	--	AF, ES	1.95E+00	2.6%, 4
Mg	AR/AF, ES	2.09E-01	3%, 8	AR/AF, ES	1.50E-01	5.4%, 8	AR/AF, ES	7.58E-02	4.5%, 8
Mn	AR/AF, ES	2.66E+00	3%, 8	AR/AF, ES	2.08E+00	7.8%, 8	AR/AF, ES	9.72E-01	6.2%, 8
Mo	AR, ES	<7.7E-03	--	AR, ES	3.76E-03	1.9%, 4	AR, ES	<2.3E-03	--
Na	AR, ES	1.66E+01	1%, 4	AR, ES	1.28E+01	0.5%, 4	AR, ES	6.09E+00	4.6%, 4
Nd	AR, MS	1.27E-01	4%, 4	AR, MS	9.62E-02	0.5%, 4	AR, MS	4.30E-02	4.6%, 4
Ni	AR/AF, ES	2.74E-01	5%, 8	AR/AF, ES	2.03E-01	4.0%, 8	AR/AF, ES	9.83E-02	8.0%, 8
P	AF, ES	<9.3E-02	--	AR/AF, ES	5.09E-02	8.4%, 8	AR, ES	2.52E-02	6.0%, 4
Pb	AR, MS	9.32E-03	3%, 4	AR, MS	6.63E-03	1.0%, 4	AR, MS	3.25E-03	3.7%, 4
Pd	AR, MS	1.45E-03	6%, 4	AR, MS	1.21E-03	1.5%, 4	AR, MS	5.54E-04	3.5%, 4
Pr	AR, MS	3.28E-02	4%, 4	AR, MS	2.58E-02	1.1%, 4	AR, MS	1.12E-02	6.3%, 4
Pu	AR, multi	2.40E-03	--	AR, multi	1.10E-02	--	AR, multi	5.30E-03	--
Rh	AR, MS	8.70E-03	4%, 4	AR, MS	6.37E-03	1.4%, 4	AR, MS	2.98E-03	4.5%, 4
Ru	AR, MS	4.24E-02	4%, 4	AR, MS	3.17E-02	0.7%, 4	AR, MS	1.46E-02	3.8%, 4
S	AR, ES	3.79E-01	2%, 4	AR/AF, ES	2.90E-01	2.7%, 8	AR/AF, ES	1.37E-01	3.1%, 8
Sb	AR, MS	3.12E-04	9%, 4	AR, MS	2.09E-04	2.1%, 4	AR, MS	9.42E-05	3.2%, 4
Si	AF, ES	4.24E-01	2%, 4	AF, ES	3.39E-01	13%, 4	AF, ES	1.76E+01	3.3%, 4
Sn	AR, MS	5.06E-02	6%, 4	AR, MS	4.07E-02	1.1%, 4	AR, MS	1.86E-02	3.7%, 4
Sr	AR/AF, ES	1.63E-02	5%, 8	AR/AF, ES	1.67E-02	3.7%, 8	AR, ES	7.59E-03	4.2%, 4
Ta	AR, MS	1.25E-04	15%, 4	AR, MS	<6.5E-05	--	AR, MS	<2.1E-05	--
Tb	AR, MS	1.66E-04	3%, 4	AR, MS	1.35E-04	0.7%, 8	AR, MS	6.31E-05	6.4%, 4
Tc	AR, MS	1.90E-03	3%, 4	AR, MS	1.38E-03	0.6%, 4	AR, MS	6.52E-04	2.7%, 4
Th	AR/AF, ES	1.73E+00	3%, 8	AR/AF, ES	1.26E+00	8.7%, 8	AR/AF, ES	5.96E-01	8.9%, 8
	AR, MS	1.75E+00	3%, 4	AR, MS	1.36E+00	2.1%, 4	AR, MS	5.42E-01	4%, 4
Ti	AR, ES	<1.4E-02	--	AR/AF, ES	1.04E-02	6.9%, 8	AR, ES	<5.6E-03	--
U	AR/AF, ES	2.22E+00	5%, 8	AR/AF, ES	1.62E+00	6.2%, 8	AR/AF, ES	7.75E-01	9.2%, 8
	AR, MS	2.46E+00	3%, 4	AR, MS	1.90E+00	0.8%, 4	AR, MS	7.49E-01	4.4%, 4
V	AF, ES	<3.4E-03	--	AR, ES	<1.3E-03	--	AR, ES	<6.2E-03	--
Zn	AR/AF, ES	1.75E-02	5%, 8	AR/AF, ES	1.31E-02	3.1%, 8	AR/AF, ES	6.50E-03	9.6%, 8
Zr	AR, ES	1.13E-01	3%, 4	AR, ES	8.49E-02	0.6%, 4	AR, ES	3.97E-02	4.7%, 4

Table 3-34. Replicate Measurements of Fe, Gd, Mn, and Th in SRAT Receipt, SRAT Product, and SME Product, in wt% of TS, σ = 10%

	SRAT Receipt		SRAT Product		SME Product	
	AR	AF	AR	AF	AR	AF
Fe by ES	8.93E+00	8.33E+00	6.52E+00	5.78E+00	2.97E+00	2.85E+00
	8.33E+00	8.20E+00	6.62E+00	5.93E+00	3.06E+00	2.62E+00
	8.60E+00	8.67E+00	6.52E+00	5.98E+00	3.22E+00	2.77E+00
	8.47E+00	8.53E+00	6.62E+00	5.83E+00	3.22E+00	2.77E+00
Gd by ES	4.47E-02	4.61E-02	3.70E-02	3.70E-02	1.71E-02	<3.2E-02
	4.19E-02	4.31E-02	3.71E-02	3.57E-02	1.75E-02	<2.1E-02
	4.37E-02	4.75E-02	3.70E-02	3.94E-02	1.87E-02	<3.6E-02
	4.21E-02	4.63E-02	3.69E-02	3.44E-02	1.87E-02	<3.3E-02
Gd by MS	5.23E-02	--	3.73E-02	--	1.64E-02	--
	4.90E-02	--	3.78E-02	--	1.66E-02	--
	4.96E-02	--	3.75E-02	--	1.76E-02	--
	4.67E-02	--	3.71E-02	--	1.78E-02	--
Mn by ES	2.81E+00	2.60E+00	2.22E+00	1.89E+00	9.69E-01	9.65E-01
	2.64E+00	2.53E+00	2.24E+00	1.96E+00	9.85E-01	8.84E-01
	2.73E+00	2.65E+00	2.23E+00	1.98E+00	1.06E+00	9.29E-01
	2.67E+00	2.64E+00	2.24E+00	1.91E+00	1.05E+00	9.36E-01
Th by ES	1.83E+00	1.70E+00	1.35E+00	1.21E+00	6.03E-01	5.72E-01
	1.69E+00	1.65E+00	1.38E+00	1.18E+00	6.36E-01	5.22E-01
	1.77E+00	1.75E+00	1.32E+00	1.22E+00	6.61E-01	5.51E-01
	1.73E+00	1.73E+00	1.35E+00	1.07E+00	6.63E-01	5.61E-01
Th by MS	1.79E+00	--	1.34E+00	--	5.16E-01	--
	1.68E+00	--	1.40E+00	--	5.36E-01	--
	1.78E+00	--	1.34E+00	--	5.59E-01	--
	1.74E+00	--	1.34E+00	--	5.55E-01	--

Table 3-35 contains the results for select radionuclides analyzed for in the SRAT and SME Product slurries. Results are reported in both concentration units (wt% of TS) and activity units (Ci/gal). ^{99}Tc, ^{232}Th, ^{237}Np, uranium isotopes, ^{239}Pu, and ^{240}Pu were determined from ICP-MS measurements of the AR digestion preparations. The remaining Pu isotopes, plus Am and Cm isotopes, were determined from radiochemical counting methods. The analogous results for radionuclides in SRAT Receipt slurry are contained in Table 3-19.

Table 3-35. Select Radioisotopes in the SRAT and SME Product Slurries, n=4

Analyte	Method	SRAT Receipt Slurry				SRAT Product Slurry				SME Product Slurry			
		Average		σ	RSD	Average		σ	RSD	Average		σ	RSD
		wt% of TS	Ci/gal			wt% of TS	Ci/gal			wt% of TS	Ci/gal		
^{99}Tc	MS	1.90E-03	2.01E-04	20%	3.3%	1.38E-03	2.02E-04	20%	0.6%	6.52E-04	2.85E-04	20%	2.7%
^{232}Th	MS	1.75E+00	1.20E-06	20%	2.9%	1.36E+00	1.29E-06	20%	2.1%	5.42E-01	1.53E-06	20%	3.7%
^{233}U	MS	5.11E-04	3.09E-05	20%	3.5%	3.58E-04	2.99E-05	20%	0.4%	1.74E-04	4.33E-05	20%	4.0%
^{234}U	MS	5.93E-04	2.31E-05	20%	1.1%	4.93E-04	2.66E-05	20%	7.1%	2.25E-04	3.62E-05	20%	6.6%
^{235}U	MS	2.93E-02	3.96E-07	20%	3.7%	2.47E-02	4.60E-07	20%	0.7%	1.16E-02	6.45E-07	20%	5.1%
^{236}U	MS	1.96E-03	7.92E-07	20%	4.4%	1.45E-03	8.12E-07	20%	0.9%	7.16E-04	1.19E-06	20%	4.7%
^{237}Np	MS	1.11E-03	4.89E-06	20%	4.2%	7.82E-04	4.76E-06	20%	1.0%	3.79E-04	6.89E-06	20%	3.8%
^{238}U	MS	2.43E+00	5.10E-06	20%	2.9%	1.87E+00	5.43E-06	20%	0.8%	7.36E-01	6.38E-06	20%	4.4%
^{238}Pu	RC	3.76E-04	4.02E-02	5%	6.8%	2.60E-04	3.84E-02	6%	15%	1.30E-04	5.72E-02	8%	10%
^{239}Pu	MS	1.45E-02	5.61E-03	20%	3.8%	9.97E-03	5.36E-03	20%	1.4%	4.78E-03	7.66E-03	20%	4.1%
^{240}Pu	MS	1.15E-03	1.64E-03	20%	3.6%	7.87E-04	1.55E-03	20%	2.4%	3.76E-04	2.21E-03	20%	3.9%
^{241}Pu	RC	3.00E-05	1.93E-02	15%	16%	1.79E-05	1.59E-02	15%	15%	9.34E-06	2.48E-02	16%	10%
^{241}Am	RC	2.83E-04	6.07E-03	5%	6.0%	2.05E-04	6.07E-03	5%	7.2%	1.05E-04	9.27E-03	5%	6.0%
^{242m}Am	RC	6.78E-08	4.11E-06	27%	15%	4.23E-08	3.56E-06	43%	51%	2.00E-08	5.01E-06	21%	20%
^{243}Am	RC	4.02E-05	5.01E-05	6%	4.4%	1.60E-04	2.76E-04	9%	12%	9.34E-06	4.80E-05	5%	11%
^{242}Cm	RC	1.65E-10	3.40E-06	27%	15%	1.03E-10	2.94E-06	43%	51%	4.86E-11	4.14E-06	21%	20%
^{244}Cm	RC	1.99E-06	1.00E-03	15%	27%	1.85E-06	1.29E-03	15%	10%	9.12E-07	1.90E-03	15%	7.8%
mass 241	MS	2.77E-04	--	20%	4.1%	1.88E-04	--	20%	0.8%	9.25E-05	--	20%	5.5%
mass 242	MS	8.90E-05	--	20%	3.8%	6.04E-05	--	20%	1.8%	2.94E-05	--	20%	3.9%

Table 3-36 contains results of the IC and wet chemistry measurements on the SRAT Product and SME Product slurries. Weighted dilutions with water were performed as the nominal preparation. CQ preparation was performed, nominally for more accurate glycolate and oxalate measurement of the slurries by IC. Upon inspection of the results, it appeared that CQ also provided better IC detection limits for the SRAT Product in this case, and thus was used to also report formate concentration and several detection limit values in the SRAT Product.

Nitrite was destroyed to below the detection limit (130 mg/kg in the SRAT, 570 mg/kg in the SME) during processing. As expected, large concentrations of nitrate and glycolate were noted due to the acid addition in the SRAT. Formate, oxalate, sulfate, and TOC were also quantified in the SRAT and SME Products. Chloride was below the detection limit of 130 mg/kg in the SRAT and 470 mg/kg in the SME, versus the SRAT Receipt supernate and slurry chloride measurement of 38 mg/L and <310 mg/kg, respectively. Although the chloride analysis in the SME Product did not meet the requested detection limit of 200 ppm, chloride is not added to the SRAT or the SME and can be estimated from the chloride result in the SRAT Receipt. Ammonium was not detected in the SRAT and SME Products (detection limits of 350 mg/kg and 470 mg/kg), respectively. Based on the nitrate, glycolate, and sulfate analysis, the SME Product slurry appears approximately 13 to 20 percent more concentrated on a mass basis than the SRAT Product slurry.

Table 3-36. Anions, TOC, and Ammonium in the SRAT and SME Product Slurries, n=4, σ nominally 10%

Analyte	Analytical Method	SRAT Product Slurry		SME Product Slurry	
		Average (mg/kg slurry)	RSD	Average (mg/kg slurry)	RSD
NO_3^-	ICA	5.28E+04	0.9%	6.33E+04	1.2%
NO_2^-	ICA	<1.3E+02 †	--	<5.7E+02	--
$C_2H_3O_3^-$	ICA-G	4.63E+04 †	2.0%	5.25E+04 †	3.2%
CHO_2^-	ICA	3.91E+02 †	0.4%	6.90E+02	2.3%
$C_2O_4^{2-}$	ICA	2.26E+03 †	1.9%	3.04E+03 †	2.6%
SO_4^{2-}	ICA	1.73E+03	1.3%	1.98E+03	1.3%
PO_4^{3-}	ICA	<1.3E+02 †	--	<4.7E+02	--
CO_3^{2-}	TIC/TOC	<1.6E+03	--	<7.0E+03	--
Cl^-	ICA	<1.3E+02 †	--	<4.7E+02	--
F^-	ICA	<1.3E+02 †	--	<4.7E+02	--
Br^-	ICA	<6.3E+02 †	--	<2.3E+03	--
NH_4^+	ICC	<3.5E+02	--	<4.7E+02	
TOC	TIC/TOC	1.66E+04	2.3%	1.79E+04	0.7%

† caustic quench preparation

Based on the nitrate, glycolate, formate, oxalate, and TS results in the SME Product slurry and using the predictive REDOX equation reported elsewhere,[48] the predicted iron REDOX for the SB10 SME Product is 0.10. This matches the targeted iron REDOX of 0.1.

Table 3-37, Table 3-38, and Table 3-39 contain results for the soluble portions of the SRAT Receipt and SRAT and SME Products. SRAT Receipt was prepared as undiluted supernate for most analyses while SRAT and SME Products were prepared as filtrate that had to subsequently be diluted based on the higher dose rates. Results for the soluble elemental analysis are on a mg/L of solution basis. Radioisotope analysis are reported in both mg/L and Ci/gal of solution. IC analysis is reported in mol/L of solution.

As seen in Table 3-37 and Table 3-38,the SRAT Receipt has relatively low concentrations of most soluble metals, with the exception of sodium, aluminum, and mercury. After addition and heating in the SRAT, the SRAT and SME Products show soluble concentrations of some of the metals and radionuclides increasing by many times, even by orders of magnitude. This solubility increase has been seen in previous tests of the NFA and NGA flowsheets, but is most pronounced with the NGA flowsheet at excess stoichiometric acid addition (as was this qualification test). A subsequent section explores the fraction of these components that are soluble in the SRAT and SME Products.

Table 3-37. Elemental Analysis of the SRAT Receipt Supernatant and the SRAT and SME Product Filtrate, n=4, σ nominally 10% (20% for Hg)

Analyte	Analytical Method	SRAT Receipt Supernate		SRAT Product Filtrate		SME Product Filtrate	
		Average (mg/L)	RSD	Average (mg/L)	RSD	Average (mg/L)	RSD
Ag	MS	≤1.9E-02	8.0%	7.95E-01	17%	3.73E-01	6.9%
Al	ES	1.21E+03	3.4%	9.86E+02	4.4%	1.87E+03	6.0%
B	ES	<2.1E+01	--	<7.9E+00	--	1.36E+02	6.0%
Ba	ES	<1.5E-01	--	<1.5E+00	--	<2.3E+00	--
Be	ES	<1.1E-01	--	<2.2E-01	--	<5.1E-01	--
Ca	ES	<4.3E+00	--	8.73E+02	4.4%	8.62E+02	6.1%
Cd	ES	<3.0E-01	--	2.32E+00	5.5%	<4.6E+00	--
Ce	MS	<2.0E-03	--	1.15E+02	3.2%	2.24E+02	6.1%
Co	MS	<2.0E-03	--	3.20E+00	2.1%	5.75E+00	5.8%
Cr	ES	5.35E+01	2.9%	1.83E+02	4.4%	3.12E+02	5.8%
Cu	ES	<1.7E+00	--	<5.2E+01	--	<1.0E+02	--
Eu	MS	<2.0E-03	--	2.11E+00	2.7%	3.97E+00	5.6%
Fe	ES	<1.3E+00	--	7.22E+02	4.4%	1.27E+03	5.9%
Gd	ES	<5.3E-01	--	8.08E+01	4.5%	1.46E+02	5.9%
	MS	<5.0E-03	--	8.37E+01	3.4%	1.48E+02	5.4%
Hg	DMA	3.42E+02	1.1%	2.60E+02	2.3%	7.89E+01	6.5%
K	ES	<5.5E+01	--	<1.4E+02	--	<4.7E+02	--
La	ES	<3.6E-01	--	3.95E+01	4.6%	7.12E+01	5.9%
	MS	<1.0E-03	--	4.20E+01	3.0%	7.72E+01	5.9%
Li	ES	<2.0E+00	--	<3.7E+01	--	<5.1E+02	--
Mg	ES	<3.2E-01	--	3.49E+02	5.0%	5.91E+02	6.0%
Mn	ES	<8.1E-01	--	4.77E+03	4.3%	8.28E+03	5.9%
Mo	ES	<4.1E+00	--	<7.2E+00	--	1.14E+01	5.8%
Na	ES	2.37E+04	4.2%	3.11E+04	3.5%	5.31E+04	6.3%
Nd	MS	<6.0E-03	--	1.47E+02	3.0%	2.65E+02	5.1%
Ni	ES	<3.8E+00	--	3.42E+02	4.4%	5.61E+02	5.7%
P	ES	<6.9E+00	--	<1.1E+01	--	<7.1E+01	--
Pb	MS	<3.0E-03	--	<6.6E-02	--	7.93E-02	17%
Pd	MS	7.06E-02	3.8%	n.d.	--	<1.9E-02	--
Pr	MS	<1.0E-03	--	3.63E+01	2.6%	6.73E+01	5.8%
Pu	multi	n.d.	n.d.	7.93E+00	n.d.	1.65E+01	n.d.
Rh	MS	4.46E-02	1.3%	9.69E+00	2.6%	1.74E+01	4.9%
Ru	MS	1.03E-01	1.1%	4.18E+01	2.7%	7.20E+01	6.2%
S	ES	6.38E+02	3.2%	6.44E+02	4.7%	1.15E+03	5.9%
Sb	MS	<2.0E-03	--	1.83E-01	4.9%	3.81E-01	5.0%
Si	ES	<1.0E+01	--	1.19E+02	6.0%	<3.3E+02	--
Sn	MS	1.71E-01	0.7%	7.13E+01	2.3%	1.17E+02	5.0%
Sr	ES	<6.9E-01	--	2.16E+01	4.7%	3.71E+01	6.1%
Ta	MS	<2.3E-03	--	4.34E-02	7.5%	6.84E-02	5.9%
Tb	MS	<1.0E-03	--	1.89E-01	3.7%	4.18E-01	5.1%
Tc	MS	6.90E-01	0.8%	1.60E+00	2.5%	3.54E+00	5.7%
Th	ES	<3.2E+00	--	3.62E+02	4.6%	7.71E+02	5.9%
	MS	<1.0E-02	--	3.61E+02	1.9%	6.91E+02	6.0%
Ti	ES	<1.4E+00	--	<5.5E+00	--	<1.0E+01	--
U	ES	<6.8E+00	--	4.09E+03	4.6%	7.05E+03	5.9%
	MS	4.48E+00	0.6%	4.30E+03	1.6%	7.51E+03	6.4%
V	ES	<1.4E+00	--	<1.2E+01	--	<7.6E+00	--
Zn	ES	≤1.7E+00	--	<8.1E+00	--	<2.3E+01	--
Zr	ES	<2.1E-01	--	<7.0E+01	--	1.29E+02	6.0%

Table 3-38. Select Radioisotopes in the SRAT Receipt Supernatant and the SRAT and SME Product Filtrate, n=4

Analyte	Method	SRAT Receipt Supernate				SRAT Product Filtrate				SME Product Filtrate			
		Average		σ	RSD	Average		σ	RSD	Average		σ	RSD
		mg/L	Ci/gal			mg/L	Ci/gal			mg/L	Ci/gal		
^{99}Tc	MS	6.90E-01	4.43E-05	20%	0.8%	1.60E+00	0.0%	20%	2.5%	3.54E+00	2.27E-04	20%	5.7%
^{232}Th	MS	<1.0E-02	<4.2E-12	--	--	3.61E+02	1.50E-07	20%	1.9%	6.91E+02	2.87E-07	20%	6.0%
^{233}U	MS	3.36E-03	1.23E-07	20%	2.1%	5.81E-01	2.13E-05	20%	2.7%	1.07E+00	3.93E-05	20%	5.9%
^{234}U	MS	4.84E-03	1.14E-07	20%	4.1%	1.02E+00	2.42E-05	20%	8.4%	1.91E+00	4.52E-05	20%	3.3%
^{235}U	MS	4.02E-01	3.28E-09	20%	1.0%	5.50E+01	4.50E-07	10%	3.2%	9.56E+01	7.82E-07	20%	6.3%
^{236}U	MS	1.78E-02	4.35E-09	20%	1.7%	3.84E+00	9.41E-07	20%	2.8%	7.69E+00	1.88E-06	20%	6.2%
^{237}Np	MS	<1.0E-03	<2.7E-09	--	--	1.10E+00	2.93E-06	20%	2.9%	2.01E+00	5.37E-06	20%	5.9%
^{238}U	MS	4.05E+00	5.15E-09	20%	0.6%	4.24E+03	5.40E-06	20%	1.6%	7.41E+03	9.42E-06	20%	6.4%
^{238}Pu	RC	n.m.	n.m.	--	--	1.33E-01	8.65E-03	7%	25%	2.67E-01	1.73E-02	7%	5.4%
^{239}Pu	MS	<8.0E-03	<1.9E-06	--	--	7.24E+00	1.70E-03	20%	2.4%	1.51E+01	3.56E-03	20%	5.9%
^{240}Pu	MS	<2.0E-03	<1.7E-06	--	--	5.48E-01	4.72E-04	20%	2.9%	1.08E+00	9.31E-04	20%	5.9%
^{241}Pu	RC	n.m.	n.m.	--	--	1.45E-02	5.64E-03	15%	15%	2.45E-02	9.56E-03	16%	13%
^{241}Am	RC	n.m.	n.m.	--	--	1.83E-01	2.38E-03	5%	1.1%	5.33E-01	6.93E-03	5%	6%
^{242m}Am	RC	n.m.	n.m.	--	--	4.88E-05	1.80E-06	62%	50%	1.18E-04	4.35E-06	13%	23%
^{243}Am	RC	n.m.	n.m.	--	--	1.46E-02	1.10E-05	14%	12%	4.79E-02	3.62E-05	5%	7%
^{242}Cm	RC	n.m.	n.m.	--	--	1.19E-07	1.49E-06	62%	50%	2.88E-07	3.60E-06	13%	23%
^{244}Cm	RC	n.m.	n.m.	--	--	2.04E-03	6.25E-04	15%	2.5%	5.20E-03	1.59E-03	15%	8%
mass 241	MS	<1.0E-03	--	--	--	2.62E-01	--	20%	2.1%	5.20E-01	--	20%	5.8%
mass 242	MS	<1.0E-03	--	--	--	3.93E-02	--	20%	5.2%	7.80E-02	--	20%	7.2%

Table 3-39. Anions in the SRAT and SME Product Filtrate, n=4, σ nominally 10%

Analyte	Analytical Method	SRAT Product Filtrate		SME Product Filtrate	
		Average (mol/L)	RSD	Average (mol/L)	RSD
NO_3^-	ICA	1.03E+00	2.9%	1.78E+00	1.6%
NO_2^-	ICA	<2.9E-03	--	<1.2E-02	--
$C_2H_3O_3^-$	ICA-G	7.29E-01	2.2%	1.17E+00	2.4%
CHO_2^-	ICA	7.30E-03	3.4%	2.33E-02	1.0%
$C_2O_4^{2-}$	ICA	2.73E-02	2.3%	4.91E-02	1.7%
SO_4^{2-}	ICA	2.23E-02	2.7%	3.47E-02	1.7%
PO_4^{3-}	ICA	<1.4E-03	--	<6.0E-03	--
Cl^-	ICA	<3.8E-03	--	<1.6E-02	--
F^-	ICA	<7.1E-03	--	<3.0E-02	--
Br^-	ICA	<8.4E-03	--	<7.1E-03	--

Apart from the addition of frit in the SME, the SME Product appears more concentrated than the SRAT Product due to additional dewater, and the degree of concentration can be estimated by comparing the concentrations of anions that are expected to be nearly fully soluble in the SRAT and SME Products. Based on the nitrate, glycolate, and sulfate analysis in Table 3-39, the SME Product filtrate appears approximately 60% to 70% more concentrated on a mass basis than the SRAT Product slurry. Caustic quench preparation was not used for supernate analysis, as it is primarily recommended for slurry analysis.

The target waste loading for this test was 36%. Waste loading can be estimated by comparing the concentration of a component that is not contained in the frit before and after frit addition. To do this, we compare the concentration in the SRAT product as wt% of CS to the concentration in the SME Product as wt% of CS. Typically, waste loading is calculated based on iron concentration. Based on SRAT Product iron of 11.0 wt% of CS and SME Product iron of 3.74 wt% of CS, the waste loading is calculated as 33.9%. Table 3-40 contains the waste loading calculations based on other applicable SRAT Product components present at greater than or equal to 0.1 wt% of CS as quantified by ICP-ES. Calculated waste loading values for the 8 applicable components ranged from 33.6% (Mn basis) to 37.1% (Ca basis) and averaged 34.4%. Calcium was excluded from this analysis due to its relatively high RSD in the SME Product. Based on this ensemble of values, the SB10 qualification test SME Product was approximately 34%, which is approximately 2 percentage points lower than the waste loading target of 36%.

Table 3-40. Waste Loading Estimated from ICP-ES Analysis

Analyte	SRAT Product wt% of CS	SME Product wt% of CS	Waste Loading
Fe	1.10E+01	3.74E+00	33.9%
Mg	2.66E-01	9.67E-02	36.3%
Mn	3.69E+00	1.24E+00	33.6%
Ni	3.60E-01	1.25E-01	34.8%
S	5.15E-01	1.75E-01	34.0%
Th	2.23E+00	7.61E-01	34.0%
U	2.88E+00	9.89E-01	34.3%
Average	--	--	34.4%

Table 3-41 contains a summary of the anion conversions during the SB10 qualification test, as calculated from the SRAT and SME Product slurry analyses. The acid calculation used the recommended inputs from the simulant testing, which are shown in Table 3-41 in the column labelled “Projected”.[44] Nitrite was destroyed to below the detection limit during the SRAT cycle. Thus, nitrite destruction and nitrite-to-nitrate conversion are not applicable (N/A) to the SME cycle. Nitrite-to-nitrate conversion in the SRAT was higher than projected (69% vs. 17.8%) and glycolate destruction across the SRAT and SME was lower than projected (9.0% and 5.5% vs. 24.6%). The net impact was that both the nitrate and glycolate concentrations were increased in the SME Product versus predicted levels, but the impacts on predicted SME Product REDOX are in the opposite direction and are roughly cancelled out. Glycolate-to-formate and glycolate-to-oxalate conversions were generally consistent with expectations. Nitrate conversion, a metric that is only applicable to the SME Product, showed neither an increase nor decrease in nitrate in the SME Product from that in the SRAT Product.

Table 3-41. Anion Conversions for the SB10 Qualification SRAT and SME Cycles

Anion Conversions	Projected[44]	SRAT Result	SME Result
Nitrite Destruction	100%	>98.7%	N/A
Nitrite-to-Nitrate Conversion	17.8%	69%	N/A
Nitrate Conversion	N/A	N/A	0.0%
Glycolate Destruction	24.6%	9.0%	5.5%
Glycolate-to-Formate Conversion	1.6%	>1.3%	0.7%
Glycolate-to-Oxalate Conversion	1.5%	2.8%	0.5%

Several factors in the way that the SC-19 qualification test was performed may have caused the differences between the anion conversions noted in this testing and the anion conversions projected from SB10 simulant testing. Amongst these factors are the additional reflux period immediately after acid addition, the lower acid addition rate, and the timing of the SRAT and SME cycles.

A possible factor contributing to the anion conversions differing from those projected from simulant testing is the addition of a reflux period immediately after acid addition and for the first 4.1 hours of boiling. In most testing of the NGA flowsheet, dewater was initiated immediately after acid addition and for the first period boiling. During a dewater period, condensate would be collected in the Slurry Mix Evaporator Condensate Tank (SMECT), while during a reflux period, the condensate would be recycled back to the SRAT. Since nitrate in condensate is thought to be highest during early boiling, adding an early reflux cycle could lead to higher nitrate in the SRAT Product (and thus higher apparent nitrite-to-nitrate conversion). Supporting this factor, and as seen in Section 3.2.6, the SRAT dewater condensate had a nitrate concentration that was atypically low when compared with typical tests where SRAT dewater occurs immediately after acid addition.

Another possible factor contributing to the anion conversions differing from those projected from simulant testing was the lower acid addition rate used in SRAT Cycle Part 2. As part of the potential mitigation for the foaming that caused the suspension of SRAT Cycle Part 1, the target acid addition rate was cut in half, reduced from a scaled 4.5 gpm to 2.25 gpm for nitric acid addition and from a scaled 4 gpm to 2 gpm for glycolic acid addition.[6] Additionally, due to drift in the acid pump calibration, the glycolic acid addition rate for SRAT Cycle Part 2 were likely lower than 2 gpm. The lowering of the acid addition rate, specifically the rate for glycolic acid addition, likely led to slower NO_x production and caused the observation noted in Section 3.2.4.1 that the off-gas did not approach anoxic conditions. This influences the off-gas chemistry species and ultimately may impact the nitrite-to-nitrate conversion. This observation may provide helpful information for the transition from the NFA flowsheet to the NGA flowsheet, where initial batches may use acid addition rates as low as the NFA flowsheet reducing acid addition rate of 2 gpm. Most testing during development of the NGA flowsheet used a reducing scaled acid addition rate of 4 gpm.

Additionally, the relatively long period between the SRAT and the SME cycles for the qualification run, when compared to typical facility timing and simulant test timing, may have contributed to the observation of glycolate destruction, glycolate-to-formate conversion, and glycolate-to-oxalate conversion during the SME cycle (which is based on the difference from the SRAT Product sample taken immediately at the completion of the SRAT cycle). It is feasible that the three week interruption in the nitric acid addition (between SRAT Cycle Part 1 and SRAT Cycle Part 2) might have impacted the nitrite to nitrate conversion, but the interruption occurred at a point in the process prior to when significant nitrate destruction is expected.

For the NGA flowsheet, which has a higher nitric acid addition than the NFA flowsheet, nitrite-to-nitrate conversion is expected to have a higher variability.

3.2.6 Condensate Analytical Results

Table 3-42 contains results for ion chromatography, mercury speciation, VOA, and semivolatile organics analysis (SVOA) analysis for the condensates during the SRAT and SME cycles. The SRAT condensate had a relatively small volume (as this was not coupled processing) and was taken near the start of boiling (just after the initial wash period to remediate the foamover). Because the SME condensate had a much larger volume and was collected in several bottles, a composite sample was created for analysis by subsampling the individual bottles of condensate for each of the decontamination water addition and frit water addition boiling periods. Note that no special effort was taken to representatively subsample insoluble mercury (primarily immiscible elemental mercury).

The SRAT condensate was identified to contain 99.0 mg/L nitrate, 32.2 mg/L glycolate, and 3.43 mg/L of total mercury. The SME condensate was identified to contain 152 mg/L nitrate, 60.5 mg/L glycolate, 15.7 mg/L formate, and 36.3 mg/L of total mercury. Glycolate concentrations in SRAT and SME condensates were similar to the concentrations in the comparable condensates from the SB10 simulant flowsheet tests (>10 mg/L to 90.5 mg/L in SRAT and SME condensates, averaging 44 mg/L in SRAT condensate) and the SC-18 SB9 NGA qualification test (>10 mg/L to 17.7 mg/L in the SRAT and SME dewater condensates).[48, 53] Even though formate concentration is lower in the SRAT and SME slurries for the NGA flowsheet compared with the NFA flowsheet, formate is often noted in the NGA condensates (<10 mg/L to 37 mg/L in the SB10 simulant flowsheet test condensates, 266 mg/L in the SC-18 SRAT dewater condensate, and <10 mg/L in the SC-18 SME dewater condensates).[48, 53]

Elemental mercury and methylmercury were detected in both the SRAT and SME condensates. The SME condensate also contained ionic (inorganic) mercury. No detectable ethyl mercury or dimethyl mercury was found in either condensate. The calculated mercury species fraction of total for the SRAT condensate is low at only 18%. However, there is a relatively low concentration of total mercury of 3.4 mg/L. The calculated mercury species fraction of total for the SME condensate was higher at 47%, considering the three detectable Hg species vs. the total mercury concentration. There is likely considerable uncertainty in this mercury species fraction calculation due to the relatively low precision (high RSD) reported for the quadruplicate ionic mercury and elemental mercury analyses.

Table 3-42. SRAT and SME Condensate Analysis , $n = 4$

Analyte	Method	SRAT Condensate		SME Condensate	
		Average (mg/L)	RSD	Average (mg/L)	RSD
F^-	ICA	<1.0E+01	--	<1.0E+01	--
CHO_2^-	ICA	<1.0E+01	--	1.57E+01	1.2%
Cl^-	ICA	<1.0E+01	--	<1.0E+01	--
NO_2^-	ICA	<1.0E+01	--	<1.0E+01	--
NO_3^-	ICA	9.90E+01	0.5%	1.52E+02	0.6%
PO_4^{3-}	ICA	<1.0E+01	--	<1.0E+01	--
SO_4^{2-}	ICA	<1.0E+01	--	<1.0E+01	--
$C_2O_4^{2-}$	ICA	<1.0E+01	--	<1.0E+01	--
Br^-	ICA	<5.0E+01	--	<5.0E+01	--
$C_2H_3O_3^-$	ICA-G	3.22E+01	0.8%	6.05E+01	0.3%
NH_4^+	ICC	<1.0E+01	--	<1.0E+01	--
HMDSO	VOA	<2.5E-01	--	<2.5E-01	--
TMSOH	SVOA	<1.0E+00	--	<1.0E+00	--
Propanal	VOA	<2.5E-01	--	<2.5E-01	--
Other VOA	VOA	<2.5E-01	--	<2.5E-01	--
Other SVOA	SVOA	<1.0E+00	--	<1.0E+00	--
Hg	DMA	3.43E+00	13%	3.63E+01	1.6%
CH_3Hg^+	CVAFS	1.36E-01	1.9%	8.36E+00	15%
$Hg^{+/2+}$	CVAFS	<1.0E-02	--	6.77E+00	87%
Hg^0	CVAFS	4.90E-01	33%	2.59E+00	92%

3.2.7 Mercury Speciation Results

Total mercury and other mercury species measured in the supernates of the SRAT Receipt slurry, the SRAT Product slurry and the SME Product slurry were measured from diluted supernates from these three slurries. Table 3-43 shows the various mercury species average concentrations and the 1-sigma standard deviations and calculated percent relative standard deviation. Total mercury values decreased from the SRAT Receipt supernate (341 mg/L) to the SRAT Product supernate (260 mg/L). Total mercury concentration in the SME Product (79 mg/L) was below 100 mg/L. No methylmercury was present in the SRAT Receipt supernate, but was present at ~62 mg/L in the SRAT Product supernate and at only ~2 mg/L in the SME Product supernate. One explanation for the lower methylmercury observed in the SME Product supernate versus the SRAT Product supernate is that there is more steam stripping in the SME. Methylmercury was analyzed at ~8.4 mg/L in the SME condensate vs. Only ~0.14 mg/L in the SRAT condensate. Another explanation for observed lower methylmercury in the SME Product supernate versus the SRAT Product supernate is the potential formation of methane seen in the SME cycle versus the SRAT cycle (discussed below). No ethyl mercury or dimethyl mercury was detected in any of the supernate samples. For both ionic mercury and elemental mercury, the starting concentrations were highest in the starting SRAT Receipt supernate and decreased in concentration through the SRAT Product supernate and then the SME Product supernate. The percent recovery of calculated total mercury shown in the last column, which is the sum of the mercury species divided by the total soluble mercury and reported as a percentage, indicates that good recovery is demonstrated for both the SRAT Receipt and SRAT Product. Higher precision was achieved in the replicate analysis of the SRAT Receipt supernate for ionic and elemental mercury versus the SRAT Product supernate. All of the RSD values for measured total mercury for all three samples show good precision in the range of 1.1% RSD to 6.5% RSD. Species fraction of total mercury for the SME Product supernate is lower than SRAT Receipt and SRAT Product supernates, with total recovery only at ~50% of measured total mercury.

Table 3-43. Total Mercury and Mercury Species for SRAT Receipt, SRAT Product and SME Product Supernatants, *n*=4

		Total Hg (mg/L)	Methyl Hg (mg/L)	Ethyl Hg (mg/L)	Ionic Hg ($Hg^{+/2+}$) (mg/L)	Purgeable Hg (mg/L)	Dimethyl Hg (mg/L)	Percent Recovery for Calculated Total Hg
SRAT Receipt Supernate	Avg.	341.5	<1.0E+00	<1.0E+00	204.5	132.8	<2.0E+00	99%-100%
	RSD	1.1%	--	--	2.8%	8.6%	--	--
SRAT Product Supernate	Avg.	260.3	61.8	<1.0E+00	80.6	107.8	<2.4E+00	94%-96%
	RSD	2.3%	2.1%	--	27%	49%	--	--
SME Product Supernate	Avg.	78.9	1.6	<1.0E+00	14.6	19.3	<2.5E+00	45%-49%
	RSD	6.5%	6.2%	--	40%	19%	--	--

Mercury data from the radioactive SB10 demonstration can be compared to similar testing with simulants.[48, 54] The simulant studies included total mercury and methylmercury analyses. General conclusions from the simulant studies include 1) observation of methylmercury during the SRAT and SME cycles, 2) likely generation of methylmercury in the SRAT and SME kettles (i.e., not exclusively formed in condensate or off-gas), 3) either consumption of methylmercury in the SRAT or SME, or volatilization of methylmercury allowing it to vaporize and enter condensate/off-gas streams during CPC processing, 4) methylmercury is likely predominately formed at the beginning of the SRAT cycle when mercury is present at the highest concentration. Mercury analyses from the radioactive tests are in general agreement with the above conclusions. Table 3-44 shows comparison of the various supernate and condensate total mercury and methylmercury data for the previous simulant tests and current radioactive testing. Similar to the simulant testing, methylmercury is observed in both the SRAT and SME cycles (product supernates and condensates) for radioactive testing. Since radioactive SRAT Product supernate methylmercury is higher in the SRAT Product supernates compared to the radioactive SME Product supernates, methylmercury is likely generated predominately early in the radioactive SRAT cycle. This conclusion assumes lack of significant destruction of methylmercury in the SME slurry during the SME cycle. One potential evidence of methylmercury destruction during the SME cycle is the observation of higher peak methane results during the radioactive SME than the radioactive SRAT, i.e., assuming the source of methane observed in the radioactive SRAT and SME involves methylmercury as a precursor to methane formation. Radioactive testing shows methylmercury is likely formed in the SRAT kettle (62 mg/L) vs. exclusively in the off-gas chemistry (0.14 mg/L).

Some differences are observed between the simulant and radioactive testing. The ratio of methylmercury for simulant SRAT condensate to simulant SRAT Product supernate is higher in simulant testing than rad testing. For instance, in every simulant SRAT cycle, the condensate methyl mercury concentration is higher than in the respective SRAT product supernate. However, in the radioactive testing the SRAT condensate methylmercury (0.14 mg/L) is at least an order of magnitude lower than in the SRAT Product supernate (62 mg/L). Another difference is observed in the much higher concentrations of both total mercury (260 mg/L) and methylmercury (62 mg/L) in the radioactive SRAT Product supernates vs. the respective total mercury and methylmercury values in the simulant SRAT Product supernates. This is also evident in the higher concentration of total mercury (79 mg/L) in the radioactive SME Product supernates vs. the simulant (maximum 19 mg/L). Methylmercury concentrations were similar between radioactive and simulant SME Product supernates. Lastly, it should be noted that observation of methylmercury in the SRAT and SME cycle product supernates and condensates for both recent simulant and current radioactive sample testing indicates that, like previous testing at DWPF for the NFA flowsheet and prior antifoams,[55] methylmercury is being formed in the NGA flowsheet with the new Momentive™ Y-17112 antifoam.

Table 3-44. Comparison of Total Mercury and Methylmercury Concentrations in the SRAT and SME for Simulant Testing and Radioactive Testing

	Product Supernate		Condensate(Rad.) or Dewater(Sim.)	
	Total Hg (mg/L)	Methylmercury (mg/L)	Total Hg (mg/L)	Methylmercury (mg/L)
SRAT				
Simulant	<1 - 13[a]	0.3 - 3.3[b]	64 - 658[c]	1 - 25[b]
Radioactive	260 ± 6	62 ± 1	3.4 ± 0.5	0.14 ± 0.003
SME				
Simulant	<1 - 19[d]	0.09 - 1.44[b]	0.7 - 15.5[e]	0.02 - 1.78[b]
Radioactive	79 ± 5	1.6 ± 1	36 ± 1	8.4 ± 1.3

a) Tables 3-10 & 3-11 from SRNL-STI-2021-00349
b) Table 3-3 from SRNL-STI-2022-00079
c) Tables 3-27 & 3-28 from SRNL-STI-2021-00349; (Avg. 376 mg/L +/- 212 mg/L)
d) Table 3-12 from SRNL-STI-2021-00349
e) Table 3-33 from SRNL-STI-2021-00349

3.2.8 Solubility Comparison

The waste slurries processed in DWPF are mixtures of insoluble solid particles and a soluble aqueous phase. The partitioning of components between solid and liquid phases of the SRAT Receipt, SRAT Product, and SME Product are reflected by the results in Table 3-45 through Table 3-47. The columns labelled “Total” contain the data for the slurry analysis on a slurry basis. The columns labelled “Soluble” contain the data for the supernate or filtrate converted to a slurry basis. The columns labelled “Insoluble” contain the difference between the total and soluble data on a slurry basis, providing an estimate of the concentration in the solid phase on a slurry basis. The percent soluble is calculated as the soluble concentration divided by the total concentration, multiplied by 100%. For components that are almost completely soluble, the measured soluble concentration occasionally exceeds the total concentration due to analytical uncertainty, which leads to negative calculated concentrations in the solid phase (shown as insoluble concentrations of “0” wt% slurry in Table 3-45 through Table 3-47). For those cases, the calculated percent soluble, or partitioning into the liquid phase, is calculated to be >100%. Both a negative insoluble concentration and a percent soluble of >100% are physical impossibilities.

Table 3-45. Total, Insoluble, and Soluble Concentrations and Percent Soluble for SRAT Receipt and SRAT and SME Products; 1 of 3

Analyte	Method	SRAT Receipt				SRAT Product				SME Product			
		Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)
Ag	MS	1.05E-03	1.05E-03	≤1.7E-06	0.2%	9.60E-04	8.91E-04	6.92E-05	7.2%	9.24E-04	9.02E-04	2.18E-05	2.4%
Al	ES	1.86E+00	1.76E+00	1.05E-01	5.6%	1.80E+00	1.71E+00	8.58E-02	4.8%	2.26E+00	2.15E+00	1.09E-01	4.9%
B	ES	<1.2E-02	--	<1.8E-03	--	<1.1E-03	--	<6.9E-04	--	5.67E-01	5.59E-01	7.92E-03	1.4%
Ba	ES	6.89E-03	≥6.9E-03	<1.3E-05	<0.2%	7.20E-03	≥7.1E-03	<1.3E-04	<1.8%	9.41E-03	≥9.3E-03	<1.4E-04	<1.5%
Ca	ES	7.95E-02	≥7.9E-02	<3.8E-04	<0.5%	1.23E-01	4.72E-02	7.60E-02	62%	1.52E-01	1.02E-01	5.04E-02	33%
Ce	MS	1.81E-02	≥1.8E-02	<1.7E-07	<0.001%	1.83E-02	8.2E-03	1.00E-02	55%	2.01E-02	7.0E-03	1.31E-02	65%
Co	MS	3.77E-04	≥3.8E-04	<1.7E-07	<0.05%	3.87E-04	1.08E-04	2.79E-04	72%	4.39E-04	1.03E-04	3.36E-04	77%
Cr	ES	2.41E-02	1.95E-02	4.64E-03	19%	2.31E-02	7.2E-03	1.59E-02	69%	2.81E-02	9.9E-03	1.82E-02	65%
^{133}Cs	MS	1.46E-04	8.14E-05	6.41E-05	44%	1.50E-04	3.1E-05	1.19E-04	79%	1.60E-04	3.4E-05	1.26E-04	79%
Cu	ES	5.28E-03	≥5.1E-03	<1.5E-04	<2.8%	<6.8E-03	--	<4.5E-03	--	<7.9E-03	--	<5.9E-03	--
Eu	MS	2.76E-04	≥2.8E-04	<1.7E-07	<0.1%	2.76E-04	9.2E-05	1.84E-04	67%	2.99E-04	6.8E-05	2.32E-04	77%
Fe	ES	1.28E+00	≥1.3E+00	<1.2E-04	<0.01%	1.25E+00	1.19E+00	6.28E-02	5.0%	1.41E+00	1.34E+00	7.40E-02	5.2%
Gd	ES	6.66E-03	≥6.6E-03	<4.6E-05	<0.7%	7.37E-03	3.4E-04	7.03E-03	95%	8.66E-03	1.3E-04	8.53E-03	99%
	MS	7.41E-03	≥7.4E-03	<4.3E-07	<0.006%	7.52E-03	2.3E-04	7.29E-03	97%	8.22E-03	0	8.64E-03	105%
Hg	DMA	4.27E-01	3.97E-01	2.96E-02	6.9%	1.54E-01	1.32E-01	2.27E-02	15%	7.52E-02	7.06E-02	4.61E-03	6.1%
La	ES	5.07E-03	≥5.0E-03	<3.1E-05	<0.6%	5.29E-03	1.85E-03	3.44E-03	65%	5.99E-03	1.83E-03	4.16E-03	69%
	MS	5.79E-03	≥5.8E-03	<8.7E-08	<0.001%	6.12E-03	2.46E-03	3.66E-03	60%	6.33E-03	1.82E-03	4.51E-03	71%
Li	ES	<9.4E-04	--	<1.7E-04	--	<5.5E-03	--	<3.2E-03	--	9.37E-01	≥9.1E-01	<3.0E-02	<3.2%
Mg	ES	3.13E-02	≥3.1E-02	<2.8E-05	<0.1%	3.02E-02	0	3.04E-02	101%	3.65E-02	1.9E-03	3.45E-02	95%
Mn	ES	3.99E-01	≥4.0E-01	<7.0E-05	<0.02%	4.18E-01	3E-03	4.15E-01	99%	4.68E-01	0	4.84E-01	103%
Na	ES	2.49E+00	4.4E-01	2.05E+00	82%	2.57E+00	0	2.71E+00	106%	2.93E+00	0	3.10E+00	106%
Nd	MS	1.90E-02	≥1.9E-02	<5.2E-07	<0.003%	1.93E-02	6.5E-03	1.28E-02	66%	2.07E-02	5.2E-03	1.55E-02	75%

Table 3-46. Total, Insoluble, and Soluble Concentrations and Percent Soluble for SRAT Receipt and SRAT and SME Products; 2 of 3

Analyte	Method	SRAT Receipt				SRAT Product				SME Product			
		Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)
Ni	ES	4.11E-02	≥4.1E-02	<3.3E-04	<0.8%	4.08E-02	1.11E-02	2.97E-02	73%	4.73E-02	1.45E-02	3.28E-02	69%
P	ES	<1.4E-02	--	<6.0E-04	--	1.02E-02	≥9.2E-03	<9.9E-04	<10%	1.21E-02	≥7.9E-03	<4.2E-03	<34%
Pb	MS	1.40E-03	--	<2.6E-07	0.0002	1.33E-03	--	<5.8E-06	<0.4%	1.57E-03	1.56E-03	4.64E-06	0.3%
Pd	MS	2.17E-04	2.11E-04	6.11E-06	2.8%	2.42E-04	--	n.d.	--	2.66E-04	≥2.7E-04	<1.1E-06	<0.4%
Pr	MS	4.92E-03	≥4.9E-03	<8.7E-08	<0.002%	5.19E-03	2.03E-03	3.16E-03	61%	5.39E-03	1.46E-03	3.93E-03	73%
Pu	multi	2.40E-03	--	n.d.	--	2.22E-03	1.53E-03	6.91E-04	31%	2.55E-03	1.58E-03	9.63E-04	38%
Rh	MS	1.31E-03	1.30E-03	3.86E-06	0.3%	1.28E-03	4.36E-04	8.44E-04	66%	1.44E-03	4.2E-04	1.01E-03	71%
Ru	MS	6.35E-03	6.34E-03	8.94E-06	0.1%	6.36E-03	2.72E-03	3.63E-03	57%	7.05E-03	2.84E-03	4.21E-03	60%
S	ES	5.68E-02	1.5E-03	5.53E-02	97%	5.83E-02	2.3E-03	5.60E-02	96%	6.60E-02	0	6.71E-02	102%
Sb	MS	4.68E-05	≥4.7E-05	<1.7E-07	<0.4%	4.20E-05	2.61E-05	1.60E-05	38%	4.53E-05	2.30E-05	2.23E-05	49%
Si	ES	6.36E-02	≥6.3E-02	<8.7E-04	<1.4%	6.80E-02	5.77E-02	1.04E-02	15%	8.46E+00	≥8.4E+00	<1.9E-02	<0.2%
Sn	MS	7.58E-03	7.57E-03	1.48E-05	0.2%	8.18E-03	1.98E-03	6.20E-03	76%	8.97E-03	2.13E-03	6.84E-03	76%
Sr	ES	2.44E-03	≥2.4E-03	<6.0E-05	<2.4%	3.36E-03	1.48E-03	1.88E-03	56%	3.65E-03	1.48E-03	2.17E-03	59%
^{88}Sr	MS	2.34E-03	2.34E-03	6.66E-07	0.03%	2.33E-03	9.5E-04	1.37E-03	59%	2.63E-03	9.0E-04	1.74E-03	66%
Ta	MS	1.88E-05	≥1.9E-05	<2.0E-07	<1.1%	<1.3E-05	--	3.78E-06	>29%	<1.0E-05	--	4.00E-06	>40%
Tb	MS	2.48E-05	≥2.5E-05	<8.7E-08	<0.3%	2.71E-05	1.06E-05	1.65E-05	61%	3.03E-05	5.9E-06	2.44E-05	81%
Th	ES	2.60E-01	≥2.6E-01	<2.8E-04	<0.1%	2.53E-01	2.22E-01	3.15E-02	12%	2.87E-01	2.42E-01	4.50E-02	16%
Ti	ES	<2.1E-03	--	<1.2E-04	--	2.09E-03	≥1.6E-03	<4.8E-04	<23%	<2.7E-03	--	<6.1E-04	--
U	ES	3.33E-01	≥3.3E-01	<5.9E-04	<0.2%	3.26E-01	0	3.56E-01	109%	3.73E-01	0	4.12E-01	110%
	MS	3.69E-01	3.68E-01	3.88E-04	0.1%	3.81E-01	6E-03	3.75E-01	98%	3.60E-01	0	4.39E-01	122%
Zn	ES	2.62E-03	≥2.5E-03	<1.5E-04	<5.6%	2.64E-03	≥1.9E-03	<7.0E-04	<27%	3.13E-03	≥1.8E-03	<1.4E-03	<43%
Zr	ES	1.69E-02	≥1.7E-02	<1.8E-05	<0.1%	1.71E-02	≥1.1E-02	<6.1E-03	<36%	1.91E-02	1.15E-02	7.55E-03	40%

Table 3-47. Total, Insoluble, and Soluble Concentrations and Percent Soluble for SRAT Receipt and SRAT and SME Products; 3 of 3

Analyte	Method	SRAT Receipt				SRAT Product				SME Product			
		Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)	Total (wt% slurry)	Insoluble (wt% slurry)	Soluble (wt% slurry)	Soluble (%)
^{99}Tc	MS	2.85E-04	2.25E-04	5.97E-05	21%	2.76E-04	1.37E-04	1.39E-04	50%	3.14E-04	1.07E-04	2.07E-04	66%
^{232}Th	MS	2.62E-01	≥2.6E-01	<8.7E-07	<0.001%	2.72E-01	2.41E-01	3.14E-02	12%	2.61E-01	2.20E-01	4.04E-02	15%
^{233}U	MS	7.66E-05	7.63E-05	2.91E-07	0.4%	7.18E-05	2.12E-05	5.06E-05	70%	8.36E-05	2.09E-05	6.26E-05	75%
^{234}U	MS	8.90E-05	8.85E-05	4.19E-07	0.5%	9.89E-05	9.7E-06	8.92E-05	90%	1.08E-04	0	1.12E-04	103%
^{235}U	MS	4.40E-03	4.36E-03	3.48E-05	0.8%	4.95E-03	1.7E-04	4.78E-03	97%	5.57E-03	0	5.59E-03	100%
^{236}U	MS	2.94E-04	2.92E-04	1.54E-06	0.5%	2.92E-04	0	3.35E-04	115%	3.45E-04	0	4.49E-04	130%
^{237}Np	MS	1.67E-04	≥1.7E-04	<8.7E-08	<0.1%	1.57E-04	6.12E-05	9.58E-05	61%	1.83E-04	6.5E-05	1.18E-04	64%
^{238}U	MS	3.64E-01	3.64E-01	3.51E-04	0.1%	3.75E-01	6E-03	3.69E-01	98%	3.54E-01	0	4.33E-01	122%
^{238}Pu	RC	5.64E-05	n.d.	n.d.	n.d.	5.22E-05	4.05E-05	1.16E-05	22%	6.24E-05	4.67E-05	1.56E-05	25%
^{239}Pu	MS	2.17E-03	≥2.2E-03	<6.9E-07	<0.03%	2.00E-03	1.37E-03	6.30E-04	31%	2.30E-03	1.42E-03	8.83E-04	38%
^{240}Pu	MS	1.73E-04	≥1.7E-04	<1.7E-07	<0.1%	1.58E-04	1.10E-04	4.77E-05	30%	1.81E-04	1.18E-04	6.31E-05	35%
^{241}Pu	RC	4.51E-06	n.d.	n.d.	n.d.	3.59E-06	2.34E-06	1.26E-06	35%	4.49E-06	3.06E-06	1.43E-06	32%
^{241}Am	RC	4.25E-05	n.d.	n.d.	n.d.	4.11E-05	2.51E-05	1.60E-05	39%	5.04E-05	1.92E-05	3.12E-05	62%
^{242m}Am	RC	1.02E-08	n.d.	n.d.	n.d.	8.50E-09	4.25E-09	4.25E-09	50%	9.62E-09	2.70E-09	6.91E-09	72%
^{243}Am	RC	6.03E-06	n.d.	n.d.	n.d.	3.22E-05	3.09E-05	1.27E-06	4.0%	4.49E-06	1.69E-06	2.80E-06	62%
^{242}Cm	RC	2.47E-11	n.d.	n.d.	n.d.	2.06E-11	1.03E-11	1.03E-11	50%	2.34E-11	6.6E-12	1.68E-11	72%
^{244}Cm	RC	2.98E-07	n.d.	n.d.	n.d.	3.72E-07	1.94E-07	1.78E-07	48%	4.38E-07	1.34E-07	3.04E-07	69%
mass 241	MS	4.15E-05	≥4.1E-05	<8.7E-08	<0.2%	3.78E-05	1.50E-05	2.28E-05	60%	4.45E-05	1.41E-05	3.04E-05	68%
mass 242	MS	1.34E-05	≥1.3E-05	<8.7E-08	<0.6%	1.21E-05	8.70E-06	3.42E-06	28%	1.41E-05	9.57E-06	4.55E-06	32%
^{235}U(eq$_{SLU}$)	MS	4.50E-03	4.47E-03	3.52E-05	0.8%	5.05E-03	2.0E-04	4.86E-03	96%	5.68E-03	9E-06	5.67E-03	100%
^{239}Pu(eq)	multi	5.10E-03	n.d.	n.d.	n.d.	5.29E-03	1.50E-03	3.79E-03	72%	6.00E-03	1.43E-03	4.57E-03	76%

Table 3-48. Calculation of Ratios of Poisons to Equivalent Fissile in the SRAT Receipt, SRAT Product, and SME Product

	SRAT Receipt			SRAT Product			SME Product		
	Total	Insoluble	Soluble	Total	Insoluble	Soluble	Total	Insoluble	Soluble
Concentrations (wt% slurry)									
Fe	1.28E+00	1.28E+00	<1.2E-04	1.25E+00	1.19E+00	6.28E-02	1.41E+00	1.34E+00	7.40E-02
Gd (ES)	6.66E-03	≥6.62E-03	<4.6E-05	7.37E-03	3.4E-04	7.03E-03	8.66E-03	1.3E-04	8.53E-03
Gd (MS)	7.41E-03	7.41E-03	<4.3E-07	7.52E-03	2.3E-04	7.29E-03	8.22E-03	0	8.64E-03
Mn	3.99E-01	3.99E-01	<7.0E-05	4.18E-01	3E-03	4.15E-01	4.68E-01	0	4.84E-01
U (ES)	3.33E-01	3.33E-01	<5.9E-04	3.26E-01	0	3.56E-01	3.73E-01	0	4.12E-01
U (MS)	3.69E-01	3.68E-01	3.88E-04	3.81E-01	6E-03	3.75E-01	3.60E-01	0	4.39E-01
^{235}U	4.40E-03	4.36E-03	3.48E-05	4.95E-03	1.7E-04	4.78E-03	5.57E-03	0	5.59E-03
$^{235}U(eq_{SLU})$	4.50E-03	4.47E-03	3.52E-05	5.05E-03	2.0E-04	4.86E-03	5.68E-03	9E-06	5.67E-03
^{239}Pu	2.17E-03	2.17E-03	6.93E-07	2.00E-03	1.37E-03	6.30E-04	2.30E-03	1.42E-03	8.83E-04
$^{239}Pu(eq)$	5.10E-03	5.08E-03	2.29E-05	5.29E-03	1.50E-03	3.79E-03	6.00E-03	1.43E-03	4.57E-03
Concentrations (g/L)									
$^{235}U(eq_{SLU})$	4.96E-02	--	4.06E-04	5.74E-02	--	5.58E-02	8.05E-02	--	9.71E-02
^{239}Pu	2.38E-02	--	<8.0E-06	2.28E-02	--	7.93E-03	3.26E-02	--	1.65E-02
$^{239}Pu(eq)$	5.61E-02	--	2.64E-04	6.01E-02	--	4.35E-02	8.49E-02	--	7.83E-02
^{235}U Enrichment (%) and Contribution to ^{239}Pu(eq) (%)									
$^{235}U/U$	1.2%	1.2%	9.0%	1.3%	--	1.3%	1.5%	--	1.3%
U in $^{239}Pu(eq)$	57%	--	>97%	62%	--	83%	62%	--	81%
Pu in $^{239}Pu(eq)$	43%	--	<3%	38%	--	17%	38%	--	19%
Poison Mass Ratios (poison mass:fissile mass)									
Fe:Gd	190:1	190:1	<2.5:1	170:1	3500:1	8.9:1	160:1	10000:1	10:1
Gd:$^{235}U(eq_{SLU})$	1.5:1	1.5:1	<1.3:1	1.5:1	1.7:1	1.4:1	1.5:1	--	1.5:1
Gd:$^{239}Pu(eq)$	1.3:1	1.3:1	<2.0:1	1.4:1	0.2:1	1.9:1	1.4:1	0.09:1	1.9:1
Gd:^{239}Pu	3.1:1	3.1:1	<67:1	3.7:1	0.2:1	11:1	3.8:1	0.09:1	9.7:1
Mn:$^{235}U(eq_{SLU})$	89:1	89:1	<2.0:1	83:1	15:1	85:1	82:1	--	85:1
Mn:$^{239}Pu(eq)$	78:1	79:1	<3.1:1	79:1	2.0:1	110:1	78:1	--	110:1
Fe:$^{239}Pu(eq)$	250:1	250:1	5.1:1	240:1	790:1	17:1	240:1	940:1	16:1
Fe:^{239}Pu	590:1	590:1	170:1	620:1	860:1	100:1	610:1	940:1	84:1

Table 3-48 contains calculations from the data that may have application to criticality evaluations. The top section of Table 3-48 summarizes results from Table 3-45 and Table 3-46 for fissiles and neutron poisons for the total slurry, the soluble liquid phase, and the particulate solid phase. The second section lists the

^{235}U(eq$_{SLU}$), ^{239}Pu, and ^{239}Pu(eq)[d] in units of g/L of slurry for the total concentration and g/L of supernate for the liquid concentration. Solid concentration results in mass per volume of solids are not calculated because they would require assumptions about the density of the particulates. The third section of Table 3-48 shows the ^{235}U enrichment and demonstrates the contribution of U and Pu to the calculated ^{239}Pu(eq). The bottom section provides various poison mass to fissile mass results.

Uranium, plutonium, iron, manganese, and gadolinium showed relatively low solubilities in the SRAT Receipt. Uranium, manganese, and gadolinium showed increased solubilities in the SRAT and SME Products. High solubilities of these components were expected from recent solubility testing of the NGA flowsheet.[56-57] Uranium measured by ICP-ES appeared to be completely soluble in the SRAT and SME Products, while uranium measured by ICP-MS appeared nearly soluble in the SRAT Product (98%) and completely soluble in the SME Product. Gadolinium was 95% (ICP-ES) and 97% (ICP-MS) soluble in the SRAT Product and 99% (ICP-MS) and fully soluble in the SME Product. Manganese was 99% soluble in the SRAT Product and fully soluble in the SME Product. When uncertainty is considered, the results for uranium, gadolinium, and manganese solubility in the SRAT and SME Products are consistent with full solubility. With the method uncertainties of 10% for ICP-ES and 20% form ICP-MS, it is difficult to determine with precision concentrations of components in the insoluble phase that are apparently >80% soluble, or to determine with precision how close to 100% soluble a component is.

Variations in the measurement of low levels of ^{233}U led to the calculation of ^{235}U(eq$_{SLU}$) in the SRAT Product being slightly less than fully soluble, even when ^{235}U was calculated to be fully soluble. For this reason, poison to ^{235}U(eq$_{SLU}$) ratios cannot be accurately represented for the SME Product and thus are not reported. The apparent in the ^{233}U solubility may have been caused by thorium interference in the ICP-MS measurement.

Plutonium is very insoluble in the SRAT Receipt, with <0.03% soluble based on the detection limit for ^{239}Pu in the SRAT Receipt supernate. Iron also showed very low solubility in the SRAT Receipt, <0.01% soluble. In the SRAT Product and SME Product, plutonium was 31% and 38% soluble, respectively, and iron was 5.0% and 5.2% soluble, respectively. These solubilities are consistent with relatively high stoichiometric acid addition cases in recent solubility studies.[56-57] Those recent solubility studies did not perform a full SRAT and SME cycle and did not heat to boiling, so this information aids in validating those studies.

The ^{235}U enrichment (^{235}U/U) was measured to be 1.2% in the SRAT Receipt, 1.3% in the SRAT Product, and 1.5% in the SME Product. The soluble portions of the SRAT and SME Products had enrichments that measured to be 1.3%. Thus, overall, the enrichment of the material used in the qualification appeared to be approximately 1.3%. An exception where enrichment strayed from the average enrichment was in the SRAT Receipt supernate, with an apparent 9.0% enrichment. Although the 9.0% enrichment of the SRAT Receipt supernate is a relatively high enrichment compared to the bulk sludge (1.2% to 1.5%), it occurs with a relatively low soluble uranium concentration of 4.5 mg/L.

The likely cause for the higher enrichment in the SRAT Receipt supernate when compared with the SRAT Receipt slurry is related to some aspect of the addition of freshly precipitated SRE material. The Tank 51 sample (HTF-51-19-114 and HTF-51-20-15) had an enrichment of 0.57%,[37] while the H Canyon SRE Tanks 16.3 and 16.4 material had an enrichment of 52% and 37%, respectively.[22] The solids in the Tank 51 material had been aging in the Tank Farm for decades while the solids in the SRE material were freshly precipitated. See Section 3.1.2 for a description of the SRE addition. The Tank 51 and SRE material portions may have had different uranium crystal phases, different uranium particle sizes, different equilibrium soluble uranium concentrations, and different degrees of sub- or super-saturation of uranium. Ultimately,

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[d] ^{239}Pu(eq) = ^{239}Pu + ^{241}Pu + ^{244}Cm + 15(^{245}Cm) + 35(^{242m}Am) + 0.65(^{235}U(eq$_{SLU}$)), where all species are on a mass basis.

any of these differences could lead to a higher contribution of enriched uranium from the SRE material to SRAT Receipt supernate uranium. Additionally, the results for the SRAT Receipt supernate ^{235}U enrichment would not be expected to exactly match the Tank Farm SB10 preparation because the specific mixing conditions and addition timing of the SRE during the SRNL SRE precipitation and sludge washing did not exactly match the H Canyon and Tank Farm processes.

A similar increase in SRAT Receipt (i.e., test feed) supernate ^{235}U enrichment was noted in the Gd-related solubility testing with a different H Canyon enriched uranium stream.[56] The analogous measurement (test feed supernate analysis) was not measured during the Mn-related solubility testing with SRE material.[56-57] A Tank 51 sample taken during Tank Farm preparation of SB10 Decant S showed a supernate ^{235}U enrichment of 2.9%,[58] where the expected slurry ^{235}U enrichment is the same as the sludge used in this report (1.2% to 1.5%). This shows that the phenomenon of higher SRAT Receipt supernate enrichment is occurring in actual SB10 washing, but to a lesser extent than occurred during the SRNL SB10 washing.

Also, in the third section of Table 3-48, the contribution of uranium and plutonium to ^{239}Pu(eq) are shown. Because the ^{235}U(eq$_{SLU}$) contributes to ^{239}Pu(eq), approximately 60% of the ^{239}Pu(eq) in the slurry is due to uranium and the remaining 40% is due to plutonium. The remaining Am and Cm components contribute to less than 1% of the ^{239}Pu(eq). Due to solubility differences between uranium and plutonium, the relative contributions of uranium and plutonium to ^{239}Pu(eq) differ in the solid and liquid phases from that in the slurry. Uranium contributed to over 80% of the ^{239}Pu(eq) in the liquid phase SRAT and SME Products and nearly all of the ^{239}Pu(eq) in the liquid phase of the SRAT Receipt. Likewise, although a calculation is not provided, the contribution of plutonium to ^{239}Pu(eq) will be increased in the solid phases.

In the SRAT Receipt, almost all uranium, plutonium, iron, manganese, and gadolinium are in the solid phase (with low or below detectable levels in the liquid phase). Thus, in the bottom section of Table 3-48, the SRAT Receipt total (slurry) and insoluble (solid) poison to fissile mass ratios are nearly identical and the soluble (liquid) ratios cannot be accurately determined. In the SRAT Product and SME Product, almost all uranium, manganese, and gadolinium are in the liquid phase. Thus, in the bottom section of Table 3-48, the insoluble (solid) Mn and Gd to ^{235}U(eq$_{SLU}$) ratios cannot be accurately determined. The increase in Fe:^{239}Pu(eq) and Fe:^{239}Pu in the insoluble (solid) phase over the slurry (total) SRAT and SME Products indicates that iron is a very good poison for the insoluble portion of the SRAT and SME Products.

The total (slurry) Mn:^{235}U(eq$_{SLU}$) was 89:1 in the SRAT Receipt, 83:1 in the SRAT Product, and 82:1 in the SME Product. These slurry values should be the same number value in all three cases and differences can be attributed to analytical uncertainty. As explained earlier, a portion of both the Mn and ^{235}U were added to the Tank 51 sample material to represent the additions from H Canyon after the samples were collected.

3.2.9 Rheology

As described in Section 2.1.3, rheological properties of each sample were measured twice; material was not removed from the instrument between measurements. Properties were determined from a linear fit of the data from the down curves (600 to 50 s^{-1} for the SRAT Receipt and Product and 300 to 50 s^{-1} for the SME Product. The slope of the linear fit is the Bingham Plastic viscosity. The slope in Pa·s is multiplied by 1000 to convert to cP). The y-intercept of the line is the Bingham Plastic yield stress. The SLOPE and INTERCEPT functions of Excel were used to determine slope and intercept. The numerical results are shown in Table 3-49. Flow curves with the line fits shown for reference are presented in Figure 3-18, Figure 3-19, and Figure 3-20.

Table 3-49. SRAT Receipt, SRAT Product, and SME Product Rheology Results

Sample	Bingham Plastic Viscosity (cP)		Bingham Plastic Yield Stress (Pa)	
	Replicate Results	Average	Replicate Results	Average
SRAT Receipt (TS = 15.0 wt%)	8.80	8.0	8.10	8.3
	7.25		8.43	
SRAT Product (TS = 20.1 wt%)	3.83	3.9	0.84	0.83
	3.90		0.83	
SME Product (TS = 48.1 wt%)	9.47	10.0	2.33	2.3
	10.54		2.29	

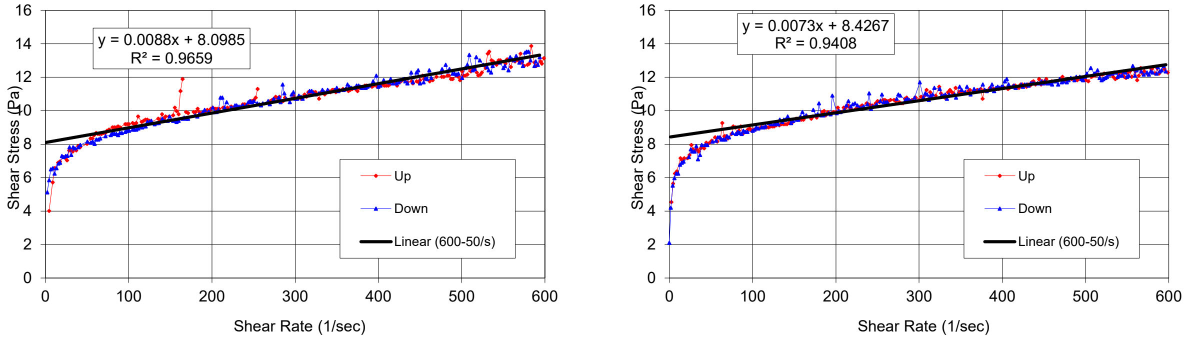

Figure 3-18. Flow Curves SRAT Receipt

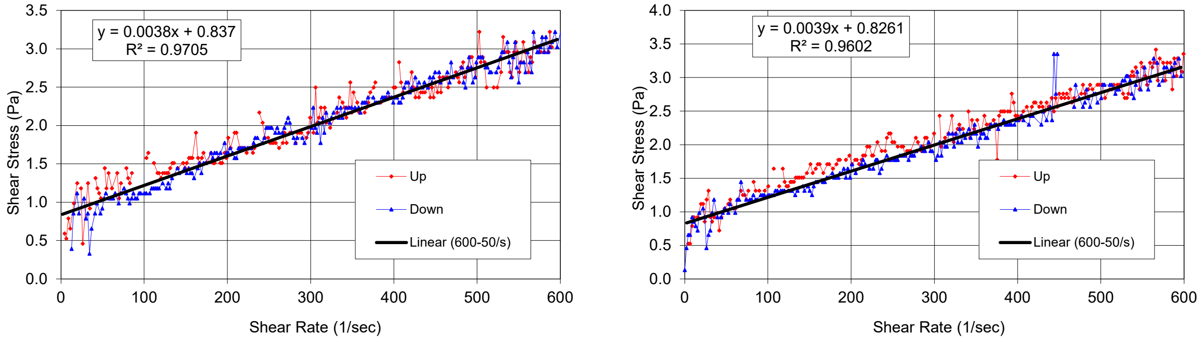

Figure 3-19. Flow Curves SRAT Product

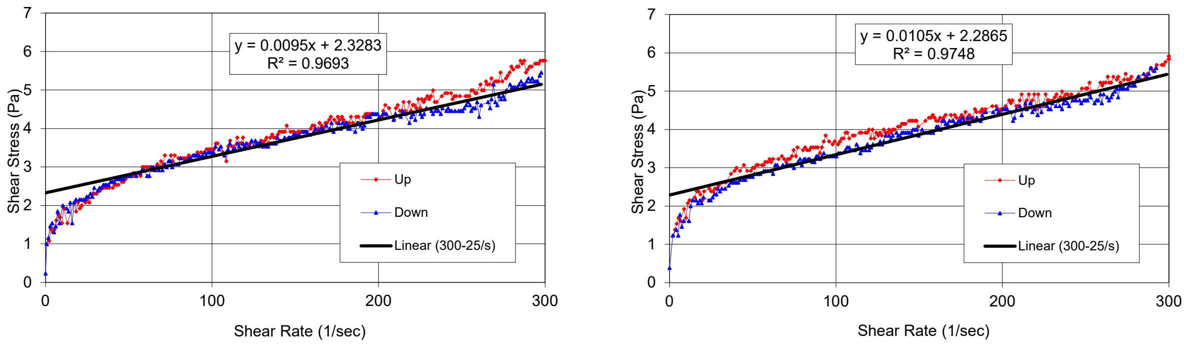

Figure 3-20. Flow Curves SME Product

The SRAT Receipt Bingham-Plastic yield stress averaged 8.3 Pa and viscosity averaged 8.0 cP. The SRAT Product yield stress averaged 0.83 Pa with a viscosity of 3.9 cP. This is below the lower range of the SRAT Product design basis of 1.5 Pa and 5 cP, which would be expected in this case where the TS content of the SRAT Product was below the target. The SME Product yield stress averaged 2.3 Pa with a viscosity of 10.0 cP. This was very close to the lower limit of the SME Product design basis of 2.5 Pa and 10 cP. SB10 qualification SME Product was significantly closer to the DWPF design basis rheology range than the similar NGA qualification for SB9.

3.3 Glass Fabrication and Analysis

3.3.1 Visual Examination

The glass was visually examined from the outside of the shielded cell window. The glass appeared dark and shiny without the presence of any visible salt layers or crystals.

3.3.2 Chemical Composition

A detectable value of Ca was measured for the blank sample submitted with the ICP-ES AR dissolutions. The measured Ca concentration was insignificant (<1% of the measured glass sample values) and does not impact the overall results and conclusions of this study.

3.3.2.1 Multi-Element Standard Solution

Table 3-50 and Table 3-51 provide a comparison of measured and reference concentrations for the multi-element standard solutions measured with the AF glass samples.[28-29] The measured concentrations of each analyte were within 5% of the reference values except for S, which had a 7% difference. These data, along with similar internal standards analyzed by analytical personnel performing the measurements, indicate that the ICP-ES analyses are of sufficient accuracy and within the analytical uncertainty.

Table 3-50. Multi-Element Standard Solution Results (SM-744-013)

Analyte	Measured (mg/L)	Reference[28] (mg/L)	% Difference
Al	3.80	4	-5.0%
B	19.8	20	-1.0%
Fe	4.16	4	4.0%
Li	9.82	10	-1.8%
Si	51.0	50	2.0%

Table 3-51. Multi-Element Standard Solution Results (SM-744-063)

Analyte	Measured (mg/L)	Reference[29] (mg/L)	% Difference
Al	48.9	50	-2.2%
Fe	50.0	50	0.0%
Mn	20.1	20	0.5%
Ni	10.2	10	2.0%
S	10.7	10	7.0%

3.3.2.2 ARG-1 Glass Standard

Table 3-52 provides a comparison of the published and measured composition of the ARG-1 glass standard. The measured value is the average of two replicates from the AF dissolution data as noted in the table. The measured concentrations for the major glass components (>0.5 wt%) values are consistent with the published values. These data indicate proper performance of the glass dissolutions, and the ICP-ES analyses are of sufficient accuracy and within the analytical uncertainty. Some of the minor components (<0.5 wt%), such as P_2O_5 and ZrO_2, exhibit higher variation, which is expected based on previous results and does not impact the overall conclusions of this study.

Table 3-52. Comparison of the Published and Measured Compositions of the ARG-1 Glass Standard

Oxide	Published[26] (wt%)*	Measured (wt%)	% Difference	Dissolution Method
Al_2O_3	4.73	4.88	3.3	AF
B_2O_3	8.67	8.95	3.2	AF
BaO	0.088	0.086	-1.9	AR
CaO	1.43	1.40	-2.2	AR
Cr_2O_3	0.093	0.095	1.8	AR
CuO	0.004	0.0038	-5.2	AR
Fe_2O_3	14	14.30	2.1	AF
K_2O	2.71	2.65	-2.2	AR
Li_2O	3.21	3.27	1.9	AR
MgO	0.86	0.88	2.1	AF
MnO	1.89	1.90	0.8	AF
Na_2O	11.5	11.80	2.6	AR
NiO	1.05	1.053	0.3	AF
P_2O_5	0.22	0.27	20.8	AR
SiO_2	47.9	46.64	-2.6	AF
SrO	0.0037	0.0035	-4.4	AR
TiO_2	1.15	1.22	5.8	AF
ZnO	0.02	0.019	-0.1	AF
ZrO_2	0.13	0.07	-43.2	AR
Total	99.66	99.49	-0.2	---

*The published ARG-1 composition represents the mean of the Corning, Inc. Glass Composition. ARG-1 has a reported concentration of 2.31 wt% MnO_2 and a total oxide concentration of 100.08 wt%. For consistency, the MnO_2 value was converted to MnO and the total oxide concentration was adjusted accordingly.

3.3.2.3 SB10 SC-19 Glass

Table 3-53 lists the oxide concentrations calculated from the detectable measured elemental concentrations of the SB10 SC-19 Qualification Glass. The value for each oxide is the average of four replicates from the noted dissolution method. The RSD values for a majority of the major glass components (> 0.5 wt%) are less than 10%, indicating good precision in the results. The higher variation exhibited by the minor glass components (<0.5 wt%) is expected based on previous results and does not impact the overall conclusions of this study.

Table 3-53. Average Measured Compositions of the SB10 SC-19 Qualification Glass

Element	Measured (wt%)	Oxide	Measured (wt%)	RSD (%)	Dissolution Method
Al	6.49	Al_2O_3	12.26	1.3	AF
B	1.62	B_2O_3	5.21	1.2	AF
Ba	0.02	BaO	0.03	0.6	AR
Ca	0.28	CaO	0.39	1.5	AR
Cr	0.10	Cr_2O_3	0.15	11.2	AR
Fe	4.22	Fe_2O_3	6.03	2.3	AF
Gd	0.02	Gd_2O_3	0.03	1.8	AR
La	0.02	La_2O_3	0.02	1.7	AF/AR
Li	2.39	Li_2O	5.14	1.6	AR
Mg	0.11	MgO	0.19	1.9	AF
Mn	1.38	MnO	1.78	0.9	AF
Mo	0.004	MoO_3	0.005	4.7	AR
Na	10.43	Na_2O	14.05	1.4	AR
Nd	0.06	Nd_2O_3	0.07	27.0	AR*
Ni	0.15	NiO	0.19	1.2	AF
P	0.03	P_2O_5	0.08	2.5	AR
S	0.12	SO_4^{2-}	0.36	1.8	AR
Si	24.20	SiO_2	51.77	6.1	AF
Sr	0.01	SrO	0.01	1.6	AR
Th	0.81	ThO_2	0.93	4.1	AF/AR
U	1.11	U_3O_8	1.31	1.9	AF/AR
Y	0.008	Y_2O_3	0.01	1.9	AR*
Zn	0.01	ZnO	0.01	2.4	AF
Zr	0.03	ZrO_2	0.04	20.4	AR
---	---	Sum of Oxides	100.05	---	---

*Analyzed by ICP-MS.

3.3.3 DWPF PCCS Evaluation

PCCS is a statistical process control system used in DWPF to assess the acceptability of the melter feed composition against various processing, product quality and solubility constraints.[46] The primary glass properties of interest (viscosity, liquidus temperature and durability) cannot be measured in-situ during DWPF processing and must be predicted using models that relate these properties to the glass composition. The average measured composition of the SB10 SC-19 glass was evaluated against the PCCS constraints and the results are shown in Table 3-54. Note that the durability constraints represent Product Consistency Test (PCT) releases that are two standard deviations below the reported mean releases from the Environmental Assessment (EA) benchmark glass, as required per the Waste Acceptance Product Specifications (WAPS) product consistency acceptance criterion.[46, 59-60] All of the PCCS criteria were met confirming that the SB10 SC-19 glass would be acceptable for processing at DWPF. Note that SB10 qualification was performed using Tank 51 sludge-only material and thus a below detect value for TiO_2 is expected. During actual processing, the MST/sludge solids stream from SWPF will be added, which will increase TiO_2 and impact other property values shown in Table 3-54 (e.g. reduce viscosity).

Table 3-54. PCCS Evaluation Results

Name	Constraint	SB10 SC-19 Glass
Sum of Oxides*	95-105 wt%	99.64 wt%
Durability (PCT)	B release < 14.251 g/L Li release < 8.095 g/L Na release < 11.542 g/L	0.55 g/L 0.60 g/L 0.56 g/L
Homogeneity	$Al_2O_3 \geq 4.0$ wt%	12.26 wt%
Liquidus Temperature	< 1050 °C	829 °C
TiO_2	< 6.0 wt%	below detect
Viscosity	20-110 Poise	95 Poise
Cr_2O_3	< 0.3 wt%	0.15 wt%
Cu	< 0.5 wt%	below detect
Nepheline	$\frac{SiO_2}{SiO_2+Na_2O+Al_2O_3} > 0.62$	0.66
All PCCS criteria met		YES

*Gd_2O_3, SO_4^{2-} and SrO are not included in the PCCS sum of oxides.

3.3.4 REDOX Preparation and Measurement

Five crucibles were prepared from the SC-19 SME Product, of which three remained sealed during vitrification.[e] Once cooled, the five crucibles were broken in half and the glasses were examined; images of glass surfaces and cross-sections from each crucible that remained sealed are shown in Figure 3-21 and regions of focus are highlighted.

As has been observed with many glasses produced from the NGA flowsheet previously, the glass appears to be dark brown in color and have minor striations (i.e. "swirls"). The surface images (images A, C, and E) show no visible signs of crystallization in the glass; the small white specks visible are dust and other small particles from handling the material in the Shielded Cells and the large white areas are either glare from the surrounding lights or the light-colored background behind the samples. The cross-section images (images B, D, and F) show the homogeneity of the brown color throughout the samples with only minor swirls where mixing may have been less than ideal (most visible in image B). Again, the white specks are dust and other small particles and the light areas are either glare or visible background material; the manipulator finger holding the glass is visible at the top of image F.

---

[e] The risk of the sealed lids popping off the crucible during vitrification is an inherent risk in the procedure. The lids must be sealed in such a way as to allow slow venting of gases to prevent pressure build-up within the crucible during heat-up but completely seal to the outside atmosphere once off-gassing has completed and the dried material is vitrifying. Additional lid weights may be used to assist sealing.

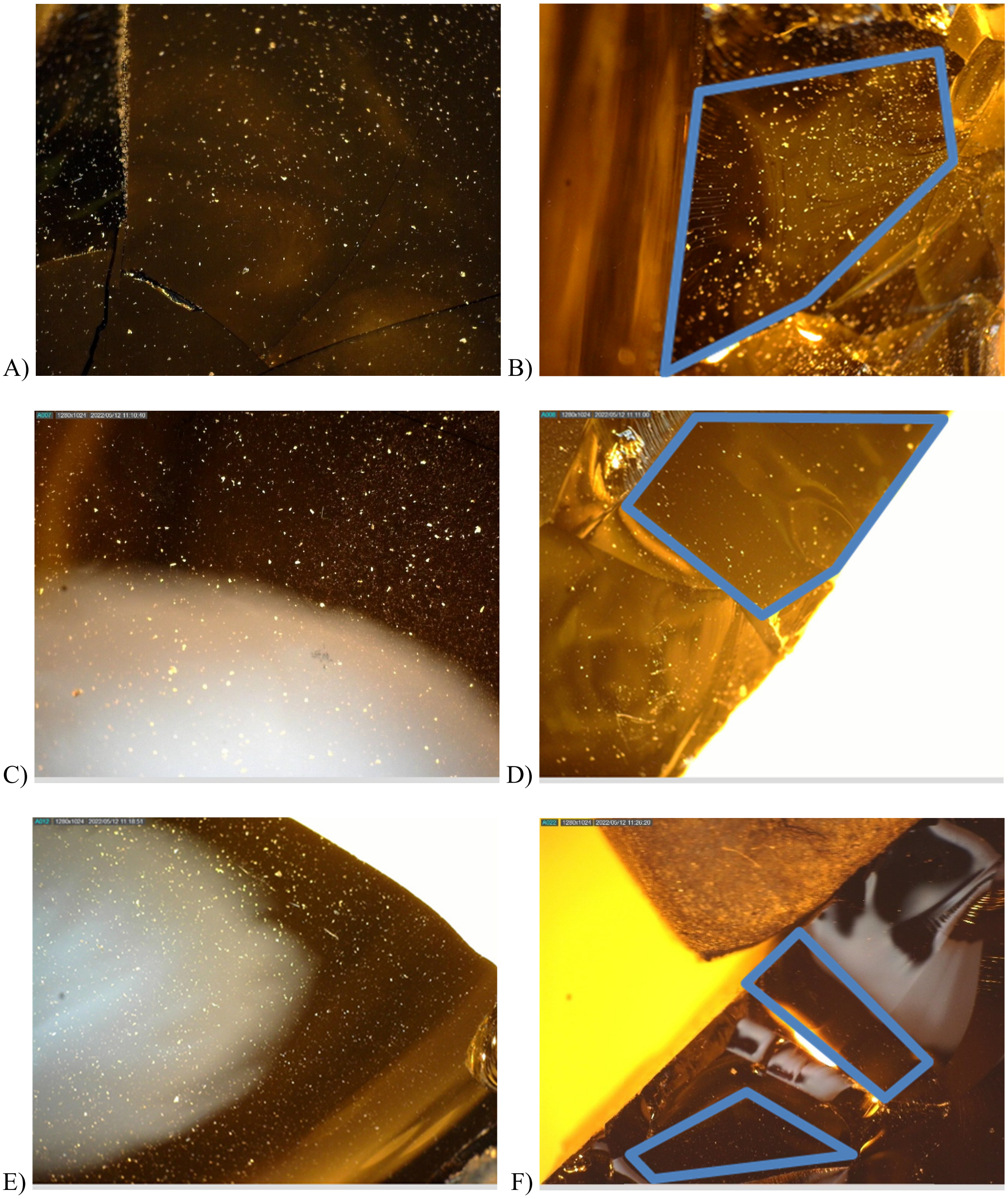

Figure 3-21. Surface (A, C, and E) and Cross-section (B, D, and F) Images of SB10 REDOX Glass Sample Replicates (respectively Crucibles 1, 4, and 5)

Table 3-55 details the UV/vis measurements made on the glass standard and samples taken from the closed sealed crucibles that remained sealed during thermal processing. The Fe(II)/ΣFe ratio that was targeted based in the acid calculation for the SRAT/SME processing was 0.1; the Fe(II)/ΣFe ratio that was predicted based on the SME Product composition was 0.10. The average Fe(II)/ΣFe ratio for the glass samples measured in the Shielded Cells was 0.062.

Table 3-55. Absorbance Values and Calculated REDOX Ratios for SB10 Glass Samples.

Sample	Source	Measured Fe(II)/∑Fe	Average
SC19-1	Crucible 1	0.037	0.091
SC19-2	Crucible 1	0.079	
SC19-3	Crucible 1	0.136	
SC19-4	Crucible 1	0.111	
SC19-5	Crucible 4	0.035	0.036
SC19-6	Crucible 4	0.029	
SC19-7	Crucible 4	0.043	
SC19-8	Crucible 5	0.053	0.052
SC19-9	Crucible 5	0.068	
SC19-10	Crucible 5	0.034	
EA-A	EA Glass Standard	0.177	0.182 (Target: 0.18)
EA-B	EA Glass Standard	0.188	

4.0 Conclusions

Based on the results of this radioactive-waste testing, SRNL has qualified SB10 with the NGA flowsheet. SRNL demonstrated acceptable mercury stripping, slurry rheology, off-gas composition, and glass REDOX at a single acid addition amount to a Tank 51 material. Specific recommendations for processing SB10 are based on a series of simulant tests, and are published in a separate document.[48] An additional recommendation resulting from this SB10 qualification test are given in Section 5.0.

Prior to the addition of antifoam, foaming was encountered in the SRAT cycle during the addition of nitric acid to the extent that sludge foamed over into the off-gas system. Based on observations during simulant testing, the plan involved addition of Momentive™ Y-17112 antifoam after the completion of nitric acid addition and prior to glycolic acid addition. The foaming during nitric acid addition was likely due to the release of carbon dioxide from the acid reacting with carbonate. After a time out (3 weeks), the SRAT cycle was resumed with an antifoam addition prior to resumption of nitric acid addition. Foaming was successfully mitigated during the remainder of the SRAT and SME cycles.

TS measurements of the SRAT and SME Products were 20.1 wt% and 48.1 wt%, respectively. CS measured 11.3 wt% in the SRAT Receipt and SRAT Product, and 37.7 wt% in the SME Product. Waste loading in the SME Product was 33.9 wt% based on iron concentration in the SRAT and SME Products, versus the target of 36% waste loading. When other elements were considered, the waste loading averaged 34.4%.

The pH stayed below 4.0 during SRAT and SME processing during the qualification test. Nitrite was destroyed to below the detection limit (<130 mg/L) during the SRAT cycle. Glycolate destruction was 9.0% during the SRAT cycle and 5.5% during the SME cycle.

The SRAT Receipt Bingham-Plastic yield stress averaged 8.3 Pa and viscosity averaged 8.0 cP. The SRAT Product yield stress averaged 0.83 Pa with a viscosity of 3.9 cP. This is below the lower range of the SRAT Product design basis of 1.5 Pa and 5 cP, which would be expected in this case where the total dried solids content of the SRAT Product was below the target. The SME Product yield stress averaged 2.3 Pa with a viscosity of 10.0 cP. This was very close to the lower limit of the SME Product design basis of 2.5 Pa and 10 cP. SB10 qualification SME Product was significantly closer to the DWPF design basis rheology range than the similar NGA qualification for SB9.

The off-gas peak observed hydrogen generation rate scaled to 6,000 gallons of SRAT and SME feed was 2.7×10^{-4} lb/h in both the SRAT and SME cycles, which is approximately 90-times less than the DWPF

NGA flowsheet limit of 2.4×10^{-2} lb/h. Methane was sporadically detected at low concentrations in both the SRAT and SME cycles.

A series of glasses were prepared from dried SB10 SME Product in sealed crucibles in order to determine the glass REDOX ratio (Fe(II)/ΣFe). Ten total analyses of three separate sealed crucible glasses yielded an average iron REDOX of 0.062 versus the predicted REDOX from the SME Product slurry analysis of 0.10 and the target REDOX of 0.1.

Additional glass was produced from the SME Product, digested and chemically analyzed. All PCCS criteria were met.

Both SRAT and SME condensate were generally dilute, but both contained measurable quantities of nitrate, glycolate, total mercury, methylmercury, and elemental mercury, with the SME condensate being generally more concentrated than the SRAT condensate. Formate was additionally present in the SME condensate. Glycolate concentrations in the SRAT and SME condensates averaged 32.2 mg/L and 60.5 mg/L, respectively. Ammonium was less than 100 mg/L in the SRAT concentrate and was 30 mg/L in the SME condensate. No antifoam degradation products seen with the previous antifoam (i.e., hexamethyldisiloxane, trimethylsilanol, and propanal) were noted in either condensate.

Final mercury concentration in the SRAT Product was 0.77 wt% of TS, which met the current target of <0.8 wt% mercury in the SRAT Product TS. Mercury was further removed to 0.16 wt% of the SME Product TS. Mercury speciation in the SRAT Receipt, SRAT Product, and SME Product identified soluble methylmercury, ionic mercury, and elemental mercury in the supernatant associated with each slurry.

5.0 Recommendations

Due to the foaming encountered during nitric acid addition (which was prior to the addition of antifoam for this test), it is recommended that antifoam be added prior to nitric acid addition.

6.0 Acknowledgements

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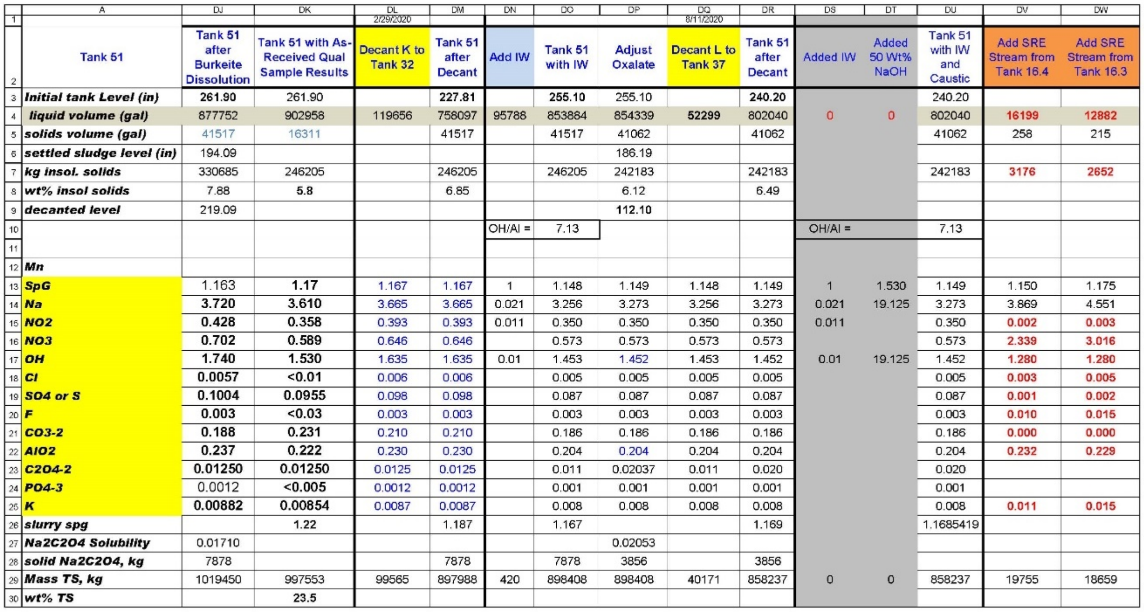
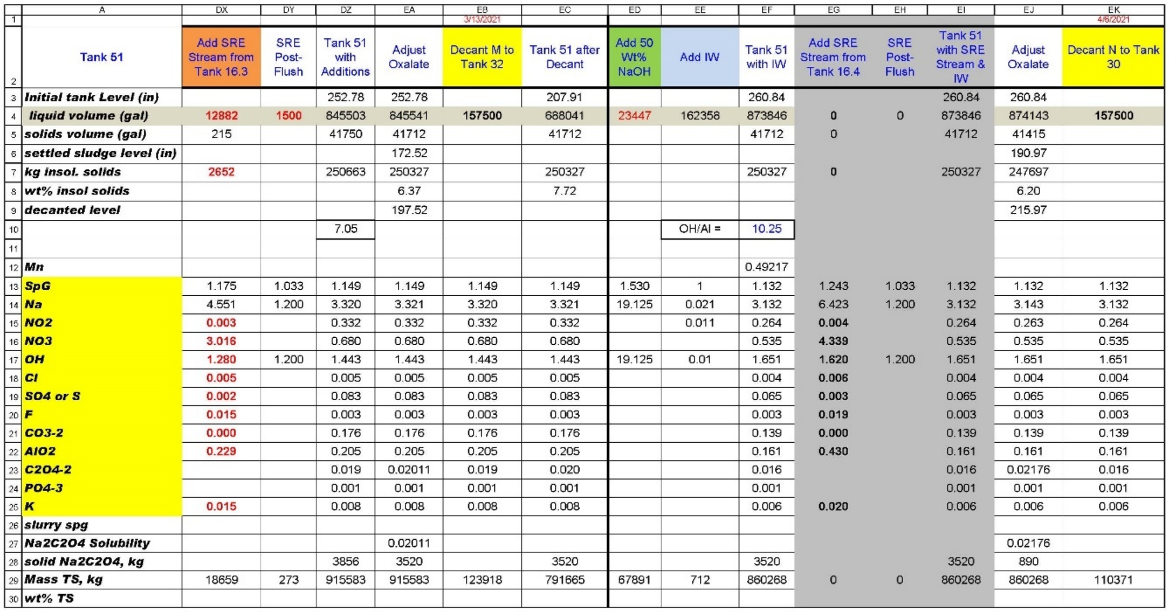
Appendix A. Excerpts From SRMC Washing Spreadsheet

Exhibit A-1. Wash Plans of September 24, 2020

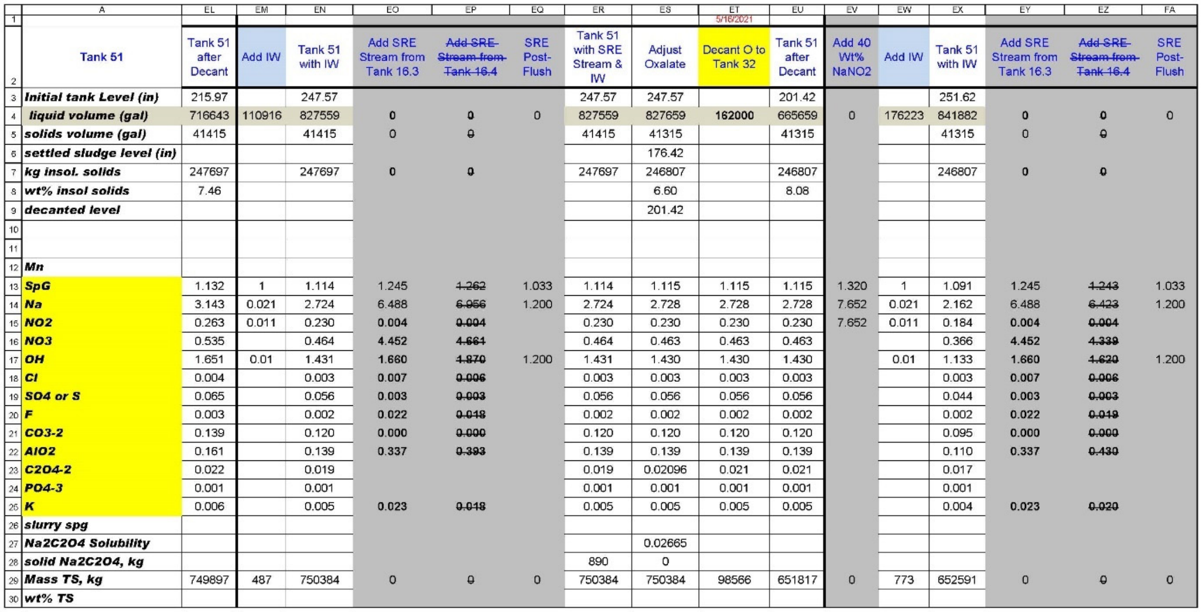


	A	DJ	DK	DL	DM	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW
1				2/29/2020					8/11/2020						
2	Tank 51	Tank 51 after Burkeite Dissolution	Tank 51 with As-Received Qual Sample Results	Decant K to Tank 32	Tank 51 after Decant	Add IW	Tank 51 with IW	Adjust Oxalate	Decant L to Tank 37	Tank 51 after Decant	Added IW	Added 50 Wt% NaOH	Tank 51 with IW and Caustic	Add SRE Stream from Tank 16.4	Add SRE Stream from Tank 16.3
3	**Initial tank Level (in)**	**261.90**	261.90		**227.81**		**255.10**	255.10		**240.20**			240.20		
4	**liquid volume (gal)**	877752	902958	119656	758097	95788	853884	854339	**52299**	802040	0	0	802040	**16199**	**12882**
5	**solids volume (gal)**	41517	16311		41517		41517	41062		41062			41062	258	215
6	**settled sludge level (in)**	194.09						186.19							
7	**kg insol. solids**	330685	246205		246205		246205	242183		242183			242183	**3176**	**2652**
8	**wt% insol solids**	7.88	**5.8**		6.85			6.12		6.49					
9	**decanted level**	219.09						**112.10**							
10						OH/Al =	7.13				OH/Al =		7.13		
11															
12	**Mn**														
13	**SpG**	1.163	**1.17**	1.167	1.167	1	1.148	1.149	1.148	1.149	1	1.530	1.149	1.150	1.175
14	**Na**	**3.720**	**3.610**	3.665	3.665	0.021	3.256	3.273	3.256	3.273	0.021	19.125	3.273	3.869	4.551
15	**NO2**	**0.428**	**0.358**	0.393	0.393	0.011	0.350	0.350	0.350	0.350	0.011		0.350	**0.002**	**0.003**
16	**NO3**	**0.702**	**0.589**	0.646	0.646		0.573	0.573	0.573	0.573			0.573	**2.339**	**3.016**
17	**OH**	**1.740**	**1.530**	1.635	1.635	0.01	1.453	1.452	1.453	1.452	0.01	19.125	1.452	**1.280**	**1.280**
18	**Cl**	**0.0057**	**<0.01**	0.006	0.006		0.005	0.005	0.005	0.005			0.005	**0.003**	**0.005**
19	**SO4 or S**	**0.1004**	**0.0955**	0.098	0.098		0.087	0.087	0.087	0.087			0.087	**0.001**	**0.002**
20	**F**	**0.003**	**<0.03**	0.003	0.003		0.003	0.003	0.003	0.003			0.003	**0.010**	**0.015**
21	**CO3-2**	**0.188**	**0.231**	0.210	0.210		0.186	0.186	0.186	0.186			0.186	**0.000**	**0.000**
22	**AlO2**	**0.237**	**0.222**	0.230	0.230		0.204	0.204	0.204	0.204			0.204	**0.232**	**0.229**
23	**C2O4-2**	**0.01250**	**0.01250**	0.0125	0.0125		0.011	0.02037	0.011	0.020			0.020		
24	**PO4-3**	0.0012	**<0.005**	0.0012	0.0012		0.001	0.001	0.001	0.001			0.001		
25	**K**	**0.00882**	**0.00854**	0.0087	0.0087		0.008	0.008	0.008	0.008			0.008	**0.011**	**0.015**
26	**slurry spg**		1.22		1.187		1.167			1.169			1.1685419		
27	**Na2C2O4 Solubility**	0.01710						0.02053							
28	**solid Na2C2O4, kg**	7878			7878		7878	3856		3856					
29	**Mass TS, kg**	1019450	997553	99565	897988	420	898408	898408	40171	858237	0	0	858237	19755	18659
30	**wt% TS**		**23.5**												

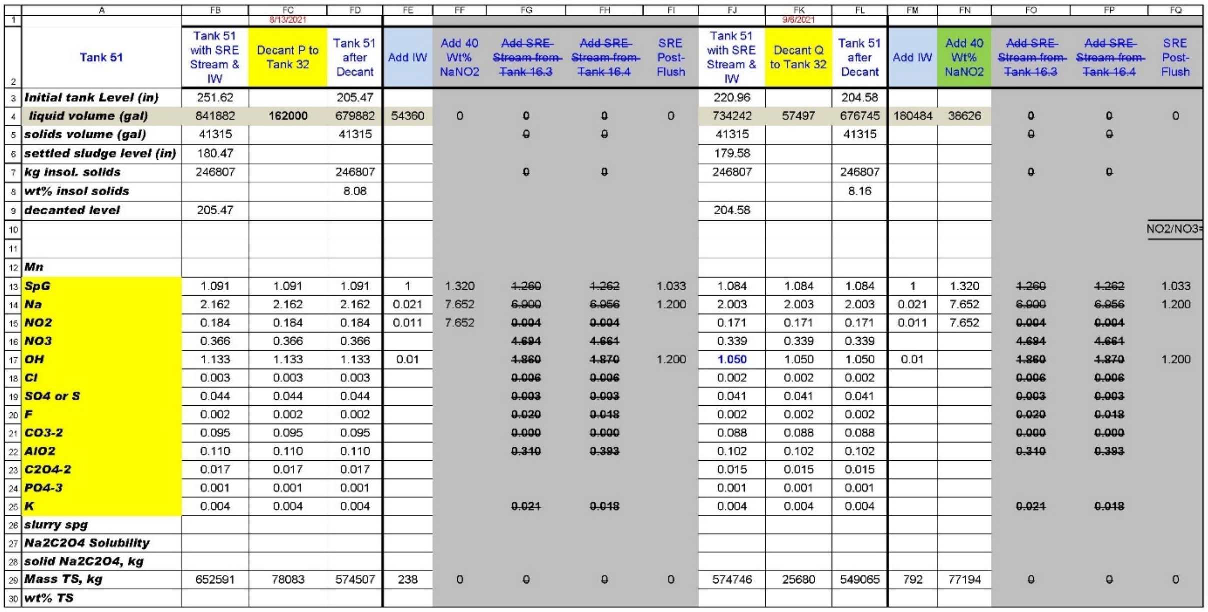


	A	DX	DY	DZ	EA	EB	EC	ED	EE	EF	EG	EH	EI	EJ	EK
1						3/13/2021									4/6/2021
2	Tank 51	Add SRE Stream from Tank 16.3	SRE Post-Flush	Tank 51 with Additions	Adjust Oxalate	Decant M to Tank 32	Tank 51 after Decant	Add 50 Wt% NaOH	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.4	SRE Post-Flush	Tank 51 with SRE Stream & IW	Adjust Oxalate	Decant N to Tank 30
3	**Initial tank Level (in)**			252.78	252.78		207.91			260.84			260.84	260.84	
4	**liquid volume (gal)**	**12882**	**1500**	845503	845541	**157500**	688041	23447	162358	873846	**0**	0	873846	874143	**157500**
5	**solids volume (gal)**	215		41750	41712		41712			41712	0		41712	41415	
6	**settled sludge level (in)**				172.52									190.97	
7	**kg insol. solids**	**2652**		250663	250327		250327			250327	**0**		250327	247697	
8	**wt% insol solids**				6.37		7.72							6.20	
9	**decanted level**				197.52									215.97	
10				7.05					OH/Al =	10.25					
11															
12	**Mn**									0.49217					
13	**SpG**	1.175	1.033	1.149	1.149	1.149	1.149	1.530	1	1.132	1.243	1.033	1.132	1.132	1.132
14	**Na**	4.551	1.200	3.320	3.321	3.320	3.321	19.125	0.021	3.132	6.423	1.200	3.132	3.143	3.132
15	**NO2**	**0.003**		0.332	0.332	0.332	0.332		0.011	0.264	**0.004**		0.264	0.263	0.264
16	**NO3**	**3.016**		0.680	0.680	0.680	0.680			0.535	**4.339**		0.535	0.535	0.535
17	**OH**	**1.280**	1.200	1.443	1.443	1.443	1.443	19.125	0.01	1.651	**1.620**	1.200	1.651	1.651	1.651
18	**Cl**	**0.005**		0.005	0.005	0.005	0.005			0.004	**0.006**		0.004	0.004	0.004
19	**SO4 or S**	**0.002**		0.083	0.083	0.083	0.083			0.065	**0.003**		0.065	0.065	0.065
20	**F**	**0.015**		0.003	0.003	0.003	0.003			0.003	**0.019**		0.003	0.003	0.003
21	**CO3-2**	**0.000**		0.176	0.176	0.176	0.176			0.139	**0.000**		0.139	0.139	0.139
22	**AlO2**	**0.229**		0.205	0.205	0.205	0.205			0.161	**0.430**		0.161	0.161	0.161
23	**C2O4-2**			0.019	0.02011	0.019	0.020			0.016			0.016	0.02176	0.016
24	**PO4-3**			0.001	0.001	0.001	0.001			0.001			0.001	0.001	0.001
25	**K**	**0.015**		0.008	0.008	0.008	0.008			0.006	**0.020**		0.006	0.006	0.006
26	**slurry spg**														
27	**Na2C2O4 Solubility**				0.02011									0.02176	
28	**solid Na2C2O4, kg**			3856	3520		3520			3520			3520	890	
29	**Mass TS, kg**	18659	273	915583	915583	123918	791665	67891	712	860268	0	0	860268	860268	110371
30	**wt% TS**														

Exhibit A -1. Wash Plans of September 24, 2020 (cont.)

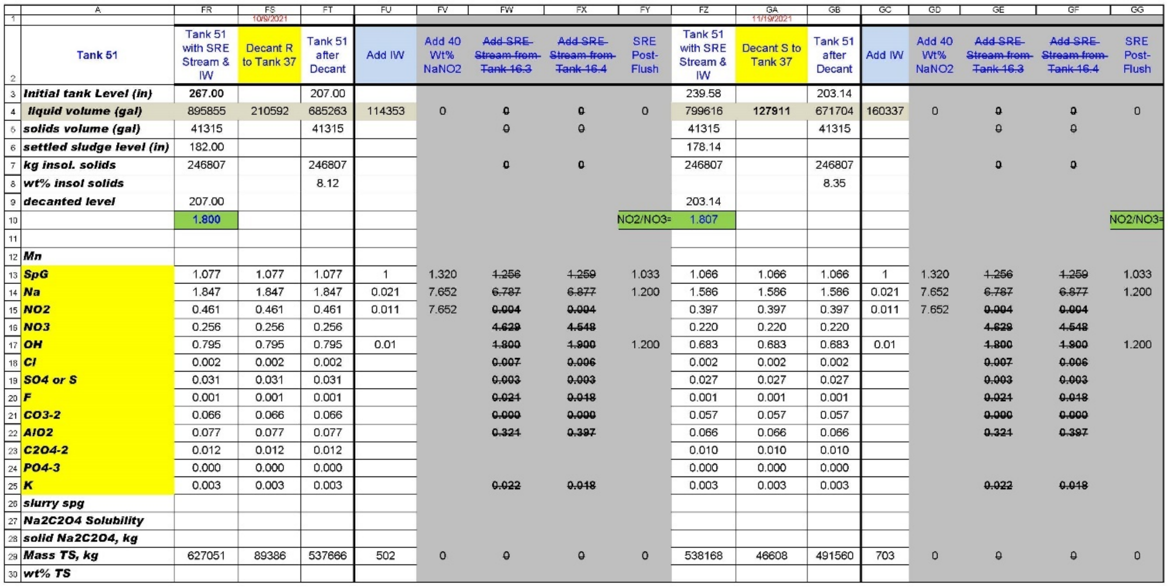


	A	EL	EM	EN	EO	EP	EQ	ER	ES	ET	EU	EV	EW	EX	EY	EZ	FA
1										5/16/2021							
2	Tank 51	Tank 51 after Decant	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.3	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Adjust Oxalate	Decant O to Tank 32	Tank 51 after Decant	Add 40 Wt% NaNO2	Add IW	Tank 51 with IW	Add SRE Stream from Tank 16.3	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	215.97		247.57				247.57	247.57		201.42			251.62			
4	***liquid volume (gal)***	716643	110916	827559	0	~~0~~	0	827559	827659	162000	665659	0	176223	841882	0	~~0~~	0
5	***solids volume (gal)***	41415		41415	0	~~0~~		41415	41315		41315			41315	0	~~0~~	
6	***settled sludge level (in)***								176.42								
7	***kg insol. solids***	247697		247697	0	~~0~~		247697	246807		246807			246807	0	~~0~~	
8	***wt% insol solids***	7.46							6.60		8.08						
9	***decanted level***								201.42								
10																	
11																	
12	***Mn***																
13	***SpG***	1.132	1	1.114	1.245	~~1.262~~	1.033	1.114	1.115	1.115	1.115	1.320	1	1.091	1.245	~~1.243~~	1.033
14	***Na***	3.143	0.021	2.724	6.488	~~6.956~~	1.200	2.724	2.728	2.728	2.728	7.652	0.021	2.162	6.488	~~6.423~~	1.200
15	***NO2***	0.263	0.011	0.230	0.004	~~0.004~~		0.230	0.230	0.230	0.230	7.652	0.011	0.184	0.004	~~0.004~~	
16	***NO3***	0.535		0.464	4.452	~~4.661~~		0.464	0.463	0.463	0.463			0.366	4.452	~~4.339~~	
17	***OH***	1.651	0.01	1.431	1.660	~~1.870~~	1.200	1.431	1.430	1.430	1.430		0.01	1.133	1.660	~~1.620~~	1.200
18	***Cl***	0.004		0.003	0.007	~~0.006~~		0.003	0.003	0.003	0.003			0.003	0.007	~~0.006~~	
19	***SO4 or S***	0.065		0.056	0.003	~~0.003~~		0.056	0.056	0.056	0.056			0.044	0.003	~~0.003~~	
20	***F***	0.003		0.002	0.022	~~0.018~~		0.002	0.002	0.002	0.002			0.002	0.022	~~0.019~~	
21	***CO3-2***	0.139		0.120	0.000	~~0.000~~		0.120	0.120	0.120	0.120			0.095	0.000	~~0.000~~	
22	***AlO2***	0.161		0.139	0.337	~~0.393~~		0.139	0.139	0.139	0.139			0.110	0.337	~~0.430~~	
23	***C2O4-2***	0.022		0.019				0.019	0.02096	0.021	0.021			0.017			
24	***PO4-3***	0.001		0.001				0.001	0.001	0.001	0.001			0.001			
25	***K***	0.006		0.005	0.023	~~0.018~~		0.005	0.005	0.005	0.005			0.004	0.023	~~0.020~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***								0.02665								
28	***solid Na2C2O4, kg***							890	0								
29	***Mass TS, kg***	749897	487	750384	0	~~0~~	0	750384	750384	98566	651817	0	773	652591	0	~~0~~	0
30	***wt% TS***																

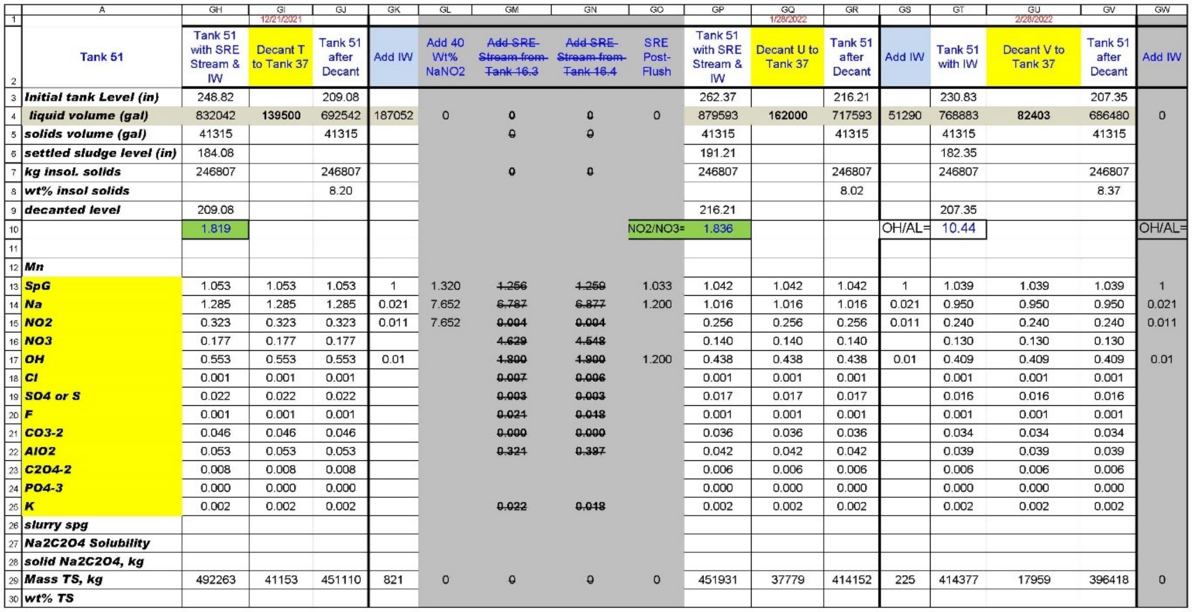


	A	FB	FC	FD	FE	FF	FG	FH	FI	FJ	FK	FL	FM	FN	FO	FP	FQ
1			8/13/2021								9/6/2021						
2	Tank 51	Tank 51 with SRE Stream & IW	Decant P to Tank 32	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant Q to Tank 32	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	251.62		205.47						220.96		204.58					
4	***liquid volume (gal)***	841882	162000	679882	54360	0	~~0~~	~~0~~	0	734242	57497	676745	180484	38626	~~0~~	~~0~~	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315			~~0~~	~~0~~	
6	***settled sludge level (in)***	180.47								179.58							
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807			~~0~~	~~0~~	
8	***wt% insol solids***			8.08								8.16					
9	***decanted level***	205.47								204.58							
10																	NO2/NO3=
11																	
12	***Mn***																
13	***SpG***	1.091	1.091	1.091	1	1.320	~~1.260~~	~~1.262~~	1.033	1.084	1.084	1.084	1	1.320	~~1.260~~	~~1.262~~	1.033
14	***Na***	2.162	2.162	2.162	0.021	7.652	~~6.900~~	~~6.956~~	1.200	2.003	2.003	2.003	0.021	7.652	~~6.900~~	~~6.956~~	1.200
15	***NO2***	0.184	0.184	0.184	0.011	7.652	~~0.004~~	~~0.004~~		0.171	0.171	0.171	0.011	7.652	~~0.004~~	~~0.004~~	
16	***NO3***	0.366	0.366	0.366			~~4.694~~	~~4.661~~		0.339	0.339	0.339			~~4.694~~	~~4.661~~	
17	***OH***	1.133	1.133	1.133	0.01		~~1.860~~	~~1.870~~	1.200	**1.050**	1.050	1.050	0.01		~~1.860~~	~~1.870~~	1.200
18	***Cl***	0.003	0.003	0.003			~~0.006~~	~~0.006~~		0.002	0.002	0.002			~~0.006~~	~~0.006~~	
19	***SO4 or S***	0.044	0.044	0.044			~~0.003~~	~~0.003~~		0.041	0.041	0.041			~~0.003~~	~~0.003~~	
20	***F***	0.002	0.002	0.002			~~0.020~~	~~0.018~~		0.002	0.002	0.002			~~0.020~~	~~0.018~~	
21	***CO3-2***	0.095	0.095	0.095			~~0.000~~	~~0.000~~		0.088	0.088	0.088			~~0.000~~	~~0.000~~	
22	***AlO2***	0.110	0.110	0.110			~~0.310~~	~~0.393~~		0.102	0.102	0.102			~~0.310~~	~~0.393~~	
23	***C2O4-2***	0.017	0.017	0.017						0.015	0.015	0.015					
24	***PO4-3***	0.001	0.001	0.001						0.001	0.001	0.001					
25	***K***	0.004	0.004	0.004			~~0.021~~	~~0.018~~		0.004	0.004	0.004			~~0.021~~	~~0.018~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	652591	78083	574507	238	0	~~0~~	~~0~~	0	574746	25680	549065	792	77194	~~0~~	~~0~~	0
30	***wt% TS***																

Exhibit A -1. Wash Plans of September 24, 2020 (cont.)

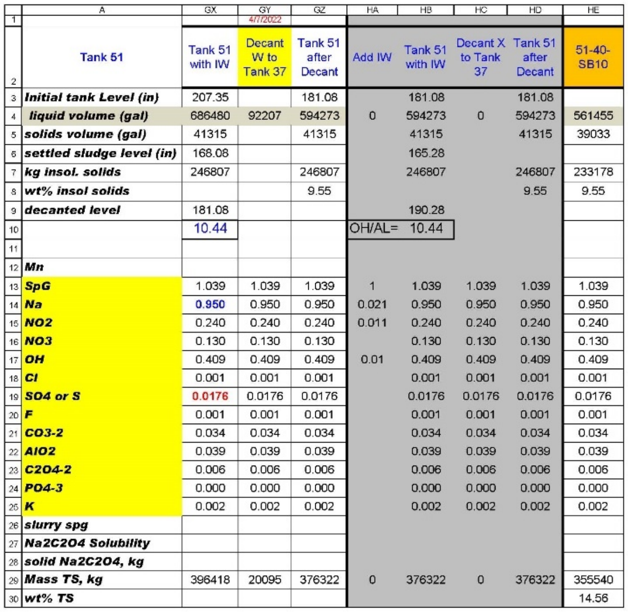


	A	FR	FS	FT	FU	FV	FW	FX	FY	FZ	GA	GB	GC	GD	GE	GF	GG
1			10/9/2021								11/19/2021						
2	Tank 51	Tank 51 with SRE Stream & IW	Decant R to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant S to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush
3	***Initial tank Level (in)***	**267.00**		207.00						239.58		203.14					
4	***liquid volume (gal)***	895855	210592	685263	114353	0	~~0~~	~~0~~	0	799616	**127911**	671704	160337	0	~~0~~	~~0~~	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315			~~0~~	~~0~~	
6	***settled sludge level (in)***	182.00								178.14							
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807			~~0~~	~~0~~	
8	***wt% insol solids***			8.12								8.35					
9	***decanted level***	207.00								203.14							
10		1.800							NO2/NO3=	1.807							NO2/NO3=
11																	
12	***Mn***																
13	***SpG***	1.077	1.077	1.077	1	1.320	~~1.256~~	~~1.259~~	1.033	1.066	1.066	1.066	1	1.320	~~1.256~~	~~1.259~~	1.033
14	***Na***	1.847	1.847	1.847	0.021	7.652	~~6.787~~	~~6.877~~	1.200	1.586	1.586	1.586	0.021	7.652	~~6.787~~	~~6.877~~	1.200
15	***NO2***	0.461	0.461	0.461	0.011	7.652	~~0.004~~	~~0.004~~		0.397	0.397	0.397	0.011	7.652	~~0.004~~	~~0.004~~	
16	***NO3***	0.256	0.256	0.256			~~4.629~~	~~4.548~~		0.220	0.220	0.220			~~4.629~~	~~4.548~~	
17	***OH***	0.795	0.795	0.795	0.01		~~1.800~~	~~1.900~~	1.200	0.683	0.683	0.683	0.01		~~1.800~~	~~1.900~~	1.200
18	***Cl***	0.002	0.002	0.002			~~0.007~~	~~0.006~~		0.002	0.002	0.002			~~0.007~~	~~0.006~~	
19	***SO4 or S***	0.031	0.031	0.031			~~0.003~~	~~0.003~~		0.027	0.027	0.027			~~0.003~~	~~0.003~~	
20	***F***	0.001	0.001	0.001			~~0.021~~	~~0.018~~		0.001	0.001	0.001			~~0.021~~	~~0.018~~	
21	***CO3-2***	0.066	0.066	0.066			~~0.000~~	~~0.000~~		0.057	0.057	0.057			~~0.000~~	~~0.000~~	
22	***AlO2***	0.077	0.077	0.077			~~0.321~~	~~0.397~~		0.066	0.066	0.066			~~0.321~~	~~0.397~~	
23	***C2O4-2***	0.012	0.012	0.012						0.010	0.010	0.010					
24	***PO4-3***	0.000	0.000	0.000						0.000	0.000	0.000					
25	***K***	0.003	0.003	0.003			~~0.022~~	~~0.018~~		0.003	0.003	0.003			~~0.022~~	~~0.018~~	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	627051	89386	537666	502	0	~~0~~	~~0~~	0	538168	46608	491560	703	0	~~0~~	~~0~~	0
30	***wt% TS***																



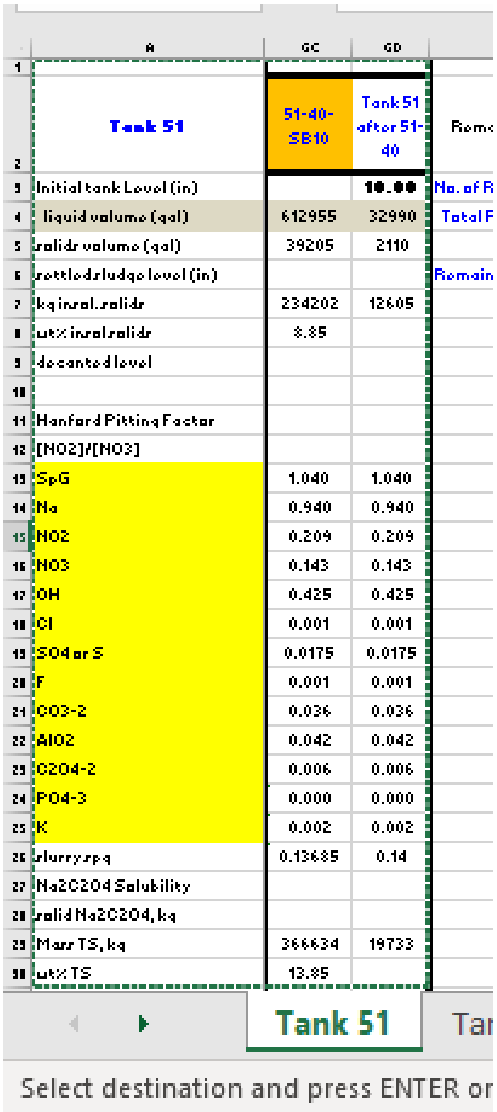
	A	GH	GI	GJ	GK	GL	GM	GN	GO	GP	GQ	GR	GS	GT	GU	GV	GW
1			12/21/2021								1/28/2022				2/28/2022		
2	Tank 51	Tank 51 with SRE Stream & IW	Decant T to Tank 37	Tank 51 after Decant	Add IW	Add 40 Wt% NaNO2	~~Add SRE Stream from Tank 16.3~~	~~Add SRE Stream from Tank 16.4~~	SRE Post-Flush	Tank 51 with SRE Stream & IW	Decant U to Tank 37	Tank 51 after Decant	Add IW	Tank 51 with IW	Decant V to Tank 37	Tank 51 after Decant	Add IW
3	***Initial tank Level (in)***	248.82		209.08						262.37		216.21		230.83		207.35	
4	***liquid volume (gal)***	832042	**139500**	692542	187052	0	~~0~~	~~0~~	0	879593	**162000**	717593	51290	768883	**82403**	686480	0
5	***solids volume (gal)***	41315		41315			~~0~~	~~0~~		41315		41315		41315		41315	
6	***settled sludge level (in)***	184.08								191.21				182.35			
7	***kg insol. solids***	246807		246807			~~0~~	~~0~~		246807		246807		246807		246807	
8	***wt% insol solids***			8.20								8.02				8.37	
9	***decanted level***	209.08								216.21				207.35			
10		1.819							NO2/NO3=	1.836			OH/AL=	10.44			OH/AL=
11																	
12	***Mn***																
13	***SpG***	1.053	1.053	1.053	1	1.320	~~1.256~~	~~1.259~~	1.033	1.042	1.042	1.042	1	1.039	1.039	1.039	1
14	***Na***	1.285	1.285	1.285	0.021	7.652	~~6.787~~	~~6.877~~	1.200	1.016	1.016	1.016	0.021	0.950	0.950	0.950	0.021
15	***NO2***	0.323	0.323	0.323	0.011	7.652	~~0.004~~	~~0.004~~		0.256	0.256	0.256	0.011	0.240	0.240	0.240	0.011
16	***NO3***	0.177	0.177	0.177			~~4.629~~	~~4.548~~		0.140	0.140	0.140		0.130	0.130	0.130	
17	***OH***	0.553	0.553	0.553	0.01		~~1.800~~	~~1.900~~	1.200	0.438	0.438	0.438	0.01	0.409	0.409	0.409	0.01
18	***Cl***	0.001	0.001	0.001			~~0.007~~	~~0.006~~		0.001	0.001	0.001		0.001	0.001	0.001	
19	***SO4 or S***	0.022	0.022	0.022			~~0.003~~	~~0.003~~		0.017	0.017	0.017		0.016	0.016	0.016	
20	***F***	0.001	0.001	0.001			~~0.021~~	~~0.018~~		0.001	0.001	0.001		0.001	0.001	0.001	
21	***CO3-2***	0.046	0.046	0.046			~~0.000~~	~~0.000~~		0.036	0.036	0.036		0.034	0.034	0.034	
22	***AlO2***	0.053	0.053	0.053			~~0.321~~	~~0.397~~		0.042	0.042	0.042		0.039	0.039	0.039	
23	***C2O4-2***	0.008	0.008	0.008						0.006	0.006	0.006		0.006	0.006	0.006	
24	***PO4-3***	0.000	0.000	0.000						0.000	0.000	0.000		0.000	0.000	0.000	
25	***K***	0.002	0.002	0.002			~~0.022~~	~~0.018~~		0.002	0.002	0.002		0.002	0.002	0.002	
26	***slurry spg***																
27	***Na2C2O4 Solubility***																
28	***solid Na2C2O4, kg***																
29	***Mass TS, kg***	492263	41153	451110	821	0	~~0~~	~~0~~	0	451931	37779	414152	225	414377	17959	396418	0
30	***wt% TS***																

Exhibit A -1. Wash Plans of September 24, 2020 (cont.)



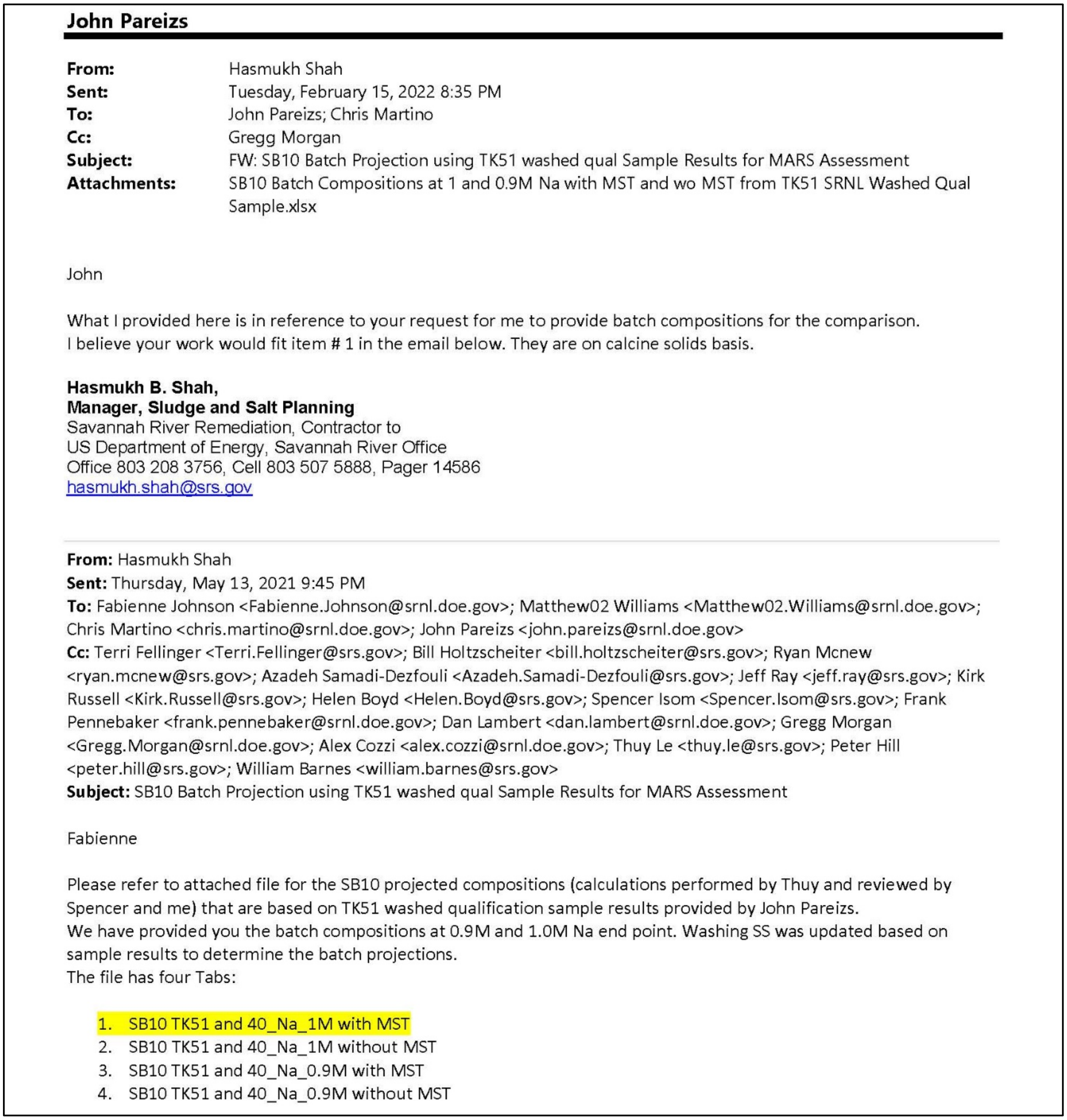
	A	GX	GY	GZ	HA	HB	HC	HD	HE
1			4/7/2022						
2	Tank 51	Tank 51 with IW	Decant W to Tank 37	Tank 51 after Decant	Add IW	Tank 51 with IW	Decant X to Tank 37	Tank 51 after Decant	51-40-SB10
3	***Initial tank Level (in)***	207.35		181.08		181.08		181.08	
4	***liquid volume (gal)***	686480	92207	594273	0	594273	0	594273	561455
5	***solids volume (gal)***	41315		41315		41315		41315	39033
6	***settled sludge level (in)***	168.08				165.28			
7	***kg insol. solids***	246807		246807		246807		246807	233178
8	***wt% insol solids***			9.55				9.55	9.55
9	***decanted level***	181.08				190.28			
10		10.44			OH/AL=	10.44			
11									
12	***Mn***								
13	***SpG***	1.039	1.039	1.039	1	1.039	1.039	1.039	1.039
14	***Na***	**0.950**	0.950	0.950	0.021	0.950	0.950	0.950	0.950
15	***NO2***	0.240	0.240	0.240	0.011	0.240	0.240	0.240	0.240
16	***NO3***	0.130	0.130	0.130		0.130	0.130	0.130	0.130
17	***OH***	0.409	0.409	0.409	0.01	0.409	0.409	0.409	0.409
18	***Cl***	0.001	0.001	0.001		0.001	0.001	0.001	0.001
19	***SO4 or S***	**0.0176**	0.0176	0.0176		0.0176	0.0176	0.0176	0.0176
20	***F***	0.001	0.001	0.001		0.001	0.001	0.001	0.001
21	***CO3-2***	0.034	0.034	0.034		0.034	0.034	0.034	0.034
22	***AlO2***	0.039	0.039	0.039		0.039	0.039	0.039	0.039
23	***C2O4-2***	0.006	0.006	0.006		0.006	0.006	0.006	0.006
24	***PO4-3***	0.000	0.000	0.000		0.000	0.000	0.000	0.000
25	***K***	0.002	0.002	0.002		0.002	0.002	0.002	0.002
26	***slurry spg***								
27	***Na2C2O4 Solubility***								
28	***solid Na2C2O4, kg***								
29	***Mass TS, kg***	396418	20095	376322	0	376322	0	376322	355540
30	***wt% TS***								14.56

Exhibit A-2. Tank Farm Wash Endpoint – February 16, 2021



	A	GC	GD	
1				
2	**Tank 51**	51-40-SB10	Tank 51 after 51-40	Rem
3	Initial tank Level (in)		**10.00**	No. of R
4	liquid volume (gal)	612955	32990	Total F
5	solids volume (gal)	39205	2110	
6	settled sludge level (in)			Remain
7	kg insol. solids	234202	12605	
8	wt% insol solids	8.85		
9	decanted level			
10				
11	Hanford Pitting Factor			
12	[NO2]/[NO3]			
13	SpG	1.040	1.040	
14	Na	0.940	0.940	
15	NO2	0.209	0.209	
16	NO3	0.143	0.143	
17	OH	0.425	0.425	
18	Cl	0.001	0.001	
19	SO4 or S	0.0175	0.0175	
20	F	0.001	0.001	
21	CO3-2	0.036	0.036	
22	AlO2	0.042	0.042	
23	C2O4-2	0.006	0.006	
24	PO4-3	0.000	0.000	
25	K	0.002	0.002	
26	slurry spg	0.13685	0.14	
27	Na2C2O4 Solubility			
28	solid Na2C2O4, kg			
29	Mass TS, kg	366634	19733	
30	wt% TS	13.85		

Exhibit A-3. **Liquid Waste contractor Projected Elemental Composition of SB10 Washed Tank 51**
The Liquid Waste Contractor provided SRNL with the projected elemental composition of SB10 Washed Tank 51 via email. An excerpt from the email and the applicable excerpt from the spreadsheet attached to the email, for comparison to SRNL results, is given here.



John Pareizs

From: Hasmukh Shah
Sent: Tuesday, February 15, 2022 8:35 PM
To: John Pareizs; Chris Martino
Cc: Gregg Morgan
Subject: FW: SB10 Batch Projection using TK51 washed qual Sample Results for MARS Assessment
Attachments: SB10 Batch Compositions at 1 and 0.9M Na with MST and wo MST from TK51 SRNL Washed Qual Sample.xlsx

John

What I provided here is in reference to your request for me to provide batch compositions for the comparison. I believe your work would fit item # 1 in the email below. They are on calcine solids basis.

Hasmukh B. Shah,
Manager, Sludge and Salt Planning
Savannah River Remediation, Contractor to
US Department of Energy, Savannah River Office
Office 803 208 3756, Cell 803 507 5888, Pager 14586
hasmukh.shah@srs.gov

From: Hasmukh Shah
Sent: Thursday, May 13, 2021 9:45 PM
To: Fabienne Johnson <Fabienne.Johnson@srnl.doe.gov>; Matthew02 Williams <Matthew02.Williams@srnl.doe.gov>; Chris Martino <chris.martino@srnl.doe.gov>; John Pareizs <john.pareizs@srnl.doe.gov>
Cc: Terri Fellinger <Terri.Fellinger@srs.gov>; Bill Holtzscheiter <bill.holtzscheiter@srs.gov>; Ryan Mcnew <ryan.mcnew@srs.gov>; Azadeh Samadi-Dezfouli <Azadeh.Samadi-Dezfouli@srs.gov>; Jeff Ray <jeff.ray@srs.gov>; Kirk Russell <Kirk.Russell@srs.gov>; Helen Boyd <Helen.Boyd@srs.gov>; Spencer Isom <Spencer.Isom@srs.gov>; Frank Pennebaker <frank.pennebaker@srnl.doe.gov>; Dan Lambert <dan.lambert@srnl.doe.gov>; Gregg Morgan <Gregg.Morgan@srnl.doe.gov>; Alex Cozzi <alex.cozzi@srnl.doe.gov>; Thuy Le <thuy.le@srs.gov>; Peter Hill <peter.hill@srs.gov>; William Barnes <william.barnes@srs.gov>
Subject: SB10 Batch Projection using TK51 washed qual Sample Results for MARS Assessment

Fabienne

Please refer to attached file for the SB10 projected compositions (calculations performed by Thuy and reviewed by Spencer and me) that are based on TK51 washed qualification sample results provided by John Pareizs.
We have provided you the batch compositions at 0.9M and 1.0M Na end point. Washing SS was updated based on sample results to determine the batch projections.
The file has four Tabs:

1. SB10 TK51 and 40_Na_1M with MST
2. SB10 TK51 and 40_Na_1M without MST
3. SB10 TK51 and 40_Na_0.9M with MST
4. SB10 TK51 and 40_Na_0.9M without MST

Exhibit A-4. Elemental composition as wt% of calcined solids.

SB10 Projection using TK51 Washed Sample Results

SB10 TK51_40_Na 1M_with MST

STATION	SB9 Tk 40	SB10.prep	SB10.Start
DESCRIPTION	Tk 40 SB9 at WASP samples	Tank 51 SB10 end of washing	Tk40, SB-10 Blend READY
Calcine Solids Mass, kg	208,067	279,640	390,740
wt%, Al	8.54	17.53	13.32
B	0.04	0.00	0.01
Ba	0.10	0.06	0.06
Ca	1.45	0.74	0.74
Ce	0.31	0.12	0.13
Cr	0.11	0.23	0.17
Cu	0.04	0.05	0.04
Fe	22.18	11.91	11.63
K	0.11	0.08	0.08
La	0.06	0.03	0.03
Li	0.06	0.02	0.02
Mg	0.30	0.29	0.25
Mn	7.21	3.72	3.68
Na	19.58	24.44	25.74
Ni	1.46	0.38	0.49
Pb	0.06	0.02	0.02
S	0.35	0.54	0.53
Si	1.75	0.59	0.68
Th	1.13	2.42	1.82
Ti	0.03	0.02	4.45
U	4.08	3.08	2.95
Zn	0.04	0.02	0.02
Zr	0.04	0.16	0.11
Hg (total solids basis)	2.22	3.23	2.78

Appendix B. Calculation of Elements on a wt% of Calcined Solids Basis

- Elements not detected by SRNL are not used.
- Only elements given by the Liquid Waste Contractor (see Appendix A) are considered in this calculation.
- Hg is not considered in the calcine calculation as it is volatilized in the calcine process, and is not present in the final glass waste form.
- wt% of total dried solids.
- Oxide to element conversion factor is:

$$Ox\ El\ Conv\ Fact = \frac{MW_{oxide}}{MW_{El} \times N}$$

Where

MW_{oxide} = molecular weight of oxide
MW_{El} = molecular weight of element
N = number of moles of element in the oxide

Oxide is the oxide form of the element in the final glass waste form.

Oxide (% of total solids) is the elemental wt% of total dried solids times the oxide to element conversion factor, essentially the amount of oxide one would expect if one calcined 100 g of dried solids.

Normalized Oxide (wt%) is the calculated percent oxides in the material if it were calcined (nothing but elements and oxides).

Normalized Element (wt%) is the normalized oxide (wt%) divided by the oxide to element conversion factor.

Element	wt% of TS	Element to Oxide Conversion Factor	Oxide	Oxide (% of TS)	Normalized Oxide (wt%)	Normalized Element (wt%)
Al	1.24E+01	1.8895	Al2O3	2.34E+01	33.41	17.7
B	<8.3E-02	3.2199	B2O3	NA	NA	NA
Ba	4.59E-02	1.1165	BaO	5.12E-02	0.07	0.0655
Ca	5.30E-01	1.3992	CaO	7.42E-01	1.06	0.756
Ce	9.72E-02	1.2284	CeO2	1.19E-01	0.17	0.139
Cr	1.61E-01	1.4616	Cr2O3	2.35E-01	0.34	0.230
Cu	3.52E-02	1.2518	CuO	4.41E-02	0.06	0.0502
Fe	8.51E+00	1.4297	Fe2O3	1.22E+01	17.35	12.1
K	<7.6E-02	1.2046	K2O	NA	NA	NA
La	3.38E-02	1.1728	La2O3	3.96E-02	0.06	0.0482
Li	<6.3E-03	4.3051	Li2O	NA	NA	NA
Mg	2.09E-01	1.6583	MgO	3.47E-01	0.49	0.298
Mn	2.66E+00	1.2912	MnO	3.43E+00	4.90	3.79
Na	1.66E+01	1.3480	Na2O	2.24E+01	31.91	23.7
Ni	2.74E-01	1.2726	NiO	3.49E-01	0.50	0.391
Pb	9.32E-03	1.0772	PbO	1.00E-02	0.01	0.0133
S	3.79E-01	2.9958	SO4	1.14E+00	1.62	0.540
Si	4.24E-01	2.1393	SiO2	9.07E-01	1.29	0.605
Th	1.73E+00	1.1379	ThO2	1.97E+00	2.81	2.47
Ti	<1.4E-02	1.6683	TiO2	NA	NA	NA
U	2.22E+00	1.1792	U3O8	2.62E+00	3.73	3.17
Zn	1.75E-02	1.2447	ZnO	2.18E-02	0.03	0.0250
Zr	1.13E-01	1.1754	ZrO	1.33E-01	0.19	0.161
Hg	2.85E+00	NA	NA	NA	NA	NA
			Total	7.01E+01	100.00	

Appendix C. Previously Published Off-gas Plots

Following are individual plots of concentrations and generation rates which have been published previously.[f] .

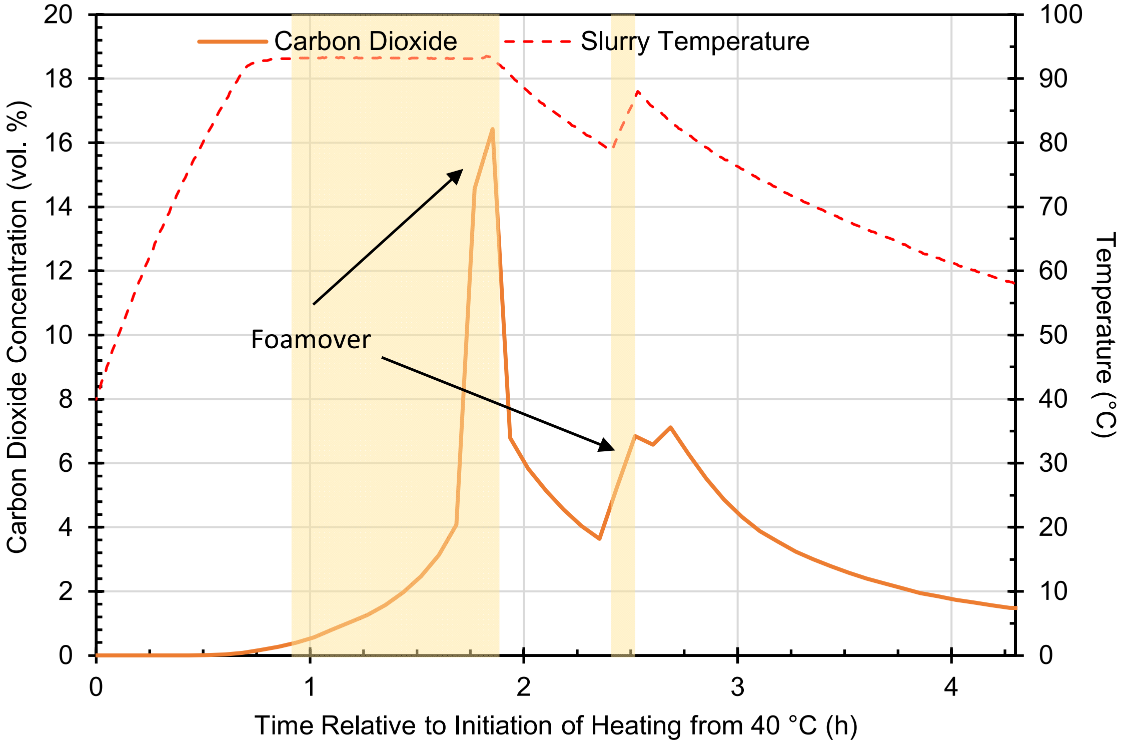


Figure C-1. SRAT Part 1 Carbon Dioxide Concentration (shaded areas show nitric acid addition)

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[f] J.M. Pareizs, “SRNL Sludge Batch 10 Qualification SRAT and SME Off-Gas Results,” Savannah River National Laboratory, Aiken, SC, SRNL-TR-2022-00071, Rev. 0, 2022.

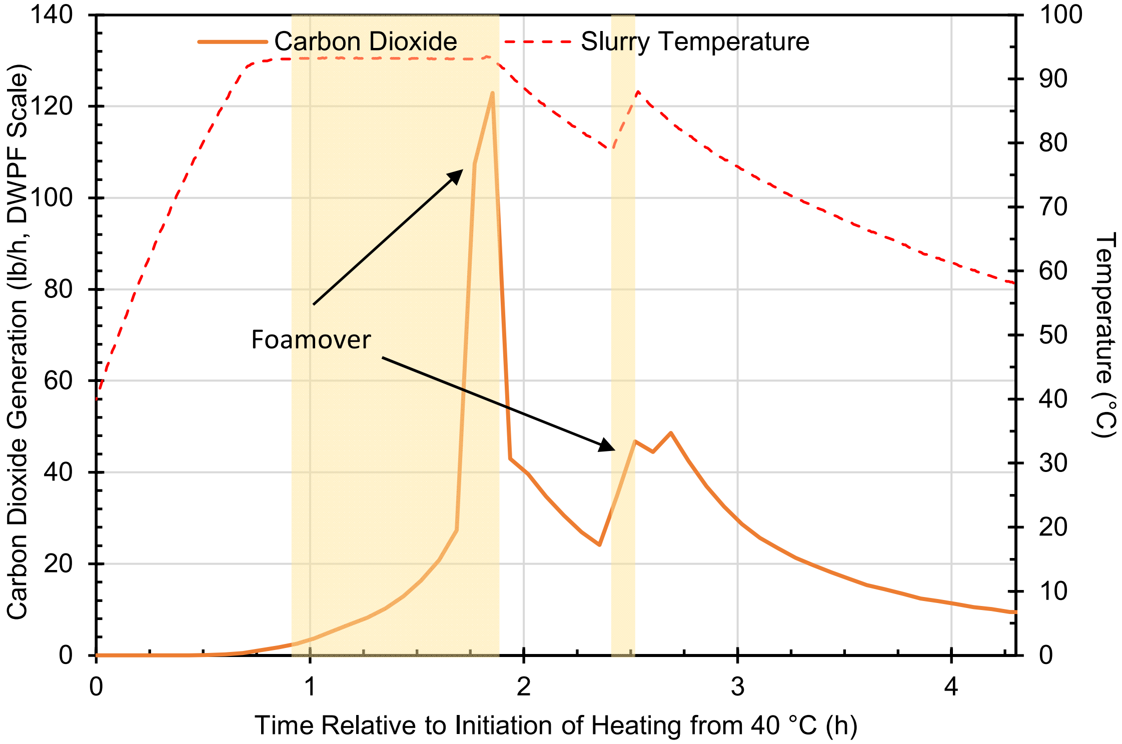

Figure C-2. SRAT Part 1 Carbon Dioxide Generation (shaded areas show nitric acid addition)

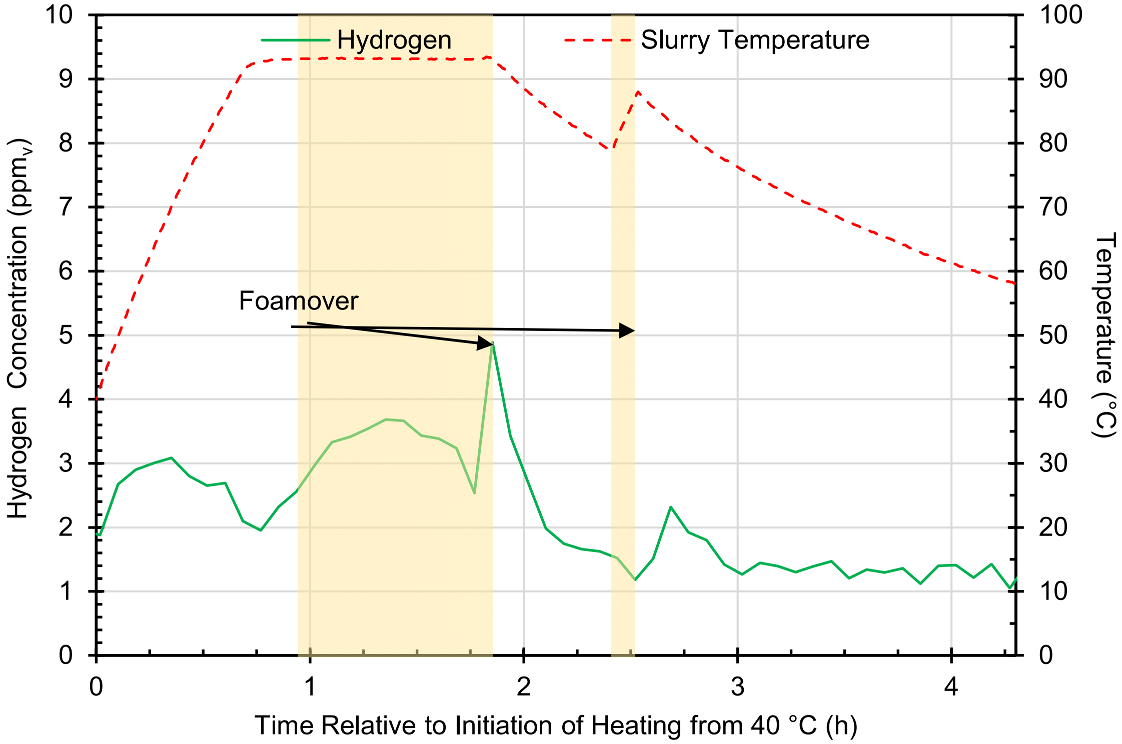

Figure C-3. SRAT Part 1 Hydrogen Concentration (shaded areas show nitric acid addition)

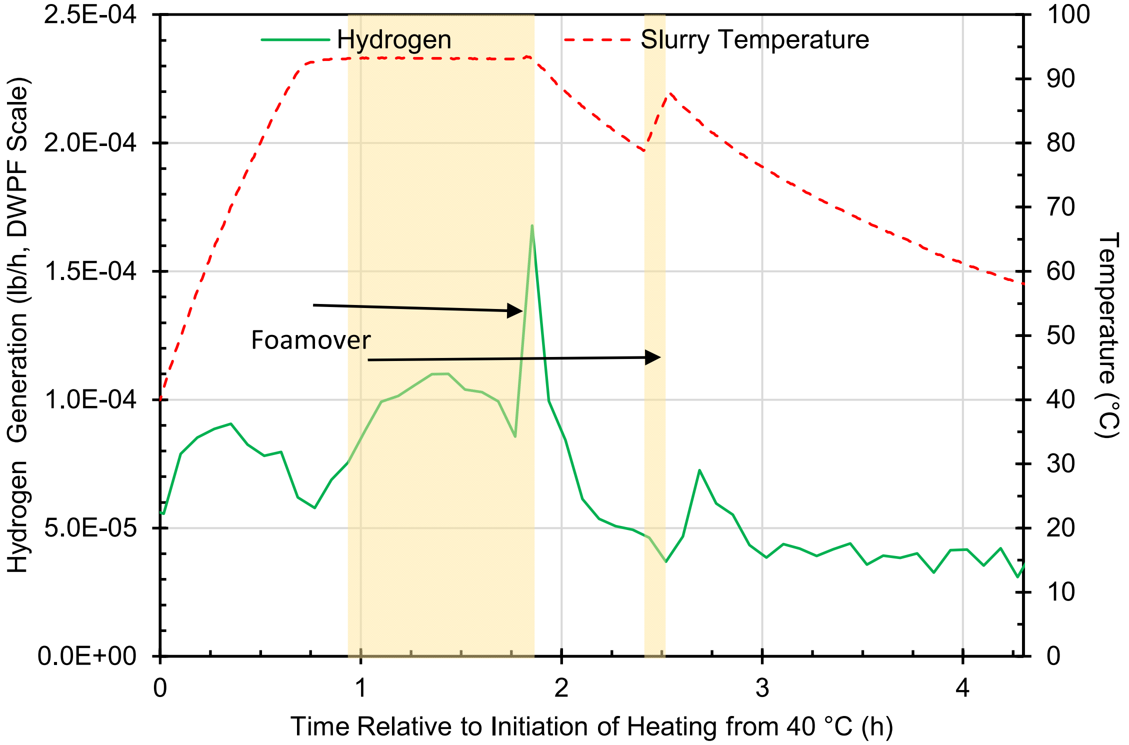

Figure C-4. SRAT Part 1 Hydrogen Generation Rate (shaded areas show nitric acid addition)

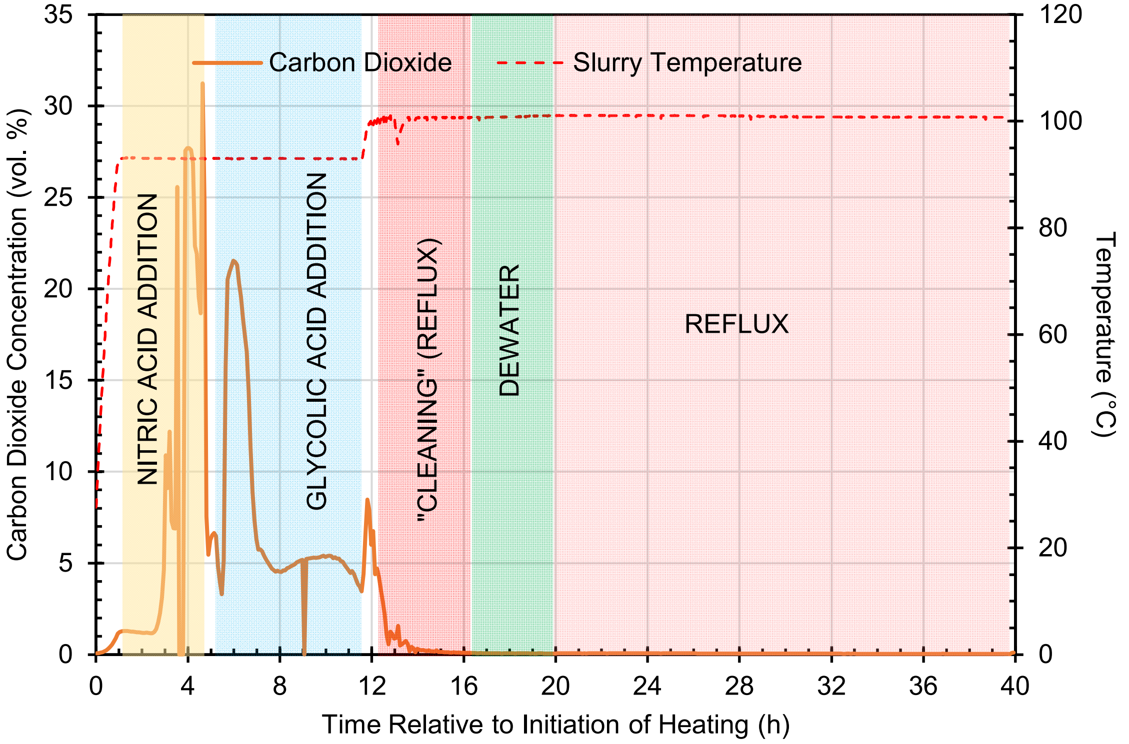

Figure C-5. SRAT Part 2 Carbon Dioxide Concentration

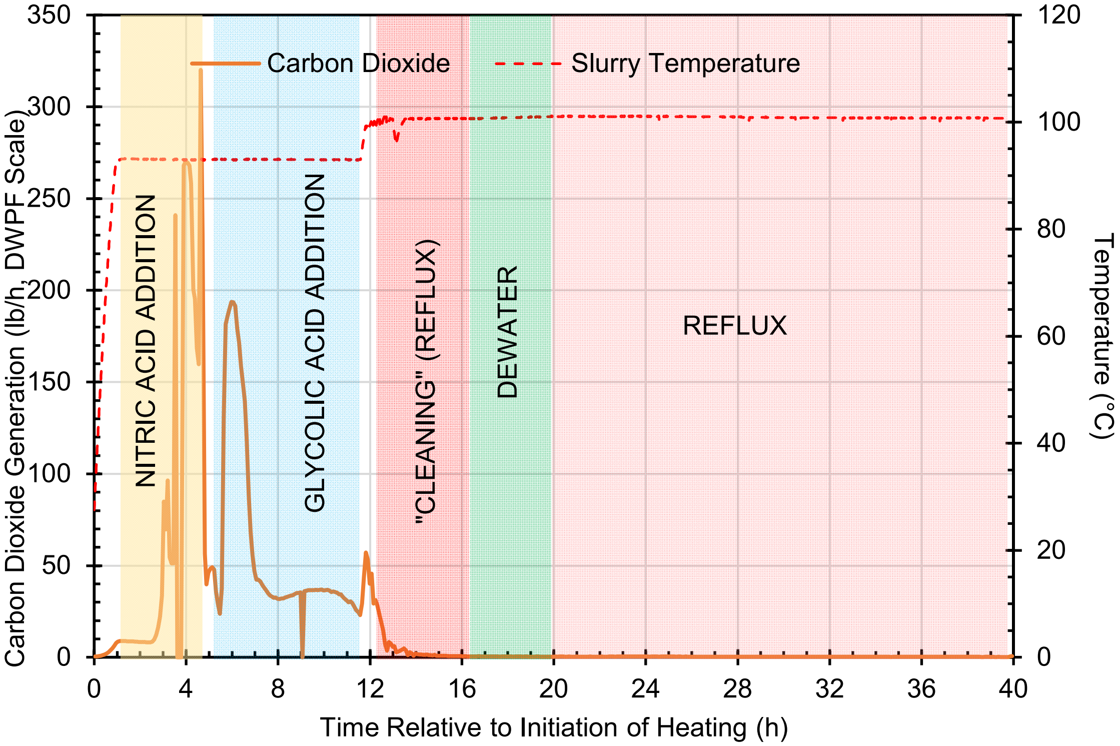

Figure C-6. SRAT Part 2 Carbon Dioxide Generation Rate

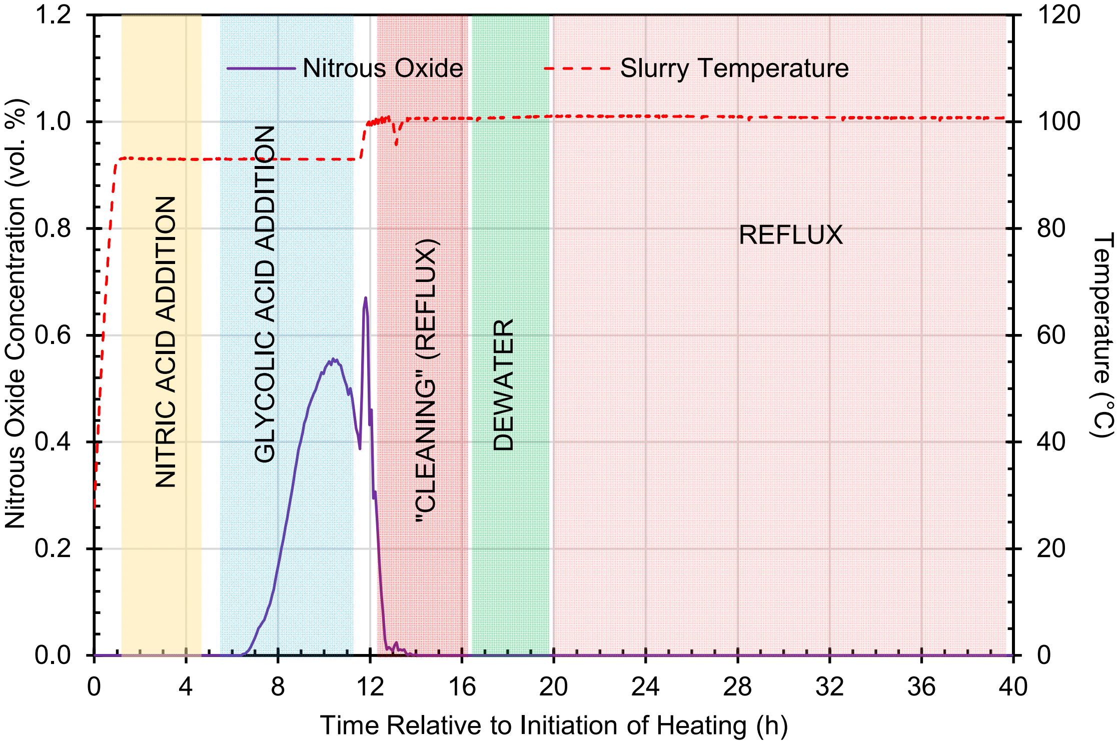

Figure C-7 SRAT Part 2 Nitrous Oxide Concentration

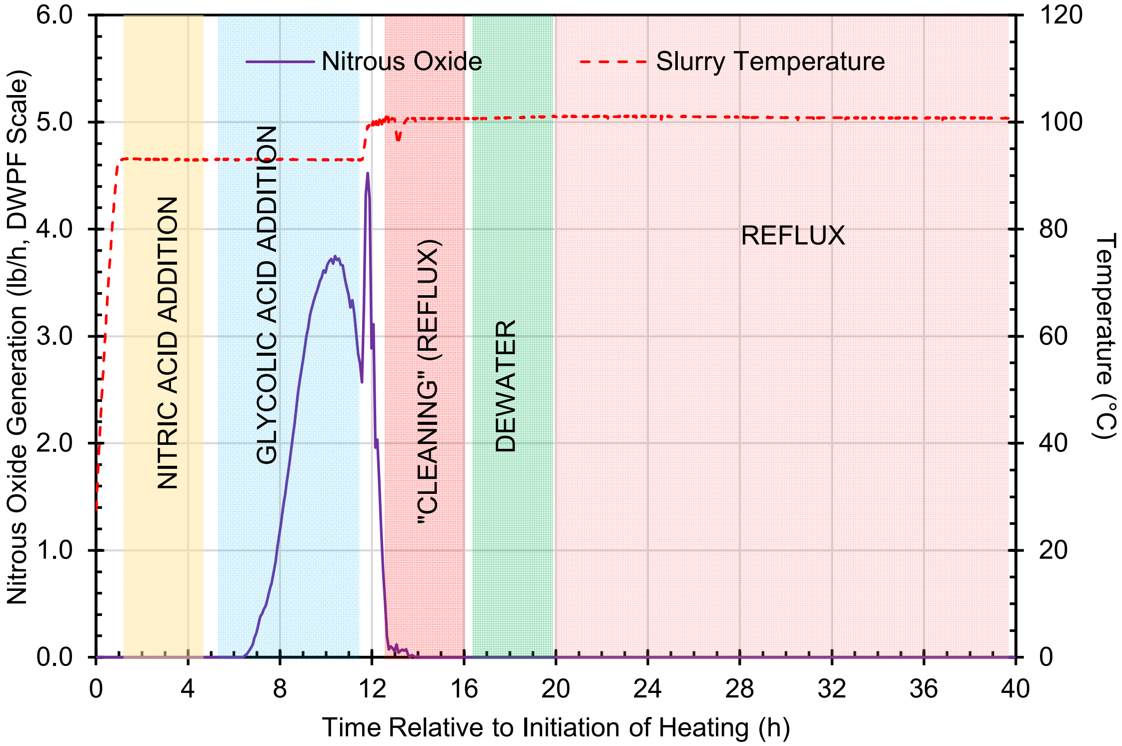

Figure C-8. SRAT Part 2 Nitrous Oxide Generation Rate

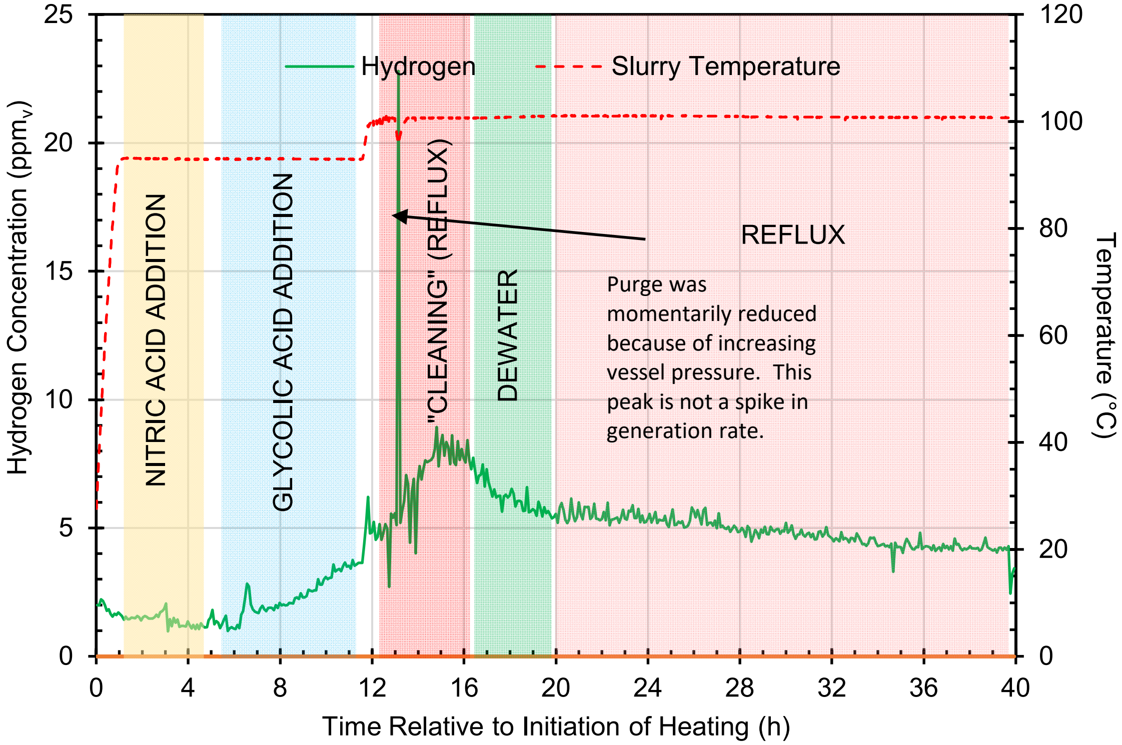

Figure C-9. SRAT Part 2 Hydrogen Concentration

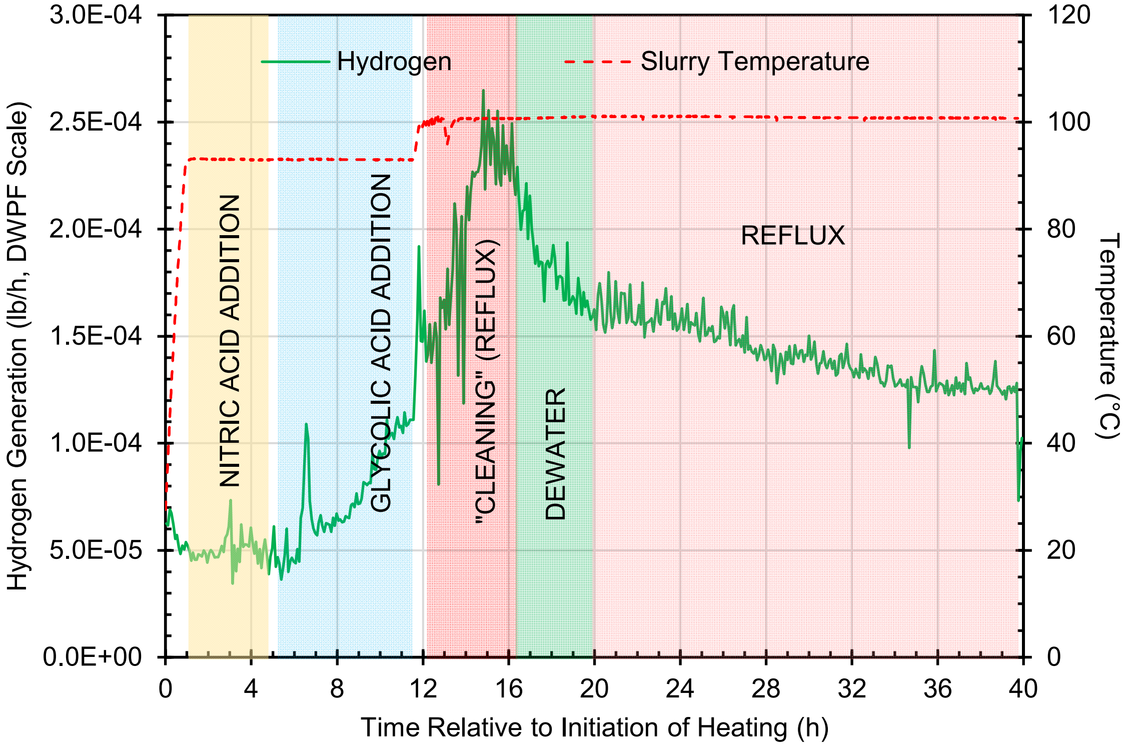

Figure C-10. SRAT Part 2 Hydrogen Generation Rate

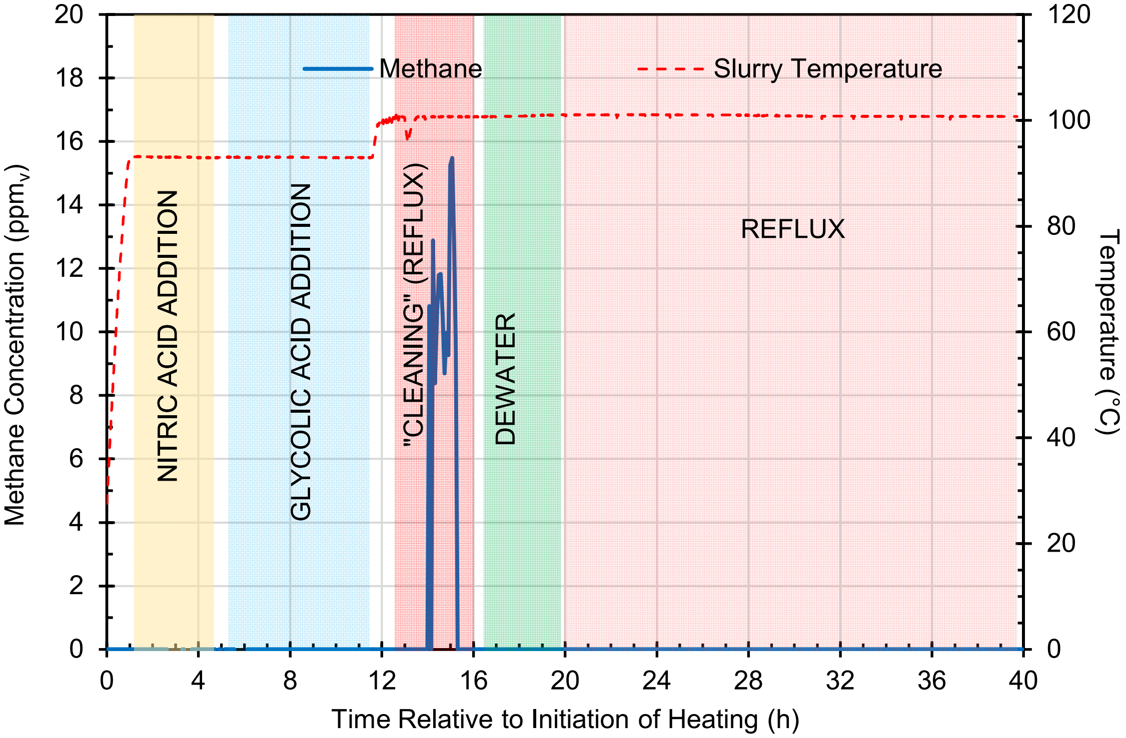

Figure C-11. SRAT Part 2 Methane Concentration

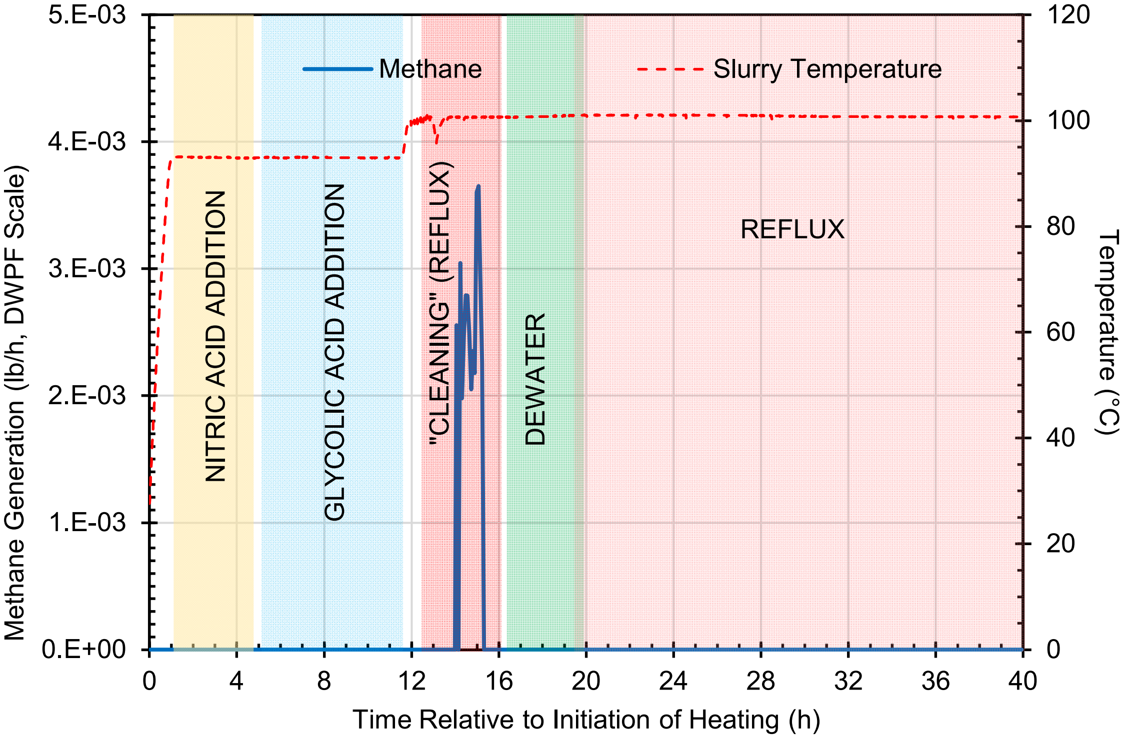

Figure C-12. SRAT Part 2 Methane Generation

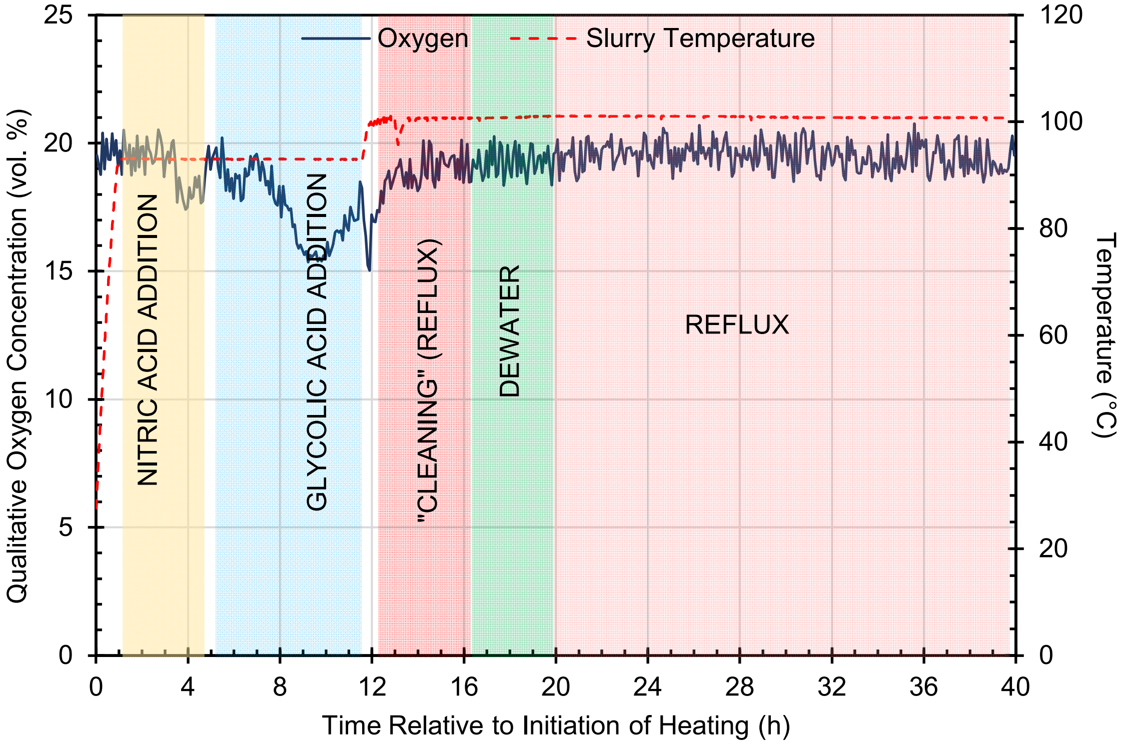

Figure C-13. SRAT Part 2 Qualitative Oxygen Concentration

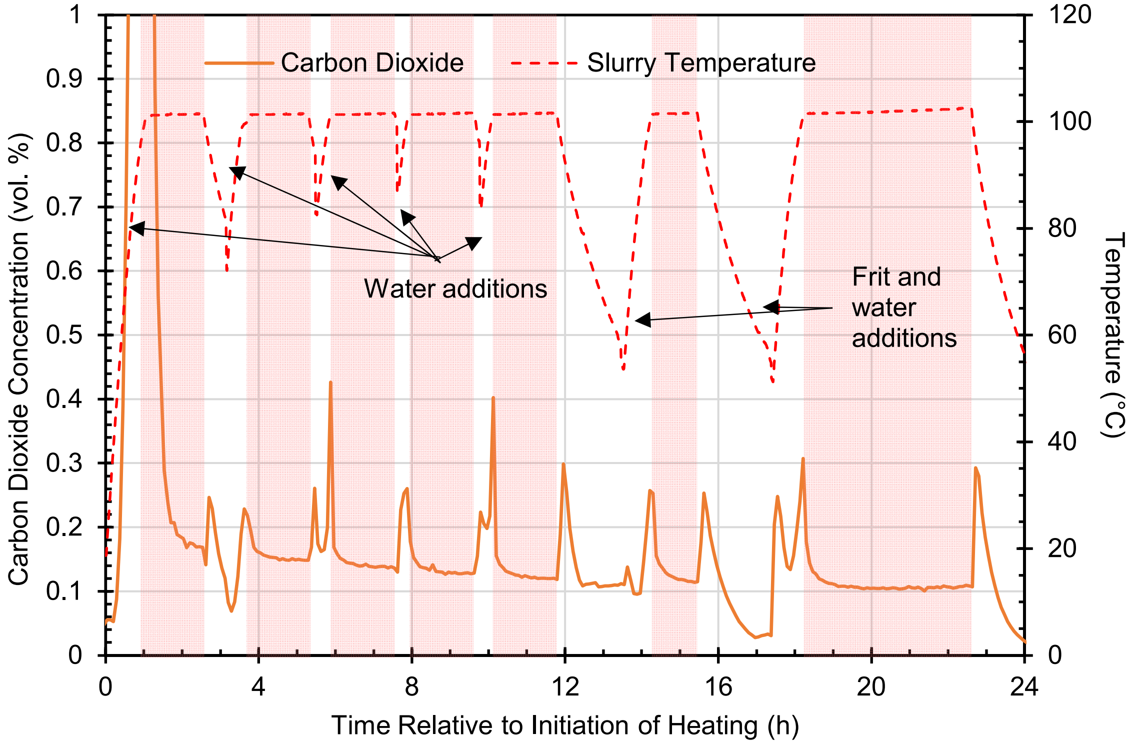

Figure C-14. SME Cycle Carbon Dioxide Concentration (shaded areas represent boiling)

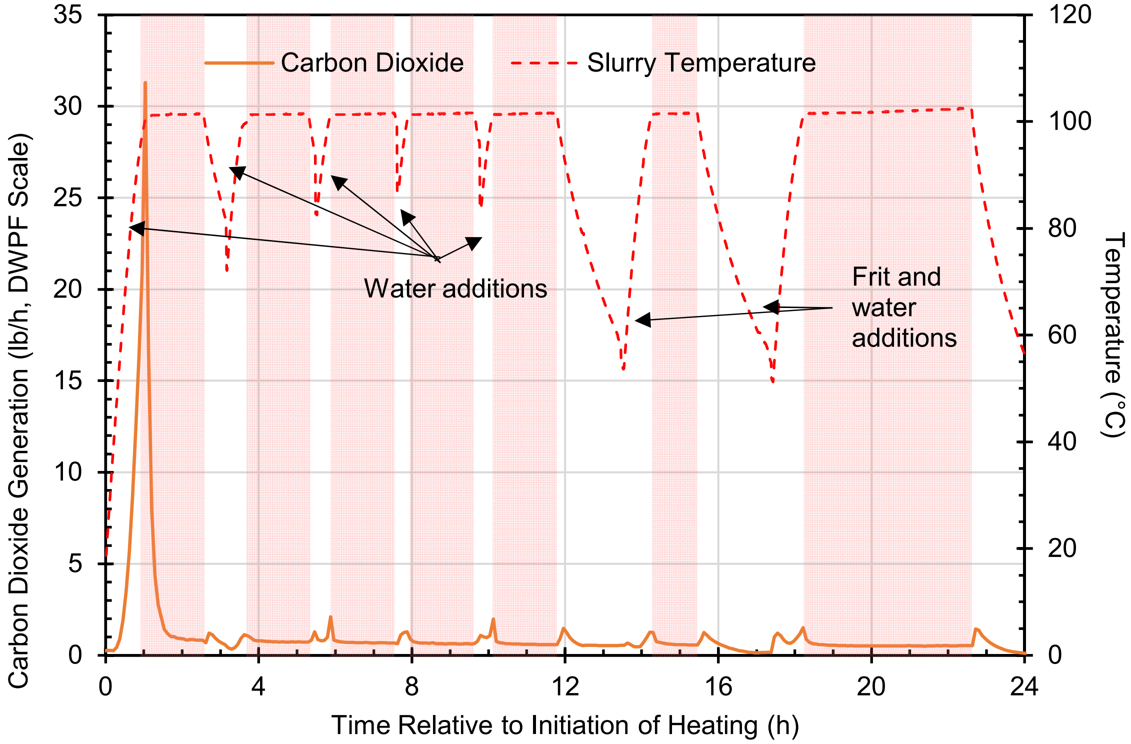

Figure C-15. SME Cycle Carbon Dioxide Generation (shaded areas represent boiling)

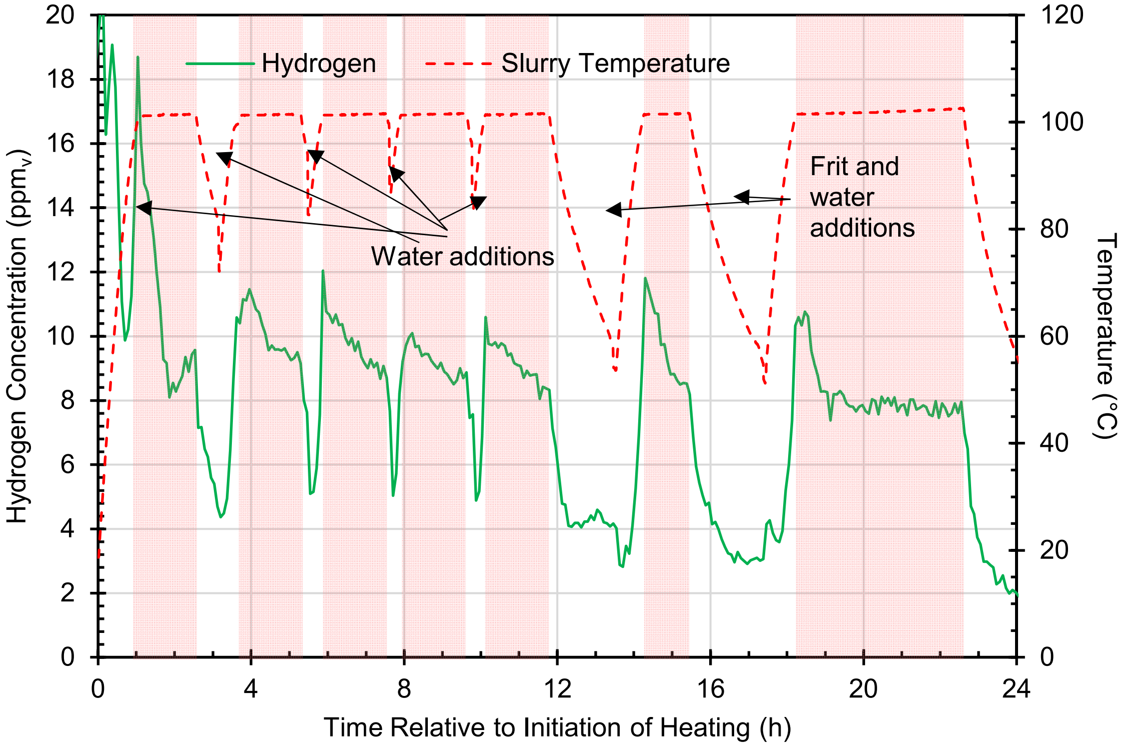

Figure C-16. SME Cycle Hydrogen Concentration (shaded areas represent boiling)

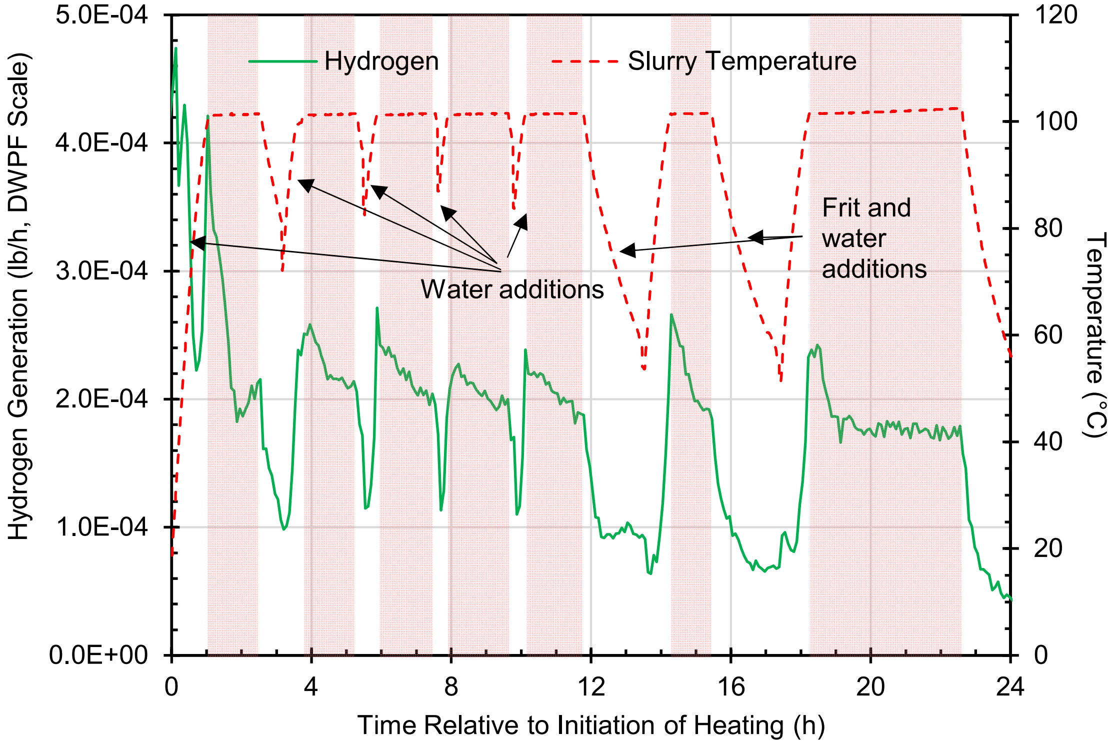

Figure C-17. SME Cycle Hydrogen Generation Rate (shaded areas represent boiling)

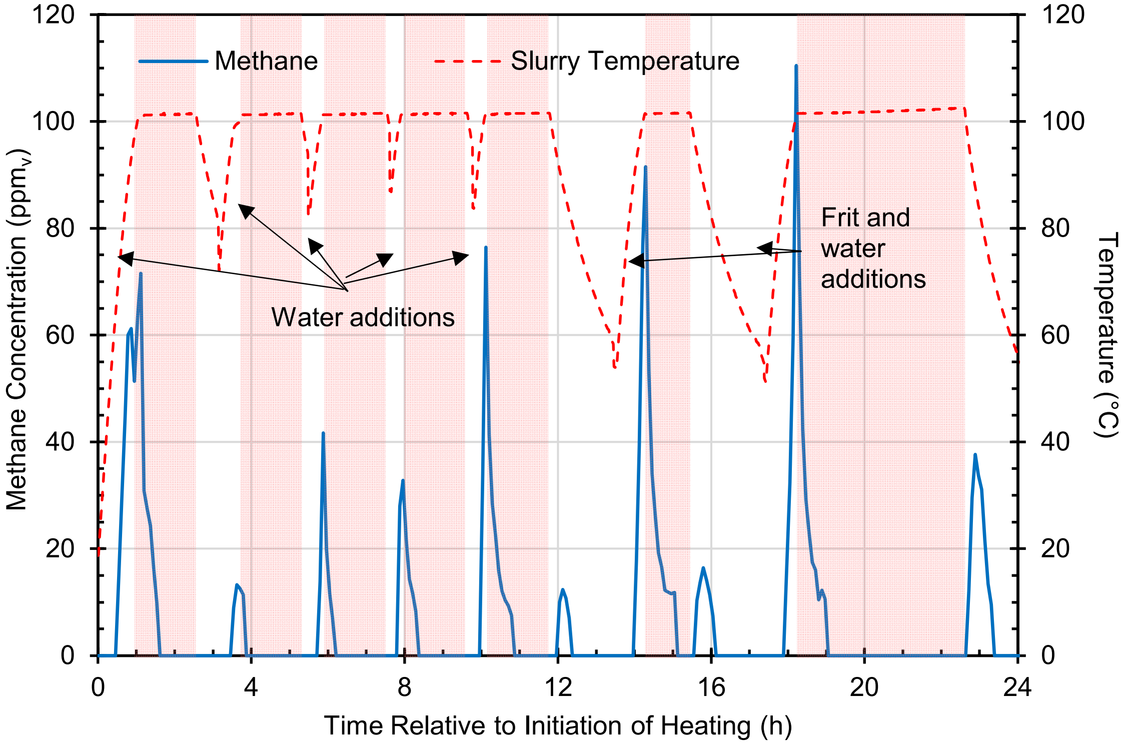

Figure C-18. SME Cycle Methane Concentration (shaded areas represent boiling)

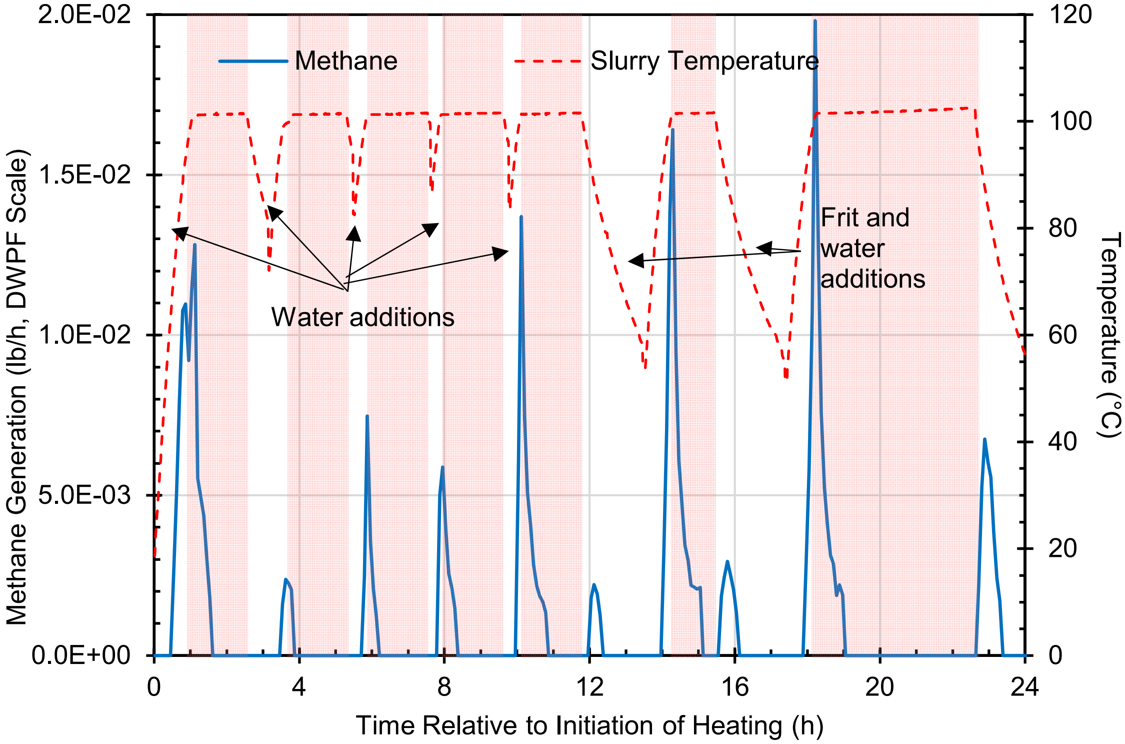

Figure C-19. SME Cycle Methane Generation (shaded areas represent boiling)

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