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Actual Waste Demonstration of the Nitric-Glycolic Flowsheet for Sludge Batch 9 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 is processable. Testing performed by the Savannah River National Laboratory has shown glycolic acid to be effective in replacing the function of formic acid in the DWPF chemical process. The nitric-glycolic flowsheet reduces mercury, significantly lowers the catalytic generation of hydrogen and ammonia which could allow purge reduction in the Sludge Receipt and Adjustment Tank (SRAT), stabilizes the pH and chemistry in the SRAT and the Slurry Mix Evaporator (SME), allows for effective rheology adjustment, and is favorable with respect to melter flammability.

In order to implement the new flowsheet, SRAT and SME cycles, designated SC-18, were performed using a Sludge Batch (SB) 9 slurry blended from SB8 Tank 40H and Tank 51H samples. The SRAT cycle involved adding nitric and glycolic acids to the sludge, refluxing to steam strip mercury, and dewatering to a targeted solids concentration. Data collected during the SRAT cycle included offgas analyses, process temperatures, heat transfer, and pH measurements. The SME cycle demonstrated the addition of glass frit and the replication of six canister decontamination additions. The demonstration concluded with dewatering to a targeted solids concentration. Data collected during the SME cycle included offgas analyses, process temperatures, heat transfer, and pH measurements. Slurry and condensate samples were collected for subsequent analysis.

Chemical Process Cell (CPC) demonstrations utilizing the alternate reductant flowsheet were performed at an acid stoichiometry of 78.0% Koopman Minimum Acid basis (87.1% Hsu basis) for the SRAT cycle. The scope specified by the technical task request has been successfully completed. Highlights of the testing results are summarized in the paragraphs below.

The total solids measurements of the SRAT and SME products were 24.6 and 49.0 weight percent (wt%), respectively, which matched the targets of 25 wt% and 48 wt% acceptably. The consistency and yield stress of the SRAT receipt material fall within the DWPF design basis. The SRAT product yield stress and consistency (0 Pa and 2.8 cP, respectively) are below the DWPF design basis. The SME product yield stress and consistency were 1.0 Pa and 16.0 cP, respectively. The SME product yield stress is below the DWPF design basis. It should be noted that similar rheological properties were encountered during SB5 processing at DWPF. DWPF successfully processed SB5 by implementing process control software to mitigate issues related to the melter feed. This software is still in use at DWPF.

Based on the comparison of the iron in the SRAT product and SME product analyses, the waste loading of the SME product is 37.6%. The current DWPF target for waste loading is 36%. Calculations based on several of the other major sludge components (Al, Ca, Mn, Ni, and U) indicated waste loadings ranging from 36.6% to 37.9% and are in good agreement with the Fe-based result. Based on the measured composition of the SC-18 glass, all of the predicted properties met the Product Composition Control System (PCCS) Measurement Acceptability Region (MAR) criteria.

In the SRAT and SME cycles, nitrite was destroyed to 304 mg/kg of SRAT product slurry and 380 mg/kg of SME product slurry. After the application of uncertainty, the measured peak concentrations of carbon

dioxide and nitrous oxide in the SRAT were 441 lb/hr and 0.73 vol%, respectively, and 34 lb/hr and 0.20 vol% in the SME, respectively. Hydrogen was not detected above the detection limit of 0.0014 lb/hr in the SRAT and the SME, which is well below the design basis limit for the formic acid flowsheet. Unlike the nitric-formic flowsheet, results from the nitric-glycolic flowsheet testing has shown there is no correlation between acid stoichiometry and hydrogen production.

The final concentration of mercury in the SRAT product was 0.71 wt% of the total solids, which is below the current target for mercury removal of 0.8 wt% of the total solids. With the additional mercury stripping during the SME cycle and the dilution of total solids with frit, the mercury concentration in the SME product dropped to 0.21 wt% of the total solids. When the SME product is put back on the same basis as the SRAT product total solids, the mercury concentration of the SME product would correspond to 0.46 wt% of SRAT total solids, near the target mercury removal of 0.45 wt% of total solids. It is expected that additional boiling during the SRAT cycle would remove mercury to levels below the target.

Antifoam 747 was added at a DWPF equivalent of 0.15 gal after nitric acid addition and 0.10 gal prior to SRAT boiling. In the SME, the DWPF equivalent of 0.25 gal of antifoam was added prior to canister decontamination water additions and frit additions. No significant foaming was observed throughout the SRAT and SME cycles.

This testing confirmed with actual waste that recommended caustic quench (CQ) preparation is acceptable for anion measurement by ion chromatography (IC) for SRAT and SME product slurries. CQ preparation also appeared to be acceptable for use with IC analysis of SRAT receipt slurry. CQ preparation successfully replicated the results of the acid digestion oxalate preparation, indicating that CQ preparation is also valid for use in oxalate measurement of SRAT receipt and SRAT and SME product slurries.

Based on the results of this actual-waste qualification and previous simulant studies, SRNL recommends implementation of the nitric-glycolic acid flowsheet in DWPF. Other recommendations resulting from this demonstration are reported in section 5.0.

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LIST OF ABBREVIATIONS

AA	Atomic Absorption
AD	Analytical Development
AR	Aqua Regia
ARG	Analytical Reference Glass
CC	Cold Chemistry
CPC	Chemical Process Cell
CVAA	Cold Vapor Atomic Absorption
DAC	Data Acquisition and Control
DI	De-ionized
DWPF	Defense Waste Processing Facility
FAVC	Formic Acid Vent Condenser
GC	Gas Chromatograph
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-AES-S	Inductively Coupled Plasma – Atomic Emission Spectroscopy - Sulfur
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
KMA	Koopman Minimum Acid
LTD	Less Than Detectable
M&TE	Measurement and Test Equipment
MA	Mixed Acid
MAR	Measurement Acceptability Region
MDL	Minimum Detection Limit
MOG	Melter Offgas
MWWT	Mercury Water Wash Tank
n	Number of replicates
NIST	National Institute of Standards and Technology
NG	Nitric/Glycolic
PCCS	Product Composition Control System
PF	Peroxide Fusion
REDOX	REDuction/OXidation
RSD	Relative Standard Deviation
SB	Sludge Batch
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory

SRR	Savannah River Remediation
SRS	Savannah River Site
SME	Slurry Mix Evaporator
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TTQAP	Task, Technical, and Quality Assurance Plan
VOA	Volatile Organic Analysis
WAC	Waste Acceptance Criteria
WAPS	Waste Acceptance Product Specification

1.0 Introduction

Savannah River Remediation (SRR) is evaluating the use of glycolic acid to replace the formic acid currently used in the Defense Waste Processing Facility (DWPF) to reduce facility hazards and improve processing times.

Testing performed by the Savannah River National Laboratory (SRNL) has shown glycolic acid to be effective in replacing the function of formic acid in the DWPF chemical process. The nitric/glycolic flowsheet reduces mercury, significantly lowers the catalytic generation of hydrogen and ammonia which could allow purge reduction in the Sludge Receipt and Adjustment Tank (SRAT), stabilizes the pH and chemistry in the SRAT and the Slurry Mix Evaporator (SME), allows for effective rheology adjustment, and is favorable with respect to melter flammability.

SRR requested the SRNL perform flowsheet testing to determine the impacts of replacing the reducing acid at DWPF [1]. Tasks were defined in a Technical Task and Quality Assurance Plan (TTQAP) [2], the main objectives of which were:

1. Use a Sludge Batch (SB) 9 sludge slurry blended from SB8 Tank 40H and Tank 51H with a comparable composition to the slurry used for the SB9 nitric-formic flowsheet demonstrations,
2. Measure and report the H_2 and N_2O concentrations for the SRAT and SME cycles,
3. Provide chemical composition data for the SRAT and SME and evaluate them for potential use in the Melter offgas (MOG) flammability evaluation. The MOG evaluation is performed and documented independently of this report, and
4. Report processing behavior and rheological properties.

In order to implement the new flowsheet, SRAT and SME cycles, designated SC-18, were performed using a SB9 slurry blended from SB8 Tank 40H and Tank 51H samples. The SRAT cycle involved adding nitric and glycolic acids to the sludge, refluxing to steam strip mercury, and dewatering to a targeted solids concentration. Data collected during the SRAT cycle included offgas analyses, process temperatures, heat transfer, mixing data, and pH measurements. Slurry and condensate samples were collected for subsequent analysis. The SME cycle demonstrated the addition of glass frit and the replication of six canister decontamination additions. The demonstration concluded with dewatering to a targeted solids concentration. Data collected during the SME cycle included offgas analyses, process temperatures, heat transfer, mixing data, and pH measurements. Slurry and condensate samples were collected for subsequent analysis. Data collected from the testing were compared to applicable metrics outlined in the DWPF Waste Acceptance Criteria (WAC) [3].

2.0 Experimental Procedure

2.1 Sample Analytical Details

2.1.1 Sampling

Slurry and condensate samples were collected throughout the SRAT cycle at strategic intervals to investigate process chemistry according to Table 2-1. Samples collected for mercury, caustic quench, and supernatant/filtrate were slurry samples. The slurry samples were collected through a 3/8 inch (outer diameter) glass sample tube connected to a sample bottle via polyethylene tubing. A stopper was fitted to the sample bottle through which a vacuum was created using a Masterflex pump. The vacuum inside the sample bottle pulled the sample through the sample tube until a stopcock on the glass sample tube was closed. Prior to sampling, the glass sample tube was flushed with air, and new polyethylene tubing was used for each sample. Condensate/Dewater samples were collected from the mercury water wash tank. Of the samples listed in Table 2-1, this report contains the results of the End of SRAT samples and Condensate/Dewater samples; other samples are covered under a separate TTR [4] and will be reported separately.

Table 2-1: Sample Schedule for SRAT Cycle

Process Step	Hg Sample	Slurry Caustic Quench	Supernatant/Filtrate	Condensate/Dewater
Post Nitric Acid Addition				✓
Mid Glycolic Acid Addition		✓	✓	
Post Glycolic Acid Addition	✓	✓	✓	
Heat to boiling				✓
1.5 hr into Dewater		✓	✓	
3.0 hr into Dewater		✓	✓	
End of Dewater	✓			✓
5.0 hr into Reflux		✓	✓	
8.0 hr into Reflux	✓			
10.0 hr into Reflux		✓	✓	
End of Reflux				✓
End of SRAT	✓	✓	✓	✓

2.1.2 Density

Density measurements on the Tank 40 slurry and supernatant were conducted at a temperature of approximately 19 °C; density measurements on the SRAT receipt slurry and supernatant were conducted at a temperature of 26 °C. The SRAT product slurry and supernatant densities were measured at 26 °C. The SME product slurry and supernatant densities were measured at 25 and 23 °C, respectively. These temperatures were governed by the Shielded Cells conditions at the time of the measurements. Densities were measured using weight-calibrated balances and 8-9 mL volume-calibrated plastic test tubes. Multiple individual slurry aliquots and individual supernatant aliquots were utilized in the measurements. Supernatant was generated as a filtrate by passing slurry through a 0.45 μm filtration membrane (note that this generation method was utilized for all of the supernatant analyses – not just those used for determining density). The density of a deionized water standard was determined along with the slurry and supernatant determinations to demonstrate measurement accuracy.

2.1.3 Solids Distribution

Total solids and dissolved solids determinations were performed by evaporating water from slurry and supernatant aliquots (respectively) at a nominal temperature of 110 °C until a steady-state weight was achieved. Four individual slurry aliquots and four individual supernatant aliquots were utilized in the measurements. The mass of each aliquot was approximately 3.0 g. Insoluble and soluble solids concentrations were calculated based on the total solids and dissolved solids measurements [5]. Calcined solids were then generated by heating the dried slurry aliquots (from the total solids measurements) to a temperature of 1100 °C.

2.1.4 Elemental Analysis of Slurry Solids

In preparation for elemental analyses (prior to submittal), four slurry aliquots were digested by the aqua regia (AR) method and four slurry aliquots were digested by the peroxide fusion (PF) method. Note that the AR method utilized a sealed vessel to prevent loss of volatile constituents. Applicable blanks were also processed through the digestion methods, and multi-element standards were submitted along with the digest solutions, where applicable, for quality assurance purposes. The total solids mass of each sample aliquot was ~0.25 g, and the volume of each final digest solution was 100 mL.

The PF method was performed by weighing approximately 1.5 g of sludge into a zirconium crucible followed by evaporation of the water at 115 °C in a drying oven. Approximately 2.5 g of sodium peroxide were added to the dry sample inside the crucible which was then placed inside a muffle furnace pre-heated to 675 °C. The crucible was heated for 15 minutes after the temperature equilibrated. The crucible was then removed from the furnace and allowed to cool for 10 minutes before dissolving the fusion residue with de-ionized water and concentrated HNO_3. The solution was diluted to 100 mL in a plastic volumetric flask. No solids were apparent after the PF method. Fifteen milliliter aliquots of solution were removed from the shielded cells and transferred to the applicable Analytical Development (AD) laboratories. A blank and Analytical Reference Glass (ARG) standard were prepared concurrently with the SRAT samples.

The AR method was performed by weighing approximately 1.5 g of sludge into a thick-walled Teflon pressure vessel and then adding 9 mL of concentrated HCl and 3 mL of concentrated HNO_3. The Teflon pressure vessel was capped and sealed with a mechanical capping station. After all the samples, blanks, and standard glass samples had been prepared, the vessels were placed in a drying oven pre-heated to 115 °C. The vessels were heated for two hours after the oven temperature equilibrated to 115 °C. After cooling for 30 minutes, the Teflon pressure vessel was opened and the solution inside completely transferred to a 100 mL plastic volumetric flask. The flask was filled to the mark with de-ionized water to achieve the dissolution volume of 100 mL. The AR dissolutions appeared clear when observed through the shielded cell window. 15 mL aliquots of the solutions were removed from the shielded cells for transport to applicable AD laboratories. A blank and ARG standard were prepared concurrently with the SRAT samples.

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was performed on both the AR and PF digest solutions along with the applicable blanks and a multi-element standard solution for quality assurance purposes. The ICP-AES measurements provided quantification of most of the elemental constituents reported in this document. The ICP-AES axial sulfur method (ICP-AES-S) was performed on the AR digest solutions for quantifying sulfur. Cold vapor atomic absorption (CVAA) spectroscopy was performed on the AR digest solutions (along with the AR blank) for the purpose of quantifying mercury. Atomic absorption (AA) spectroscopy was performed on the AR digest solutions (along with the blank) for the purpose of quantifying arsenic and selenium. Inductively coupled plasma mass spectrometry (ICP-MS) was performed on the AR digest solutions (along with the AR blank) for the purpose of quantifying neodymium, palladium, rhodium, ruthenium, and silver. Dilution-correction of the results was performed by AD prior to reporting. Elemental analyses were performed on both the Tank 40 slurry, the SRAT receipt, the SRAT product, and the SME product.

The elemental results determined through ICP-AES analyses were based either solely on the AR digest solutions, solely on the PF digest solutions, or on both the AR and PF digest solutions, depending on the following factors: potential for interference, magnitude of "blank values," magnitude of minimum detection limits, consistency of data, and apparent anomalies. Applicable digestion method(s) feeding the results is identified in the table providing the results.

In the case of the SRAT receipt slurry, the integrities of the fourth aqua regia digest solution (AR4) and the first peroxide fusion digest solution (PF1) were compromised, as determined through the quality assurance measures. In the case of the SRAT product slurry, the integrity of the fourth aqua regia digest solution (AR4) was also compromised. As such, the ICP-AES, IPC-AES-S, and ICP-MS results associated with these digest solutions were excluded from reporting.

The elemental results determined through ICP-MS analyses were based on sums of specific isotope results. For neodymium, the isotope results that were summed were those associated with mass numbers 143-146, 148, and 150. For rhodium, the result was based solely on the isotope associated with mass number 103. For ruthenium, the isotope results that were summed were those associated with mass numbers 101, 102, and 104. For palladium and silver, the results were calculated utilizing fission yields and measurements based on mass numbers 105-110, per the method documented by Bibler in 2005 [6].

2.1.5 Elemental Analysis of Supernatant

In preparation for the elemental analyses (prior to submittal), four supernatant aliquots were each diluted by a factor of ~26 (on a volume basis), using 0.5 M HNO_3. Use of the 0.5 M HNO_3 diluent resulted in a final solution pH of 1-2, which was considered beneficial for minimizing loss of constituents through sorption to the walls of the sample submittal vessels and through potential precipitation reactions. An applicable "acid blank" and a multi-element standard were submitted along with the acidified/diluted supernatant for quality assurance purposes.

ICP-AES, ICP-AES-S, CVAA, and ICP-MS were performed on the acidified/diluted supernatant aliquots to quantify routine elemental constituents; sulfur; mercury, and palladium, rhodium, ruthenium, silver, and uranium. Note that prior to the supernatant mercury measurements, AD performed permanganatepersulfate digestions on the acidified/diluted sample aliquots. For palladium, rhodium, ruthenium, and silver, the results were calculated utilizing measurements based on mass numbers 101-110, per the method documented by Bibler [6]. For uranium, the results were calculated by summing the measurements of mass numbers 233-236 and 238. Dilution-correction of all results was performed prior to reporting. Supernatant elemental analyses were performed on the SRAT receipt, the SRAT product, and the SME product. Supernatant elemental analyses were not performed on the sample HTF-40-13-124.

2.1.6 Anions in the Slurry

In preparation for the anion analyses (prior to submittal), four slurry aliquots were each diluted by a factor of 25 to 50 (on a volume basis), using de-ionized water. The diluted slurry aliquots were agitated for a minimum of 30 seconds, and then passed through a 0.45 µm filtration membrane, to remove insoluble solids. An applicable de-ionized water blank was also prepared.

Ion chromatography (IC) was performed on the filtrate aliquots, to quantify bromide, chloride, fluoride, formate, glycolate, nitrate, nitrite, oxalate, phosphate, and sulfate. Dilution-correction of the results was performed prior to reporting. This method was utilized to quantify anions in both the SB9 Alternate Reductant blend slurry and the Tank 40 slurry.

Two additional methods were used to prepare the SB9 Alternate Reductant blend slurry for IC analyses – the caustic quench (CQ) method and the acid digestion method.

The CQ method was developed as an IC preparation for acidic slurries to stop oxidation reactions, to free up chelated organic acids by converting chelated metal compounds to metal oxides, and to adjust the pH of the sample to match the IC eluent. The CQ preparation and analysis were performed as described in the test report [7]. In the CQ method, ~2 g of 50 wt% NaOH solution was mixed with ~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 were run on the IC between each analytical sample. For the SRAT receipt analysis, a slight variation was performed on the CQ preparation method where all four 1 mL aliquots of a single CQ mixture were each diluted to 100 mL with de-ionized water.

In the acid digestion (oxalate preparation) method, ~2 mL of concentrated HCl and ~2 mL of concentrated HNO_3 were mixed with ~1 g of slurry, and the mixture was then diluted to 250 mL using de-ionized water. This was repeated four times to produce four different acid digested aliquots. These aliquots, along with an acid digestion blank, were analyzed by IC for oxalate. The advantage of the acid digestion approach is that it dissolves oxalate present in the form of the calcium salt, whereas the standard water dilution and caustic quench methods do not. A disadvantage of the acid digestion method is that the acids can destroy oxalate over time and as such, the analyses must be performed as soon as possible following digestion to minimize analytical bias associated with oxalate destruction.

2.1.7 Anions in the Supernatant

In preparation for the anion analyses (prior to submittal), four supernatant aliquots were each diluted by a factor of ~26 (on a volume basis), using de-ionized (DI) water.

In the case of the SRAT receipt, the diluted supernatant aliquots were analyzed by: a) IC to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, and sulfate; b) total inorganic carbon (TIC) to quantify carbonate; c) base titration to quantify free hydroxide; and d) ICP-AES to quantify aluminate, under the assumption that 100% of the aluminum was present as aluminate. For the SRAT product and SME product, the diluted supernate aliquots were analyzed by a) IC to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, sulfate, and glycolate and b) TIC to quantify bicarbonate. Additionally for the SRAT and SME product supernatants, CQ preparation (without filtration of the diluted preparations) and IC analysis were performed. In the case of sample HTF-40-13-124, the diluted supernatant aliquots were analyzed solely by base titration to quantify free hydroxide. Dilution-correction of the results was performed prior to reporting.

2.1.8 Select Radioisotopes in the Slurry Solids

The same AR and PF digestion methods that were used for the slurry elemental analyses were utilized for preparing the slurry aliquots for the select radioisotope analyses (four slurry aliquots digested by AR, four slurry aliquots digested by PF, plus an AR blank and a PF blank for quality assurance purposes). ICP-MS was performed on the AR digest solutions to quantify Tc-99, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-239, and Pu-240. Radiometric (counting) methods were performed on the PF digest solutions to quantify Pu-238, Pu-239/240, Pu-241, Am-241, Am-242m, Am-243, Cm-242, Cm-244, and Cm-245. Dilution-correction of the results was performed prior to reporting. Note that the radioisotope analyses were limited to the SB9 Alternate Reductant blend case (radioisotope analyses were not performed on the Tank 40 sample).

As previously identified, the integrities of the fourth aqua regia digest solution (AR4) and the first peroxide fusion digest solution (PF1) were compromised, as determined through the quality assurance measures. As such, the ICP-MS and radiometric results associated with these digest solutions were excluded from reporting.

2.1.9 Select Radioisotopes in the Supernatant

The same acid dilution method that was used for the supernatant elemental analyses was utilized for preparing the supernatant aliquots for the select radioisotope analyses (four supernatant aliquots acidified and diluted using 0.5 M nitric acid plus a 0.5 M acid blank for quality assurance purposes). ICP-MS and radiometric methods were performed to quantify the same series of radioisotopes that was determined for the slurry solids. Dilution-correction of the results was performed prior to reporting. As in the case of the slurry solids, the supernatant radioisotope analyses were performed for the SRAT receipt, SRAT product, and SME product (radioisotope analyses were not performed on sample HTF-40-13-124).

2.1.10 TIC, Total Organic Carbon (TOC), and Volatile Organics in the Slurry

The same water dilution method that was used for the slurry anion analyses was utilized for preparing the slurry aliquots for the TIC/TOC and volatile organic analyses. However, in contrast to the preparation approach for the slurry anion analyses, **no** filtration of the diluted slurry was performed prior to submitting the diluted slurry aliquots for the TIC/TOC and volatile organic analyses. This was done to assure that all insoluble and soluble compounds would be measured. Dilution-correction of the results was performed prior to reporting. Note that the TIC/TOC analyses were performed on the SRAT receipt slurry, SRAT product slurry, SME product slurry, and the Tank 40 slurry – however, the volatile organic analysis (VOA) was performed solely on the SRAT receipt slurry and the SME product slurry.

2.1.11 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 analytical accuracy. In general, the one sigma analytical uncertainty reported by Analytical Development 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-AES, ICP-AES-S, ICP-MS, and TIC/TOC analyses; and b) ~20% for CVAA analyses and VOA; and c) highly variable for radiometric (counting) methods, but often in the 20-30% range. As such, only one to two of the leading digits reported for the AD analysis results should be considered significant.

2.1.12 Assessment of the Results

Multiple approaches were used to assess the validity of the analytical data being reported. The primary goal of this was to demonstrate that the reported results were both reasonable and consistent with expectations. Discussion of the assessment approaches and results is included in Section 3.0. Note that when characterization results were compared, percent differences were calculated as follows:

% Difference = 100 × [(absolute value of the difference between results) ÷ (the average result)]

2.1.13 DWPF Analytical Methods

The sludge samples were dissolved in quadruplicate in the SRNL Shielded Cells facility in a manner similar to the DWPF Cold Chemistry (CC) method, and by PF and AR digestion methods. The CC method digestion (see DWPF Procedure SW4-15.201) was performed by adding 25 mL of concentrated HF to 2.7-3.0 g of the sludge and stirring this mixture for 1 hour. This step was followed by addition of 25 mL of concentrated HNO_3 and this mixture was stirred for an additional 30 minutes. Fine, dark, slow-settling solids remained in each digestion bottle after the acid additions and stirring. Each sample was then diluted with DI water to 250 mL in a plastic volumetric flask. The mixture was suspended just prior to taking a 15 mL aliquot for removal from the shielded cells and transport to the AD ICP-AES and AA laboratories.

Dissolution blanks for each of the methods were created by concurrently performing each of the dissolutions methods with only the reagents.

For mercury, the DWPF method consisted of weighing 10.0 ± 0.5 g of sludge into the bottom of a Teflon pressure vessel followed by dropwise addition of 5 mL concentrated H_2SO_4. When it was apparent that no additional reaction with the acid was taking place, 2.5 mL of concentrated HNO_3 was added. Fifteen milliliters of 0.4 M $KMnO_4$ was added and the Teflon pressure vessel set aside for 15 minutes to allow any reaction to proceed. Ten milliliters of $Na_2S_2O_8$ was then added and the Teflon pressure vessel was capped and placed in an oven that had been pre-heated to 95 °C. After heating for two hours, the vessels were cooled for 30 minutes. Fifteen milliliters of 3.6 M sodium chloride-hydroxylamine hydrochloride was added to ensure that the purple color from $KMnO_4$ had dissipated. Five milliliters concentrated HNO_3 was added. The solution was diluted to 250 mL in a plastic volumetric flask and an aliquot of this solution removed from the cells for analysis.

The ARG was dissolved concurrently with the SRAT samples and the blanks. Glass is not an ideal standard to validate a dissolution method for sludge since glass and sludge have much different physical properties. However, the ARG analysis does provide information on the overall process of weighing samples in the cells, carrying out the dissolution, and elemental analysis by ICP-AES of a material with similar elemental composition to sludge.

A multi-element standard solution that did not go through the dissolution process was also analyzed concurrently with each set of SRAT elemental analyses.

2.2 SRAT Receipt Preparation

The SB9 SRAT receipt for the alternate reductant qualification testing was prepared by blending slurry samples from Tank 51H and Tank 40H, HTF-51-15-130 and HTF-40-13-124, respectively. The same blending strategy used for the SC-17 nitric/formic qualification was implemented for the alternate reductant qualification testing [8]; sample blending was performed on an insoluble solids basis targeting a ratio of 56:44, Tank 40H:Tank 51H.

2.3 Process Equipment

SRAT and SME cycles were performed in a 4-L glass vessel with a stainless steel lid. Both SRAT and SME cycles utilized the same equipment. Heating was provided using two 0.5 inch diameter stainless steel heating rods powered by an automated direct current power supply (TDL 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 Model 647 Multi Gas Controller. Values for pH were acquired using a Mettler-Toledo probe coupled to a Thermo Scientific Orion Star™ pH meter.

A SRAT condenser/mercury water wash tank (MWWT) combination that allowed the condensate to drip vertically into the MWWT at a point below the gas-liquid interface inside the MWWT was used during testing. During the SRAT cycle, the MWWT was used to reflux condensate back to the SRAT vessel, whereas during the SME it was used as a decanter. Offgas exiting the condenser passed through a vapor trap (i.e. cold finger) cooled by an aluminum block mounted on a Torrey Pines Scientific electronic ice cube before continuing to the gas chromatograph (GC). An ammonia scrubber was not used, as it has no impact on the SRAT and SME chemistry being investigated during these tests.

Acids were metered into the 4-L vessel through an injection tube below the surface of the slurry using a Series R valveless piston pump head (manufactured by Fluid Metering, Inc.) mounted to a Masterflex Series 77000 pump.

The SRAT condenser was cooled 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. Flow rate was measured using a direct read rotameter.

The experiment had a dedicated Agilent 3000A dual column micro GC. Column-A collected data related to He, H_2, O_2, and N_2, while column-B collected data related to CO_2, and N_2O. Due to low GC sensitivity, other oxides of nitrogen and carbon were not analyzed. The GCs were calibrated with a standard calibration gas containing He, H_2, O_2, N_2, CO_2 and N_2O. The calibration was verified prior to starting the SRAT cycle and after completing the SME cycle. Raw chromatographic data were acquired by the GC from the offgas stream samples using a separate computer interfaced to the data acquisition computer. Sampling frequency was approximately one chromatogram every four minutes.

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.

A schematic depicting the equipment used during flowsheet testing in the shielded cells is presented in Figure 2-1, and a photograph of the equipment in the shielded cells is presented in Figure 2-2.

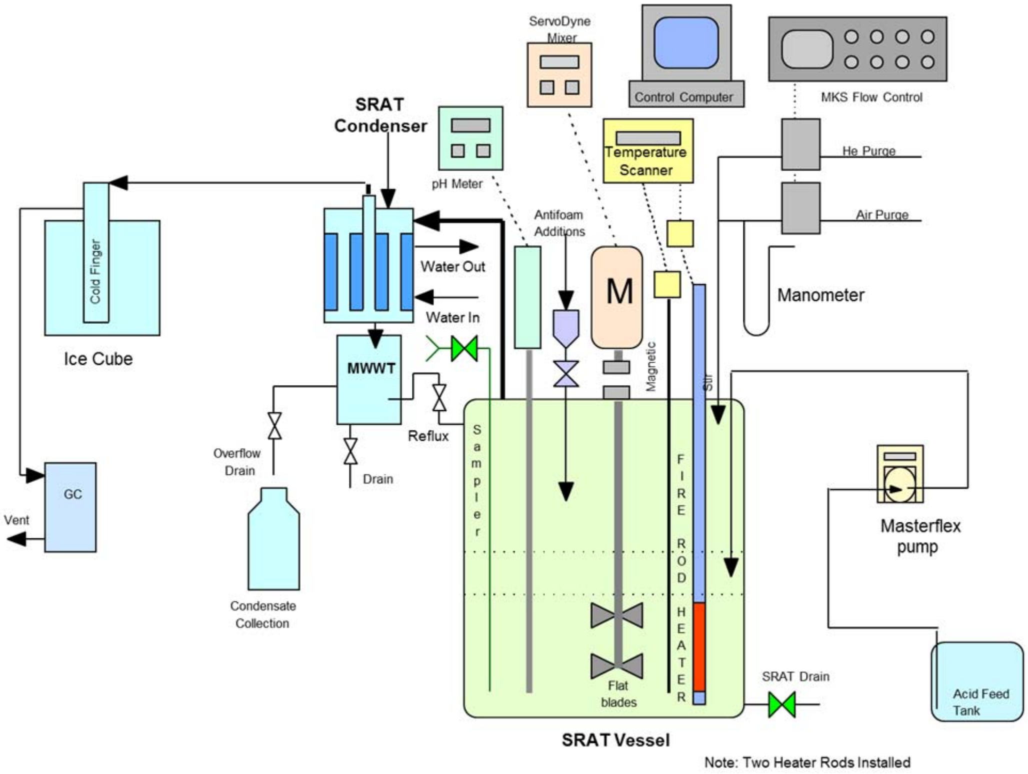

Figure 2-1: Shielded Cells 4-L Process Equipment

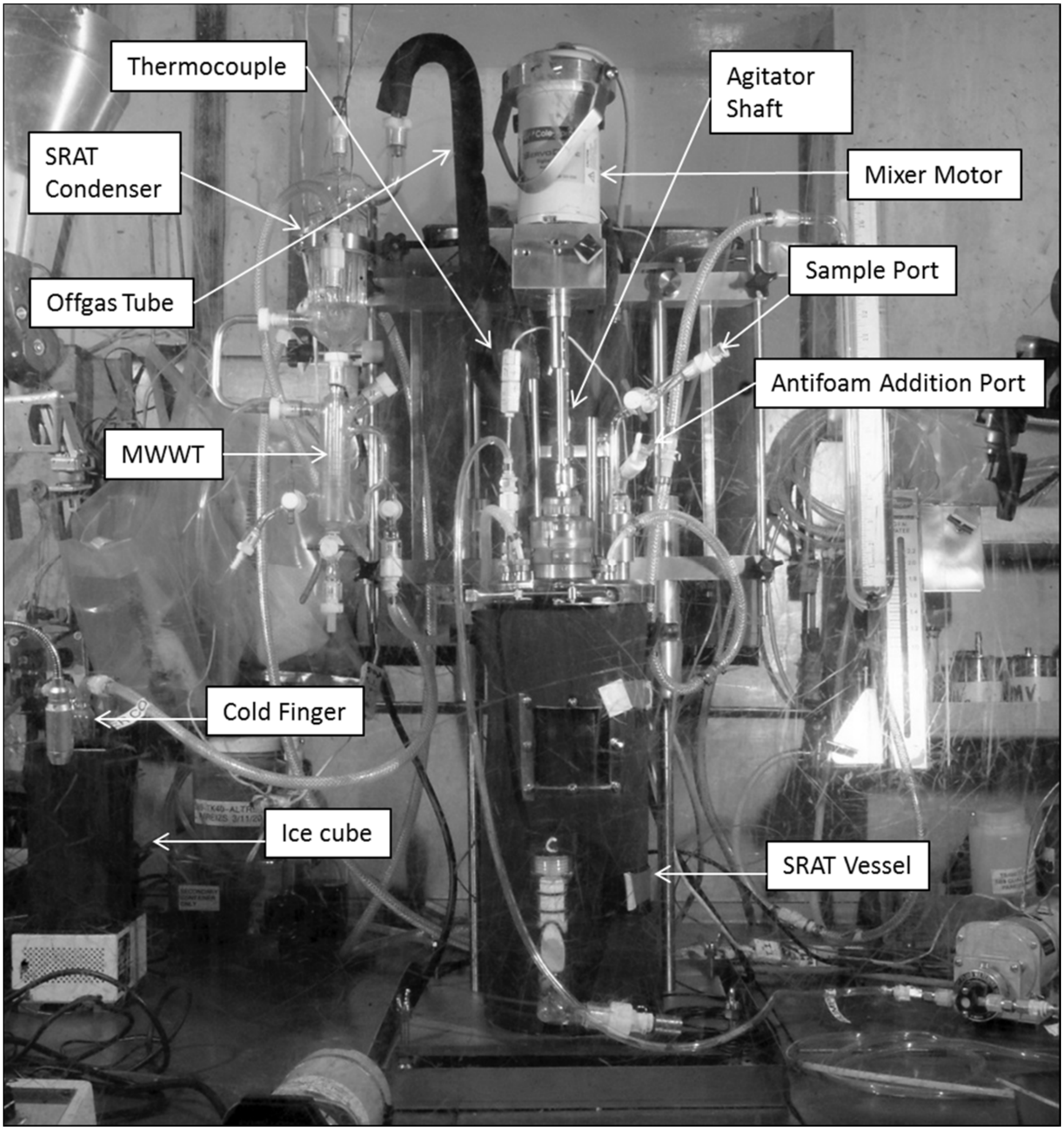

Figure 2-2: Photograph of 4-L CPC Equipment in A Block Shielded Cell 2

2.4 Glass Fabrication and Compositional Analysis

In preparation for glass fabrication, approximately 120 g of SB9 SC-18 SME product was divided into four nearly equal portions, placed into high-purity alumina crucibles, and dried overnight at 110 °C. After thorough drying, the first portion was transferred to an open Pt/Au crucible and rapidly heated to 1150 °C in an electrically heated furnace. Upon reaching 1150 °C, the sample was held at temperature for approximately 30 minutes. The remaining portions were then added incrementally, allowing the crucible to rapidly return to temperature between each addition, resulting in a total time at the melting temperature of 1150 °C of 3.5 hours. The crucible was removed from the furnace while at temperature and bottom quenched (cooled) in a shallow pan of water, making sure no water contacted the glass during the process. The resulting glass appeared dark and shiny, without the presence of a visible salt layer or crystals present.

To support compositional analysis, a portion of the SB9 SC-18 Qualification Glass had to be dissolved. In order to enhance dissolution, approximately 4 g of the glass was crushed and ground using agate cups, balls and caps in a mechanical pulverizing mixer mill. The glass was sieved and only the portion that passed through a 200 mesh (<75 µm) brass sieve was used for the dissolutions. Four replicate samples (nominally 0.25 g each) of the sieved glass were dissolved remotely by two different methods to ensure that all the elements of interest were dissolved and could be analyzed in at least one of the preparations. The two methods were a sodium peroxide fusion [9] at 675 °C followed by a HNO_3 uptake, and a mixed acid dissolution (MA) [10] in sealed vessels at 115 °C using a combination of HF, HCl, and HNO_3 acids. Boric acid was added to this latter dissolution method to complex excess fluoride. Concurrent with each set of dissolutions in the Shielded Cells, three samples of the ARG [11] were also dissolved to confirm complete dissolution of the SB9 SC-18 Qualification glass. The solutions of the dissolved glass were diluted to known volumes so that approximately 15 mL aliquots could be safely removed from the Shielded Cells without exposing personnel to excess radiation. The aliquots from both dissolution methods were then submitted to AD where they were analyzed by ICP-AES and by ICP-MS. A multi-element standard containing known concentrations of Al, B, Fe, K, Li, Na, and Si was also submitted with each set of samples sent to AD to confirm the accuracy of the measurements[1].

2.5 Rheology

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

The shear rate Newtonian fluid is geometrically determined using the equations of change (continuity and motion). This assumption also presumes that the velocity distribution is fully developed and that the flow is in the laminar regime. The shear rate can be calculated for non-Newtonian fluids using the measured data and fitting these data to the rheological model or corrected as recommended by Darby [12]. In either case, for shear thinning non-Newtonian fluids typical of Savannah River Site (SRS) sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a mathematically thinner fluid. Correcting the flow curves will not be performed in this task, resulting in calculations giving slightly more viscous fluid parameters.

The bob typically used for measuring tank sludge or SRAT product is the MV I rotor. For SME product, the MV II rotor is used to perform the measurements, due to the larger frit particles that are present in the

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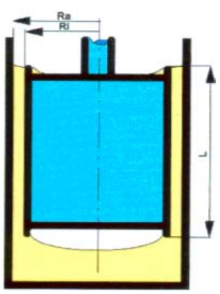
[1] ICP multi-element custom solution, product number SM-744-013, High Purity Standards, Charleston, SC.

SME product. The MV II has a larger gap to accommodate the larger frit particles. The shape, dimensions, and geometric constants for the MV I and MV II rotors are provided in Table 2-2.

Table 2-2: 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

Prior to the measurements being performed, the rotors and cups are inspected for physical damage. The torque/speed sensors and temperature bath are verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard, using the MV I rotor. The resulting flow curves are then fitted as a Newtonian fluid and this calculated viscosity must be within ± 10% of the reported NIST viscosity at a given temperature for the system to be considered functionally operable. An N10 oil standard was used to verify system operability prior to the sludge measurements.

The flow curves for the sludge were fitted to the down curves using the Bingham Plastic rheological model, Equation 2-1, where τ is the measured stress (Pa), τ_o is the Bingham Plastic yield stress (Pa), μ_∞ is the plastic viscosity (Pa·sec), and $\dot{\gamma}$ is the measured shear rate (sec^{-1}). During all these measurements, the sample remained in the cup for the 2nd measurement, due to the limited sample availability.

Equation 2-1:

$$\tau = \tau_o + \mu_\infty \dot{\gamma}$$

2.6 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

3.1 Characterization of Washed Tank 51 Sample

A sample of washed Tank 51H slurry (HTF-51-15-130) was received by SRNL for characterization and preparation of the SB9 Alternate Reductant blend for the qualification test. The sample was taken after in-tank washing of Tank 51H slurry for SB9 preparation had been completed. Analytical results of the slurry have been previously reported [13, 14]. Key results from the reported analyses are reproduced below in Table 3-1 through Table 3-5.

Table 3-1: Densities and Solids Contents of the Washed Tank 51 Sample

Measurement	Result	%RSD, *n*
Slurry density, g/mL	1.14	0.7, 4
Supernatant density, g/mL	1.05	0.3, 4
Total solids, wt% of slurry	18.9	0.7, 4
Insoluble solids, wt% of slurry	13.7	N/A
Soluble solids, wt% of slurry	5.2	N/A
Calcined solids, wt% of slurry	14.7	1.2, 3
Dissolved solids, wt% of supernatant	6.0	0.4, 4

Table 3-2: Elemental Analysis of the Washed Tank 51 Slurry Solids (Shading Indicates Concentrations > 0.1 wt%)

Constituent	Digestion Method(s)	Concentration, wt% solids	%RSD, *n*
Ag	AR	7.02E-03	2.6, 4
Al	AR & PF	7.00E+00	4.0, 7
B	AR	6.80E-03	25, 4
Ba	AR & PF	5.76E-02	3.6, 7
Be	AR	3.15E-04	1.3, 4
Ca	AR	1.04E+00	1.2, 4
Cd	AR	1.51E-02	3.1, 4
Ce	AR	< 2.8E-03	N/A
Co	AR	7.16E-03	1.5, 4
Cr	AR	7.34E-02	0.9, 4
Cu	AR	3.87E-02	0.7, 4
Fe	AR & PF	1.70E+01	3.8, 7
Gd	AR & PF	7.20E-02	7.8, 7
Hg	AR	2.60E+00	3.8, 4
K	AR	6.52E-02	14, 4
La	AR	2.39E-02	0.7, 4
Li	AR	9.17E-02	1.5, 4
Mg	AR	2.16E-01	0.4, 4
Mn	AR & PF	5.73E+00	5.0, 7
Mo	AR	1.10E-02	1.4, 4
Na	AR	1.27E+01	2.9, 4
Nd	AR	1.01E-01	0.8, 4
Ni	AR & PF	7.42E-01	6.2, 7
P	AR	1.66E-01	1.0, 4
Pb	AR	3.49E-02	6.0, 4
Pd	AR	1.62E-03	21, 4
Rh	AR	8.66E-03	1.0, 4
Ru	AR	3.99E-02	1.9, 4
S	AR	1.64E-01	4.7, 4
Sb	AR	< 2.1E-02	N/A
Si	PF	1.59E+00	4.1, 3
Sn	AR	< 1.3E-02	N/A
Sr	AR	2.17E-02	0.4, 4
Th	AR & PF	8.39E-01	8.0, 7
Ti	AR	3.23E-02	0.5, 4
U	AR & PF	2.63E+00	6.5, 7
V	AR	< 4.6E-04	N/A
Zn	AR & PF	2.98E-02	4.2, 7
Zr	AR	2.28E-02	7.0, 4

Table 3-3: Elemental Analysis of the Washed Tank 51 Supernatant

Constituent	Concentration, mg/L	%RSD, *n*
Ag	< 6.0E-01	N/A
Al	1.44E+03	0.4, 4
B	1.37E+01	6.9, 4
Ba	< 1.8E-01	N/A
Be	< 5.4E-02	N/A
Ca	7.46E+00	35, 4
Cd	< 8.2E-01	N/A
Ce	< 2.4E+00	N/A
Co	< 6.4E-01	N/A
Cr	< 9.3E-01	N/A
Cu	< 6.1E-01[a]	N/A
Fe	1.30E+00	20, 4
Gd	< 3.1E+00	N/A
Hg	3.88E+01	14, 4
K	9.80E+01	2.1, 4
La	< 3.7E-01	N/A
Li	< 4.5E+00	N/A
Mg	4.66E-01	88, 4
Mn	1.00E-01[b]	3.6, 2
Mo	< 1.8E+00	N/A
Na	2.20E+04	0.5, 4
Ni	< 5.7E+00	N/A
P	< 8.7E+00	N/A
Pb	< 8.8E+00	N/A
S	2.74E+02	0.8, 4
Sb	< 1.8E+01	N/A
Si	< 3.5E+00[c]	N/A
Sn	< 1.1E+01	N/A
Sr	< 5.4E-02	N/A
Th	< 3.2E+00	N/A
Ti	< 8.7E+00	N/A
U	< 1.7E+01	N/A
V	< 3.6E-01	N/A
Zn	5.68E-01	59, 4
Zr	< 5.3E-01	N/A

[a] The Cu result in the table is based on three measurements which were less than the minimum detection limit (MDL). The fourth measurement was a detectable concentration (0.73 mg/L) that exceeded the MDL identified in the table.

[b] The Mn result in the table is based on two measurements which exceeded the MDL. The other two Mn measurements were lower than the MDL and lower than the result in the table (their concentrations were <0.075 mg/L).

[c] The Si result in the table is based on three measurements which were less than the MDL. The fourth measurement was a detectable concentration (4.4 mg/L) that exceeded the MDL identified in the table.

Table 3-4: Anions in the Washed Tank 51 Supernatant (*n* = 4)

Anion	Concentration, M	%RSD
Aluminate	5.32E-02	0.4
Bromide	<1.6E-02	N/A
Carbonate	5.44E-02	3.4
Chloride	<7.2E-03	N/A
Fluoride	<1.3E-02	N/A
Formate	<5.7E-03	N/A
Free hydroxide	2.52E-01	6.6
Nitrate	1.40E-01	1.5
Nitrite	3.42E-01	1.4
Oxalate	5.27E-02	1.2
Phosphate	<2.7E-03	N/A
Sulfate	6.86E-03	2.6

Table 3-5: Select Radioisotopes in Supernatant (*n* = 4)

Isotope	Concentration, Ci/gal supernatant	%RSD
Tc-99	3.11E-05	1.6
Cs-137	2.47E-01	3.3
Ba-137m	2.34E-01	3.3
U-235	2.08E-10	1.7
U-238	2.05E-09	9.0

3.2 Characterization of the Tank 40 Sample (HTF-40-13-124)

Three nominally 3-L samples of SB8 slurry were taken from Tank 40H in July 2013 and shipped to SRNL [15]. Based on a measurement of solids content, samples HTF-40-13-119 and HTF-40-13-124 were seen to be similar to each other and in line with the SB8 projection. Sample HTF-40-13-119 was used in the Waste Acceptance Product Specifications (WAPS) analysis and received extensive characterization. Subsequently, sample HTF-40-13-119 was used to represent SB8 Tank 40H in the SB9 blends for the nitric-formic qualification [8]. Because only solids content analysis was previously performed on sample HTF-40-13-124 that was to be used in the SB9 blend for the nitric-glycolic qualification, a reduced set of analysis was performed on HTF-40-13-124 to show how closely analogous it is to the well characterized sample HTF-40-13-119.

Densities and solids contents of sample HTF-40-13-124 are given in Table 3-6. As shown in the table, the slurry and supernatant densities are 1.11 and 1.05 g/mL, respectively, and the weight percent total solids, insoluble solids, and calcined solids are 17.1, 11.4, and 13.7, respectively. The reanalysis of solids content are consistent with the analysis performed just after sample HTF-40-13-124 was received by SRNL. These values are all relatively consistent with those of the SRAT receipt SB9 Alternate Reductant blend shown in Section 3.4, with the Tank 40H values being 0-7% lower than those of the blend. These minor differences reflect the slightly higher solids content of the blend.

Table 3-6: Densities and Solids Contents of the Tank 40 Slurry Sample HTF-40-13-124

Measurement	Previous Analysis [15]		Current Reanalysis	
	Result	%RSD, *n*	Result	%RSD, *n*
Slurry density, g/mL	NA	NA	1.11	0.2, 4
Supernatant density, g/mL	NA	NA	1.05	0.4, 4
Total solids, wt% of slurry	17.2	0.2, 3	17.1	0.9, 4
Insoluble solids, wt% of slurry	11.4	NA	11.4	NA
Soluble solids, wt% of slurry	5.80	NA	5.7	NA
Calcined solids, wt% of slurry	NA	NA	13.7	4.7, 4
Dissolved solids, wt% of supernatant	6.54	1.2, 3	6.4	0.3, 4

Concentrations of the elemental constituents in the Tank 40H slurry solids are given in Table 3-7, with results greater than 0.1 wt% shaded for easy identification. As shown in the table, the most dominant constituents in the solids include iron, sodium, aluminum, manganese, uranium, nickel and mercury, with concentrations ranging from approximately 1-17 wt% of the solids (iron has the highest concentration and mercury has the lowest concentration).

Most of the RSDs for the elemental analyses were limited to about ten percent or less, demonstrating typical analytical precision. In contrast, the RSDs applicable to boron, chromium, gadolinium, phosphorus, and zirconium were higher, ranging from 12 to 38%. These higher RSDs give an indication that the propagated analytical uncertainties associated with these five constituents are likely higher than those of the other constituents.

Concentrations of anions, TIC, and TOC in the slurry are given in Table 3-8. As shown in table, the dominant anions included nitrite and nitrate, at concentrations of ~12,000 mg/kg and 6,800 mg/kg, respectively. Oxalate was present at ~1,500 mg/kg, sulfate was present at ~1,400 mg/kg, TIC was present at ~1,300 mg/kg, and TOC was present at ~500 mg/kg (which compares well to the oxalate carbon present at 400 mg/kg). The other anions in the Tank 40 slurry were present at concentrations less than the minimum detection limits, as identified in the table. Note that the RSDs were all two percent or less, indicating high measurement precision.

The concentration of free hydroxide in the Tank 40 supernatant is 0.240 M, as given in Table 3-9. The RSD of this result is ~5%, which is typical for this type of analysis.

Table 3-7: Elemental Analysis of the Tank 40 Slurry Solids (Shading Indicates Concentrations > 0.1 wt%)

Constituent	Digestion Method(s)	Concentration, wt% solids	%RSD, *n*
Ag	AR	1.22E-02	1.8, 4
Al	AR & PF	6.59E+00	4.1, 8
B	PF	2.51E-02	12, 4
Ba	AR & PF	8.41E-02	2.3, 8
Be	AR	9.34E-04	1.9, 4
Ca	AR & PF	1.03E+00	9.4, 8
Cd	AR & PF	1.75E-02	9.8, 8
Ce	AR	4.86E-02	2.4, 4
Co	AR	1.10E-02	2.8, 4
Cr	AR & PF	7.49E-02	13, 8
Cu	AR	3.00E-02	2.3, 4
Fe	AR & PF	1.65E+01	2.4, 8
Gd	AR & PF	7.86E-02	18, 8
Hg	AR	1.47E+00	7.6, 4
K	AR	<2.2E-01	NA
La	AR	5.01E-02	1.6, 4
Li	AR	<2.3E-02	NA
Mg	AR & PF	1.98E-01	18, 8
Mn	AR & PF	5.33E+00	2.2, 8
Mo	AR	<7.9E-03	NA
Na	AR	1.45E+01	0.7, 4
Nd	AR	1.80E-01	1.4, 4
Ni	AR & PF	1.59E+00	2.9, 8
P	AR	1.16E-01	20, 4
Pb	AR	<3.5E-02	NA
Pd	AR	2.72E-03	1.6, 4
Rh	AR	1.51E-02	1.3, 4
Ru	AR	6.54E-02	1.6, 4
S	AR	3.39E-01	2.5, 4
Sb	AR	<3.7E-02	NA
Si	PF	9.96E-01	3.3, 4
Sn	AR	<2.2E-02	NA
Sr	AR & PF	3.12E-02	1.6, 8
Th	AR & PF	8.52E-01	5.2, 8
Ti	PF	<7.7E-02	NA
U	AR & PF	3.33E+00	5.1, 8
V	AR	<1.0E-03	NA
Zn	AR & PF	2.89E-02	5.6, 8
Zr	AR	9.05E-02	38, 4

Table 3-8: Anions and TIC/TOC in the Tank 40 Slurry (*n* = 4)

Constituent	Concentration, mg/kg slurry	%RSD
Bromide	<1.6E+03	NA
Chloride	<3.2E+02	NA
Fluoride	<3.2E+02	NA
Formate	<3.2E+02	NA
Nitrate	6.75E+03	1.3
Nitrite	1.23E+04	1.4
Oxalate	1.48E+03	1.1
Phosphate	<3.2E+02	NA
Sulfate	1.35E+03	1.1
TIC	1.28E+03	0.5
TOC	4.72E+02	1.9

Table 3-9: Free Hydroxide in the Tank 40 Supernatant (*n* = 4)

Concentration, M	%RSD
2.40E-01	5.2

3.3 Comparison of HTF-40-13-124 and Sludge HTF-40-13-119 Analytical Results

Comparisons of the analytical results reported for the current Tank 40 sample (HTF-40-13-124) versus those reported for the final SB8 sample [16] are given in Table 3-10 and Table 3-11. This comparison provides a basis for determining if the current Tank 40 sample is consistent with the Tank 40 material collected during SB8 characterization. Table 3-10 addresses the density and solids content measurements, while Table 3-11 addresses concentrations of the dominant slurry constituents.

Table 3-10: Comparison of Density and Solids Measurement Results for the Sludge Batch 8 Sample HTF-40-13-124 versus the Sludge Batch 8 WAPS Sample HTF-40-13-119

Measured Property and the Reporting Units	HTF-40-13-124	HTF-40-13-119	% Difference
Slurry density, g/mL	1.11	1.16	4.4
Supernatant density, g/mL	1.05	1.06	0.9
Total solids, wt% of slurry	17.1	17.2	0.6
Insoluble solids, wt% of slurry	11.4	11.3	0.9
Soluble solids, wt% of slurry	5.7	5.9	3.4
Calcined solids, wt% of slurry	13.7	13.1	4.5
Dissolved solids, wt% of supernatant	6.4	6.6	3.1

As shown in Table 3-10, the density of the Tank 40 slurry is about four percent lower than that of the final SB8 slurry, while the density of the Tank 40 supernatant is only about one percent lower than that of the final SB8 slurry. The density difference between the slurries seems significant, since the portion of the density value exceeding one is generally reflective of the impact of the solids. From this perspective, the solids contributing to the Tank 40 density appear to be significantly less than the solids contributing to the SB8 slurry. However, given the relatively good agreement between the various measured solids contents of the two slurries (all of these differences were five percent or less), there is reason to believe that at least a part of the slurry density difference may have been due to measurement uncertainties. Regardless of the slurry density differences, the solids contents of the two slurries appear to be reasonably consistent.

Table 3-11: Comparison of Analytical Results for Dominant Slurry Constituents in Sludge Batch 8 Sample HTF-40-13-124 versus the Sludge Batch 8 WAPS Sample HTF-40-13-119

Constituent	Concentration Reporting Units	HTF-40-13-124	HTF-40-13-119	% Difference
Al	wt% solids	6.59E+00	6.98E+00	5.7
Ca		1.03E+00	9.68E-01	6.2
Fe		1.65E+01	1.69E+01	2.4
Hg		1.47E+00	1.86E+00	23
Mg		1.98E-01	2.29E-01	15
Mn		5.33E+00	5.29E+00	0.7
Na		1.45E+01	1.45E+01	0.0
Nd		1.80E-01	1.79E-01	0.6
Ni		1.59E+00	1.64E+00	3.1
P		1.16E-01	1.40E-01	19
S		3.39E-01	3.44E-01	1.5
Si		9.96E-01	1.10E+00	9.9
Th		8.52E-01	8.16E-01	4.3
U		3.33E+00	3.74E+00	12
Nitrate	mg/kg slurry	6.75E+03	7.08E+03	4.8
Nitrite		1.23E+04	1.17E+04	5.0
Oxalate		1.48E+03	1.62E+03	9.0
Sulfate		1.35E+03	1.23E+03	9.3
TIC		1.28E+03	1.18E+03	8.1
TOC		4.72E+02	4.48E+02	5.2

As shown in Table 3-11, most of the concentrations of dominant slurry constituents of the Tank 40 sample were reasonably consistent with those of the final SB8 sample. For the most dominant elemental constituents (Fe, Na, Al, and Mn), the differences ranged from 0-6%, which is considered small relative to the expected magnitudes of analytical uncertainties. The largest differences were observed for elements Hg, P, and Mg, where the differences were 23, 19, and 15%, respectively. A 23% difference for Hg is not unreasonable, given that the expected one sigma analytical uncertainty for the Hg measurement is 20%. Similarly, the differences for P and Mg should not be considered extreme given that the expected one sigma analytical uncertainty for these elements is 10%. On the whole, the differences between the elemental concentrations were considered reasonable, with an average difference of ~7%.

The anion, TIC, and TOC differences ranged from ~5-9%, which is considered good, given that the expected one sigma uncertainty for these analyses is 10%.

Based on the comparisons presented in Table 3-10 and Table 3-11, the composition of the current Tank 40 sample is considered to be consistent with that of the final SB8 sample.

3.4 Characterization of the SRAT Receipt (SB9 Alternate Reductant Blend)

Densities and solids contents of the SB9 alternate reductant blend are given in Table 3-12. As shown in the table, the slurry and supernatant densities are 1.12 and 1.05 g/mL, respectively, and the weight percent total solids, insoluble solids, and calcined solids are 17.6, 12.2, and 13.8, respectively. These values are consistent with expectations based upon the results observed for the washed Tank 51 sample and the Tank 40 sample. In all cases, the density and solids values for the SB9 alternate reductant blend were within the range of the values determined for the Tank 51 and 40 samples (1.11-1.14 g/mL for slurry density; 1.05-1.05 g/mL for supernatant density; 17.1-18.9 wt% for total solids; 11.4-13.7 wt% for insoluble solids; 5.2-5.7 wt% for soluble solids; 13.7-14.7 wt% for calcined solids; and 6.0-6.4 wt% for dissolved solids). All of the RSDs for the densities and solids content measurements of the blend were low (ranging from 0.5 to 2.1%), demonstrating high measurement precision.

Table 3-12: Densities and Solids Contents of the SRAT Receipt SB9 Alternate Reductant Blend (*n* = 4)

Measurement	Result	%RSD
Slurry density, g/mL	1.12	2.1
Supernatant density, g/mL	1.05	0.7
Total solids, wt% of slurry	17.6	0.5
Insoluble solids, wt% of slurry	12.2	NA
Soluble solids, wt% of slurry	5.4	NA
Calcined solids, wt% of slurry	13.8	0.6
Dissolved solids, wt% of supernatant	6.1	1.7

Concentrations of the elemental constituents in the slurry solids are given in Table 3-13, with results greater than 0.1 wt% shaded for easy identification. As shown in the table, the most dominant constituents in the solids include iron, sodium, aluminum, manganese, uranium, and mercury, with concentrations ranging from approximately 2-17 wt% of the solids (iron has the highest concentration and mercury has the lowest concentration). These concentrations are consistent with expectations based on the elemental compositions observed for the washed Tank 51 sample and the Tank 40 sample. They are also consistent with expectations based on knowing that a significant fraction of the SB9 material was H-Modified (HM) Tank 12 sludge that had been processed through aluminum dissolution.

Most of the RSDs for the elemental analyses of the SB9 alternate reductant blend were limited to about ten percent or less, demonstrating typical analytical precision. In contrast, the RSDs applicable to beryllium, cadmium, gadolinium, thorium, zinc, and zirconium were higher, ranging from 13 to 19%.

These higher RSDs give an indication that the propagated analytical uncertainties associated with these constituents are likely higher than those of the other constituents.

Concentrations of select radioisotopes in the slurry solids are given in Table 3-14, both on a mass concentration basis (wt% solids) and a slurry activity basis (Ci/gallon slurry). As expected, U-238 was the isotope present at the highest mass concentration, at 3.3 wt%. In contrast, the mass concentrations of the other radioisotopes were one or more orders of magnitude lower. On an activity basis, the Pu-238 concentration was highest (~1E-01 Ci/gal) and the U-235 concentration (~5E-07 Ci/gal) was lowest, which was reasonable, given the relatively high specific activity of Pu-238 and the very low specific activity and low isotopic abundance of U-235.

A comparison of the uranium mass concentration presented in Table 3-14 (3.33 wt% total for all uranium isotopes) versus that presented in Table 3-13 (3.05 wt%) shows relatively good agreement between the results of the ICP-MS measurements and the results of the ICP-AES measurements. Specifically, the uranium results from the two methods differed by about 9%, which is reasonable, given that 10% is the estimated one sigma analytical uncertainty of each of these methods.

Table 3-13: Elemental Analysis of the SRAT Receipt SB9 Alternate Reductant Blend Slurry Solids (Shading Indicates Concentrations > 0.1 wt%)

Constituent	Digestion Method(s)	Concentration, wt% solids	%RSD, *n*
Ag	AR	1.18E-02	1.5, 3
Al	AR & PF	7.29E+00	9.5, 6
As	AR	<1.1E-03	NA
B	PF	3.53E-02	1.1, 3
Ba	AR & PF	7.55E-02	6.7, 6
Be	PF	1.28E-03	19, 3
Ca	AR & PF	1.11E+00	11, 6
Cd	AR & PF	1.68E-02	19, 6
Ce	AR	<7.2E-03	NA
Co	AR	9.07E-03	4.4, 3
Cr	AR & PF	6.99E-02	10, 6
Cu	AR	3.15E-02	0.3, 3
Fe	AR & PF	1.71E+01	4.6, 6
Gd	AR & PF	8.15E-02	15, 6
Hg	AR	2.18E+00	6.5, 3
K	AR	<1.0E-01	NA
La	AR	4.06E-02	0.4, 3
Li	AR	9.98E-02	6.1, 3
Mg	AR & PF	2.22E-01	1.3, 6
Mn	AR & PF	5.63E+00	2.3, 6
Mo	AR	8.11E-03	9.3, 3
Na	AR	1.44E+01	0.8, 3
Nd	AR	1.46E-01	0.7, 3
Ni	AR & PF	1.31E+00	2.5, 6
P	AR	1.69E-01	6.4, 3
Pb	AR	5.01E-02	2.8, 3
Pd	AR	2.51E-03	0.3, 3
Rh	AR	1.24E-02	1.2, 3
Ru	AR	5.66E-02	2.8, 3
S	AR	2.74E-01	5.7, 3
Sb	AR	<5.4E-02	NA
Se	AR	<2.2E-03	NA
Si	PF	1.48E+00	2.8, 3
Sn	AR	<3.4E-02	NA
Sr	AR & PF	2.58E-02	3.6, 6
Th	AR & PF	7.78E-01	15, 6
Ti	AR	2.15E-02	NA
U	AR & PF	3.05E+00	7.3, 6
V	AR	<1.2E-03	NA
Zn	AR & PF	3.46E-02	13, 6
Zr	AR	9.89E-02	15, 3

Table 3-14: Select Radioisotopes in the SRAT Receipt SB9 Alternate Reductant Blend Slurry (n = 3)

Isotope	Mass Concentration, wt% solids	Activity Concentration, Ci/gal slurry	%RSD
Tc-99	1.58E-03	1.98E-04	2.6
U-233	6.65E-04	4.76E-05	2.3
U-234	7.71E-04	3.56E-05	0.4
U-235	3.18E-02	5.08E-07	0.8
U-236	1.84E-03	8.79E-07	1.5
U-238	3.29E+00	8.18E-06	1.0
Np-237	2.80E-03	1.46E-05	0.4
Pu-238	1.06E-03	1.36E-01	25
Pu-239	9.28E-03	4.27E-03	1.3
Pu-240	9.07E-04	1.53E-03	2.4
Pu-239/240	NA	6.81E-03	31
Pu-241	3.67E-05	2.82E-02	26
Am-241	5.58E-04	1.43E-02	22
Am-242m	1.75E-07	1.27E-05	7.1
Am-243	8.04E-05	1.20E-04	19
Cm-242	4.26E-10	1.05E-05	7.2
Cm-244	8.23E-06	4.96E-03	11
Cm-245	<1.1E-04	<1.4E-04	NA

Based on the isotopic results in Table 3-14, the calculated U-235 mass enrichment is about one percent. Using the isotopic results in Table 3-14, the calculated ratio of Pu-240 mass to Pu-239 plus Pu-240 mass is about 9%. This is about 40% lower than the average ratio based on waste history receipt [17] – however, still well within the normal range of ratios as indicated per the waste receipt history (most of the ratios fall between 6 and 16%, based on receipt history). These comparisons provide a measure of confidence that the radioisotope concentrations in Table 3-14 are reasonable.

Note that the RSDs for the individual uranium isotope results and the individual Pu-239 and Pu-240 results were all less than three percent, indicating high analytical precision. In contrast, the RSDs for the Pu-238, Pu-239/240, Pu-241 and americium and curium results were higher, ranging from 7-31% and indicating that the propagated analytical uncertainties associated with these results are likely higher than those of the individual uranium isotopes and the individual Pu-239 and Pu-240 isotopes. Comparison of the Pu-239/240 activity concentration determined by counting methods (6.81E-03 Ci/gal) versus the sum of the individual Pu-239 and Pu-240 activity concentrations determined via mass spectrometry (5.80E-03 Ci/gal [which is 4.27E-03 + 1.53E-03 Ci/gal]) shows a 16% difference. This difference is reasonable, given the expected uncertainties of the ICP-MS and radiometric analyses.

A comparison of the replicate slurry analysis results for the neutron poisons and actinide isotopes is given in Table 3-15. This includes results for the elements iron, gadolinium, manganese, and thorium, and select isotopes of uranium, plutonium, americium, and curium. As identified in Section 2.1.4, the integrities of the fourth AR digest solution and the first PF digest solution were compromised, as determined through the quality assurance measures. As such, the results associated with these solutions were excluded from reporting.

Table 3-15: Comparison of Replicate Slurry Analysis Results for the SRAT Receipt SB9 Alternate Reductant Blend

Constituent	Reporting Units	Measured Concentration					
		AR1	**AR2**	**AR3**	**PF2**	**PF3**	**PF4**
Fe	µg/g slurry	2.91E+04	2.91E+04	2.88E+04	3.03E+04	3.18E+04	3.18E+04
Gd		1.61E+02	1.58E+02	1.57E+02	1.39E+02	1.42E+02	1.04E+02
Mn		9.83E+03	9.82E+03	9.73E+3	9.72E+03	1.02E+04	1.02E+04
Th		1.54E+03	1.54E+03	1.53E+03	1.11E+03	1.35E+03	1.15E+03
U-233		1.18E+00	1.19E+00	1.14E+00	N/A	N/A	N/A
U-234		1.35E+00	1.36E+00	1.36E+00	N/A	N/A	N/A
U-235		5.63E+01	5.60E+01	5.54E+01	N/A	N/A	N/A
U-236		3.29E+00	3.21E+00	3.20E+00	N/A	N/A	N/A
U-238		5.83E+03	5.82E+03	5.73E+00	N/A	N/A	N/A
Pu-239		1.64E+01	1.65E+01	1.61E+01	N/A	N/A	N/A
Pu-240		1.58E+00	1.64E+00	1.57E+00	N/A	N/A	N/A
Pu-238	dpm/g slurry	N/A	N/A	N/A	6.89E+07	8.99E+07	5.41E+07
Pu-239/240		N/A	N/A	N/A	3.33E+06	4.77E+06	2.60E+06
Pu-241		N/A	N/A	N/A	1.47E+07	1.86E+07	1.10E+07
Am-242m		N/A	N/A	N/A	6.11E+03	6.99E+03	6.86E+03
Cm-244		N/A	N/A	N/A	2.54E+06	2.90E+06	2.36E+06
Cm-245		N/A	N/A	N/A	<6.0E+04	<8.7E+04	<7.7E+04

AR# = aqua regia digestion number; PF# = peroxide fusion digestion number.

Concentrations of anions, TIC/TOC, and volatile organics in the slurry are given in Table 3-16. This includes slurry results determined by water dilution, caustic quench, and acid digestion approaches. As shown in the table, the dominant anions included nitrite and nitrate, at concentrations of 12,000 – 14,000 mg/kg and 7,000 – 8,000 mg/kg, respectively, as determined by the water dilution and caustic quench methods. In contrast, oxalate was present at 2,400 – 2,600 mg/kg, sulfate was present at 1,000 – 1,200 mg/kg, TIC was present at ~1,100 mg/kg, and TOC was present at ~900 mg/kg. The other anions and the volatile organics were present at concentrations less than the minimum detection limits, as identified in the table. Note that the RSDs were all about five percent or less, indicating high measurement precision. Variations between the results of the three preparation methods (water dilution, caustic quench, and acid digestion) were in the 10-20% range, which is reasonable given the total expected analytical uncertainty (a one sigma analytical uncertainty of ~10% is expected).

Table 3-16: Anions, TIC/TOC, and VOA in the SRAT Receipt SB9 Alternate Reductant Blend Slurry (*n* = 4)

Constituent	Concentration, mg/kg slurry (%RSD)		
	Water Dilution	Caustic Quench	Acid Digestion
Bromide	<1.7E+03 (NA)	<5.6E+02 (NA)	NA
Chloride	<3.3E+02 (NA)	<5.6E+02 (NA)	NA
Fluoride	<3.3E+02 (NA)	<5.6E+02 (NA)	NA
Formate	<3.3E+02 (NA)	<5.6E+02 (NA)	NA
Glycolate	<6.7E+02 (NA)	<2.2E+03 (NA)	NA
Nitrate	7.62E+03 (1.4)	6.92E+03 (4.0)	NA
Nitrite	1.37E+04 (3.5)	1.20E+04 (0.9)	NA
Oxalate	2.61E+03 (3.5)	2.43E+03 (1.9)	2.37E+03 (4.1)
Phosphate	<3.3E+02 (NA)	<5.6E+02 (NA)	NA
Sulfate	1.20E+03 (2.6)	1.00E+03 (5.4)	NA
TIC	1.14E+03 (2.3)	NA	NA
TOC	8.80E+02 (4.9)	NA	NA
Volatile organics	<8.4E+00 (NA)	NA	NA

Concentrations of elemental constituents in the supernatant are given in Table 3-17. As shown in the table, sodium was the most dominant constituent with a concentration of 21,500 mg/L, corresponding to a molarity of slightly less than one (0.93 M). The second and third most dominant constituents were aluminum and sulfur, with concentrations of ~1,300 and 450 mg/L, respectively. Mercury and chromium came next, with concentrations of ~60 and 30 mg/L, respectively. The concentrations of uranium and silver were significantly lower, at ~1 and 0.03 mg/L, respectively. In contrast, the concentrations of all other elemental constituents were less than the minimum detection limits, which ranged from 0.6 mg/L to 550 mg/L, depending on the element.

The relative dominance of sodium, aluminum, sulfur, and mercury in the supernatant is consistent with expectations, based upon the anticipated elemental abundances, elemental solubilities, and previous characterization experiences. Note that RSDs for the sodium, aluminum, sulfur, mercury, and uranium results were all less than 8%, indicating good analytical precision. In contrast, the RSDs for silver and chromium were slightly higher, although still good, at 10 and 12%, respectively.

Concentrations of anions in the supernatant are given in Table 3-18. As shown in the table, the most dominant anions were nitrite, free hydroxide, and nitrate, at concentrations of ~0.31, 0.22, and 0.13 M, respectively. Less dominant, although still detectable, were carbonate, aluminate, oxalate, and sulfate, at concentrations of ~0.12, 0.048, 0.032, and 0.013 M, respectively. When converted to a mass concentration basis, the relative quantities of anions in the supernatant mirror those measured in the slurry (Table 3-16), where nitrite was most dominant, followed by nitrate, oxalate, sulfate, and carbonate (as TIC). Concentrations of the other anions (bromide, chloride, fluoride, formate, glycolate, and phosphate) in the supernatant were all less than the minimum detection limits, which was consistent with what was determined for the slurry. Note that the RSDs for the supernatant anion results were all less than 10%, indicating good analytical precision.

A charge balance comparison was performed to demonstrate consistency between the concentrations of primary supernatant cations and the concentrations of primary supernatant anions. For this comparison, the primary cation was assumed to be Na^+, and the primary anions were assumed to be NO_2^-, OH^-, NO_3^-, CO_3^{2-}, $Al(OH)_4^-$, $C_2O_4^{2-}$, and SO_4^{2-}. These assumptions are based upon the analytical results obtained for the respective supernatant analyses (ICP-AES results for aluminum and sodium; IC results for nitrate, nitrite, oxalate, and sulfate; base titration for free hydroxide; and TIC for carbonate).

In this comparison, molar concentrations of the respective ions were converted to equivalent concentrations, based on the applicable ionic charge (an ion charge of one for sodium, aluminate, free hydroxide, nitrate, and nitrite – and an ion charge of two for carbonate, oxalate, and sulfate). The sums of the equivalent concentrations for the cations and anions were then calculated and compared to one another, to determine consistency.

Results of the charge balance comparison are given in Table 3-19. As shown in the table, the total equivalent concentration for the cation was calculated to be 0.935 eq/L, while the total equivalent concentration for the anions was calculated to be 1.048 eq/L. The difference between these values is ~11%, a value which indicates good consistency, as it is well below the total anticipated sampling and analysis uncertainty. (Neglecting processing uncertainty, the estimated two sigma analytical uncertainty for an individual determination is approximately 20%).

Table 3-17: Elemental Analysis of the SRAT Receipt SB9 Alternate Reductant Blend Supernatant

Constituent	Concentration, mg/L	%RSD, *n*
Ag	2.90E-02	10, 4
Al	1.31E+03	0.6, 4
B	<2.6E+01	NA
Ba	<1.5E+00	NA
Be	<6.3E-01	NA
Ca	<1.6E+01	NA
Cd	<1.9E+01	NA
Ce	<6.1E+01	NA
Co	<2.9E+01	NA
Cr	2.65E+01[a]	12, 2
Cu	<7.1E+01	NA
Fe	<3.1E+01	NA
Gd	<1.5E+01	NA
Hg	6.38E+01	2.9, 4
K	<3.8E+02	NA
La	<1.2E+01	NA
Li	<4.8E+01	NA
Mg	<3.1E+00	NA
Mn	<2.7E+00	NA
Mo	<6.1E+01	NA
Na	2.15E+04	7.6, 4
Ni	<3.3E+01	NA
P	<2.7E+02	NA
Pb	<2.7E+02	NA
Pd	1.93E-01	9.2, 4
Rh	3.43E-02	2.6, 4
Ru	1.48E-01	3.5, 4
S	4.50E+02	1.7, 4
Sb	<5.5E+02	NA
Si	<2.6E+02	NA
Sn	<1.7E+02	NA
Sr	<1.4E+00	NA
Th	<1.3E+02	NA
Ti	<1.2E+02	NA
U	1.24	4.4, 4
V	<7.9E+00	NA
Zn	<7.7E+00	NA
Zr	<9.2E+00	NA

[a] The Cr value in the table is based on two results that exceeded the MDL. The other two results were less than the MDL (<20 mg/L).

Table 3-18: Anions in the SRAT Receipt SB9 Alternate Reductant Blend Supernatant (*n* = 4)

Anion	Concentration, M	%RSD
Aluminate	4.84E-02	0.6
Bromide	<1.6E-02	NA
Carbonate	1.20E-01	1.0
Chloride	<7.4E-03	NA
Fluoride	<1.4E-02	NA
Formate	<5.8E-03	NA
Free hydroxide	2.25E-01	9.6
Glycolate	<7.0E-03	NA
Nitrate	1.34E-01	2.7
Nitrite	3.10E-01	3.0
Oxalate	3.23E-02	0.8
Phosphate	<2.8E-03	NA
Sulfate	1.31E-02	0.4

Table 3-19: The SRAT Receipt SB9 Alternate Reductant Blend Supernatant Charge Balance Comparison

Ion	Equivalent Concentration, eq/L		% Difference
	Cationic	Anionic	
Na^+	0.935	N/A	
$Al(OH)_4^-$	N/A	0.048	
CO_3^{2-}	N/A	0.240	
OH^-	N/A	0.225	
NO_3^-	N/A	0.134	
NO_2^-	N/A	0.310	
$C_2O_4^{2-}$	N/A	0.065	
SO_4^{2-}	N/A	0.026	
	Σ = 0.935	Σ = 1.048	11

Concentrations of select radioisotopes in the supernatant are given in Table 3-20. On a mass basis, the concentrations varied over eight or more orders of magnitude, from a low of <9E-09 mg/L for Cm-242 to a high of ~1E+00 mg/L for U-238. In contrast, on an activity basis, the concentrations varied over five or more orders of magnitude, with the lowest detectable concentration being ~1E-10 Ci/gal for U-235, the highest detectable concentration being ~5E-05 Ci/gal for Tc-99, and several of the isotope concentrations being less than the MDLs. These variations are consistent with expectations, given: a) the relatively high specific activity and low solubility of Cm-242; b) the extremely low specific activity and high isotopic abundance of U-238; c) the low specific activity and low isotopic abundance of U-235; and d) the relatively high fission product yield and high solubility of Tc-99. Note that the RSDs ranged from ~1% to ~20%, which was considered normal for these types of analyses.

A comparison of the replicate supernatant analysis results for the neutron poisons and actinide isotopes is given in Table 3-21. As shown in the table, most of the constituent concentrations were less than the minimum detection limits. Exceptions included all four analytical results for U-235, U-238, Pu-239, and Pu-239/240, and one of the four results for Cm-244.

Table 3-20: Select Radioisotopes in the SRAT Receipt SB9 Alternate Reductant Blend Supernatant (*n* = 4)

Isotope	Mass Concentration, mg/L supernatant	Activity Concentration, Ci/gal supernatant	%RSD
Tc-99	8.56E-01	5.49E-05	0.7
U-233	<2.6E-03	<9.5E-08	N/A
U-234	<2.6E-03	<6.2E-08	N/A
U-235	1.47E-02	1.20E-10	2.0
U-236	<2.6E-03	<6.4E-10	N/A
U-238	1.22E+00	1.55E-09	4.4
Np-237	4.09E-03	1.09E-08	3.8
Pu-238	<7.8E-05	<5.1E-06	N/A
Pu-239	3.48E-03	8.18E-07	7.2
Pu-239/240	N/A	9.46E-07	22
Pu-241	<1.2E-05	<4.8E-06	N/A
Am-241	<1.8E-04	<2.3E-06	N/A
Am-242m	<3.9E-06	<1.4E-07	N/A
Am-243	<1.9E-03	<1.4E-06	N/A
Cm-242	<9.4E-09	<1.2E-07	N/A
Cm-244	5.62E-07*	1.72E-07*	N/A
Cm-245	<6.3E-03	<4.1E-06	N/A

*This value refers to a single result exceeding the MDL. The other three results were less than the MDL (<4.0E-07 mg/L or <1.2E-07 Ci/gal).

Table 3-21: Comparison of Replicate Supernatant Results for the SRAT Receipt SB9 Alternate Reductant Blend

Constituent	Reporting Units	Measured Concentration			
		Dilution 1	Dilution 2	Dilution 3	Dilution 4
Fe	mg/L	<3.1E+01	<3.1E+01	<3.1E+01	<3.1E+01
Gd		<1.5E+01	<1.5E+01	<1.5E+01	<1.5E+01
Mn		<2.8E+00	<2.8E+00	<2.7E+00	<2.7E+00
Th		<1.3E+02	<1.3E+02	<1.3E+02	<1.3E+02
U-233		<2.6E-03	<2.6E-03	<2.6E-03	<2.6E-03
U-234		<2.6E-03	<2.6E-03	<2.6E-03	<2.6E-03
U-235		1.46E-02	1.45E-02	1.45E-02	1.51E-02
U-236		<2.6E-03	<2.6E-03	<2.6E-03	<2.6E-03
U-238		1.20E+00	1.20E+00	1.18E+00	1.30E+00
Pu-239		3.36E-03	3.52E-03	3.22E-03	3.80E-03
Pu-240		<2.6E-03	<2.6E-03	<2.6E-03	<2.6E-03
Pu-238	dpm/mL	<2.9E+03	<3.3E+03	<3.1E+03	<2.7E+03
Pu-239/240		7.38E+02	4.65E+02	4.99E+02	5.19E+02
Pu-241		<9.2E+02	<3.2E+03	<3.2E+03	<4.1E+03
Am-242m		<1.3E+02	<5.4E+01	<9.0E+01	<6.3E+01
Cm-244		<1.0E+02	<3.9E+01	<7.7E+01	1.01E+02
Cm-245		<2.8E+03	<2.2E+03	<1.9E+03	<2.6E+03

3.5 SRAT and SME Cycles

Prior to qualification of the nitric/glycolic flowsheet, simulant studies were performed. These studies provided data that were used to formulate a processing recommendation for the shielded cells demonstration [18]. SRNL uses the Koopman Minimum Acid (KMA) equation [19] to calculate the stoichiometric amount of acid required to destroy nitrite, reduce mercuric oxide to elemental mercury, reduce manganese, and neutralize the slurry.

Subsequent to completing the SRAT cycle, it was determined that the amount of acid and antifoam added were less than the targeted amounts provided by the acid calculation. Analytical data generated from the SRAT cycle were reviewed and compared with previous SB9 nonradioactive simulant studies [18]. These studies investigated SB9 processing over a range of acid stoichiometries, 77% to 123% KMA. Hydrogen generation during these tests was very low compared to testing with the nitric-formic acid flowsheet. Unlike the nitric-formic acid flowsheet, there is no correlation between acid stoichiometry and hydrogen generation for the nitric-glycolic flowsheet [18, 20]. The acid stoichiometry utilized during the SC-18 SRAT cycle (78% KMA) falls within the range of SB9 simulant testing and compares well with the NG-58 flowsheet demonstration performed at 77% KMA. The under addition of antifoam resulted in no impact to processing; no foaming was observed during processing of the sludge slurry (see Section 3.6). Furthermore, the under addition did not significantly impact the solids content or processing times and volume. Antifoam degradation products were measured during simulant testing; thus, it was not part of the scope for the actual-waste test [1].

Upon review of the data generated during the SRAT cycle, it was determined that the SRAT cycle resulted in a successful demonstration of the flowsheet: nitrite was destroyed below the acceptable limit, no hydrogen generation was observed, rheology was similar to simulant tests, and calculated REDOX was similar to simulant tests.

3.5.1 Acid Calculation inputs

Inputs to the acid calculation, along with assumptions for anion destruction and conversion, are presented in Table 3-22. Inputs and outputs annotated with an asterisk represent values that deviated from the original acid calculation.

Table 3-22: SRAT Receipt Acid Calculation Inputs

SRAT Receipt Mass	3,453	g slurry
SRAT Receipt Volume	3,091	mL slurry
SRAT Receipt Weight % Total Solids	17.61	wt%
SRAT Receipt Weight % Calcined Solids	13.75	wt%
SRAT Receipt Weight % Insoluble Solids	12.24	wt%
SRAT Receipt Density	1.117	kg / L slurry
SRAT Receipt Supernatant Density	1.050	kg / L supernate
SRAT Receipt Nitrite	13,700	mg/kg slurry
SRAT Receipt Nitrate	7,610	mg/kg slurry
SRAT Receipt Oxalate	2,610	mg/kg slurry
SRAT Receipt Slurry TIC (treated as carbonate)	1,140	mg/kg slurry
SRAT Receipt Supernatant TIC (treated as carbonate)	1,440	mg/L supernate
SRAT Receipt Hydroxide (Base Equivalents) pH = 7	0.505	mol/L slurry
SRAT Receipt Manganese	7.12	wt % calcined basis
SRAT Receipt Mercury	2.18	wt% dry basis
SRAT Receipt Magnesium	0.286	wt % calcined basis
SRAT Receipt Calcium	1.29	wt % calcined basis
Destruction of Nitrite in SRAT and SME Cycle	100	% of starting nitrite destroyed
Net Conversion of Glycolic Acid to Oxalate	1	% glycolate converted to C2O4
Net Conversion of Glycolic Acid to Formate	1	% glycolate converted to COOH
Percent Acid in Excess Stoichiometric Ratio (Koopman Min Acid Eq) – for Total acid to add determination *	78.0	%
Water to rinse sample bottle	99.8	g
Total water added to flush nitric and glycolic acid lines/bottles	20	g
SRAT Product Target Solids	25	wt%
REDOX Target *	0.07	Fe^{2+} /ΣFe
Antifoam Addition prior to Glycolic Acid Addition *	0.15	gal (DWPF Scale)
Antifoam Addition Prior to Initial SRAT Boiling *	0.1	gal (DWPF Scale)
Antifoam Addition During SRAT Boiling *	0.1	gal (DWPF Scale)
Water Flush volume after each antifoam addition	100	gal (DWPF Scale)
SRAT air purge	93.7	scfm (DWPF Scale)
DWPF Acid addition Rate	179	mol per minute (DWPF Scale)
Nitric Acid Molarity	10.47	Molar
Glycolic Acid Molarity	11.96	Molar
SRAT boil-up rate	5,000	lbs/hr (DWPF Scale)
SRAT Mercury Product Target Concentration	0.45	wt % total solids basis
SRAT Steam Stripping Factor	750	(g steam/g mercury)
Frit Type	803	
Sludge Oxide Contribution in SME (Waste Loading)	38.00	%
Target SME Solids total Wt%	45.0	wt%
Number of frit additions in SME Cycle	2	
# DWPF Canister decons simulated	6	
Volume of water per deconed can	1,000	gal at DWPF scale
SME air purge	72	scfm
SME boil-up rate	5000	lbs/hr

Results of the acid calculation are presented in Table 3-23. DWPF currently uses the Hsu acid equation for determining acid demand, whereas SRNL utilizes the KMA equation [19]. The differences between the two equations are: 1) Hsu has a coefficient of 0.75 for nitrite, while KMA has a factor of 1.0; 2) Hsu has a factor of 1.2 for Mn, while KMA has a factor of 1.5; 3) KMA includes Ca and Mg in the acid demand calculation; and 4) Hsu uses slurry TIC, while KMA uses supernate TIC as can be seen in Equation 3-1 and Equation 3-2.

Equation 3-1: Hsu Acid Equation

$$\frac{\text{moles acid}}{\text{L slurry}} = \text{base equivalents} + 2 * \text{total TIC} + 0.75 * \text{nitrite} + 1.2 * \text{Mn} + \text{Hg}$$

Equation 3-2: Koopman Minimum Acid Equation

$$\frac{\text{moles acid}}{\text{L slurry}} = \text{base equivalents} + \text{Hg} + \text{soluble TIC} + 1.5 * (\text{Ca} + \text{Mg}) + 1.0 * \text{nitrite} + 1.5 * \text{Mn}$$

Table 3-23: Acid Calculation Outputs

Headspace Volume to Sludge Volume Ratio	0.586	
Conversion of Nitrite to Nitrate in SRAT Cycle *	50.49	gmol NO_3^-/100 gmol NO_2^-
Destruction of Glycolic acid charged in SRAT *	26.33	% glycolate converted to CO_2 etc.
Hsu Total Stoichiometric Acid required	3.79	mol
Koopman Minimum Stoichiometric Acid required	4.24	mol
Total Acid to Add based on Koopman equation stoichiometric factor	3.304	mol
Stoichiometric Acid Hsu Eqn (%) *	87.1	%
Fraction of Glycolic Acid *	0.5743	moles glycolic acid / mole total acid
Nitric acid required *	176.1	g
Mol nitric acid required *	1.41	mol
Glycolic acid required *	200.6	g
Mol glycolic acid required *	1.90	mol
Prototypical nitric acid feed time *	58	min
Prototypical glycolic acid feed time *	80	min
SRAT Dewater Mass (including condensate samples)	971	g
SRAT conflux time	1410	min
Total frit to add in the SME	658	g
Final dewater in the SME	641	g

Table 3-24 presents the DWPF to SRNL scale factor and targeted processing parameters for the SRAT and SME cycles. The scale factor is based on a DWPF SRAT receipt volume of 6000 gallons. The SRNL and DWPF scaled amounts for antifoam reflect the actual amounts added during testing.

Table 3-24: DWPF and SRNL-Scale Processing Parameters

Scale 6000 gal DWPF SRAT Receipt to 3,091 mL SRNL SRAT Receipt		
Volume Based Scale Factor	7348	
	DWPF Scale	**SRNL Scale**
SRAT air purge	93.7 scfm	361 sccm (includes He tracer)
Nitric acid addition rate	179 mol/min, ~4.0 gpm	2.33 mL/min
Glycolic acid addition rate	179 mol/min, ~4.5 gpm	2.04 mL/min
Boil up rate	5000 lb steam/hr	5.14 g/min
Antifoam Addition (prior to glycolic acid addition) *	0.15 gal	0.077 g
Antifoam Addition (added prior to boiling, every 12 hours during boiling, and as needed) *	0.1 gal	0.052 g
Antifoam Flush (added after any antifoam addition)	100 gal	51.51g
Scale 6000 gal DWPF SRAT Receipt to 1,711 mL SRNL SRAT Product		
SME air purge	72 scfm	153 sccm (includes He tracer)
Volume Based Scale Factor in SME	13300	
Antifoam Addition added after canister decon	0.25 gal	0.071g
Antifoam Addition added after frit addition	0.25 gal	0.071g
Antifoam Flush (added after any antifoam addition)	100 gal	28.5 g

3.5.2 SRAT and SME Cycle Data

The data collected from the CPC demonstrations using the SB9 blended sample are presented in this section. The SB9 SRAT cycle began by heating the sludge to 93 °C while adding nitric acid. Upon the slurry reaching 93 °C, glycolic acid was added. Acids were added at the scaled DWPF rate presented in Table 3-24. Following acid addition, the vessel contents were heated to boiling and water was removed to a targeted solids concentration of 25 wt%. The boilup rate for both SRAT and SME was scaled to the DWPF rate of 5000 lbs steam/hr.

For the SME cycle, six canister decontamination water additions and removals were simulated. Two Frit 803 additions were performed with an equivalent amount of water to simulate at 50 wt% solution. A sludge waste loading of 38% was targeted. The DWPF currently targets waste loading of 38% to ensure that the contract target of 36% waste loading is achieved. No formic acid was included with the frit additions. Following the final frit addition, the slurry was then concentrated to a targeted final total solids concentration of 48 wt%.

3.5.2.1 Offgas Analysis

Figure 3-1 presents the carbon dioxide, nitrous oxide, and oxygen concentrations in the SRAT cycle offgas. Hydrogen was not detected; hydrogen measurements are discussed below. Offgas concentrations correlate well with acid additions. Carbon dioxide peaked during glycolic acid addition (between -1.7 and 0 hours) as carbonates were neutralized. Nitrous oxide concentration peaked shortly after acid addition as nitrite was destroyed by the added acids. Oxygen nearly reached zero (<1 vol%) during nitrous oxide generation. Other nitrogen oxide compounds were also produced, consuming oxygen, which are not quantified by the GC. Nitric oxide, which is detected by the GC but not quantified, correlated well with nitrous oxide. Prior to the application of uncertainty, the measured peak concentrations of carbon dioxide and nitrous oxide were 35.6 vol% and 0.57 vol%, respectively. The measured peak nitrous oxide concentration was below the DWPF limit of 15 vol% [21]. Carbon dioxide and nitrous oxide generation rates have been calculated from the measured concentrations. The calculation is based on scaling of the initial laboratory SRAT slurry volume to 6000 gallons of DWPF-scale SRAT receipt. Results are shown in Figure 3-2.

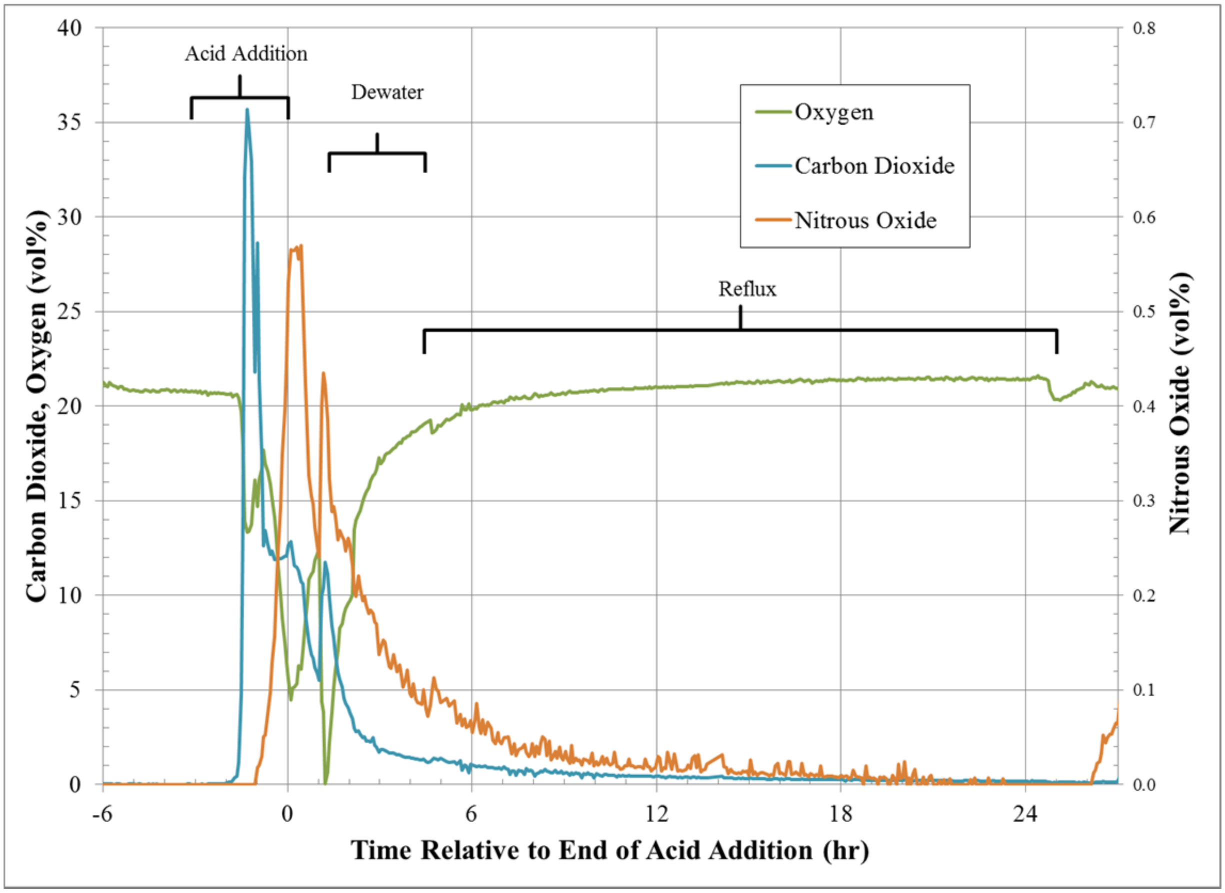


Figure 3-1: Concentrations of Carbon Dioxide, Oxygen, and Nitrous Oxide in the SC-18 SRAT Cycle Offgas

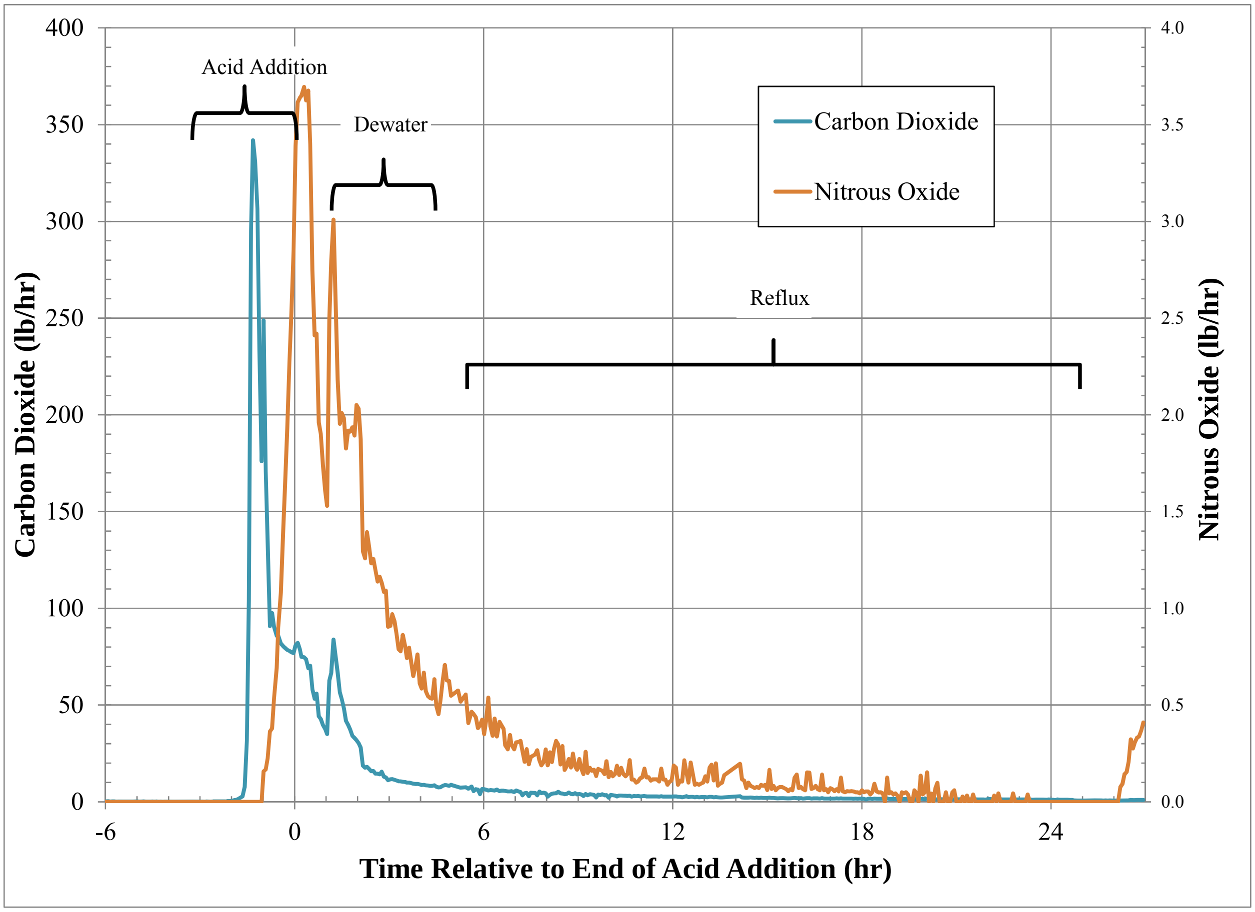


Figure 3-2: DWPF-Scale Generation Rates of Carbon Dioxide and Nitrous Oxide in the SC-18 SRAT Cycle Offgas

Figure 3-3 shows the concentration of helium and nitrogen during the SRAT cycle. Both gasses are inert in the SRAT process. As expected, helium and nitrogen concentrations track each other well. During peak offgas generation (during acid addition and initial dewatering), both gasses drop in concentration. Drops in helium without corresponding drops in nitrogen indicate a breach in the vessel. The first drop (at the approximately three hour mark) corresponds to an antifoam and flush water addition. The second drop at just before the six hour mark follows the change from dewatering to reflux; the reflux valve between the MWWT and the vessel is opened.

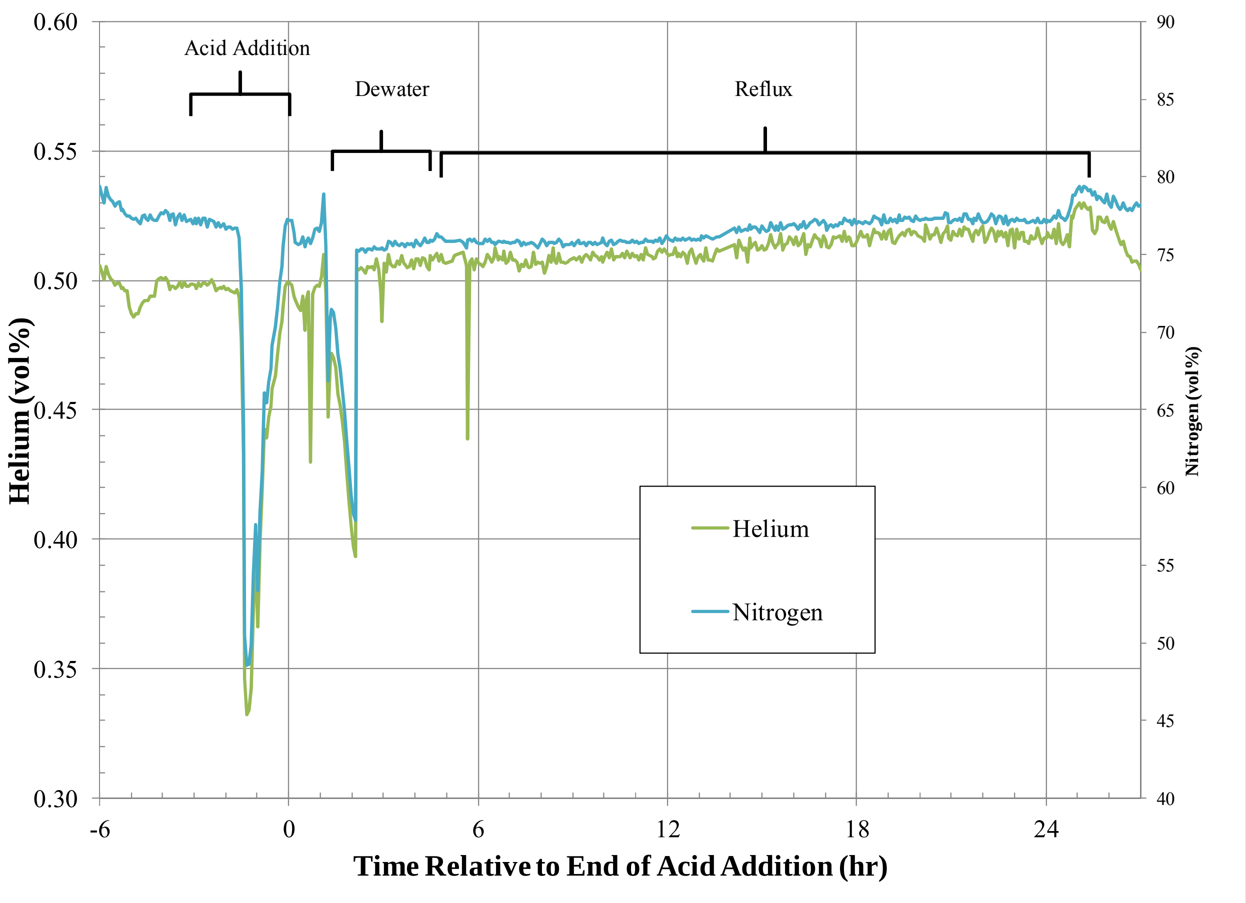


Figure 3-3: Helium and Nitrogen Concentrations Measured During the SC-18 SRAT Cycle as Quantified by GC

As stated above, hydrogen was not detected during the SRAT cycle. Hydrogen was detected when mixing began (see Figure 3-4). Mixing was initiated two days after charging the vessel. This is radiolytic hydrogen retained in the sludge slurry. Hydrogen concentration quickly peaked and then slowly decreased as the vapor space was replaced with hydrogen-free purge gas. The hydrogen data can be used to estimate the hydrogen detection limit for the SRAT cycle. The lowest detected concentration of hydrogen was measured to be 0.004 mol% at approximately -4.25 hours. This corresponds to a DWPF-scale hydrogen generation rate of 0.0011 lb/hr.

This retained hydrogen data cannot be compared directly to the DWPF SRAT due to differences in vessel size, vessel geometry, and mixing. Radiolytic hydrogen is related to absorbed dose, which is highly dependent on vessel geometry. Hydrogen retention is also related to vessel geometry. Finally, hydrogen release is dependent on mixing; SRNL mixing is not scaled to DWPF mixing. Also, with a four minute GC sampling time, the actual peak may have been missed.

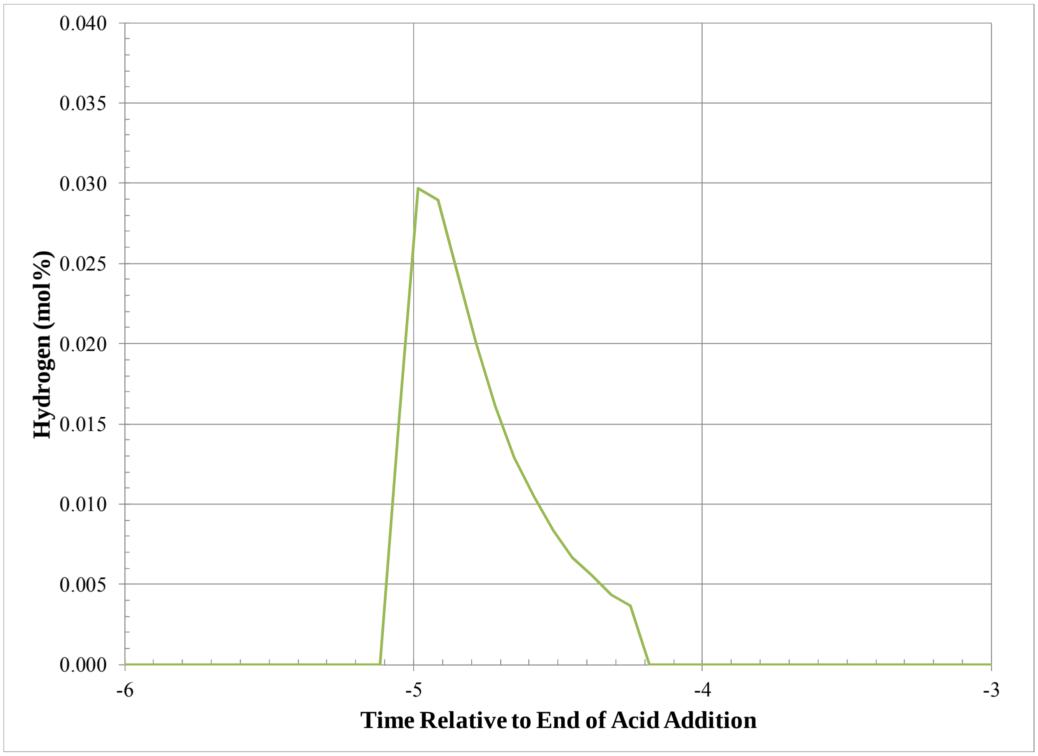


Figure 3-4: Release of Hydrogen Prior to Initiation of SRAT Cycle

Figure 3-5 and Figure 3-6 contain plots of SME cycle offgas concentrations and generation rates, respectively. The plots only include carbon dioxide and nitrous oxide. Hydrogen was not detected. Generation rates are adjusted to the DWPF scale and are reported in pounds-per-hour. The adjustment is based on scaling of the initial laboratory SME slurry volume to 6000 gallons of DWPF-scale SME receipt [22].

The peak production and release of carbon dioxide and nitrous oxide during the SME were upon the initiation of boiling. Prior to the application of uncertainty, the measured peak concentrations of carbon dioxide and nitrous oxide were 4.0 vol% and 0.0778 vol%, respectively.

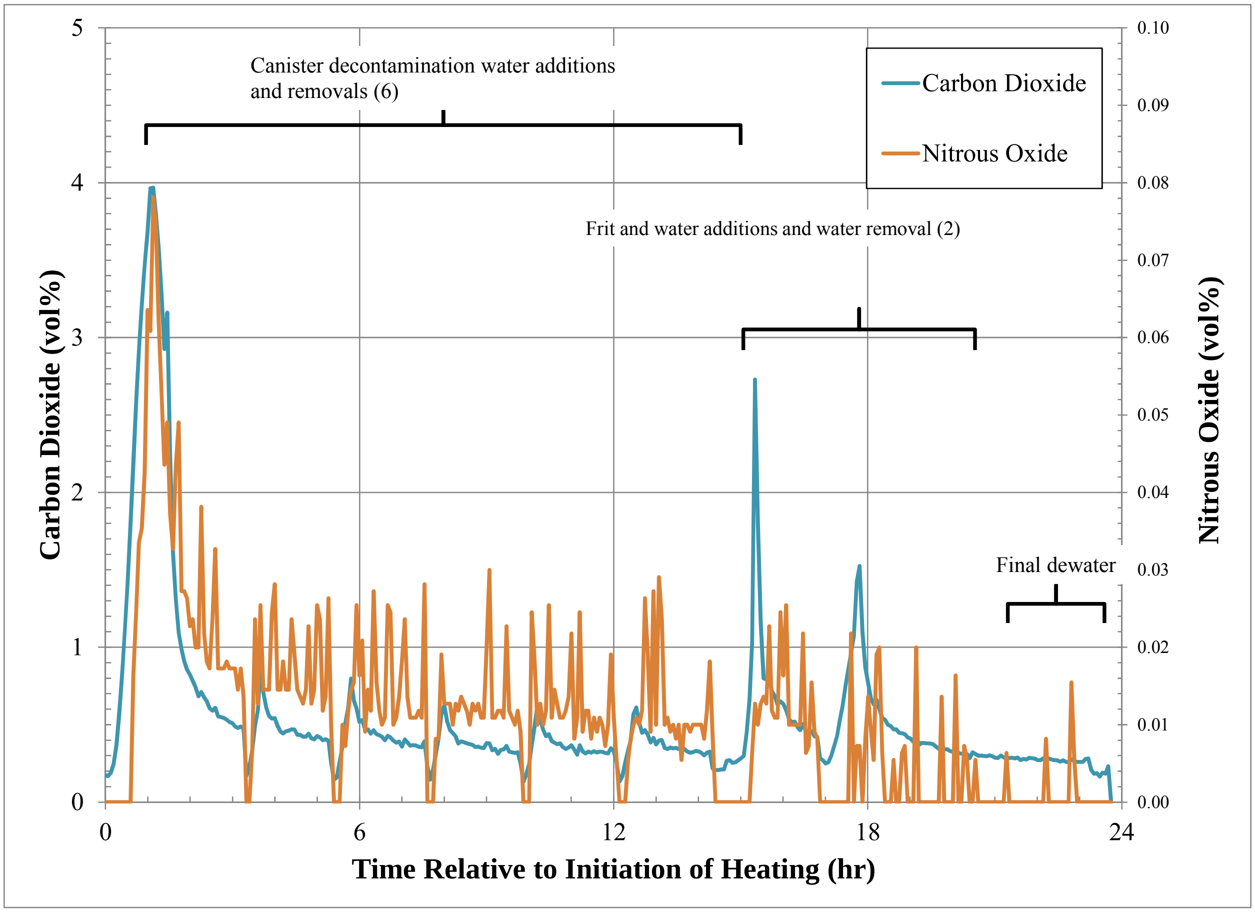


Figure 3-5: Carbon Dioxide and Nitrous Oxide Concentrations Measured During the SC-18 SME Cycle as Quantified by GC

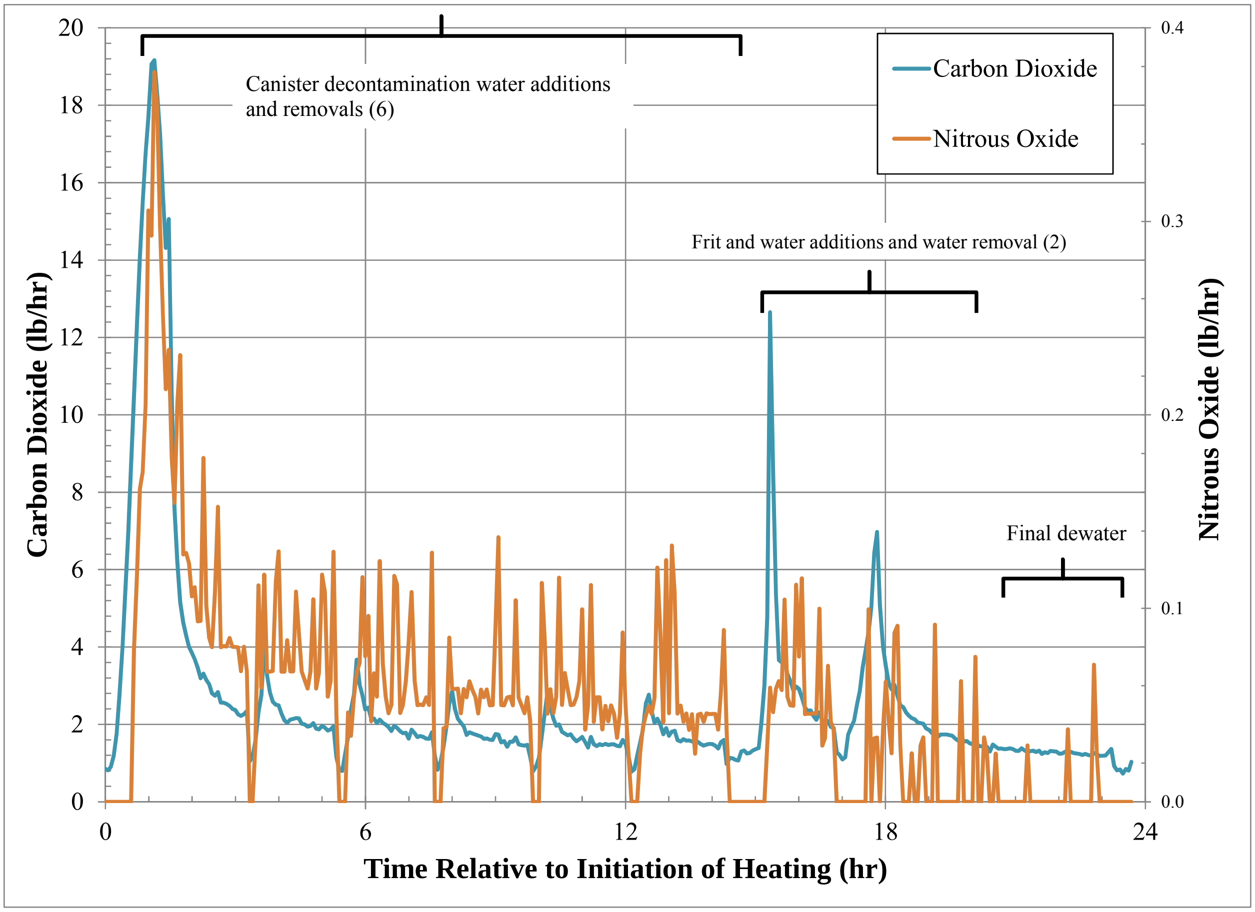


Figure 3-6: Carbon Dioxide and Nitrous Oxide Generation Rates During the SC-18 SME Cycle

Figure 3-7 shows the helium, oxygen, and nitrogen concentrations during the SME cycle. The drops in helium correspond to breaches in the vessel: five decontamination water additions plus two frit/water additions. Note that the first of the six total decontamination water additions was made prior to heating. Unlike the SRAT cycle, there is no indication of oxygen being consumed in any reactions. Oxygen is nearly constant during the SME cycle, as expected.

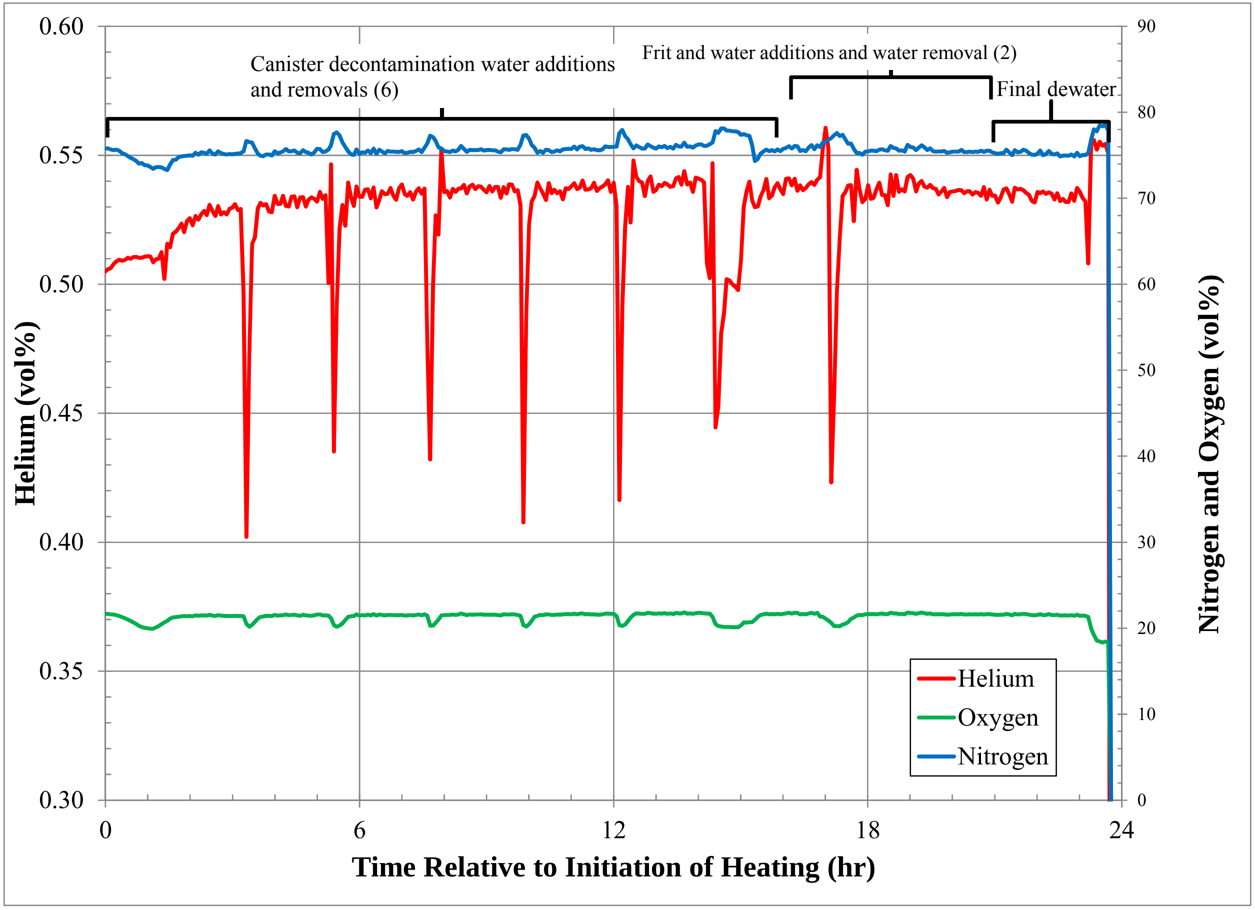


Figure 3-7: Helium, Oxygen, and Nitrogen Concentrations Measured During the SME Cycle as Quantified by GC

Like the SRAT cycle, hydrogen was not detected during the SME cycle. Hydrogen was detected when mixing began (see Figure 3-8). Mixing was initiated 21 days after the SRAT cycle was completed. Similar to the SRAT, hydrogen concentration quickly peaked and then slowly decreased as the vapor space was replaced with hydrogen-free purge gas. The hydrogen data can be used to estimate the hydrogen detection limit for the SRAT cycle. The lowest detected concentration of hydrogen was measured to be 0.003 mol% at approximately -3.6 hours. This corresponds to a DWPF-scale hydrogen generation rate of 0.0008 lb/hr.

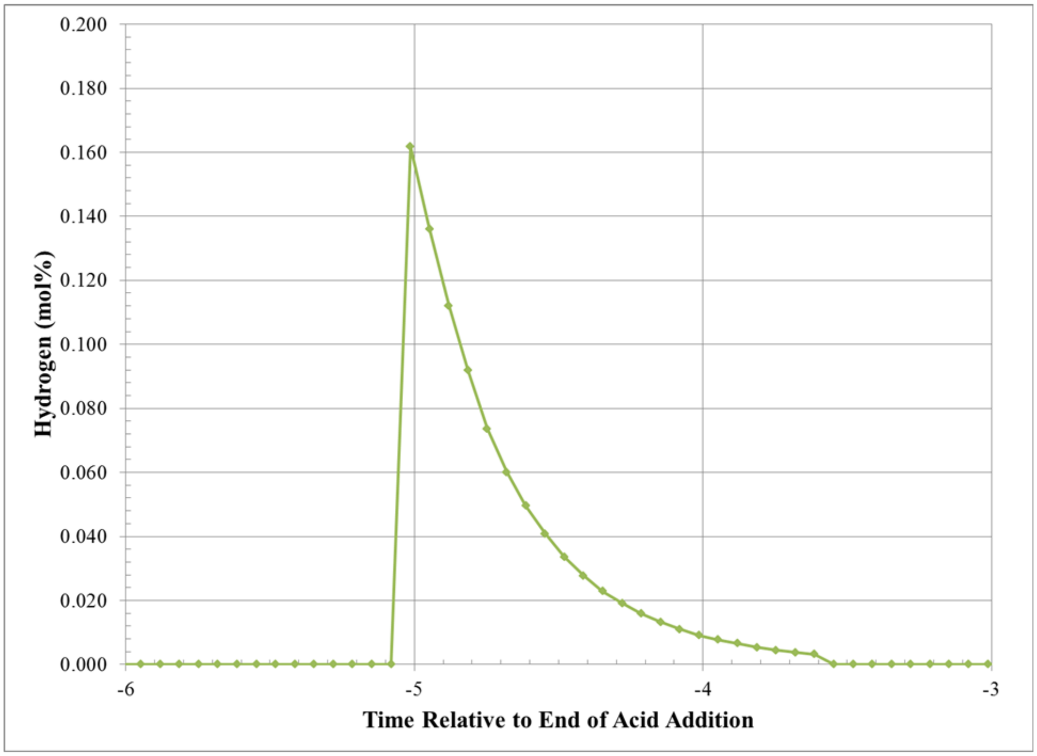


Figure 3-8: Release of Hydrogen Prior to Initiation of SME Cycle

SRNL performed an uncertainty analysis in order to place an upper bound (with 95% confidence) on the reported maximum carbon dioxide and nitrous oxide generation rates; maximum carbon dioxide and nitrous oxide concentrations and corresponding generation rates encountered during the SB9 nitric-glycolic demonstration. For hydrogen, which was below the detectable level during the SRAT and SME cycles, the upper bound was calculated for the detection limit concentration and generation rate.

Several factors contribute to uncertainty in the carbon dioxide, nitrous oxide, and hydrogen measurements and generation rates for the SB9 shielded cells qualification run. The factors considered in this analysis include the following:

- Uncertainty in the air purge flowrate
- Uncertainty in the helium tracer flowrate
- Uncertainty in the carbon dioxide and nitrous oxide concentrations in the GC calibration gasses
- Uncertainty due to variance in GC measurements
- Bias due to drift in the calibration during the run

The SRNL air and helium purge flowrates were determined by scaling the SRNL SRAT and SME volumes to a 6,000 gallon DWPF basis. The uncertainty associated with these setpoints is not included in this analysis. Scaling factors are based on weights and densities and the uncertainties associated with these measurements are expected to be small compared to the uncertainties in the flow meter and offgas measurements.

The MKS flow meter / flow controllers used for the flowrates of the air purge and helium tracer had tolerances of 2% of full scale and were tracked in the Measurement and Test Equipment (M&TE) program. The standards used to calibrate the GC for gas concentrations have an analysis certification to 2% of the reported concentrations. The variance in the GC measurements is estimated from the data collected during the instrument calibration check. The bias due to the calibration drift is handled by processing the calibration of the GC in a manner to provide conservatively large carbon dioxide and nitrous oxide generation measurements. The pre- and post-run calibration check information is compared, and the sets of calibration data are used that would maximize the instrument-measured carbon dioxide and nitrous oxide concentrations and minimize the helium tracer concentration. Because the detection limit for hydrogen was determined by the minimum quantifiable hydrogen that was detected prior to starting the SRAT and SME cycles, the pre-run calibration data is used for hydrogen detection limit uncertainty.

Gas of known concentration (calibration gas) is run through the GC and multiple measurements are made. A response factor is determined as the relationship between the integrated chromatogram area with the concentration in the gas standard ($C^{std}/area^{std}$). The response factors are then used to determine concentration of gasses in the SRAT and SME offgas. Uncertainty can be applied to the carbon dioxide, nitrous oxide, and hydrogen concentrations by the following equations: Equation 3-3, Equation 3-4, Equation 3-6 and Equation 3-6. The concentrations of carbon dioxide, nitrous oxide, hydrogen, and helium (C_{CO_2} , $C_{\mathrm{N_2O}}$, $C_{\mathrm{H_2}}$, and C_{He}) are in mole fraction (mol/mol). The flowrates of air and helium purges at lab scale (F_{air} and F_{He}) and $F_{\mathrm{SRNL\text{-}purge}}$ are the SRNL purge rates in standard cubic centimeters per minute (sccm). While the ratio ($F_{\mathrm{SRNL\text{-}purge}}$ / (F_{air} + F_{He})) is by definition equal to 1 (the sum of the He and air flowrates are set to equal the SRNL purge rate), the equations allow one to account for the uncertainty in the helium and air flow controllers. The GC response for carbon dioxide, nitrous oxide, hydrogen, and helium are in terms of an area (e.g. $area_{H_2}$). Although helium is used as a tracer rather than being produced as an offgas, it is necessary to include it in the uncertainty analysis because it is used in the calculation of the carbon dioxide and nitrous oxide generation rates.

Equation 3-3

$$C_{CO_2} = area_{CO_2}\left(\frac{C^{std}_{CO_2}}{area^{std}_{CO_2}}\right)\left(\frac{F_{\mathrm{SRNL\text{-}purge}}}{F_{\mathrm{air}} + F_{\mathrm{He}}}\right)$$

Equation 3-4

$$C_{\mathrm{N_2O}} = area_{\mathrm{N_2O}}\left(\frac{C^{std}_{\mathrm{N_2O}}}{area^{std}_{\mathrm{N_2O}}}\right)\left(\frac{F_{\mathrm{SRNL\text{-}purge}}}{F_{\mathrm{air}} + F_{\mathrm{He}}}\right)$$

Equation 3-5

$$C_{\mathrm{H_2}} = area_{\mathrm{H_2}}\left(\frac{C^{std}_{\mathrm{H_2}}}{area^{std}_{\mathrm{H_2}}}\right)\left(\frac{F_{\mathrm{SRNL\text{-}purge}}}{F_{\mathrm{air}} + F_{\mathrm{He}}}\right)$$

$$C_{\mathrm{He}} = area_{\mathrm{He}}\left(\frac{C^{std}_{\mathrm{He}}}{area^{std}_{\mathrm{He}}}\right)\left(\frac{F_{\mathrm{SRNL\text{-}purge}}}{F_{\mathrm{air}} + F_{\mathrm{He}}}\right)$$

Equation 3-6

The values calculated by Equation 3-7, Equation 3-8, and Equation 3-9 are the DWPF-scale generation rates of carbon dioxide, nitrous oxide, and hydrogen, respectively, in lb/hr scaled from the results of the shielded cells SB9 qualification run. The flowrates of air and helium purges at lab scale (F_{air} and F_{He}) and the flowrate of purge at DWPF-scale ($F_{\text{DWPF-purge}}$) are in sccm. The DWPF-scale flowrates are based on 186 standard cubic feet per minute (scfm) purge in the SRAT and 72 scfm in the SME. MW values are the molecular weights of the gasses and A_{constant} is a combination of multiple unit conversions (including the ideal gas molar volume at 70 °F and 1 atmosphere). Per these equations, the helium tracer concentration is used to correct the offgas data for the unknown total offgas flowrate.

Equation 3-7
$$CO_{2(\text{DWPF-scale})}(\text{lb/hr}) = \frac{C_{CO_2}}{C_{He}} * F_{He} * \frac{F_{\text{DWPF-purge}}}{F_{air} + F_{He}} * MW_{CO_2} * A_{\text{constant}}$$

Equation 3-8
$$\text{N}_2\text{O}_{(\text{DWPF-scale})}(\text{lb/hr}) = \frac{C_{\text{N}_2\text{O}}}{C_{He}} * F_{He} * \frac{F_{\text{DWPF-purge}}}{F_{air} + F_{He}} * MW_{\text{N}_2\text{O}} * A_{\text{constant}}$$

Equation 3-9
$$\text{H}_{2(\text{DWPF-scale})}(\text{lb/hr}) = \frac{C_{\text{H}_2}}{C_{He}} * F_{He} * \frac{F_{\text{DWPF-purge}}}{F_{air} + F_{He}} * MW_{\text{H}_2} * A_{\text{constant}}$$

Results were processed using the statistical package GUM Workbench [23] to propagate the uncertainty in the measurements to the calculated results. Table 3-25 and Table 3-26 contain the results of the uncertainty analysis for the DWPF-scale maximum concentrations and generation rates, respectively. The expanded uncertainties are the half-widths of the two-sided 95% confidence intervals (CI) on the average analytical measurements adjusted to DWPF scale. The upper 95% bounds are the sum of the averages and the half-widths of the CI. Thus, these upper 95% bounds are the maximum values adjusted for the uncertainty based on the SB9 qualification demonstration. The values reported for hydrogen concentration and generation rate, which are preceded by "<", are the detection limits because hydrogen was not detected during the SRAT and SME cycles. While additional measurement uncertainty does not typically need to be applied to instrument detection limits, they are applied in this case due to the influence of the uncertainty in other factors (i.e., purge flow rates and helium concentration) on the hydrogen detection limit.

For an ideal gas mixture, mol% and 100*(mol/mol) are considered to be equivalent to vol%.

Table 3-25: Maximum Concentrations of Carbon Dioxide and Nitrous Oxide in the SC-18 SRAT and SME Cycles

	Maximum concentration prior to applying uncertainty (mol%)	Expanded uncertainty, half-width of 95% CI (mol%)	Maximum concentration, upper 95% bound (mol%)
SRAT Carbon Dioxide	36	10	46
SRAT Nitrous Oxide	0.57	0.16	0.73
SRAT Hydrogen	< 0.004	0.001	< 0.005
SME Carbon Dioxide	4.0	3.0	7.0
SME Nitrous Oxide	0.08	0.12	0.20
SME Hydrogen	< 0.003	0.003	< 0.006

Table 3-26: Peak DWPF-Scale Generation Rates of Carbon Dioxide and Nitrous Oxide in the SC-18 SRAT and SME cycles

	DWPF-scale rate prior to applying uncertainty (lb/hr)	Expanded uncertainty, half-width of 95% CI (lb/hr)	DWPF-scale rate, upper 95% bound (lb/hr)
SRAT Carbon Dioxide	342	99	441
SRAT Nitrous Oxide	3.7	1.1	4.8
SRAT Hydrogen	< 0.0011	0.0003	< 0.0014
SME Carbon Dioxide	19	15	34
SME Nitrous Oxide	0.38	0.57	0.95
SME Hydrogen	< 0.0008	0.0006	< 0.0014

3.5.2.2 Condensate Analysis

Table 3-27 and Table 3-28 contain the results of the IC anions and cations analyses of the condensate samples taken during the SRAT and SME cycles. Nitrate, nitrite, formate, glycolate and ammonium were quantified in many of the samples. The remainder of the anions were below the detection limits in all condensate samples: <50 mg/L for fluoride and bromide, and <10 mg/L for chloride, phosphate, sulfate and oxalate. Reported one sigma analytical uncertainties of these analyses are 10%.

The MWWT samples are samples of the transient condensate composition and the other dewater samples are bulk samples accumulated over longer periods of time. The MWWT sample collected at the completion of glycolic acid addition had the highest level of formate of any condensate sample and relatively high levels of nitrate and glycolate as well. The cold finger samples are similar to what might be expected in the Formic Acid Vent Condenser (FAVC). The material collected from the cold finger at the end of the SRAT and SME cycles contained the largest concentrations of nitrate and glycolate when compared to other condensate samples for the respective cycle.

In general, nitrate concentration in the condensate trended downward during the SRAT and SME cycles. Glycolate and formate concentration in the condensate shared a similar downward trend. Nitrite was not seen in the condensate during the SRAT cycle, but was present in the SME cycle condensates with a downward trend in each subsequent dewater sample. Ammonium concentrations (typically 10 to 20 mg/L) were not noted until the condensate sample from the end of the SRAT reflux and persisted during the SME cycle.

Table 3-27: Anion and Cation Analysis of SC-18 SRAT Dewater Samples

SRAT Condensate Sample	Nitrate (mg/L)	Nitrite (mg/L)	Formate (mg/L)	Glycolate (mg/L)	Ammonium (mg/L)
MWWT after glycolic acid addition	6.28E+04	<1.0E+01	1.54E+03	7.00E+01	<5.0E+00
MWWT at end of dewater	4.75E+03	<1.0E+01	1.90E+01	1.46E+01	<5.0E+00
Total dewater	8.96E+03	<1.0E+01	2.66E+02	1.77E+01	<5.0E+00
MWWT at end of reflux	1.47E+03	<1.0E+01	<1.0E+01	1.51E+01	1.77E+01
Cold finger (FAVC) contents	3.44E+05	<1.0E+01	2.63E+02	2.15E+02	1.27E+01

Table 3-28: Anion and Cation Analysis of SC-18 SME Dewater Samples

SME Condensate Sample	Nitrate (mg/L)	Nitrite (mg/L)	Formate (mg/L)	Glycolate (mg/L)	Ammonium (mg/L)
Canister decontamination dewater 1	2.97E+01	5.41E+01	<1.0E+01	1.27E+01	2.41E+01
Canister decontamination dewater 2	4.02E+01	4.73E+01	<1.0E+01	1.21E+01	2.18E+01
Canister decontamination dewater 3	3.56E+01	4.08E+01	<1.0E+01	1.08E+01	1.83E+01
Canister decontamination dewater 4	2.51E+01	3.50E+01	<1.0E+01	<1.0E+01	1.54E+01
Canister decontamination dewater 5	1.91E+01	3.12E+01	<1.0E+01	<1.0E+01	1.32E+01
Canister decontamination dewater 6	2.09E+01	2.61E+01	<1.0E+01	<1.0E+01	1.14E+01
Frit dewater 1	1.26E+01	1.35E+01	<1.0E+01	<1.0E+01	9.90E+00
Frit dewater 2	1.43E+01	1.14E+01	<1.0E+01	<1.0E+01	1.03E+01
Final dewater	<1.0E+01	1.07E+01	<1.0E+01	<1.0E+01	1.11E+01
Cold finger (FAVC) contents	2.98E+04	1.16E+01	8.43E+01	8.28E+01	5.80E+00

3.5.2.3 SRAT and SME Product Analyses

Densities and solids content of the SRAT and SME products are given in Table 3-29. The total solids measurements of the SRAT and SME products were 24.6 and 49.0 wt%, respectively, which match the targets of 25 wt% and 48 wt% acceptably. The SRAT and SME product slurry densities are 1.23 and 1.43 mg/L, respectively, which are consistent with the overall solids content attained. The SC-18 SRAT product density of 1.23 g/mL is higher than the SB9 nitric-formic qualification SRAT product density of 1.15 mg/L [24]. The SC-18 SRAT and SME product supernatant densities of 1.09 g/mL and 1.13 g/mL, respectively, are equivalent to the supernate densities measured for the SRAT and SME products of the SB9 nitric-formic qualification [24].

Table 3-29: Densities and Solids Contents of SC-18 SRAT and SME Products

Measurement	SRAT Product		SME Product	
	Result	%RSD, *n*	Result	%RSD, *n*
Slurry density, g/mL	1.23	1.8, 5	1.43	1.1, 4
Supernatant density, g/mL	1.09	0.2, 4	1.13	2.0, 4
Total solids, wt% of slurry	24.6	0.2, 4	49.0	1.7, 4
Insoluble solids, wt% of slurry	13.2	NA	38.4	NA
Soluble solids, wt% of slurry	11.4	NA	10.6	NA
Calcined solids, wt% of slurry	16.5	1.0, 4	40.8	2.4, 4
Dissolved solids, wt% of supernatant	13.1	0.2, 4	17.2	0.2, 4

Concentrations of the elemental constituents in the SRAT and SME product slurries are given in Table 3-30 and Table 3-31, respectively, with results greater than 0.1 wt% shaded for easy identification. These tables contain analytical results for the primary elemental constituents measured by ICP-AES, sulfur by ICP-AES-S, mercury by CVAA, and uranium, thorium, neodymium, palladium, rhodium, ruthenium, and silver by ICP-MS. ICP-AES, ICP-AES-S, and ICP-MS have a 1-σ uncertainty of 10% and CVAA has a 1-σ uncertainty of 20%.

SRAT product results are consistent with the SRAT receipt analysis. Most concentrations of major components of the SRAT product are approximately 80% (76–84%) of their concentration in the SRAT receipt on a total solids basis due to the acid additions and reactions during SRAT processing. Mercury is an exception, where the concentration in the SRAT product is 32% of the concentration in the SRAT receipt due to the intentional reduction and stripping during the SRAT cycle. The other exceptions are Na, U, and S, where the reduction of concentration in the SRAT product is 90 to 92% of the concentration in the SRAT. These three components are not thought to be added or removed during SRAT processing, so the differences in the change between these and the other major sludge components is thought to be due to the analytical uncertainty. For Zr, SRAT product results are higher than SRAT receipt results because the CC preparation is better for Zr measurement than aqua regia preparation.

SME supernate results are consistent with the SRAT receipt and product analyses. The sodium result for the SME is not accurate because sodium cannot be measured by the sodium peroxide fusion digestion and frit cannot be fully dissolved by aqua regia digestion. The sodium result appears to have little or none of the sodium that was contained in the frit addition. For SME sodium analysis, the result for the glass analysis should be used (Section 3.7.1), which calculates to 8.32 wt% sodium in SME product on a total solids basis. The SME product analysis results for peroxide fusion digestions show the expected increase of Si, B, and Li due to the addition of frit and the decrease of sludge components on a total solids basis due to the dilution effect. In the SME product, most major sludge components were approximately 38% (35–42%) of their concentration in the SRAT receipt and approximately 46% (76–84%) of their concentration in the SRAT product on a total solids basis.

The final concentration of mercury in the SRAT product is 0.71 wt% of the total solids, which is below the target for mercury removal of 0.8 wt% of the total solids [25]. With the additional mercury stripping during the SME cycle and the dilution of total solids with frit, the mercury concentration in the SME product dropped to 0.21 wt% of the total solids. When the SME product is put back on the same basis as the SRAT product total solids, the mercury concentration of the SME product would correspond to 0.46 wt% of SRAT total solids, near the target mercury removal to 0.45 wt% total solids. It is expected that additional boiling would further remove mercury to levels below the target.

Based on the comparison of the iron in the SRAT product and SME product analysis, the waste loading of the SME product is 37.6%, which compares acceptably with the targeted value of 38% waste loading for this test. Calculations based on several of the other major sludge components (Al, Ca, Mn, Ni, and U) indicated waste loadings ranging from 36.6% to 37.9%, in good agreement with the Fe-based result.

Concentrations of select radioisotopes in the SRAT and SME slurry solids are given in Table 3-32 and Table 3-33, respectively, both on a mass concentration basis (wt% solids) and a slurry activity basis (Ci/gallon slurry). Relative isotopic makeup of the SRAT and SME products are consistent with the SRAT receipt. Pu-241 concentrations and activities were calculated from the measured Pu-238 activities in the SRAT and SME products and assuming the same ratio of Pu-241 to Pu-238 activity as in the SRAT receipt.

Table 3-30: Elemental Analysis of SRAT Product Slurry Solids (Shading Indicates Concentrations > 0.1 wt%)

Constituent	Digestion Method(s)	Concentration, wt% TS	%RSD, *n*
Ag	AR	9.03E-03	0.2, 3
Al	PF	5.86E+00	1.7, 4
As	AR	<6.5E-04	NA
B	AR	<5.7E-03	NA
Ba	AR & PF	6.18E-02	4.3, 7
Be	AR	1.48E-03	0.8, 3
Ca	AR	8.49E-01	1.0, 3
Cd	PF	1.35E-02	6.4, 4
Ce	AR	2.79E-02	5.0, 3
Co	AR	8.70E-03	0.3, 3
Cr	PF	7.25E-02	3.4, 4
Cu	AR	2.64E-02	3.2, 3
Fe	AR & PF	1.41E+01	1.4, 7
Gd	AR	7.69E-02	0.5, 3
Hg	AR	7.08E-01	2.1, 3
K	AR	<1.5E-01	NA
La	PF	3.13E-02	1.5, 4
Li	PF	2.98E-02	2.1, 4
Mg	PF	1.73E-01	1.7, 4
Mn	AR & PF	4.67E+00	1.1, 7
Mo	AR	<5.5E-03	NA
Na	AR	1.29E+01	0.9, 3
Nd	AR	1.20E-01	1.2, 3
Ni	AR & PF	1.06E+00	4.3, 7
P	AR	1.38E-01	4.1, 3
Pb	AR	4.38E-02	5.1, 3
Pd	AR	1.94E-03	1.0, 3
Rh	AR	1.01E-02	0.7, 3
Ru	AR	4.59E-02	1.2, 3
S	AR	2.52E-01	5.8, 3
Sb	AR	<5.0E-02	NA
Se	AR	<1.3E-03	NA
Si	PF	1.17E+00	2.5, 4
Sn	AR	<1.5E-02	NA
Sr	AR & PF	2.28E-02	3.9, 7
Th	AR	6.55E-01	0.6, 3
Ti	AR	1.80E-02	0.6, 3
U	AR	2.81E+00	0.7, 3
V	AR	<7.2E-04	NA
Zn	PF	2.13E-02	1.3, 4
Zr	CC	1.16E-01	0.3, 4

Table 3-31: Elemental Analysis of SME Product Slurry Solids (Shading Indicates Concentrations > 0.1 wt%)

Constituent	Digestion Method(s)	Concentration, wt% TS	%RSD, *n*
Ag	AR	4.37E-03	3.5, 4
Al	PF	2.72E+00	3.3, 4
As	AR	<4.1E-04	NA
B	PF	1.27E+00	2.9, 4
Ba	AR & PF	2.92E-02	4.2, 8
Be	AR	<1.2E-03	NA
Ca	AR	3.92E-01	2.8, 4
Cd	AR	6.23E-03	3.1, 4
Ce	AR & PF	9.29E-02	7.0, 8
Co	AR	<6.0E-03	NA
Cr	AR & PF	3.12E-02	5.6, 8
Cu	AR	1.17E-02	3.2, 4
Fe	AR & PF	6.55E+00	3.4, 8
Gd	AR	3.46E-02	2.9, 4
Hg	AR	2.14E-01	6.4, 4
K	AR	1.61E-01	50, 4
La	AR	1.73E-02	3.3, 4
Li	PF	1.45E+00	2.6, 4
Mg	AR	8.90E-02	3.1, 4
Mn	AR & PF	2.19E+00	3.2, 8
Mo	AR	<2.8E-03	NA
Na **	AR	5.60E+00	2.9, 4
Nd	AR	5.80E-02	2.5, 4
Ni	AR & PF	4.91E-01	3.3, 8
P	AR	5.56E-02	8.4, 4
Pb	AR	1.39E-02	6.3, 4
Pd	AR	8.82E-04	4.3, 4
Rh	AR	4.95E-03	2.8, 4
Ru	AR	2.20E-02	5.9, 4
S	AR	1.09E-01	2.7, 4
Sb	AR	<1.3E-02	NA
Se	AR	<8.2E-04	NA
Si	PF	1.99E+01	2.4, 3
Sn	AR	<7.8E-03	NA
Sr	AR	1.12E-02	3.1, 4
Th	AR	3.20E-01	2.2, 4
Ti	AR	8.41E-03	3.3, 4
U	AR	1.27E+00	2.9, 4
V	AR	<4.2E-04	NA
Zn	AR & PF	1.00E-02	5.9, 8
Zr	AR	1.44E-02	50, 4

** Sodium in the SME product is biased low due to incomplete dissolution of frit by aqua regia.

Table 3-32: Select Radioisotopes in the SRAT Product Slurry (*n* = 4)

Isotope	Mass Concentration, wt% TS	Activity Concentration, Ci/gal slurry	%RSD
Tc-99	1.29E-03	2.51E-04	0.8
Th-232	6.55E-01	8.23E-07	0.6
U-233	5.70E-04	6.31E-05	4.0
U-234	5.73E-04	4.10E-05	0.4
U-235	2.66E-02	6.58E-07	1.5
U-236	1.52E-03	1.12E-06	1.9
U-238	2.78E+00	1.07E-05	0.7
Np-237	2.37E-03	1.91E-05	0.4
Pu-238	6.32E-04	1.24E-01	11
Pu-239	7.68E-03	5.46E-03	1.1
Pu-240	7.62E-04	1.99E-03	0.3
Pu-239/240	NA	6.39E-03	21
Pu-241 **	2.17E-05	2.56E-02	NA
Am-241	4.86E-04	1.91E-02	3.9
Am-242m	1.71E-07	1.90E-05	14
Am-243	6.72E-05	1.53E-04	8.4
Cm-242	4.16E-10	1.57E-05	14
Cm-244	7.55E-06	6.99E-03	6.8
Cm-245	<1.1E-04	<2.2E-04	NA

** Pu-241 is calculated from Pu-238 activity using ratio in SRAT receipt.

Table 3-33: Select Radioisotopes in the SME Product Slurry (*n* = 4)

Isotope	Mass Concentration, wt% TS	Activity Concentration, Ci/gal slurry	%RSD
Tc-99	6.15E-04	2.76E-04	4.0%
Th-232	3.20E-01	9.32E-07	4.2%
U-233	2.90E-04	7.44E-05	2.6%
U-234	2.83E-04	4.70E-05	3.0%
U-235	1.22E-02	6.97E-07	3.6%
U-236	7.57E-04	1.30E-06	3.7%
U-238	1.26E+00	1.12E-05	2.5%
Np-237	1.20E-03	2.24E-05	2.9%
Pu-238	3.97E-04	1.80E-01	16%
Pu-239	3.92E-03	6.46E-03	2.4%
Pu-240	3.76E-04	2.28E-03	3.4%
Pu-239/240	NA	9.99E-03	15%
Pu-241 **	1.37E-05	3.73E-02	NA
Am-241	2.01E-04	1.83E-02	2.5%
Am-242m *	7.11E-08	1.83E-05	33%
Am-243	3.02E-05	1.60E-04	4.4%
Cm-242 *	1.73E-10	1.52E-05	33%
Cm-244	3.25E-06	6.98E-03	3.6%
Cm-245	<6.8E-05	<3.1E-04	NA

* Am-242m and Cm-242 are calculated form a combination of below and above detection limit values
** Pu-241 is calculated from Pu-238 activity using ratio in SRAT receipt.

Concentrations of anions and TIC/TOC in the SRAT and SME product slurries are given in Table 3-34 and Table 3-35, respectively. This includes slurry results determined by water dilution, caustic quench, and acid digestion approaches. In general for slurry analysis (as indicated by the bold style in the table), the caustic quench preparation results should be used for the three organic anions (glycolate, formate, and oxalate) and the water dilution preparation results may be better to use for the inorganic anions (nitrite, nitrate, etc.). The caustic quench and oxalate preps appear to be equivalent when considering the 1-σ analytical uncertainty of 10% for the IC anion measurements. A more detailed comparison of the three preparation methods used for IC is contained in Section 3.5.2.7.

Changes in anions are noted when comparing the SRAT and SME products to the SRAT receipt due to the chemistry of SRAT processing. The nitric and glycolic acids added in the SRAT cycle directly contribute to an increase in the nitrate and glycolate in the SRAT and SME products from the level in the SRAT receipt. In the SRAT and SME cycles, nitrite is destroyed to a 304 mg/kg of SRAT product slurry and 380 mg/kg of SME product slurry. Formate and oxalate are also increased in the SRAT and SME products from levels in the SRAT receipt due to the process chemistry. Based on the pH of the SRAT and SME products, the TIC carbon likely corresponds to bicarbonate anion.

Table 3-34: Anions and TIC/TOC in the SRAT Product Slurry (n = 4)

Constituent (mg/kg slurry)	Water Dilution		Caustic Quench		Oxalate Prep	
	Concentration	%RSD	Concentration	%RSD	Concentration	%RSD
Glycolate	3.38E+04	0.8	**3.71E+04**	0.8	NA	NA
Formate	1.41E+03	0.9	**1.45E+03**	0.5	NA	NA
Chloride	< 1.2E+02	NA	NA	NA	NA	NA
Nitrite	**3.04E+02**	0.6	< 6.4E+02	NA	NA	NA
Bromide	< 1.2E+02	NA	< 6.4E+02	NA	NA	NA
Nitrate	**4.29E+04**	0.7	4.10E+04	0.7	NA	NA
Phosphate	< 1.2E+02	NA	< 6.4E+02	NA	NA	NA
Sulfate	**1.27E+03**	0.7	1.23E+03	0.9	NA	NA
Oxalate	2.42E+03	3.2	**4.79E+03**	1.9	**5.19E+03**	3.8
TIC	1.39E+03	1.0	NA	NA	NA	NA
TOC	1.33E+04	0.6	NA	NA	NA	NA

Table 3-35: Anions and TIC/TOC in the SME Product Slurry (n = 4)

Constituent (mg/kg slurry)	Water Dilution		Caustic Quench		Oxalate Prep	
	Concentration	%RSD	Concentration	%RSD	Concentration	%RSD
Fluoride	<1.2E+03	NA	<6.5E+02	NA	NA	NA
Glycolate	3.16E+04	1.4	**3.27E+04**	2.2	NA	NA
Formate	1.95E+03	1.2	**1.49E+03**	3.0	NA	NA
Chloride	<1.2E+02	NA	NA	NA	NA	NA
Nitrite	**3.80E+02**	1.2	<6.5E+02	NA	NA	NA
Bromide	<1.2E+03	NA	<3.3E+03	NA	NA	NA
Nitrate	**4.66E+04**	3.7	4.21E+04	3.1	NA	NA
Phosphate	<1.2E+02	NA	<6.5E+02	NA	NA	NA
Sulfate	**1.19E+03**	1.1	1.26E+03	3.1	NA	NA
Oxalate	2.36E+03	1.0	**4.92E+03**	3.0	**4.86E+03**	3.6
TIC	1.48E+03	2.8	NA	NA	NA	NA
TOC	1.19E+04	1.3	NA	NA	NA	NA
VOA	<6.22E+00	NA	NA	NA	NA	NA

Concentrations of elemental constituents in the SRAT and SME product supernatant are given in Table 3-36 and Table 3-37, respectively. Concentrations of select radioisotopes in the SRAT and SME product supernatant are given in Table 3-38 and Table 3-39. Similar to the SRAT receipt, the major soluble elemental cation in the SRAT and SME products is sodium. However, some metals become significantly more soluble during the SRAT cycle. Manganese concentration in the SRAT and SME supernate is 2600 and 2890 mg/L, respectively. Uranium concentration in the SRAT and SME supernate is 1220 and 1640 mg/L, respectively.

Table 3-36: Elemental Analysis of SRAT Product Supernatant (mg/L of Filtrate)

Constituent	Concentration, mg/L	%RSD, *n*
Ag	<5.0E-02	NA
Al	1.42E+02	5.9, 4
B	<2.6E+01	NA
Ba	<4.7E+00	NA
Be	<2.6E-01	NA
Ca	<1.1E+02	NA
Cd	<8.2E+00	NA
Ce	<2.5E+01	NA
Co	<1.2E+01	NA
Cr	1.71E+01	14, 4
Cu	<3.0E+01	NA
Fe	1.38E+01	6.6, 4
Gd	1.37E+01	17, 4
Hg	1.66E+01	0.7, 4
K	<7.1E+02	NA
La	6.48E+00	11, 4
Li	<7.7E+01	NA
Mg	1.32E+02	4.3, 4
Mn	2.60E+03	4.3, 4
Mo	<2.5E+01	NA
Na	3.30E+04	4.3, 4
Nd	8.72E+00	3.9, 4
Ni	2.79E+01	11, 4
P	<1.1E+02	NA
Pb	<1.1E+02	NA
Pd	<4.5E-02	NA
Rh	3.89E+00	4.2, 4
Ru	1.13E+01	4.3, 4
S	5.40E+02	9.1, 4
Sb	<2.3E+02	NA
Si	<1.1E+02	NA
Sn	<7.2E+01	NA
Sr	1.64E+01	4.1, 4
Th	1.53E+00	3.5, 4
Ti	<4.9E+01	NA
U	1.22E+03	4.5, 4
V	<3.3E+01	NA
Zn	<3.2E+00	NA
Zr	<3.9E+00	NA

Table 3-37: Elemental Analysis of SME Product Supernatant (mg/L of Filtrate)

Constituent	Concentration, mg/L	%RSD, *n*
Ag	<5.3E-02	NA
Al	1.54E+02	0.4, 4
B	4.34E+01	0.6, 4
Ba	4.18E+00	2.0, 4
Be	<5.0E-01	NA
Ca	3.52E+01	0.9, 4
Cd	<2.6E+00	NA
Ce	<2.3E+01	NA
Co	<7.4E+00	NA
Cr	2.08E+01	0.7, 4
Cu	<5.9E+00	NA
Fe	4.55E+01	9.9, 4
Gd	2.24E+01	0.6, 4
Hg	2.81E+00	11, 4
K	2.23E+02	1.2, 4
La	<3.3E+00	NA
Li	1.48E+02	0.5, 4
Mg	1.69E+02	0.6, 4
Mn	2.89E+03	0.2, 4
Mo	<1.0E+01	NA
Na	4.67E+04	0.2, 4
Nd	1.25E+01	0.4, 4
Ni	3.34E+01	1.7, 4
P	<1.2E+01	NA
Pb	<1.2E+01	NA
Pd	<4.7E-02	NA
Rh	5.51E+00	0.6, 4
Ru	1.36E+01	0.8, 4
S	7.85E+02	0.7, 4
Sb	<2.4E+01	NA
Si	3.84E+01	13, 4
Sn	<7.5E+00	NA
Sr	2.18E+01	0.6, 4
Th	2.46E+00	0.3, 4
Ti	<2.6E+00	NA
U	1.64E+03	0.4, 4
V	<3.5E+00	NA
Zn	<1.3E+00	NA
Zr	<2.0E+00	NA

Table 3-38: Select Radioisotopes in the SRAT Product Supernatant (*n* = 4)

Isotope	Mass Concentration, mg/L filtrate	Activity Concentration, Ci/gal filtrate	%RSD
Tc-99	1.11E+00	7.13E-05	5.2
Th-232	1.53E+00	6.34E-10	3.5
U-233	1.89E-01	6.91E-06	3.2
U-234	2.48E-01	5.87E-06	4.6
U-235	1.16E+01	9.45E-08	4.5
U-236	6.79E-01	1.66E-07	3.5
U-238	1.21E+03	1.53E-06	4.5
Np-237	1.44E+00	3.84E-06	3.4
Pu-238	1.77E-03	1.14E-04	4.6
Pu-239	1.02E-01	2.39E-05	3.7
Pu-240	<2.7E-02	<2.4E-05	NA
Pu-239/240	NA	1.07E-05	29
Pu-241 **	6.07E-05	2.37E-05	NA
Am-241	1.22E-02	1.58E-04	7.2
Am-242m	<1.0E-05	<3.7E-07	NA
Am-243	<4.5E-02	<3.4E-05	NA
Cm-242	<2.5E-08	<3.1E-07	NA
Cm-244	4.26E-04	1.31E-04	6.3
Cm-245	<1.6E-01	<1.0E-04	NA

** Pu-241 is calculated from Pu-238 activity using ratio in SRAT receipt.

Table 3-39: Select Radioisotopes in the SME Product Supernatant (*n* = 4)

Isotope	Mass Concentration, mg/L filtrate	Activity Concentration, Ci/gal filtrate	%RSD
Tc-99	1.73E+00	1.11E-04	0.8
Th-232	2.46E+00	1.02E-09	0.3
U-233	2.65E-01	9.71E-06	0.7
U-234	3.21E-01	7.59E-06	1.4
U-235	1.54E+01	1.26E-07	0.4
U-236	9.39E-01	2.30E-07	0.4
U-238	1.62E+03	2.07E-06	0.4
Np-237	2.35E+00	6.26E-06	0.6
Pu-238	2.60E-03	1.69E-04	11
Pu-239	1.77E-01	4.16E-05	1.6
Pu-240	<2.8E-02	<2.5E-05	NA
Pu-239/240	NA	1.87E-05	30
Pu-241 **	<9.0E-05	<3.5E-05	NA
Am-241	1.53E-02	1.99E-04	53
Am-242m *	<2.1E-05	<7.7E-07	NA
Am-243	<5.2E-03	<4.0E-06	NA
Cm-242 *	<4.5E-08	<5.6E-07	NA
Cm-244	3.98E-04	1.22E-04	44
Cm-245	<1.7E-02	<1.1E-05	NA

* Am-242m and Cm-242 are calculated form a combination of below and above detection limit values
** Pu-241 is calculated from Pu-238 activity using ratio in SRAT receipt.

Concentrations of anions and TIC/TOC in the SRAT and SME product supernatants are given in Table 3-40 and Table 3-41, respectively. For most components in the supernate, the caustic quench and oxalate preps appear to be equivalent when considering the 1-σ analytical uncertainty of 10% for the IC anion measurements. A more detailed comparison of the three preparation methods used for IC is contained in Section 3.5.2.7.

Most anions are more concentrated in the SME product than in the SRAT product when compared on a mg/L basis, which is consistent with the higher density and dissolved solids content of the SME product. Based on the pH of the SRAT and SME products, the soluble TIC carbon likely corresponds to bicarbonate anion. The insoluble TIC in the SRAT, and to a lesser extent the SME, is likely carbonates of the alkaline earth metals (Ca, Mg, Sr). The difference in TIC solubility between the SRAT and the SME suggests that equilibrium had not been achieved with respect to the solubility of these metal carbonates by the end of the SRAT process, though this trend was not reflected in the Ca, Mg, and Sr solubilities.

Table 3-42 contains a comparison of the anion and TIC/TOC concentrations in the SRAT and SME products by casting the data in Table 3-40 and Table 3-41 in terms of wt% of the dissolved solids. This comparison is not intended to sum to 100% because it does not include soluble cations and also includes TOC, which is partially redundant with the organic anions. From this comparison, it appears that there is no significant change in the nitrite concentration between the SRAT and the SME. There is evidence that formate and soluble TIC (and possibly oxalate) are formed between the SRAT product and the SME product. While the direct evidence of additional glycolate decomposition in the SME based on the glycolate concentration measurements is not strong, the additional formate and TIC formed is consistent with a small amount of additional glycolate decomposition in the SME. Based on the complete set of data, the nitrate measurement of the water dissolution of the SME product appears to be anomalously high.

Table 3-40: Anions, TIC, and TOC in SRAT Product Supernatant (mg/L of Filtrate, *n* = 4)

Constituent (mg/L filtrate)	Water Dilution		Caustic Quench	
	Concentration	%RSD	Concentration	%RSD
Fluoride	<1.4E+03	NA	<6.6E+02	NA
Glycolate	4.03E+04	1.8	4.19E+04	2.3
Formate	1.78E+03	2.2	1.89E+03	3.1
Chloride	<1.4E+02	NA	NA	NA
Nitrite	3.93E+02	2.7	<6.6E+02	NA
Bromide	<6.8E+02	NA	<3.3E+03	NA
Nitrate	5.40E+04	1.7	5.21E+04	2.6
Phosphate	<1.4E+02	NA	<6.6E+02	NA
Sulfate	1.55E+03	2.1	1.54E+03	3.9
Oxalate	2.80E+03	3.4	2.15E+03	3.3
TIC	2.31E+02	9.5	NA	NA
TOC	1.56E+04	1.4	NA	NA

Table 3-41: Anions, TIC, and TOC in SME Product Supernatant (mg/L of Filtrate, *n* = 4)

Constituent (mg/L filtrate)	Water Dilution		Caustic Quench	
	Concentration	%RSD	Concentration	%RSD
Fluoride	<1.4E+03	NA	<7.2E+02	NA
Glycolate	5.48E+04	11	5.40E+04	1.7
Formate	3.75E+03	11	3.75E+03	1.2
Chloride	9.71E+01*	13	NA	NA
Nitrite	6.64E+02	12	<7.2E+02	NA
Bromide	<1.4E+03	NA	<1.4E+03	NA
Nitrate	8.72E+04	10	6.96E+04	1.4
Phosphate	<2.8E+02	NA	<7.2E+02	NA
Sulfate	2.41E+03	12	2.11E+03	1.3
Oxalate	4.45E+03	7.9	3.46E+03	1.5
TIC	1.22E+03	3.6	NA	NA
TOC	2.03E+04	11	NA	NA

* excludes one chloride outlier result of 1.02E+03 mg/L of filtrate

Table 3-42: Comparison of SRAT and SME Product Supernatant Anions on a Dissolved Solids Basis

Wt% of DS in filtrate	SRAT Product		SME Product	
	WD	CQ	WD	CQ
Glycolate	28.3	29.4	28.3	27.9
Formate	1.2	1.3	1.9	1.9
Nitrite	0.3	–	0.3	–
Nitrate	37.9	36.5	45.0	35.9
Sulfate	1.1	1.1	1.2	1.1
Oxalate	2.0	1.5	2.3	1.8
TIC	0.2	–	0.6	–
TOC	10.9	–	10.5	–

Table 3-43 contains a comparison of key constituents in the filtrate to the constituents in the slurry for the SRAT and SME products. The slurry data are given as “Total” and the filtrate data are given as “Soluble”. Both types of data are given in units of mg/kg of slurry, allowing for the ratio of the soluble to the total to be the fraction that is soluble. The constituents included in the comparison are primarily the analytes where below-detection-limit values were not involved in most cases.

By this comparison, the anions glycolate, formate, nitrite, nitrate, and sulfate appear to be fully or nearly fully soluble in both the SRAT and SME products, with deviations from 100% soluble being well within the experimental uncertainty. Oxalate is approximately 50% soluble and TIC solubility is particularly low in the SRAT product.

Na was mostly soluble in the SRAT product, but incomplete frit dissolution prevented quantification of Na solubility in the SME product. The percentage of Mn soluble was 18% in the SRAT product and 15% in the SME product, while the percentage of Fe soluble in the CPC products remained relatively low (<0.1%). There was significant percent solubility of the noble metals Rh (12%) and Ru (7–8%) in the CPC products.

The percent of U soluble increased to approximately 14% in the SRAT and SME products. Tc was approximately 30% soluble in the SRAT and SME products, Np and Sr were approximately 20% soluble, and Th was less than 0.1% soluble. Pu remained less the 0.1% soluble in the CPC products, Am less than 1% soluble, and Cm less than 2% soluble. Pu-238 is a better estimate of soluble percent than Pu-239 due to smaller measurement uncertainties for the Pu-238 concentrations.

Table 3-43: Percentages of Components Soluble in the SRAT and SME Products

Constituent	SRAT Product			SME Product		
	Total	Soluble	**Soluble**	Total	Soluble	**Soluble**
	mg/kg slurry	mg/kg slurry	**%**	mg/kg slurry	mg/kg slurry	**%**
Glycolate	3.71E+04	3.33E+04	**90%**	3.27E+04	2.99E+04	**91%**
Formate	1.45E+03	1.50E+03	**104%**	1.95E+03	2.05E+03	**105%**
Nitrite	3.04E+02	3.13E+02	**103%**	3.80E+02	3.63E+02	**96%**
Nitrate	4.29E+04	4.30E+04	**100%**	4.66E+04	4.76E+04	**102%**
Sulfate	1.27E+03	1.23E+03	**97%**	1.19E+03	1.32E+03	**111%**
Oxalate	4.79E+03	2.23E+03	**46%**	4.92E+03	2.43E+03	**49%**
TIC	1.39E+03	1.84E+02	**13%**	1.48E+03	6.67E+02	**45%**
TOC	1.33E+04	1.24E+04	**93%**	1.19E+04	1.11E+04	**93%**
Al	1.44E+04	1.13E+02	**0.79%**	1.34E+04	8.39E+01	**0.6%**
B	<1.4E+01	<2.1E+01	**NA**	6.24E+03	2.37E+01	**0.4%**
Ca	2.09E+03	<8.4E+01	**<4%**	1.92E+03	1.92E+01	**1.0%**
Cr	1.78E+02	1.36E+01	**7.6%**	1.53E+02	1.14E+01	**7.4%**
Fe	3.46E+04	1.10E+01	**0.032%**	3.21E+04	2.48E+01	**0.077%**
Gd	1.89E+02	1.09E+01	**5.8%**	1.70E+02	1.22E+01	**7.2%**
K	<3.8E+02	<5.7E+02	**NA**	7.91E+02	1.22E+02	**15%**
La	7.70E+01	5.16E+00	**6.7%**	8.46E+01	<1.8E+00	**<2%**
Li	7.33E+01	<6.2E+01	**NA**	7.09E+03	8.07E+01	**1.1%**
Mg	4.25E+02	1.05E+02	**25%**	4.36E+02	9.21E+01	**21%**
Mn	1.15E+04	2.07E+03	**18%**	1.07E+04	1.58E+03	**15%**
Na	3.16E+04	2.63E+04	**83%**	2.74E+04*	2.55E+04	**NA**
Nd	2.94E+02	6.94E+00	**2.4%**	2.84E+02	6.80E+00	**2.4%**
Ni	2.60E+03	2.22E+01	**0.86%**	2.41E+03	1.83E+01	**0.8%**
Ru	1.13E+02	9.03E+00	**8.0%**	1.08E+02	7.42E+00	**6.9%**
Rh	2.49E+01	3.10E+00	**12%**	2.42E+01	3.01E+00	**12%**
S	6.20E+02	4.30E+02	**69%**	5.34E+02	4.29E+02	**80%**
Sr	5.61E+01	1.31E+01	**23%**	5.47E+01	1.19E+01	**22%**
Th	1.61E+03	1.22E+00	**0.076%**	1.57E+03	1.34E+00	**0.086%**
U	6.90E+03	9.70E+02	**14%**	6.22E+03	8.96E+02	**14%**
Tc-99	3.17E+00	8.85E-01	**28%**	3.01E+00	9.44E-01	**31%**
Np-237	5.82E+00	1.15E+00	**20%**	5.86E+00	1.28E+00	**22%**
Pu-238	1.55E+00	1.41E-03	**0.091%**	1.95E+00	1.42E-03	**0.073%**
Pu-239	1.89E+01	8.10E-02	**0.43%**	1.92E+01	9.66E-02	**0.50%**
Am-241	1.19E+00	9.70E-03	**0.81%**	9.86E-01	8.38E-03	**0.85%**
Cm-244	1.85E-02	3.39E-04	**1.8%**	1.59E-02	2.17E-04	**1.4%**

* Concentration of Na in the SME product slurry is biased low due to incomplete dissolution of frit.

3.5.2.4 Comparison of Replicate Slurry Analyses

The Technical Task Request [1] governing this work requested that all replicates of certain elemental components (Fe, Gd, Mn, and Th) be reported for the SRAT receipt, the SRAT product, and the SME product. Similarly, it was requested that all replicates be reported for uranium and plutonium isotopes, Am-242m, Cm-244, and Cm-245. This subsection contains the data on the individual results for the requested constituents for the SRAT product slurry (Table 3-44 and Table 3-45), SRAT product filtrate (Table 3-46 and Table 3-47), SME product slurry (Table 3-48 and Table 3-49), and SME product filtrate (Table 3-50 and Table 3-51). Results measured by ICP-AES and ICP-MS are presented with units of mg/kg of slurry for slurry samples and mg/L of filtrate for filtrate samples. Results measured by radiochemical analyses are presented with units of dpm/g of slurry for slurry samples and dpm/mL of filtrate for filtrate samples. Counting uncertainties are provided with the radiochemical analysis as a major element of the overall analysis uncertainty. High counting uncertainties are encountered when isotopes are at low concentrations approaching the detectable level for the method performed.

Table 3-44: Replicate Analysis of SRAT Product Slurry

Constituent (mg/kg slurry)	Method	Measured Concentration			
		Result 1	Result 2	Result 3	Result 4
Fe by AR	ICP-AES	3.48E+04	3.50E+04	3.49E+04	NA
Fe by PF	ICP-AES	3.38E+04	3.45E+04	3.43E+04	3.52E+04
Gd by AR	ICP-AES	1.90E+02	1.89E+02	1.88E+02	NA
Gd by PF	ICP-AES	1.63E+02	1.55E+02	1.42E+02	1.53E+02
Mn by AR	ICP-AES	1.15E+04	1.15E+04	1.15E+04	NA
Mn by PF	ICP-AES	1.13E+04	1.14E+04	1.14E+04	1.17E+04
Th by AR	ICP-AES	1.80E+03	1.78E+03	1.80E+03	NA
Th by PF	ICP-AES	1.68E+03	1.68E+03	1.62E+03	1.73E+03
U by AR	ICP-AES	6.68E+03	6.62E+03	6.73E+03	NA
U by PF	ICP-AES	6.65E+03	6.76E+03	6.27E+03	6.80E+03
Th-232	ICP-MS	1.61E+03	1.62E+03	1.60E+03	NA
U-233	ICP-MS	1.46E+00	1.39E+00	1.35E+00	NA
U-234	ICP-MS	1.40E+00	1.41E+00	1.41E+00	NA
U-235	ICP-MS	6.64E+01	6.44E+01	6.53E+01	NA
U-236	ICP-MS	3.80E+00	3.66E+00	3.71E+00	NA
U-238	ICP-MS	6.87E+03	6.83E+03	6.77E+03	NA
Pu-239	ICP-MS	1.87E+01	1.91E+01	1.88E+01	NA
Pu-240	ICP-MS	1.87E+00	1.88E+00	1.87E+00	NA
Mass 241	ICP-MS	1.16E+00	1.17E+00	1.19E+00	NA
Mass 242	ICP-MS	1.21E-01	1.42E-01	1.45E-01	NA
Mass 243	ICP-MS	1.45E-01	1.56E-01	1.56E-01	NA

Table 3-45: Replicate Radiochemical Analysis of SRAT Product Slurry (dpm/g of slurry)

Constituent (dpm/g slurry)	Result 1		Result 2		Result 3		Result 4	
	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.
Pu-238	6.56E+07	7.7%	5.60E+07	9.8%	5.16E+07	7.6%	6.28E+07	8.9%
Pu-239/240	3.74E+06	7.8%	2.35E+06	10.8%	2.66E+06	8.0%	3.43E+06	9.3%
Am-241	9.10E+06	5.0%	9.00E+06	5.0%	8.71E+06	5.0%	9.57E+06	5.0%
Am-242m	7.80E+03	20.1%	8.70E+03	21.6%	8.97E+03	19.1%	1.08E+04	19.5%
Am-243	6.88E+04	11.0%	7.03E+04	11.3%	7.09E+04	11.9%	8.22E+04	11.9%
Cm-242	6.45E+03	20.1%	7.19E+03	21.6%	7.41E+03	19.1%	8.94E+03	19.5%
Cm-244	3.31E+06	15.1%	3.24E+06	15.7%	3.12E+06	15.6%	3.65E+06	15.0%
Cm-245	<1.4E+05	NA	<1.3E+05	NA	<6.9E+04	NA	<9.0E+04	NA

Table 3-46: Replicate Analysis of SRAT Product Supernatant

Constituent (mg/L filtrate)	Method	Measured Concentration			
		Result 1	Result 2	Result 3	Result 4
Fe	ICP-AES	1.46E+01	1.31E+01	1.29E+01	1.46E+01
Gd	ICP-AES	1.63E+01	1.31E+01	1.07E+01	1.45E+01
Mn	ICP-AES	2.51E+03	2.54E+03	2.60E+03	2.76E+03
Th	ICP-AES	<5.56E+01	<5.68E+01	<5.39E+01	<5.4E+01
U	ICP-AES	<2.46E+03	<2.51E+03	<2.38E+03	<2.4E+03
Th-232	ICP-MS	1.49E+00	1.51E+00	1.51E+00	1.61E+00
U-233	ICP-MS	1.84E-01	1.85E-01	1.89E-01	1.97E-01
U-234	ICP-MS	2.40E-01	2.39E-01	2.50E-01	2.64E-01
U-235	ICP-MS	1.11E+01	1.13E+01	1.16E+01	1.23E+01
U-236	ICP-MS	6.58E-01	6.67E-01	6.78E-01	7.12E-01
U-238	ICP-MS	1.16E+03	1.17E+03	1.21E+03	1.28E+03
Pu-239	ICP-MS	9.82E-02	1.01E-01	1.01E-01	1.07E-01
Pu-240	ICP-MS	<2.6E-02	<2.8E-02	<2.9E-02	<2.7E-02

Table 3-47: Replicate Radiochemical Analysis of SRAT Product Supernatant (dpm/mL of filtrate)

Constituent (dpm/mL filtrate)	Result 1		Result 2		Result 3		Result 4	
	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.
Pu-238	7.10E+04	8.9%	6.68E+04	12.0%	6.34E+04	11.2%	6.72E+04	9.2%
Pu-239/240	6.76E+03	41.8%	3.94E+03	75.1%	8.39E+03	33.4%	6.01E+03	40.4%
Am-241	9.10E+04	7.9%	9.09E+04	7.0%	1.02E+05	6.4%	8.70E+04	6.3%
Am-242m	<3.0E+02	NA	<2.8E+02	NA	<1.3E+02	NA	<1.6E+02	NA
Am-243	<1.2E+04	NA	<7.1E+03	NA	<7.6E+03	NA	<5.3E+04	NA
Cm-242	<2.5E+02	NA	<2.3E+02	NA	<1.1E+02	NA	<1.3E+02	NA
Cm-244	7.43E+04	8.8%	7.55E+04	8.1%	8.36E+04	6.9%	7.29E+04	6.8%
Cm-245	<3.2E+04	NA	<1.8E+04	NA	<2.3E+04	NA	<1.6E+05	NA

Table 3-48: Replicate Analysis of SME Product Slurry

Constituent (mg/kg slurry)	Method	Measured Concentration			
		Result 1	Result 2	Result 3	Result 4
Fe by AR	ICP-AES	3.20E+04	3.40E+04	3.20E+04	3.22E+04
Fe by PF	ICP-AES	3.00E+04	3.23E+04	3.20E+04	3.23E+04
Gd by AR	ICP-AES	1.67E+02	1.77E+02	1.68E+02	1.67E+02
Gd by PF	ICP-AES	1.29E+02	1.37E+02	1.25E+02	1.29E+02
Mn by AR	ICP-AES	1.06E+04	1.13E+04	1.06E+04	1.06E+04
Mn by PF	ICP-AES	1.01E+04	1.09E+04	1.08E+04	1.09E+04
Th by AR	ICP-AES	1.62E+03	1.72E+03	1.62E+03	1.63E+03
Th by PF	ICP-AES	1.42E+03	1.49E+03	1.46E+03	1.50E+03
U by AR	ICP-AES	5.69E+03	6.04E+03	5.69E+03	5.71E+03
U by PF	ICP-AES	5.52E+03	5.67E+03	5.45E+03	5.70E+03
Th-232	ICP-MS	1.56E+03	1.62E+03	1.56E+03	1.54E+03
U-233	ICP-MS	1.36E+00	1.44E+00	1.38E+00	1.49E+00
U-234	ICP-MS	1.38E+00	1.44E+00	1.35E+00	1.38E+00
U-235	ICP-MS	5.88E+01	6.22E+01	5.82E+01	5.90E+01
U-236	ICP-MS	3.69E+00	3.91E+00	3.63E+00	3.63E+00
U-238	ICP-MS	6.08E+03	6.42E+03	6.03E+03	6.09E+03
Pu-239	ICP-MS	1.89E+01	1.99E+01	1.89E+01	1.90E+01
Pu-240	ICP-MS	1.82E+00	1.91E+00	1.83E+00	1.82E+00
Mass 241	ICP-MS	1.17E+00	1.24E+00	1.15E+00	1.19E+00
Mass 242	ICP-MS	<2.20E-01	<2.20E-01	<2.16E-01	<2.24E-01
Mass 243	ICP-MS	<2.20E-01	<2.20E-01	<2.16E-01	<2.24E-01

Table 3-49: Replicate Radiochemical Analysis of SME Product Slurry (dpm/g of slurry)

Constituent (dpm/g slurry)	Result 1		Result 2		Result 3		Result 4	
	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.
Pu-238	5.69E+07	10.1%	7.84E+07	6.7%	7.75E+07	7.0%	8.30E+07	7.9%
Pu-239/240	3.20E+06	11.1%	4.27E+06	7.5%	4.30E+06	8.1%	4.61E+06	8.8%
Am-241	7.73E+06	5.0%	7.29E+06	5.0%	7.47E+06	5.0%	7.57E+06	5.0%
Am-242m	9.03E+03	56.8%	9.75E+03	65.2%	<7.1E+03	NA	<4.2E+03	NA
Am-243	6.74E+04	12.2%	6.40E+04	18.7%	6.82E+04	11.9%	6.20E+04	11.6%
Cm-242	7.47E+03	56.8%	8.07E+03	65.2%	<5.9E+03	NA	<3.4E+03	NA
Cm-244	2.86E+06	15.2%	2.72E+06	15.3%	2.90E+06	15.2%	2.96E+06	15.4%
Cm-245	<1.1E+05	NA	<2.7E+05	NA	<8.2E+04	NA	<4.5E+04	NA

Table 3-50: Replicate Analysis of SME Product Supernatant

Constituent (mg/L filtrate)	Method	Measured Concentration			
		Result 1	Result 2	Result 3	Result 4
Fe	ICP-AES	4.68E+01	5.13E+01	4.20E+01	4.19E+01
Gd	ICP-AES	2.26E+01	2.24E+01	2.24E+01	2.22E+01
Mn	ICP-AES	2.88E+03	2.89E+03	2.89E+03	2.89E+03
Th	ICP-AES	<3.20E+01	<3.20E+01	<3.33E+01	<3.22E+01
U	ICP-AES	1.63E+03	1.64E+03	1.63E+03	1.63E+03
Th-232	ICP-MS	2.47E+00	2.45E+00	2.46E+00	2.45E+00
U-233	ICP-MS	2.65E-01	2.63E-01	2.65E-01	2.67E-01
U-234	ICP-MS	3.25E-01	3.25E-01	3.18E-01	3.15E-01
U-235	ICP-MS	1.54E+01	1.55E+01	1.54E+01	1.54E+01
U-236	ICP-MS	9.44E-01	9.37E-01	9.39E-01	9.36E-01
U-238	ICP-MS	1.62E+03	1.63E+03	1.62E+03	1.62E+03
Pu-239	ICP-MS	1.80E-01	1.74E-01	1.78E-01	1.75E-01
Pu-240	ICP-MS	<2.8E-02	<2.8E-02	<2.8E-02	<3.0E-02

Table 3-51: Replicate Radiochemical Analysis of SME Product Supernatant (dpm/mL of filtrate)

Constituent (dpm/mL filtrate)	Result 1		Result 2		Result 3		Result 4	
	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.	Measurement	1σ unc.
Pu-238	8.28E+04	9.8%	1.03E+05	10.1%	1.06E+05	8.4%	1.04E+05	9.9%
Pu-239/240	8.12E+03	41.9%	1.19E+04	31.6%	1.52E+04	16.0%	8.67E+03	34.3%
Am-241	1.50E+05	31.1%	1.88E+05	23.9%	7.37E+04	22.6%	5.66E+04	30.9%
Am-242m	<7.6E+02	NA	3.34E+02	55.5%	<2.7E+02	NA	<4.4E+02	NA
Am-243	<2.2E+03	NA	<2.7E+03	NA	<2.2E+03	NA	<2.2E+03	NA
Cm-242	<6.3E+02	NA	1.00E+02	55.5%	<2.2E+02	NA	<3.6E+02	NA
Cm-244	1.18E+05	41.3%	5.35E+04	34.3%	6.32E+04	33.0%	5.07E+04	41.6%
Cm-245	<5.9E+03	NA	<8.9E+03	NA	<5.6E+03	NA	<5.6E+03	NA

3.5.2.5 SRAT and SME pH Data

The pH probe in the laboratory-scale SRAT vessel began malfunctioning near the end of glycolic acid addition. Thus, pH was measured on the intermediate samples taken during the SRAT process and on the final SRAT and SME product samples. Figure 3-9 contains the pH data for the samples taken during the SRAT process, as well as data collected from the pH probe while functioning. The SRAT sample taken at the conclusion of glycolic acid addition had the lowest pH (approximately 4.8) while the pH of the subsequent samples trended from near 6.3 in the sample taken after less than 3 hours from the end of acid addition to near 7.0 in the SRAT product sample. The error bar on the pH result is taken as 0.10 pH units and is consistent with the pre- and post-measurement calibration check. The SME product sample pH, which is not included in Figure 3-9, was measured as 6.96.

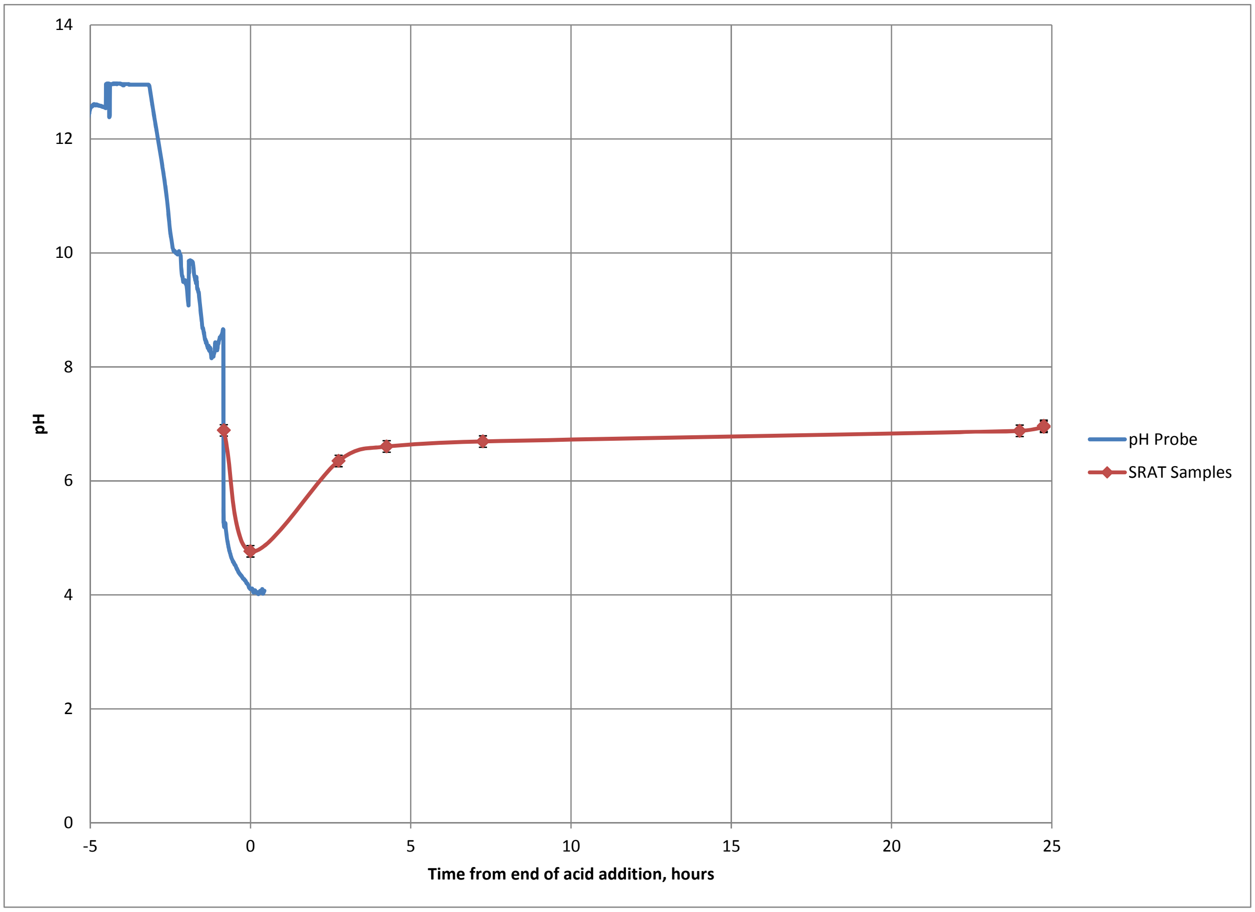


Figure 3-9: pH Profile During the SRAT as Determined from Intermediate and Final Samples

3.5.2.6 Processing Data

Processing data were collected by the DAC. Data collected included process temperatures, heat transfer coefficients, and mixer torque. Processing times for key steps during the SRAT and SME cycles are presented in Table 3-52.

Table 3-52: Processing Times for Key Steps in the SRAT and SME Cycles

SRAT	Time	Date
Start Acid Addition	0936	5/11/2016
End Acid Addition	1245	5/11/2016
Start Dewater	1358	5/11/2016
End Dewater	1714	5/11/2016
Begin Reflux	1714	5/11/2016
End of SRAT	1327	5/12/2016
SME	Time	Date
Start Canister Decon #1	2010	6/2/2016
End Canister Decon #1	2145	6/2/2016
Start Canister Decon #2	2210	6/2/2016
End Canister Decon #2	2346	6/2/2016
Start Canister Decon #3	0020	6/3/2016
End Canister Decon #3	0204	6/3/2016
Start Canister Decon #4	0230	6/3/2016
End Canister Decon #4	0416	6/3/2016
Start Canister Decon #5	0444	6/3/2016
End Canister Decon #5	0631	6/3/2016
Start Canister Decon #6	0701	6/3/2016
End Canister Decon #6	0842	6/3/2016
Frit Addition #1	0935	6/3/2016
Start Frit Dewater #1	0953	6/3/2016
End Frit Dewater #1	1121	6/3/2016
Frit Addition #2	1145	6/3/2016
Start Frit Dewater #2	1217	6/3/2016
End Frit Dewater #2	1400	6/3/2016
Start Final Dewater	1400	6/3/2016
End of SME	1742	6/3/2016

The equipment configuration used for this demonstration was similar to that of the non-rad tests. The setup utilized two heating rods to supply power instead of the external heating mantle configuration used in previous shielded cells demonstrations. The rods have internal thermocouples and known heated areas which allow for calculation of a heat transfer coefficient, h. This coefficient is calculated from the temperature differential between the heating rods and slurry, ΔT; power, Q; and known heating rod area, A(see Equation 3-10). The calculated heat transfer resistance is essentially the slurry film heat transfer coefficient on the outside rod area.

Equation 3-10: $$h = \frac{Q}{A * \Delta T}$$

Heat transfer data for the SRAT and SME cycles are presented in Figure 3-10 and Figure 3-11, respectively. The coefficient values are unreliable during periods of heating which manifests as noisy peaks at the beginning of the SRAT cycle and the larger peaks, both positive and negative, in the SME cycle. At constant boilup rates, the heat transfer coefficient values would ideally be constant. Slightly decreasing values occur as the slurry is concentrated and yield stress increases, as can be seen in both the SRAT and SME cycles. A large decrease in heat transfer would be indicative of rod fouling and was not observed in either SRAT or SME cycles.

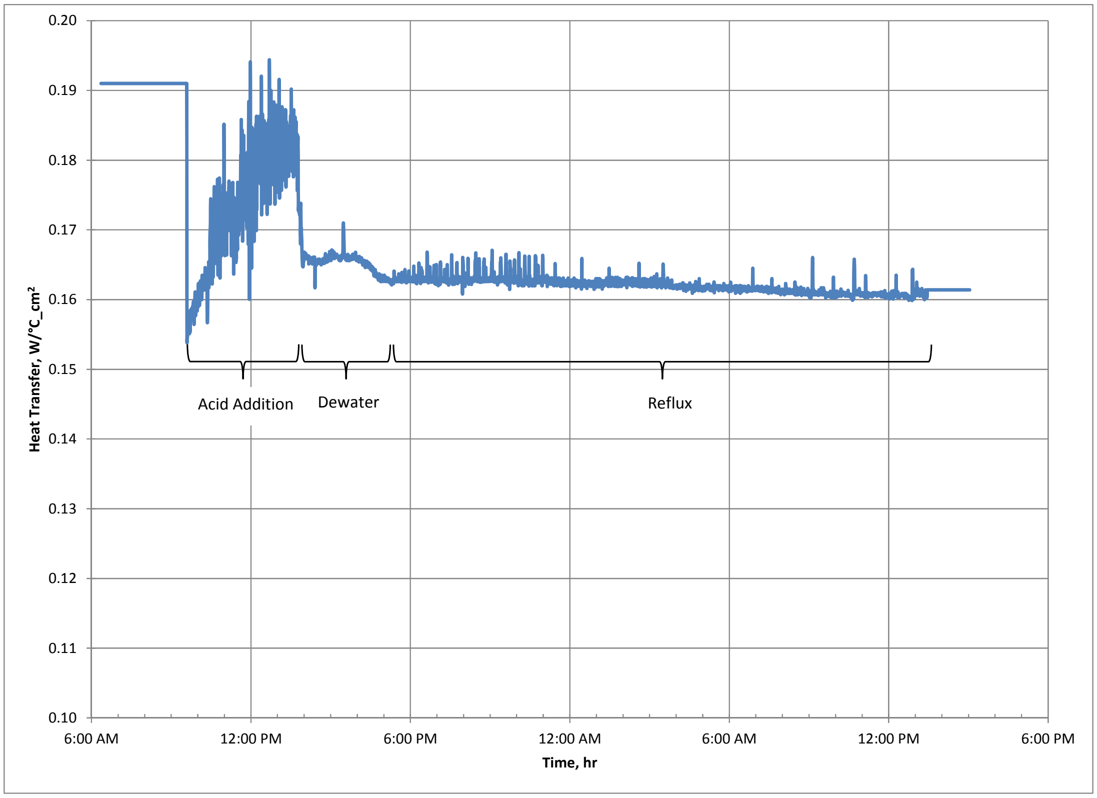


Figure 3-10: SRAT Heat Transfer

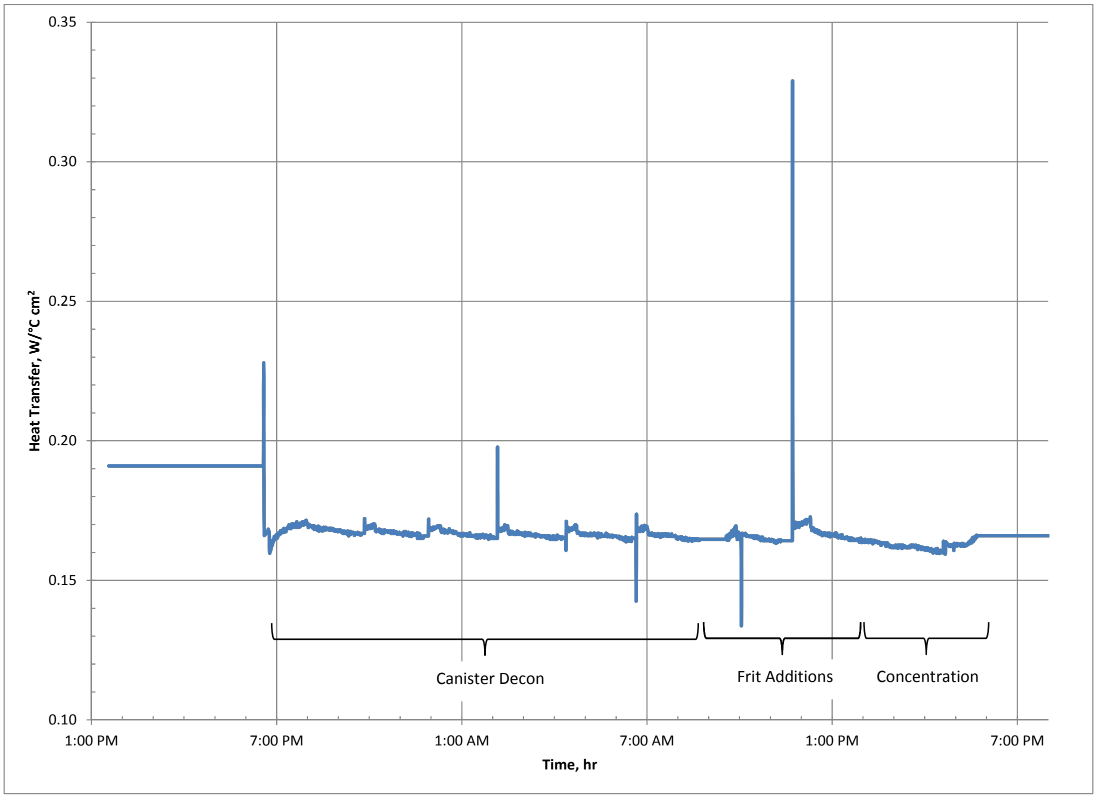


Figure 3-11: SME Heat Transfer

Process temperature data for the bulk slurry, heating rods, and offgas equipment are presented in Figure 3-12. At 0936 hr, power was applied to the heating rods resulting in an increase in heating rod and slurry temperatures. The rate at which the rod temperature increases is governed by a programmable algorithm that forms part of the DAC application. Heating was constrained so as not to exceed a 30 °C temperature differential between the slurry temperature and heating rods. As the slurry temperature approaches the targeted temperature setpoint (for acid addition, this was 93 °C) the control algorithm reduces power to the heating rods in order to not exceed the desired temperature. As can be seen in Figure 3-12, there is no temperature over-shoot for the slurry, and rod temperatures were constrained to the 30 °C temperature differential.

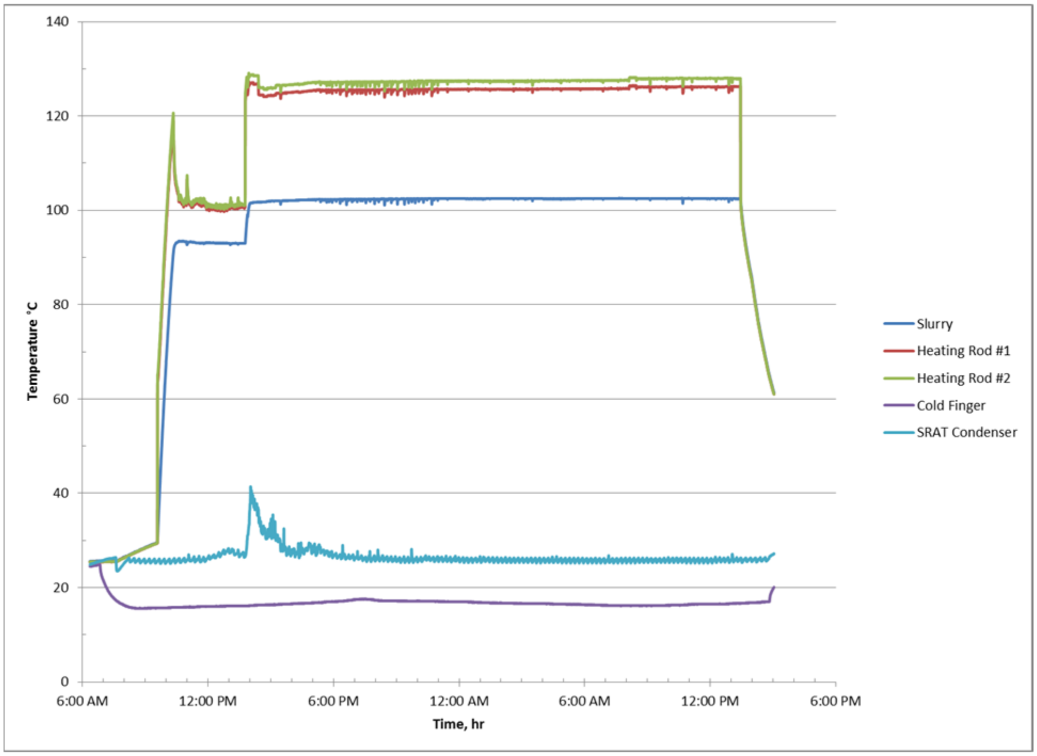


Figure 3-12: SRAT Processing Temperatures

After acid addition, additional power was added to the heating rods to increase temperature to boiling. An increase in the SRAT temperature is observed at the same time. The SRAT offgas is cooled as it passes through the SRAT condenser. The condenser was cooled using a water/glycol mixture cooled to 25 °C. As the slurry temperature increased to boiling, an expected spike in the offgas temperature is observed.

Temperature data for the SME cycle are presented in Figure 3-13**.** Power to the heating rods was turned off during the six canister decontamination water additions and two frit additions which resulted in the negative peaks observed in Figure 3-13. There was a slight over-shoot in heating rod temperature relative to the other additions that can be seen when going to boiling after the first frit addition. The heating rod temperature did not exceed the 30 °C temperature differential limit between the slurry and heating rods.

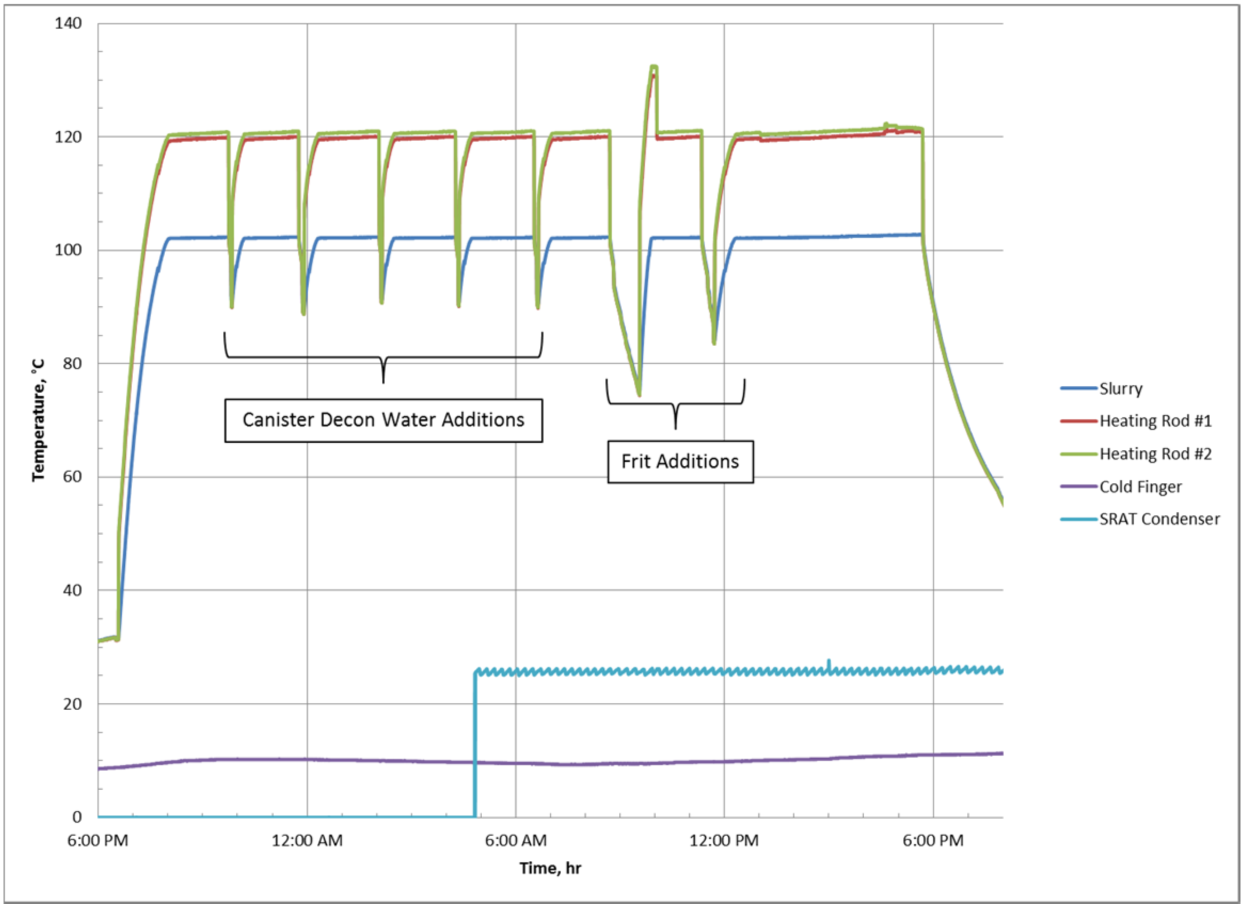


Figure 3-13: SME Process Temperatures

Data for the SRAT and SME vessel mixer torque as a function of time are presented in Figure 3-14 and Figure 3-15, respectively. Minimum mixing speed for both the SRAT and SME cycles was 700 rpm. The absolute values of torque are of less significance than the relative changes in torque throughout the flowsheet testing. Changes in torque values are observed for both the SRAT and SME cycles while transitioning to boiling temperatures. Throughout the SRAT cycle, the torque remained fairly constant at approximately 12 in_oz. Periodic increases in mixer torque observed during the SME cycle are directly related to canister decontamination water and frit additions. As expected, torque values increased as a function of total solids. At about one hour before the end of the SME cycle (6:40 p.m.), the mixing rate was increased from 700 to 800 rpm to counter the gradual decrease in heat transfer.

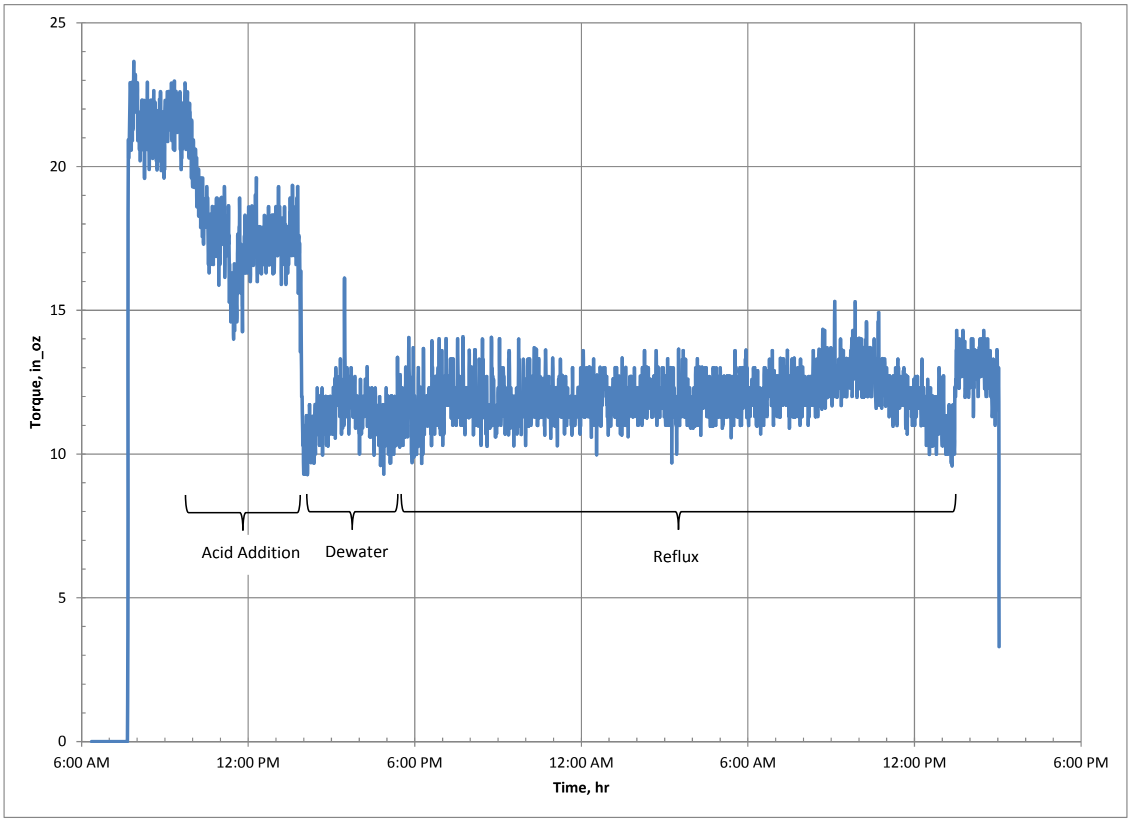


Figure 3-14: SRAT Mixer Torque

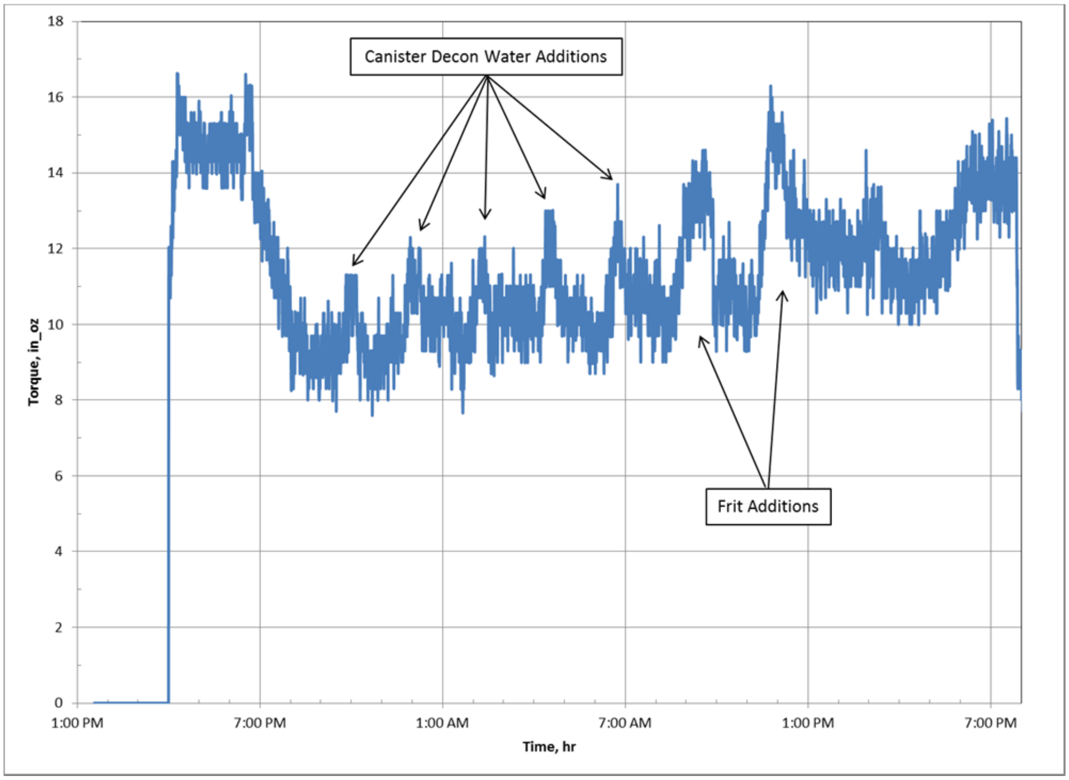


Figure 3-15: SME Mixer Torque

3.5.2.7 Comparison to DWPF Methods

For each sludge batch that is processed in the DWPF, the SRNL tests the applicability of the digestion methods used by the DWPF lab for elemental analysis of SRAT receipt samples and SRAT product process control samples. DWPF SRAT samples are typically dissolved using high concentrations of HF-HNO_3 acid at room temperatures (usually referred to as the DWPF CC Method, see DWPF Procedure SW4-15.201). The CC method is a faster and more convenient sample preparation method for elemental analysis by ICP-AES than the tandem sample preparation methods of hot (115 °C) AR [26] and PF [9] melted at 675 °C used for many years at SRNL to analyze sludge. Since no heating is employed in the CC method, it is considered to be potentially less effective than the methods based on heating. However, historically [27-32] the CC method has been shown to be effective for routine process control elemental analyses after samples from the first 10 SRAT cycles of a new sludge batch have been dissolved concurrently with the PF and CC methods to verify that the CC method has no unacceptable biases. The CC method was most recently evaluated for SB9 that had been processed as part of the waste qualification demonstration using formic acid as the mercury reductant in the SRAT [33]. The DWPF specified [1] that the CC method also be tested on the SRAT Product as part of the nitric-glycolic flowsheet demonstration to determine if the use of glycolic acid rather than formic acid as the mercury reductant in the SRAT would have a deleterious effect on the CC method.

All of the digestion results were provided in microgram of element per gram of slurry of SRAT Product material. To get the measurements to the correct basis for comparison, the results were divided by the appropriate wt% total solids: 24.57 wt% for the SRAT Product material.

JMP Pro Version 11.2.1 [34] was used to conduct the statistical analyses supporting these investigations. Table 3-53 provides the measurements of the blanks that were included in all of this analytical work. The measurements for the blanks were handled in the same manner as the SRAT Product samples to obtain the wt% results provided.

Table 3-53: Measurements of Blanks as Weight Percent of Total Solids

(Detection Limits were used to Represent Values below Detection as the Wt% Values were Computed)

Method	Aqua Regia	Peroxide Fusion	Cold Chem
LIMS	LW1171	LW1188	LW1200
Ag	<2.07E-03	<2.00E-02	<6.67E-03
Al	<5.41E-03	<1.36E-01	<2.44E-02
B	<6.55E-03	<2.62E-02	N/A
Ba	<1.16E-03	<4.64E-03	<1.37E-03
Be	<6.55E-05	<4.76E-03	<1.59E-03
Ca	<2.61E-03	1.04E-01	<4.48E-03
Cd	<4.06E-02	<8.10E-03	<5.45E-03
Ce	<6.31E-03	<2.28E-02	<1.53E-02
Co	<3.07E-03	<1.23E-02	<9.85E-03
Cr	<2.54E-03	<1.01E-02	<5.70E-03
Cu	<7.41E-03	<2.96E-02	<1.98E-02
Fe	<2.77E-03	<5.41E-02	<7.41E-03
Gd	<1.56E-03	<6.23E-03	<4.19E-03
K	<1.77E-01	<3.80E-01	<1.07E-01
La	<1.23E-03	<6.35E-03	<4.23E-03
Li	<3.69E-01	<8.47E-03	<1.33E-02
Mg	<3.23E-04	<1.29E-03	<8.63E-04
Mn	<3.83E-04	<1.14E-03	<1.03E-03
Mo	<6.31E-03	<2.53E-02	<1.69E-02
Na	<6.88E-03	N/A	<8.83E-02
Ni	<3.47E-03	<5.70E-02	<3.82E-02
P	<2.82E-02	<1.13E-01	<7.57E-02
Pb	<2.81E-02	<1.12E-01	<7.53E-02
S	<1.77E+00	<7.04E+00	N/A
Sb	<5.78E-02	<1.18E-01	<1.55E-01
Si	<2.71E-02	<1.08E-01	N/A
Sn	<1.78E-02	<7.12E-02	<4.76E-02
Sr	<1.44E-04	<1.36E-03	<3.85E-04
Th	<1.35E-02	<2.72E-03	<1.82E-03
Ti	<1.22E-02	<2.52E-02	<1.69E-02
U	<5.94E-02	<2.39E-01	<1.60E-01
V	<8.26E-04	<3.80E-03	<2.54E-03
Zn	1.34E-03	<3.22E-03	<8.18E-03
Zr	<9.61E-04	N/A	<2.61E-03
S (axial)	<8.26E-02	N/A	<1.62E-01

Table 3-54 provides the measurements (in mg/L) of the solution standards that were included in all of this analytical work. Exhibit A1 in Appendix A provides a plot of these data.

Table 3-54: Measurements as mg/L of the Solution Standard Sample SM744_063

Element	Method/LIMS		
	Aqua Regia	Peroxide Fusion	Cold Chem
	LW1177	LW1194	LW1206
Ag	<7.63E-02	<7.36E-01	<1.84E-01
Al (50)[f]	52.1	51.1	51.9
B	<2.41E-01	<9.65E-01	N/A
Ba	<4.27E-02	<1.71E-01	<3.78E-02
Be	<2.42E-03	<1.80E-01	<4.38E-02
Ca	<9.63E-02	<3.85E-01	<1.24E-01
Cd	<7.48E-02	<2.99E-01	<1.50E-01
Ce	<2.33E-01	<8.42E-01	<4.21E-01
Co	<1.13E-01	<4.53E-01	<2.72E-01
Cr	<9.35E-02	<3.74E-01	<1.57E-01
Cu	<2.73E-01	<1.09E+00	<5.46E-01
Fe (50)	50.6	50.6	50.6
Gd	<5.74E-02	<2.30E-01	<1.15E-01
K	<6.53E+00	<1.40E+01	<2.94E+00
La	5.30E-02	<2.33E-01	<1.17E-01
Li	<6.81E-01	<3.12E-01	<3.67E-01
Mg	<1.19E-02	<4.76E-02	<2.38E-02
Mn (20)	20.8	21.1	20.4
Mo	<2.33E-01	<9.32E-01	<4.66E-01
Na (150)	160	N/A	152
Ni (10)	11	9.62	9.97
P	<1.04E+00	<4.17E+00	<2.08E+00
Pb	<1.04E+00	<4.15E+00	<2.07E+00
S (10)	<6.53E+01	<2.60E+02	N/A
Sb	<2.13E+00	<4.36E+00	<4.27E+00
Si	<9.98E-01	<3.99E+00	N/A
Sn	<6.55E-01	<2.62E+00	<1.31E+00
Sr	<5.30E-03	<5.00E-02	<1.06E-02
Th	<4.96E-01	<1.00E-01	<5.02E-02
Ti	<4.48E-01	<9.29E-01	<4.65E-01
U	<2.20E+00	<8.79E+00	<4.39E+00
V	<3.05E-02	<1.40E-01	<7.00E-02
Zn	<2.96E-02	<1.18E-01	<2.25E-01
Zr	<3.53E-02	N/A	<7.20E-02
S axial (10)	10.9	N/A	11.2

[f] Reference values for elements present in the standard are shown in parentheses.

Table 3-55 provides the measurements of the ARG-1 samples that were included as part of this analytical work. The elemental reference values for the standard glass are also given in this table.

Table 3-55: Measurements (wt% of glass) for the ARG-1 Samples

Element	Reference Value for ARG-1	Method/LIMS		
		Aqua Regia LW1452	Peroxide Fusion LW1193	Cold Chem LW1205
Ag	0	<2.91E-03	<2.58E-02	<8.94E-03
Al	2.5	2.45E+00	2.59E+00	2.49E+00
B	2.69	2.68E+00	2.61E+00	N/A
Ba	0	7.93E-02	8.57E-02	8.33E-02
Be	0	4.03E-03	<6.14E-03	<2.13E-03
Ca	1.02	1.07E+00	1.25E+00	1.01E+00
Cd	0	1.16E-02	<1.05E-02	<7.26E-03
Ce	0	<8.89E-03	<2.94E-02	<2.04E-02
Co	0	8.61E-03	<1.58E-02	<1.32E-02
Cr	0.064	6.24E-02	6.69E-02	6.66E-02
Cu	0.003	<1.04E-02	<3.82E-02	<2.65E-02
Fe	9.79	9.82E+00	1.01E+01	9.86E+00
Gd	0	6.41E-03	<8.03E-03	<5.57E-03
K	2.26	2.28E+00	2.46E+00	2.16E+00
La	0	<3.44E-02	<8.16E-03	<5.67E-03
Li	1.49	1.53E+00	1.46E+00	1.51E+00
Mg	0.52	5.32E-01	5.33E-01	5.29E-01
Mn	1.46	1.49E+00	1.44E+00	1.46E+00
Mo	0	<8.89E-03	<3.26E-02	<2.26E-02
Na	8.52	9.07E+00	N/A	8.62E+00
Ni	0.827	8.90E-01	8.23E-01	7.99E-01
P	0	1.37E-01	<1.46E-01	1.16E-01
Pb	0	<3.96E-02	<1.45E-01	<1.01E-01
S	0	<2.49E+00	<9.10E+00	N/A
Sb	0	<8.14E-02	<1.52E-01	<2.07E-01
Si	22.4	3.84E-01	2.23E+01	N/A
Sn	0	<2.50E-02	<9.16E-02	<6.36E-02
Sr	0	3.11E-03	4.90E-03	3.16E-03
Th	0	<1.89E-02	<3.51E-03	<1.22E-02
Ti	0.69	5.78E-01	6.79E-01	6.78E-01
U	0	<8.39E-02	<3.07E-01	<2.13E-01
V	0	<1.16E-03	1.13E-02	1.05E-02
Zn	0	2.03E-02	1.55E-02	1.77E-02
Zr	0.096	5.66E-02	N/A	9.77E-02
S (axial)	0	<1.16E-01	N/A	<2.16E-01

Table 3-56 provides the measurements of the SRAT Product samples as wt% of total solids. Exhibit A2 in Appendix A provides a preliminary plot of these data for each element for an initial review of the results.

Exhibit A3 in Appendix A provides a more detailed and thorough statistical evaluation of the SRAT Product measurements. This exhibit provides a statistical review of the elemental measurements (as wt% of total solids) of the SRAT Product samples. Comparisons are made across all of the preparation methods that were utilized to provide the available data for the element in question. In each case, the results from a statistical test for the equality of the variances across the preparation methods for each set of measurements are provided. JMP includes several tests as part of this output. The conclusions made as part of this study are taken from the results of the Levene test. A p-value from this test that is less than or equal to 0.05 (given by the Prob>F value for this test in the exhibit) indicates a statistically significant difference in the variances of the measurements from the preparation methods (at the 5% significance level). If the p-value is greater than 0.05, then the hypothesis of equality for the variances cannot be rejected at the 5% significance level. As an example, consider the results for Al for the SRAT Product samples. Measurements from all three preparation methods were available for this element. The p-value for the Levene test is 0.0136, indicating that the hypothesis of equal variances across the three preparation methods should be rejected at the 5% significance level for the SRAT Product Al results.

Comparisons of the average values across the preparation methods are also important comparisons provided for each element as part of the JMP output of this exhibit. There are two approaches for making comparisons of the means across the preparation methods: (1) one approach is based on an assumption of equal variances for the measurements across the preparation methods and (2) the second approach is based on an assumption of unequal variances. JMP provides the appropriate statistical tests for each of these situations: the results from an analysis of variance (ANOVA) for the equal variance case and the results from Welch's test for the unequal variance case. The selection of the appropriate set of results is made by relying on the outcome of the Levene test for the element. As an example, consider once again the results for SRAT Product Al. Since the results from the Levene test did indicate that the measurement variances were different across the preparation methods, an assumption of equal variances cannot be made and the Welch's test results are used for the comparison of the means. The p-value for this analysis, indicated by the Prob>F value, is given as 0.0287 (a value less than the 0.05 for a 5% significance level). This leads to the conclusion that the means of the measurements for the SRAT Product Al differ across the three preparation methods. If Levene's test had suggested that the variances were equal, the ANOVA test would have been used. The p-value for this test is indicated by the value for Prob>F, which would be interpreted in the same manner as the ANOVA's p-value. To aid in the interpretation of these results in the exhibit, the Levene information is enhanced (i.e., bolded and shown in a larger font) as is the appropriate p-value result from the testing for the means.

Table 3-56: Measurements in Weight Percent of Total Solids of the SB9 SRAT Product Samples

Element	Aqua Regia/LIMS				Peroxide Fusion/LIMS				Cold Chem/LIMS			
	LW1173	LW1174	LW1175	LW1449	LW1189	LW1190	LW1191	LW1192	LW1201	LW1202	LW1203	LW1204
Ag	<2.19E-03	<1.95E-03	<1.79E-03	<1.82E-03	<1.99E-02	<2.21E-02	<2.39E-02	<2.34E-02	<6.72E-03	<6.47E-03	<6.76E-03	<6.76E-03
Al	5.58E+00	5.37E+00	5.58E+00	1.90E+00	5.78E+00	5.90E+00	5.78E+00	5.98E+00	5.90E+00	5.90E+00	5.90E+00	5.86E+00
B	<6.92E-03	<6.15E-03	<5.66E-03	<5.74E-03	<2.61E-02	<2.91E-02	<3.13E-02	<3.06E-02	N/A	N/A	N/A	N/A
Ba	5.90E-02	5.94E-02	5.94E-02	2.43E-02	6.19E-02	6.43E-02	6.27E-02	6.59E-02	6.15E-02	5.82E-02	6.02E-02	5.98E-02
Be	1.47E-03	1.47E-03	1.49E-03	6.11E-04	<4.76E-03	<5.29E-03	<5.70E-03	<5.54E-03	<1.60E-03	<1.54E-03	<1.60E-03	<1.61E-03
Ca	8.42E-01	8.47E-01	8.59E-01	3.41E-01	9.20E-01	9.20E-01	8.42E-01	9.56E-01	8.63E-01	8.47E-01	8.51E-01	8.55E-01
Cd	2.10E-02	1.93E-02	1.88E-02	1.08E-02	1.25E-02	1.36E-02	1.32E-02	1.45E-02	1.37E-02	1.42E-02	1.34E-02	1.44E-02
Ce	2.69E-02	2.74E-02	2.95E-02	1.18E-02	1.71E-01	1.74E-01	1.63E-01	1.81E-01	2.03E-01	1.86E-01	1.93E-01	1.89E-01
Co	8.67E-03	8.71E-03	8.71E-03	3.30E-03	<1.23E-02	<1.36E-02	<1.47E-02	<1.44E-02	<9.89E-03	<9.56E-03	<9.97E-03	<9.97E-03
Cr	6.15E-02	6.19E-02	6.23E-02	2.55E-02	7.00E-02	7.12E-02	7.57E-02	7.33E-02	7.20E-02	7.20E-02	7.12E-02	7.12E-02
Cu	2.67E-02	2.54E-02	2.70E-02	1.09E-02	<2.96E-02	<3.28E-02	<3.54E-02	<3.46E-02	2.53E-02	2.54E-02	2.47E-02	2.50E-02
Fe	1.42E+01	1.42E+01	1.42E+01	5.70E+00	1.38E+01	1.40E+01	1.40E+01	1.43E+01	1.40E+01	1.40E+01	1.40E+01	1.39E+01
Gd	7.73E-02	7.69E-02	7.65E-02	3.08E-02	6.63E-02	6.31E-02	5.78E-02	6.23E-02	7.41E-02	6.96E-02	7.33E-02	7.20E-02
K	<1.87E-01	<1.66E-01	<1.53E-01	<1.56E-01	<3.79E-01	<4.23E-01	<4.56E-01	<4.44E-01	<1.07E-01	<1.03E-01	<1.07E-01	<1.08E-01
La	3.56E-02	3.50E-02	3.56E-02	1.45E-02	3.13E-02	3.15E-02	3.18E-02	3.07E-02	3.57E-02	3.23E-02	3.41E-02	3.33E-02
Li	4.44E-02	4.15E-02	3.99E-02	2.34E-02	2.94E-02	2.92E-02	3.06E-02	3.01E-02	3.32E-02	3.20E-02	3.12E-02	3.00E-02
Mg	1.92E-01	1.92E-01	1.93E-01	7.90E-02	1.71E-01	1.73E-01	1.70E-01	1.77E-01	1.90E-01	1.90E-01	1.90E-01	1.91E-01
Mn	4.68E+00	4.68E+00	4.68E+00	1.94E+00	4.60E+00	4.64E+00	4.64E+00	4.76E+00	4.68E+00	4.72E+00	4.72E+00	4.72E+00
Mo	<6.67E-03	<5.94E-03	<5.45E-03	<5.58E-03	<2.52E-02	<2.80E-02	<3.02E-02	<2.95E-02	<1.70E-02	<1.64E-02	<1.71E-02	<1.71E-02
Na	1.29E+01	1.30E+01	1.27E+01	5.21E+00	N/A	N/A	N/A	N/A	1.19E+01	1.20E+01	1.19E+01	1.18E+01
Ni	1.10E+00	1.10E+00	1.10E+00	4.64E-01	1.01E+00	1.01E+00	1.01E+00	1.05E+00	1.01E+00	1.01E+00	1.01E+00	1.01E+00
P	1.44E-01	1.33E-01	1.36E-01	6.27E-02	1.53E-01	1.62E-01	1.63E-01	1.76E-01	2.00E-01	1.90E-01	1.97E-01	1.95E-01
Pb	4.64E-02	4.27E-02	4.23E-02	<2.47E-02	<1.12E-01	<1.25E-01	<1.34E-01	<1.31E-01	<7.57E-02	<7.29E-02	<7.61E-02	<7.61E-02
S	<1.87E+00	<1.66E+00	<1.53E+00	<1.56E+00	<7.04E+00	<7.81E+00	<8.42E+00	<8.26E+00	N/A	N/A	N/A	N/A
Sb	<6.11E-02	<5.41E-02	<5.01E-02	<5.09E-02	<1.18E-01	<1.31E-01	<1.41E-01	<1.38E-01	<1.55E-01	<1.50E-01	<1.56E-01	<1.57E-01
Si	4.01E-01	3.57E-01	3.63E-01	1.55E-01	1.16E+00	1.16E+00	1.14E+00	1.21E+00	N/A	N/A	N/A	N/A
Sn	<1.88E-02	<1.67E-02	<1.54E-02	<1.56E-02	<7.08E-02	<7.90E-02	<8.51E-02	<8.30E-02	<4.76E-02	<4.60E-02	<4.80E-02	<4.80E-02
Sr	2.20E-02	2.19E-02	2.21E-02	8.79E-03	2.31E-02	2.35E-02	2.31E-02	2.42E-02	2.25E-02	2.14E-02	2.20E-02	2.19E-02
Th	7.33E-01	7.24E-01	7.33E-01	3.02E-01	6.84E-01	6.84E-01	6.59E-01	7.04E-01	6.80E-01	6.19E-01	6.51E-01	6.39E-01
Ti	1.80E-02	1.79E-02	1.81E-02	<1.07E-02	<2.52E-02	<2.80E-02	<3.01E-02	<2.95E-02	1.79E-02	1.82E-02	1.80E-02	1.84E-02
U	2.72E+00	2.69E+00	2.74E+00	1.12E+00	2.71E+00	2.75E+00	2.55E+00	2.77E+00	2.63E+00	2.64E+00	2.62E+00	2.62E+00
V	<8.71E-04	<7.77E-04	<7.16E-04	<7.29E-04	<3.79E-03	<4.19E-03	<4.52E-03	<4.44E-03	<2.55E-03	<2.46E-03	<2.56E-03	<2.57E-03
Zn	2.76E-02	2.76E-02	2.74E-02	1.14E-02	2.16E-02	2.11E-02	2.11E-02	2.15E-02	2.32E-02	2.29E-02	2.30E-02	2.35E-02
Zr	5.41E-02	4.07E-02	4.07E-02	1.71E-02	N/A	N/A	N/A	N/A	1.17E-01	1.16E-01	1.16E-01	1.16E-01
S (axial)	2.46E-01	2.69E-01	2.42E-01	1.07E-01	N/A	N/A	N/A	N/A	2.74E-01	3.04E-01	2.75E-01	2.82E-01

The JMP output in this exhibit provides the results from one additional comparison of the means (under the assumption of equal variances) for those elements measured by all three preparations. This statistical evaluation makes comparisons across all pairs of means and highlights those that are statistically different at the 5% significance level. Consider the Al results for the SRAT Product samples. The exhibit shows:

Level			**Mean**
Cold Chem	A		5.89
Peroxide Fusion	A		5.86
Aqua Regia		B	5.51

The means of the measurements for the three preparations are given in descending order (top to bottom). They are also labeled from the largest to the smallest average value with an "A" and "B." As indicated in the exhibit, "levels not connected by same letter are significantly different." Thus, in this case the Cold Chem and Peroxide Fusion means are not statistically different from each other, but both of these means differ from (are larger than) the mean for Aqua Regia: the labels being used such that A > B.

Table 3-57 summarizes the results from Exhibit A3. This table provides the average of the elemental wt% measurements for each preparation method along with the relative standard deviation (%RSD), i.e., the standard deviation as a percentage of the average. Also, the table contains a column with the results of the Levene test for a difference in the variances of the measurements across the preparation methods for each element. (A "yes" entry designates a statistically significant, at the 5% level, difference in the variances for the measurements of the indicated element.) The results of the statistical comparisons across the means of the measurements from the preparation methods are highlighted in the last three columns of Table 3-57. The use of the letters "A", "B", and "C" in labeling the average values in this table is as described above. Note that these comparisons of the means when across all three preparations are based on an assumption of equal variances for the measurements of the given element across the preparation methods.

Table 3-57: SRAT Product Results

	Shaded Entries Represent "Less than Detection" (LTD) Values			Levene's Test: Variances Significantly Different	"Equal" Means have Connecting Letters[f]		
	Average (%Relative Standard Deviation)						
Element	Aqua Regia	Cold Chem	Peroxide Fusion		AR	CC	PF
Ag	0.002	0.007	0.022				
Al	5.510 (2.2%)	5.890 (0.3%)	5.860 (1.7%)	Y	B	A	A
B	0.006	N/A	0.029				
Ba	0.059 (0.4%)	0.060 (2.3%)	0.064 (2.8%)	N	B	B	A
Be	0.001 (0.8%)	0.002	0.005				
Ca	0.849 (1.0%)	0.854 (0.8%)	0.910[♣] (5.3%)	N	A	A	A
Cd	0.020 (5.9%)	0.014 (3.3%)	0.013 (6.2%)	N	A	B	B
Ce	0.028 (4.9%)	0.193 (3.8%)	0.172 (4.3%)	N	C	A	B
Co	0.009 (0.3%)	0.010	0.014				
Cr	0.062 (0.6%)	0.072 (0.6%)	0.073 (3.5%)	Y	B	A	A
Cu	0.026 (3.2%)	0.025 (1.3%)	0.033	N	A	B	
Fe	14.200 (0.0%)	13.975 (0.4%)	14.025 (1.5%)	N	A	A	A
Gd	0.077 (0.5%)	0.072 (2.7%)	0.062 (5.6%)	N	A	A	B
K	0.169	0.106	0.426				
La	0.035 (1.0%)	0.034 (4.2%)	0.031 (1.5%)	N	A	A	B
Li	0.042 (5.4%)	0.032 (4.3%)	0.030 (2.2%)	N	A	B	B
Mg	0.192 (0.3%)	0.190 (0.3%)	0.173 (1.8%)	N	A	A	B
Mn	4.680 (0.0%)	4.710 (0.4%)	4.660 (1.5%)	N	A	A	A
Mo	0.006	0.017	0.028				
Na	12.867 (1.2%)	11.900 (0.7%)	N/A	N	A	B	
Ni	1.100 (0.0%)	1.010 (0.0%)	1.020 (2.0%)	Y	A	B	B
P	0.138 (4.1%)	0.196 (2.1%)	0.164 (5.8%)	N	C	A	B
Pb	0.044 (5.2%)	0.075	0.126				
S	1.687	N/A	7.883				
S (axial)	0.252 (5.8%)	0.284 (4.9%)	N/A	N	B	A	
Sb	0.055	0.155	0.132				
Si	0.374 (6.4%)	N/A	1.168 (2.6%)	N	B		A
Sn	0.017	0.047	0.079				
Sr	0.022 (0.5%)	0.022 (2.1%)	0.023 (2.2%)	N	B	B	A
Th	0.730 (0.7%)	0.647 (3.9%)	0.683 (2.7%)	N	A	B	B
Ti	0.018 (0.6%)	0.018 (1.2%)	0.028	N	A	A	
U	2.717 (0.9%)	2.628 (0.4%)	2.695 (3.7%)	N	A	A	A
V	0.001	0.003	0.004				
Zn	0.028 (0.4%)	0.023 (1.1%)	0.021 (1.2%)	N	A	B	C
Zr	0.045 (17.1%)	0.116 (0.4%)	N/A	Y	B	A	

[f] AR represents Aqua Regia; CC represents Cold Chem, PF, represents Peroxide Fusion, and the comparisons of the means presented in these columns are made with the assumption of equal variances for the methods.

[♣] The slightly higher Ca value for the PF method may be due to contamination (see the results for the blanks in Table 1).

The Cold Chemical method did not completely dissolve the alternate reductant SRAT Product sludge. Fine, dark solids remained which slowly dissolved over a matter of a few days. However, elemental analyses were performed on dilutions of the initial CC dissolution that still contained solids when removed from the shielded cells. Of the nine elements that comprise at least 0.5 wt% of the alternate reductant sludge on a dry solids basis, the CC method produced statistically equivalent results to either the AR or PF methods for seven elements (Al, Ca, Fe, Mn, Ni, Th, and U). The CC Na determinations were ~7.5 % lower than the AR Na determinations. Si determinations are precluded in the CC method because strong attack of HF on the quartz components of the SRNL ICP-AES sample introduction system leads to high Si bias.

The precision of the CC determinations was excellent with less than 5% relative standard deviation of four determinations for all elements with a concentration of at least 0.5 wt% in the sludge.

Discussions of the results and statistical analysis from dissolving the SRAT receipt and SRAT product sludge using the CC, the AR, and the PF methods are provided below:

- Differences in the variances and the means were observed for Al determinations. The CC Al determinations were ~7% higher than those from the AR method. There was no statistical difference between the means of the CC and PF methods. Al concentration is ~5.9 wt% in the alternate reductant SRAT Product as measured using the CC and PF dissolution methods.
- There was no difference in either the variances or the means of Ca determinations from the three sample preparation methods. Ca concentration is ~0.9 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances of the Ce determinations from the three sample preparation methods. However, there were differences in the means. The CC Ce determinations were ~700% higher than the AR Ce determinations and ~12% higher than PF Ce determinations. Ce concentration is ~0.2 wt% in the alternate reductant SRAT product
- There was no difference in either the variances or the means of Fe determinations from the three sample preparation methods. Fe concentration is ~14.0 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances for Mg determinations. The CC and AR Mg determinations were in statistical agreement. However, the CC Mg determinations were ~10% higher than PF Mg determinations. Mg is ~0.2 wt% in the alternate reductant SRAT Product.
- There was no difference in either the variances or the means of Mn determinations from the three sample preparation methods. Mn concentration is ~4.7 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances of Na determinations between the CC and AR methods. However, there was a difference in the means with the CC Na determinations ~7.5% less than the AR Na determinations. Na cannot be determined from the PF method because of the large amount of Na introduced as the fusion reagent. Na concentration is 12.9 wt% in the alternate reductant SRAT Product as measured with the AR method.
- Differences in both the variances and the means were observed for Ni determinations. The CC and PF Ni methods were in statistical agreement and were ~9% lower than AR Ni determinations. Ni concentration is ~1 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances for P determinations. However, there were differences in the means with the CC P determinations ~42% higher than the AR P determinations and ~20% higher than PF P determinations. P concentration is ~0.2 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances of S determinations from the CC and AR methods. The CC S determinations were ~13% higher than AR S determinations. It is believed that the

unreliable PF S determinations (which were eliminated from statistical consideration) were due to deleterious effects of the high alkali on the ICP-AES performance on the analytical line for S. S concentration is ~0.3 wt% in the SRAT Product.

- Si determinations are only considered reliable from the PF method. The AR method may not dissolve all forms of Si. The CC method is generally effective for Si compounds, but the high concentration of HF reacts with the quartz ICP-AES sample introduction system resulting in a very high Si bias. Si concentration is ~1.2 wt% in the alternate reductant SRAT Product.
- There was no difference in the variances of the Th determinations from the three methods. However, there was a difference in the means with the CC Th determinations ~11% lower than AR Th determinations. The CC Th determinations were in statistical agreement with PF Th determinations. Th concentration is ~0.7 wt% in the alternate reductant SRAT Product.
- There was no difference in either the variances or the means of U determinations from the three sample preparation methods. U concentration is ~2.7 wt% in the alternate reductant SRAT Product.
- Differences in both the variances and the means were observed for Zr determinations. The CC Zr determinations were ~260% higher than the AR determinations. The PF method cannot be used to determine Zr because a Zr crucible is used in the fusion. Since HF is an effective dissolution reagent for Zr compounds, CC Zr determinations are considered to be more accurate and conservative for Zr determinations in SRS sludge. Zr concentration is ~0.1 wt% in the alternate reductant SRAT Product as determined with the CC dissolution method.

In view of the excellent precision and agreement with reference AR and PF dissolution methods for most elements, the CC method should be adequate to provide DWPF process control analyses after performing side-by-side tests of the CC and PF methods for the first 10 SRAT batches with SB9 sludge processed with the nitric-glycolic acid flowsheet. During these tests, the DWPF Laboratory will be able to accumulate enough data over a long time period to determine if the CC method produces biases and, if so, if these biases are acceptable.

Comparing the agreement of the CC dissolution method with the AR and PF dissolution methods for the nitric-glycolic SRAT product with the level of agreement for the SRAT receipt and the nitric-formic SRAT product [33], the nitric-glycolic flowsheet does not appear to have a negative impact on the use of the CC method for DWPF process control analyses.

The DWPF Laboratory monitors the mercury concentration in the SRAT Receipt and SRAT Product before and after chemical reduction of mercury compounds to elemental mercury followed by steam stripping. Mercury determinations are made using atomic absorption spectroscopy after a sample preparation step to adjust the oxidation state to Hg^{+2}. Stannous chloride is then added to the solutions to reduce Hg^{+2} to elemental mercury just prior to measurements. The sample preparation method used by the DWPF is the so called "EPA Method" used by many commercial, government, and academic laboratories in the U.S. This method uses a battery of strong acids and powerful oxidizing reagents to destroy organomercury compounds and convert them to inorganic forms. The alternative reductant demonstration in the SRNL Shielded Cell Facility afforded the opportunity to test the DWPF method in concert with the aqua regia method commonly used for high level sludge characterizations in SRNL.

All of the digestion results were provided in microgram of element per gram of slurry of SRAT Product material. To get the measurements to the correct basis for comparison, the results were divided by the appropriate wt% total solids: 24.57 for the SRAT Product material.

Table 3-58 provides the measurements of the blanks that were included in all of this analytical work. The measurements for the blanks were handled in the same manner as the SRAT Product samples to obtain the wt% results provided.

Table 3-58: Measurements of Blanks as Weight Percents of Total Solids

(Detection Limits were used to Represent Values below Detection as the Wt% Values were Computed)

Sample Name	LIMS	Method	Hg
AD Blank	LW1183	Aqua Regia	<2.98E-03
AD Blank	LW1243	EPA Hg Prep	<1.12E-03

Table 3-59 provides the Hg measurements of the SRAT Product samples as wt% of total solids. Exhibit A4 in Appendix A provides a preliminary plot of these Hg data for an initial review of the results. Note that the Hg value for the sample with LIMS # LW1451 is much lower than the values for the other samples (regardless of method). This is the questionable aqua regia-prepared sample noted above, and the corresponding Hg measurement, shaded in Table 3-59, is not included in the subsequent analyses presented in this section.

Table 3-59: Hg Measurements in Weight Percent of Total Solids of the SRAT Product Samples

Sample Name	LIMS	Method	Hg wt% of Total Solids
TS190-15-C-01000	LW1185	Aqua Regia	0.724
TS190-15-C-01001	LW1186	Aqua Regia	0.696
TS190-15-C-01002	LW1187	Aqua Regia	0.704
TS190-15-C-01390	LW1451	Aqua Regia	0.298
TS190-15-C-01027	LW1244	EPA Hg Prep	0.590
TS190-15-C-01028	LW1245	EPA Hg Prep	0.623
TS190-15-C-01029	LW1246	EPA Hg Prep	0.598
TS190-15-C-01030	LW1247	EPA Hg Prep	0.700

Exhibit A5 in Appendix A provides a more detailed and thorough statistical evaluation of the Hg measurements. Comparisons are made of the results from the two preparation methods that were utilized to provide the available data. Statistical tests for the equality of the variances across the two preparation methods are provided. JMP includes several tests as part of this output. The conclusions made as part of this study are taken from the results of the Levene test. A p-value from this test that is less than or equal to 0.05 (given by the Prob>F value for this test in the exhibit) indicates a statistically significant difference in the variances of the measurements from the preparation methods (at the 5% significance level). If the p-value is greater than 0.05, then the hypothesis of equality for the variances cannot be rejected at the 5% significance level. For the Hg measurements, the p-value for the Levene test is 0.1892 (highlighted in the exhibit), indicating that the hypothesis of equal variances across the two preparation methods cannot be rejected at the 5% significance level.

Comparisons of the averages of the Hg values across the two preparation methods are also provided as part of the JMP output of this exhibit. There are two approaches for making comparisons of the means across the preparation methods: (1) one approach is based on an assumption of equal variances for the measurements across the preparation methods and (2) the second approach is based on an assumption of unequal variances. JMP provides the appropriate statistical tests for each of these situations: the results from an ANOVA for the equal variance case and the results from Welch's test for the unequal variance case. The selection of the appropriate set of results is made by relying on the outcome of the Levene test. Since the results from the Levene test for Hg did not indicate that the measurement variances were different across the two preparation methods, an assumption of equal variance can be made and the ANOVA test results are used for the comparison of the means. The p-value for this analysis, indicated by the Prob>F value, is given as 0.0464 (a value less than the 0.05 for a 5% significance level). This leads to the conclusion that the means of the measurements for the SRAT Product Hg differ across the two preparation methods with the average of the aqua regia results being larger than the average of the EPA Hg Prep results. To aid in the interpretation of these results in the exhibit, the Levene information is enhanced (i.e., bolded and shown in a larger font) as is the p-value result from the ANOVA testing of the means.

Table 3-60 summarizes the results from Exhibit A5. This table provides the average of the Hg wt% measurements for each preparation method along with the relative standard deviation (%RSD), i.e., the standard deviation as a percentage of the average. Also, the table contains a column of notes indicating the results of Levene's test for a difference in the variances of the measurements and the results of the statistical comparison of the means of the measurements for the two preparation methods. Thus, the aqua regia values are on average about 12.7% higher than the values from the EPA Hg prep.

The 8% difference in mercury determinations after sample preparations by the DWPF method and the aqua regia method should not raise a red flag of undue concern. The DWPF method should be superior for destroying the organic compounds in the DWPF samples that can have a deleterious effect on mercury analyses by atomic absorption. The DWPF should continue with its plans to use its method for sample preparation to support mercury process control analyses.

Table 3-60: Summary Statistics for the Hg Results for the SRAT Product Samples

Method	Average Hg wt% TS	%RSD	Notes (at the 5% significance level)
Aqua Regia	0.708	2.0	Variances are not statistically different, but the means are.
EPA Hg Prep	0.628	8.0	

A method of analysis of glycolic acid by IC was developed in support of the alternate reductant flowsheet [7]. Comparisons of the CQ method and water dilution methods are presented below. Table 3-61 provides the measurements of the blanks that were included in all of this analytical work.

Table 3-61: Measurements of Blanks

Type	SRAT Product	SRAT Product	SRAT Product	SME Product	SME Product	SME Product
LIMS	LW1151	LW1166	LW1897	LW1312	LW1332	LW1897
Method	water dilution	caustic quench	oxalate prep	water dilution	caustic quench	oxalate prep
Units	mg/kg of slurry	mg/kg of slurry	mg/kg of slurry	mg/kg of slurry	mg/kg of slurry	mg/kg of slurry
Fluoride	<1.2E+03	<6.4E+03	NA	<1.2E+03	<6.6E+02	NA
Glycolate	<1.2E+02	<6.4E+02	NA	<2.5E+02	<6.6E+02	NA
Formate	<1.2E+02	<6.4E+02	NA	<1.2E+02	<6.6E+02	NA
Chloride	<1.2E+02	NA	NA	<1.2E+02	NA	NA
Nitrite	<1.2E+02	<6.4E+02	NA	<1.2E+02	<6.6E+02	NA
Bromide	<6.1E+02	<3.2E+03	NA	<1.2E+03	<3.3E+03	NA
Nitrate	<1.2E+02	<6.4E+02	NA	<1.2E+02	2.11E+03	NA
Phosphate	<1.2E+02	<6.4E+02	NA	<1.2E+02	<6.6E+02	NA
Sulfate	<1.2E+02	<6.4E+02	NA	<1.2E+02	<6.6E+02	NA
Oxalate	<1.2E+02	<6.4E+02	<2.50E+03	<1.2E+02	<6.6E+02	<2.5E+03
Type	Filtered SRAT Product	Filtered SRAT Product	Filtered SME Product	Filtered SME Product		
LIMS	LW1223	LW1238	LW1382	LW1397		
Method	water dilution	caustic quench	water dilution	caustic quench		
Units	mg/kg of filtrate	mg/kg of filtrate	mg/kg of filtrate	mg/kg of filtrate		
Fluoride	<1.2E+03	<7.1E+03	<2.5E+02	<6.1E+02		
Glycolate	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		
Formate	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		
Chloride	<1.2E+02		<2.8E+02			
Nitrite	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		
Bromide	<6.2E+02	<3.2E+03	<2.5E+02	<1.2E+03		
Nitrate	<1.2E+02	<6.5E+02	<2.5E+02	2.64E+03		
Phosphate	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		
Sulfate	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		
Oxalate	<1.2E+02	<6.5E+02	<2.5E+02	<6.1E+02		

(Measurements below the Detection Limits (i.e., less than detection, LTD) are shown with less than signs)

Table 3-62 provides the measurements of the SRAT and SME products and the measurements of the filtered SRAT and SME Products. Exhibit A6 in Appendix A provides preliminary plots of these data along with the Table 3-61 measurements for the blanks.

Table 3-62: Anion Measurements of the SRAT and SME Products and Filtered Products

Type	Sample Name	LIMS	Method	Units	Fluoride	Glycolate	Formate	Chloride	Nitrite
SRAT Product	TS190-15-C-01035	LW1152	water dilution	mg/kg of slurry	<1.2E+03	3.34E+04	1.40E+03	<1.2E+02	3.01E+02
SRAT Product	TS190-15-C-01036	LW1153	water dilution	mg/kg of slurry	<1.2E+03	3.40E+04	1.42E+03	<1.2E+02	3.06E+02
SRAT Product	TS190-15-C-01037	LW1154	water dilution	mg/kg of slurry	<1.3E+03	3.39E+04	1.42E+03	<1.3E+02	3.04E+02
SRAT Product	TS190-15-C-01038	LW1155	water dilution	mg/kg of slurry	<1.2E+03	3.37E+04	1.41E+03	<1.2E+02	3.05E+02
SRAT Product	TS190-15-C-01051	LW1167	caustic quench	mg/kg of slurry	<6.3E+03	3.68E+04	1.44E+03	NA	<6.3E+02
SRAT Product	TS190-15-C-01052	LW1168	caustic quench	mg/kg of slurry	<6.5E+03	3.73E+04	1.46E+03	NA	<6.5E+02
SRAT Product	TS190-15-C-01053	LW1169	caustic quench	mg/kg of slurry	<6.3E+03	3.73E+04	1.45E+03	NA	<6.3E+02
SRAT Product	TS190-15-C-01054	LW1170	caustic quench	mg/kg of slurry	<6.2E+03	3.69E+04	1.45E+03	NA	<6.2E+02
SRAT Product	TS190-15-C-01491	LW1898	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SRAT Product	TS190-15-C-01492	LW1899	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SRAT Product	TS190-15-C-01493	LW1900	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SRAT Product	TS190-15-C-01494	LW1901	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
Filtered SRAT Product	TS190-15-C-01075	LW1224	water dilution	mg/kg of filtrate	<1.2E+03	3.66E+04	1.62E+03	<1.2E+02	3.55E+02
Filtered SRAT Product	TS190-15-C-01076	LW1225	water dilution	mg/kg of filtrate	<1.3E+03	3.69E+04	1.62E+03	<1.3E+02	3.53E+02
Filtered SRAT Product	TS190-15-C-01077	LW1226	water dilution	mg/kg of filtrate	<1.2E+03	3.80E+04	1.69E+03	<1.2E+02	3.74E+02
Filtered SRAT Product	TS190-15-C-01078	LW1227	water dilution	mg/kg of filtrate	<1.3E+03	3.65E+04	1.61E+03	<1.3E+02	3.58E+02
Filtered SRAT Product	TS190-15-C-01091	LW1239	caustic quench	mg/kg of filtrate	<6.9E+03	3.83E+04	1.74E+03	NA	<6.3E+02
Filtered SRAT Product	TS190-15-C-01092	LW1240	caustic quench	mg/kg of filtrate	<6.3E+03	3.88E+04	1.78E+03	NA	<5.8E+02
Filtered SRAT Product	TS190-15-C-01093	LW1241	caustic quench	mg/kg of filtrate	<6.4E+03	3.93E+04	1.75E+03	NA	<5.9E+02
Filtered SRAT Product	TS190-15-C-01094	LW1242	caustic quench	mg/kg of filtrate	<6.6E+03	3.72E+04	1.65E+03	NA	<6.1E+02
SME Product	TS190-15-C-01194	LW1313	water dilution	mg/kg of slurry	<1.3E+03	3.20E+04	1.97E+03	<1.3E+02	3.85E+02
SME Product	TS190-15-C-01195	LW1314	water dilution	mg/kg of slurry	<1.3E+03	3.11E+04	1.93E+03	<1.3E+02	3.77E+02
SME Product	TS190-15-C-01196	LW1315	water dilution	mg/kg of slurry	<1.2E+03	3.19E+04	1.97E+03	<1.2E+02	3.81E+02
SME Product	TS190-15-C-01197	LW1316	water dilution	mg/kg of slurry	<1.2E+03	3.13E+04	1.94E+03	<1.2E+02	3.76E+02
SME Product	TS190-15-C-01210	LW1333	caustic quench	mg/kg of slurry	<6.6E+02	3.22E+04	1.52E+03	NA	<6.6E+02
SME Product	TS190-15-C-01211	LW1334	caustic quench	mg/kg of slurry	<6.6E+02	3.36E+04	1.49E+03	NA	<6.6E+02
SME Product	TS190-15-C-01212	LW1335	caustic quench	mg/kg of slurry	<6.2E+02	3.21E+04	1.42E+03	NA	<6.2E+02
SME Product	TS190-15-C-01213	LW1336	caustic quench	mg/kg of slurry	<6.6E+02	3.31E+04	1.52E+03	NA	<6.6E+02
SME Product	TS190-15-C-01495	LW1902	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SME Product	TS190-15-C-01496	LW1903	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SME Product	TS190-15-C-01497	LW1904	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
SME Product	TS190-15-C-01498	LW1905	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	NA
Filtered SME Product	TS190-15-C-01262	LW1383	water dilution	mg/kg of filtrate	<1.2E+03	4.57E+04	3.14E+03	9.01E+02	5.46E+02
Filtered SME Product	TS190-15-C-01263	LW1384	water dilution	mg/kg of filtrate	<1.3E+03	5.62E+04	3.86E+03	<2.9E+02	6.88E+02
Filtered SME Product	TS190-15-C-01264	LW1385	water dilution	mg/kg of filtrate	<1.2E+03	4.80E+04	3.29E+03	<2.8E+02	5.81E+02
Filtered SME Product	TS190-15-C-01265	LW1386	water dilution	mg/kg of filtrate	<1.3E+03	4.45E+04	3.03E+03	<2.9E+02	5.43E+02
Filtered SME Product	TS190-15-C-01278	LW1398	caustic quench	mg/kg of filtrate	<6.7E+02	4.79E+04	3.27E+03	NA	<6.7E+02
Filtered SME Product	TS190-15-C-01279	LW1399	caustic quench	mg/kg of filtrate	<6.2E+02	4.91E+04	3.34E+03	NA	<6.2E+02
Filtered SME Product	TS190-15-C-01280	LW1400	caustic quench	mg/kg of filtrate	<6.5E+02	4.71E+04	3.32E+03	NA	<6.5E+02
Filtered SME Product	TS190-15-C-01281	LW1401	caustic quench	mg/kg of filtrate	<6.2E+02	4.76E+04	3.36E+03	NA	<6.2E+02

(Measurements below the Detection Limits (i.e., less than detection, LTD) are shown with less than signs)

Table 3-62: Anion Measurements of the SRAT and SME Products and Filtered Products (continued)

Type	Sample Name	LIMS	Method	Units	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
SRAT Product	TS190-15-C-01035	LW1152	water dilution	mg/kg of slurry	<6.1E+02	4.24E+04	<1.2E+02	1.26E+03	2.51E+03
SRAT Product	TS190-15-C-01036	LW1153	water dilution	mg/kg of slurry	<6.0E+02	4.31E+04	<1.2E+02	1.28E+03	2.41E+03
SRAT Product	TS190-15-C-01037	LW1154	water dilution	mg/kg of slurry	<6.3E+02	4.30E+04	<1.3E+02	1.28E+03	2.43E+03
SRAT Product	TS190-15-C-01038	LW1155	water dilution	mg/kg of slurry	<6.1E+02	4.30E+04	<1.2E+02	1.27E+03	2.33E+03
SRAT Product	TS190-15-C-01051	LW1167	caustic quench	mg/kg of slurry	<3.2E+03	4.06E+04	<6.3E+02	1.24E+03	4.67E+03
SRAT Product	TS190-15-C-01052	LW1168	caustic quench	mg/kg of slurry	<3.3E+03	4.10E+04	<6.5E+02	1.24E+03	4.89E+03
SRAT Product	TS190-15-C-01053	LW1169	caustic quench	mg/kg of slurry	<3.2E+03	4.10E+04	<6.3E+02	1.23E+03	4.80E+03
SRAT Product	TS190-15-C-01054	LW1170	caustic quench	mg/kg of slurry	<3.1E+03	4.12E+04	<6.2E+02	1.22E+03	4.81E+03
SRAT Product	TS190-15-C-01491	LW1898	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	5.47E+03
SRAT Product	TS190-15-C-01492	LW1899	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	5.18E+03
SRAT Product	TS190-15-C-01493	LW1900	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	5.09E+03
SRAT Product	TS190-15-C-01494	LW1901	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	5.03E+03
Filtered SRAT Product	TS190-15-C-01075	LW1224	water dilution	mg/kg of filtrate	<6.1E+02	4.90E+04	<1.2E+02	1.41E+03	2.65E+03
Filtered SRAT Product	TS190-15-C-01076	LW1225	water dilution	mg/kg of filtrate	<6.4E+02	4.94E+04	<1.3E+02	1.42E+03	2.63E+03
Filtered SRAT Product	TS190-15-C-01077	LW1226	water dilution	mg/kg of filtrate	<5.8E+02	5.08E+04	<1.2E+02	1.46E+03	2.49E+03
Filtered SRAT Product	TS190-15-C-01078	LW1227	water dilution	mg/kg of filtrate	<6.6E+02	4.89E+04	<1.3E+02	1.39E+03	2.49E+03
Filtered SRAT Product	TS190-15-C-01091	LW1239	caustic quench	mg/kg of filtrate	<3.1E+03	4.77E+04	<6.3E+02	1.42E+03	1.98E+03
Filtered SRAT Product	TS190-15-C-01092	LW1240	caustic quench	mg/kg of filtrate	<2.9E+03	4.84E+04	<5.8E+02	1.46E+03	2.04E+03
Filtered SRAT Product	TS190-15-C-01093	LW1241	caustic quench	mg/kg of filtrate	<3.0E+03	4.89E+04	<5.9E+02	1.44E+03	2.00E+03
Filtered SRAT Product	TS190-15-C-01094	LW1242	caustic quench	mg/kg of filtrate	<3.0E+03	4.61E+04	<6.1E+02	1.33E+03	1.88E+03
SME Product	TS190-15-C-01194	LW1313	water dilution	mg/kg of slurry	<1.3E+03	4.53E+04	<1.3E+02	1.20E+03	2.39E+03
SME Product	TS190-15-C-01195	LW1314	water dilution	mg/kg of slurry	<1.3E+03	4.50E+04	<1.3E+02	1.17E+03	2.37E+03
SME Product	TS190-15-C-01196	LW1315	water dilution	mg/kg of slurry	<1.2E+03	4.75E+04	<1.2E+02	1.20E+03	2.35E+03
SME Product	TS190-15-C-01197	LW1316	water dilution	mg/kg of slurry	<1.2E+03	4.85E+04	<1.2E+02	1.18E+03	2.34E+03
SME Product	TS190-15-C-01210	LW1333	caustic quench	mg/kg of slurry	<3.3E+03	4.26E+04	<6.6E+02	1.30E+03	4.89E+03
SME Product	TS190-15-C-01211	LW1334	caustic quench	mg/kg of slurry	<3.3E+03	4.19E+04	<6.6E+02	1.27E+03	4.99E+03
SME Product	TS190-15-C-01212	LW1335	caustic quench	mg/kg of slurry	<3.1E+03	4.04E+04	<6.2E+02	1.21E+03	4.72E+03
SME Product	TS190-15-C-01213	LW1336	caustic quench	mg/kg of slurry	<3.3E+03	4.34E+04	<6.6E+02	1.28E+03	5.07E+03
SME Product	TS190-15-C-01495	LW1902	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	4.71E+03
SME Product	TS190-15-C-01496	LW1903	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	4.97E+03
SME Product	TS190-15-C-01497	LW1904	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	5.05E+03
SME Product	TS190-15-C-01498	LW1905	oxalate prep	mg/kg of slurry	NA	NA	NA	NA	4.71E+03
Filtered SME Product	TS190-15-C-01262	LW1383	water dilution	mg/kg of filtrate	<1.2E+03	7.19E+04	<2.4E+02	1.95E+03	3.61E+03
Filtered SME Product	TS190-15-C-01263	LW1384	water dilution	mg/kg of filtrate	<1.3E+03	8.87E+04	<2.6E+02	2.49E+03	4.35E+03
Filtered SME Product	TS190-15-C-01264	LW1385	water dilution	mg/kg of filtrate	<1.2E+03	7.68E+04	<2.4E+02	2.12E+03	4.00E+03
Filtered SME Product	TS190-15-C-01265	LW1386	water dilution	mg/kg of filtrate	<1.3E+03	7.21E+04	<2.6E+02	2.00E+03	3.83E+03
Filtered SME Product	TS190-15-C-01278	LW1398	caustic quench	mg/kg of filtrate	<1.3E+03	6.21E+04	<6.7E+02	1.87E+03	3.04E+03
Filtered SME Product	TS190-15-C-01279	LW1399	caustic quench	mg/kg of filtrate	<1.2E+03	6.27E+04	<6.2E+02	1.90E+03	3.13E+03
Filtered SME Product	TS190-15-C-01280	LW1400	caustic quench	mg/kg of filtrate	<1.3E+03	6.07E+04	<6.5E+02	1.85E+03	3.02E+03
Filtered SME Product	TS190-15-C-01281	LW1401	caustic quench	mg/kg of filtrate	<1.2E+03	6.13E+04	<6.2E+02	1.86E+03	3.07E+03

(Measurements below the Detection Limits (i.e., less than detection, LTD) are shown with less than signs)

Exhibit A7 in Appendix A provides a more detailed and thorough statistical evaluation of the measurements that were above the detection limits of the analytical procedures. This exhibit provides a statistical review of the anion measurements for the samples of each type of material. Comparisons are made across all of the preparation methods that were utilized to provide the available "above detect" data for the anion in question. In each case, the results from a statistical test for the equality of the variances across the preparation methods for each set of measurements are provided. JMP includes several tests as part of this output. The conclusions made as part of this study are taken from the results of the Levene test. A p-value from this test that is less than or equal to 0.05 (given by the Prob>F value for this test in the exhibit) indicates a statistically significant difference in the variances of the measurements from the preparation methods (at the 5% significance level). If the p-value is greater than 0.05, then the hypothesis of equality for the variances cannot be rejected at the 5% significance level. As an example, consider the results for Formate for the SRAT Product samples in Exhibit A7. Measurements from two preparation methods were available for this element. The p-value for the Levene test is 0.5060, indicating that the hypothesis of equal variances across the two preparation methods cannot be rejected at the 5% significance level for the SRAT Product Formate results.

Comparisons of the average values across the preparation methods are also important comparisons provided for each element as part of the JMP output of this exhibit. There are two approaches for making comparisons of the means across the preparation methods: (1) one approach is based on an assumption of equal variances for the measurements across the preparation methods and (2) the second approach is based on an assumption of unequal variances. JMP provides the appropriate statistical tests for each of these situations: the results from an analysis of variance (ANOVA) for the equal variance case and the results from Welch's test for the unequal variance case. The selection of the appropriate set of results is made by relying on the outcome of the Levene test for the element. As an example, consider once again the results in Exhibit A7 for SRAT Product formate. Since the results from the Levene test did not indicate that the measurement variances were different across the preparation methods, an assumption of equal variance can be made and the ANOVA test results can be used for the comparison of the means. The p-value for this analysis, indicated by the Prob>F value, is given as 0.0010 (a value less than the 0.05 for a 5% significance level). This leads to the conclusion that the means of the measurements for the SRAT Product formate differ across the two preparation methods. If Levene's test had suggested that the variances were equal, the Welch's test would have been used. The p-value for this test is indicated by the value for Prob>F, which would be interpreted in the same manner as the ANOVA's p-value. To aid in the interpretation of these results in the exhibit, the Levene information is enhanced (i.e., bolded and shown in a larger font) as is the appropriate p-value result from the testing for the means.

For Oxalate measurements for the SRAT and SME Product samples, the JMP output in this exhibit provides the results from one additional comparison of the means for this anion. This statistical evaluation makes comparisons across all pairs of means and highlights those that are statistically different at the 5% significance level. Consider the Oxalate results for the SRAT Product samples in Exhibit A7. The Levene's results indicate no statistically significant difference among the variances of the three methods used to complete these measurements. The ANOVA results, however, do indicate a difference among the means of these results. The exhibit also shows:

Level				Mean
oxalate prep	A			5192.5
caustic quench		B		4792.5
water dilution			C	2420

The means of the measurements for the three preparations are given in descending order (top to bottom). They are also labeled from the largest to the smallest average value with an “A”, “B”, and “C.” As indicated in the exhibit, “levels not connected by same letter are significantly different.” Thus, in this case all three means are statistically different from each other with the oxalate prep method yielding the largest mean value and the water dilution the smallest. This is an illustrative example, the SME slurry oxalate mean concentrations were not statistically different between the oxalate prep. and the caustic quench. Although the SRAT product slurry oxalate mean concentrations were statistically different between the oxalate prep. and the caustic quench, as explained later this difference was well within the method uncertainty accounting for day-to-day measurement differences.

Table 3-63 summaries the results from Exhibit A7 as well as providing information of the measurements that were LTDs. This table provides the average of the anion measurements for each method along with the relative standard deviation (%RSD), i.e., the standard deviation as a percentage of the average. For each of those anion/method combinations whose measurements were less than detection, the average of the detection limits is provided. These results have been shaded in Table 3-63. Also, the table contains a column with the results of the Levene test for a difference in the variances of the measurements across the preparation methods for each anion. (A “yes” entry designates a statistically significant, at the 5% level, difference in the variances for the measurements of the indicated element.) The results of the statistical comparisons across the means of the measurements from the preparation methods are highlighted in the last three columns of Table 3-63. The use of the letters “A”, “B”, and “C” in labeling the average values in this table is as described above.

The CQ preparation method was previously recommended as the preferred method for acidic slurry IC anions measurements for the SRAT and SME product with the nitric-glycolic acid flowsheet [7]. During the method development work, the CQ method was shown to provide higher results for glycolate than water dilution results that were known to be biased low. The CQ method values for SRAT and SME product glycolate analyses matched the expected glycolate concentration offgas measurement and mass balance within the 10% error bars. Nitrate and formate analysis were also acceptable with overlapping 10% error bars between CQ and water dilution preparations. This current testing fulfils a recommendation in the IC analysis development report that the CQ method be tested with actual waste when appropriate.

Table 3-63: Anion Results for Samples of SRAT and SME Materials

(Measurements of the SRAT and SME Products are in mg/kg of slurry, and measurements of the Filtered SRAT and SME Products are in mg/kg of filtrate.)

		Shaded Entries Represent Average of "Less than Detection" (LTD) Values			Levene's Test: Variances Significantly Different	"Equal" Means have Connecting Letters [f]		
		Average (%Relative Standard Deviation)						
	Anion	caustic quench	oxalate prep	water dilution		**CQ**	**OP**	**WD**
SRAT Product	Bromide	632.5		612.5				
SRAT Product	Chloride			122.5				
SRAT Product	Fluoride	6325		1225				
SRAT Product	Formate	1450 (0.56%)		1412.5 (0.68%)	no	A		B
SRAT Product	Glycolate	37075 (0.71%)		33750 (0.78%)	no	A		B
SRAT Product	Nitrate	40950 (0.61%)		42875 (0.75%)	no	B		A
SRAT Product	Nitrite	632.5		304 (0.71%)				
SRAT Product	Oxalate	4792.5 (1.90%)	5192.5 (3.76%)	2420 (3.06%)	no	B	A	C
SRAT Product	Phosphate	632.5		122.5				
SRAT Product	Sulfate	1232.5 (0.78%)		1272.5 (0.75%)	no	B		A
SME Product	Bromide	3200		1250				
SME Product	Chloride			125				
SME Product	Fluoride	650		1250				
SME Product	Formate	1487.5 (3.17%)		1952.5 (1.06%)	no	B		A
SME Product	Glycolate	32750 (2.21%)		31575 (1.40%)	no	A		B
SME Product	Nitrate	42075 (3.03%)		46575 (3.65%)	no	B		A
SME Product	Nitrite	650		379.75 (1.08%)				
SME Product	Oxalate	4917.5 (3.07%)	4860 (3.63%)	2362.5 (0.94%)	yes	A	A	B
SME Product	Phosphate	650		125				
SME Product	Sulfate	1265 (3.06%)		1187.5 (1.26%)	no	A		B
Filtered SRAT Product	Bromide	3000		622.5				
Filtered SRAT Product	Chloride			125				
Filtered SRAT Product	Fluoride	6550		1250				
Filtered SRAT Product	Formate	1730 (3.24%)		1635 (2.26%)	no	A		B
Filtered SRAT Product	Glycolate	38400 (2.34%)		37000 (1.86%)	no	A		B
Filtered SRAT Product	Nitrate	47775 (2.55%)		49525 (1.77%)	no	A		A
Filtered SRAT Product	Nitrite	602.5		360 (2.65%)				
Filtered SRAT Product	Phosphate	602.5		125				
Filtered SRAT Product	Oxalate	1975 (3.45%)		2565 (3.39%)	no	B		A
Filtered SRAT Product	Sulfate	1412.5 (4.06%)		1420 (2.07%)	no	A		A
Filtered SME Product	Bromide	1250		1250				
Filtered SME Product	Chloride			286.7				
Filtered SME Product	Fluoride	640		1250				
Filtered SME Product	Formate	3322.5 (1.16%)		3330 (11.1%)	no	A		A
Filtered SME Product	Glycolate	47925 (1.77%)		48600 (10.8%)	no	A		A
Filtered SME Product	Nitrate	61700 (1.43%)		77375 (10.2%)	no	B		A
Filtered SME Product	Nitrite	640		589.5 (11.5%)				
Filtered SME Product	Oxalate	3065 (1.56%)		3947.5 (7.91%)	no	B		A
Filtered SME Product	Phosphate	640		250				
Filtered SME Product	Sulfate	1870 (1.16%)		2140 (11.4%)	no	A		A

[f] CQ represents Caustic Quench; OP represents Oxalate Prep, and WD represents Water Dilution; and the comparisons of the oxalate means presented in these columns are made with the assumption of equal variances for the methods.

The ANOVA performed comparing CQ and water dilutions showed statistically significant differences between the two preparation methods for most analytes in the SRAT and SME products. There was no statistically significant difference between the SME product analysis of oxalate by CQ and by the acid digestion oxalate preparation. However, the actual differences between the methods were small when compared to the reported 10% uncertainty in the IC method. The small variances (reflected in the small %RSD in the measurements) lead to the determination that most measurements were statistically different between the preparation methods. The larger day-to-day variability in IC results would be expected to lead to much larger variance than those encountered in this analysis. Thus, when comparing results obtained on different IC batches, it would be more appropriate to take the IC method uncertainty into account even when the %RSD values are small.

The IC method development report used the criterion of overlapping of the 10% error bars to determine which constituents compared well between methods. If the one sigma uncertainty for the method was actually 10%, the roughly 95% confidence two-sigma uncertainty would actually be 20%. Using this criterion of overlapping 10% error bars, nitrate and glycolate measurements in the SRAT and SME products compare well between CQ and water dilution preparations. Error bars for formate in the SME product slurry do not overlap, with the water dilution measurement higher than the CQ method measurement and the water dilution measurement more consistent with the SME product supernatant analysis. Oxalate measurements in the SRAT and SME products compare well between the CQ method and acid digestion oxalate preparation. This testing confirmed with actual waste that recommended CQ preparation is acceptable for anion measurement by IC for SRAT and SME product slurries.

CQ preparation successfully replicated the results of the acid digestion oxalate preparation, indicating on an actual waste sample that CQ preparation is valid for use of oxalate measurement in SRAT receipt and SRAT and SME product slurries. Though not developed for caustic solutions, CQ preparation appeared to also be adequate for IC analysis of SRAT receipt slurry.

In most cases, the CQ preparation appeared adequate for IC analysis of supernatant as well as slurry. However, there appears to be no advantage to using CQ over water dilution for supernatant. For the case of oxalate analysis in SRAT and SME product supernatant, there appeared to be a difference between water dilution and CQ preparations that is greater than the day-to-day experimental uncertainty, with water dilution giving the larger measurement result for both cases.

3.5.3 Rheology

Presented in Table 3-64 are rheology measurement results of the SC-18 CPC processing samples. DWPF design basis values are included for reference [35], [36]. Flow curves are presented in Figure 3-16, Figure 3-17, and Figure 3-18. As can be seen in the table, the consistency and yield stress of the SRAT receipt material fall within the DWPF design basis, while the SRAT and SME product yield stresses are below the DWPF design basis. The SRAT product showed no measureable yield stress and low consistency. As can be seen in the SRAT product flow curves (Figure 3-17), the SRAT product has a negligible yield stress and the consistency is at the lower end of the SRNL instrument sensitivity. For each sample (SRAT receipt, SRAT product, and SME product), material remained in the rheometer; material was not removed and remixed. Figure 3-19 contains a comparison of the SME product flow curves to the melter feed design basis rheology range, as indicated by the green shaded area. The SME product results are rheologically similar to those of SB5 melter feed samples evaluated by SRNL when DWPF was having problems with melter feed loop clogging; DWPF attributed the clogging to the rheological properties of the SME product (low yield stress) [37]. DWPF was able to successfully process SB5 by implementing process control software to mitigate issues related to the melter feed. This software is still in use at DWPF [38].

Table 3-64: Rheology of Sludge Batch 9 Related Samples

Sample Description	Consistency (cP)	Yield Stress (Pa)
Washed Sludge Slurry Design Basis	4-12	2.5-10
SC-18 SRAT Receipt	5.9	3.3
SRAT Slurry Design Basis	5-12	1.5-5
SC-18 SRAT Product	2.8	0
Melter Feed Design Basis	10-40	2.5-15
SC-18 SME Product	16.0	1.0

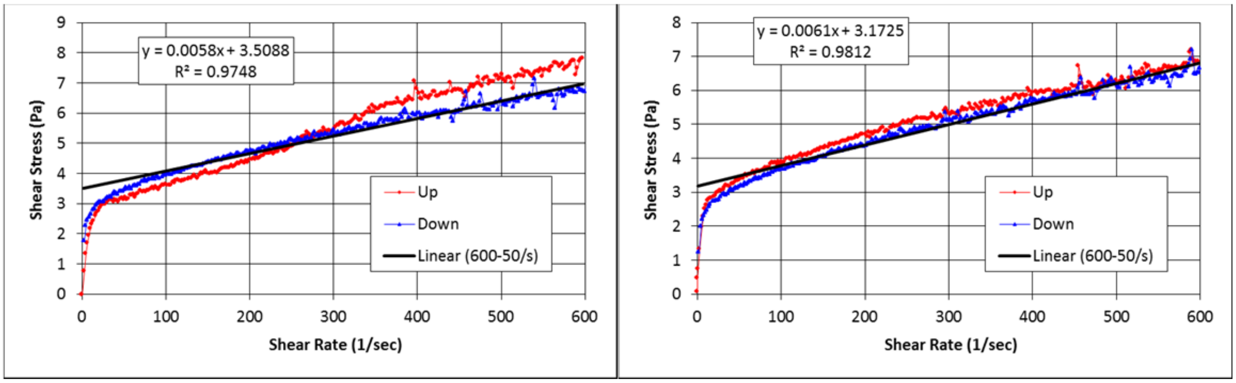


Figure 3-16: SRAT Receipt Flow Curves

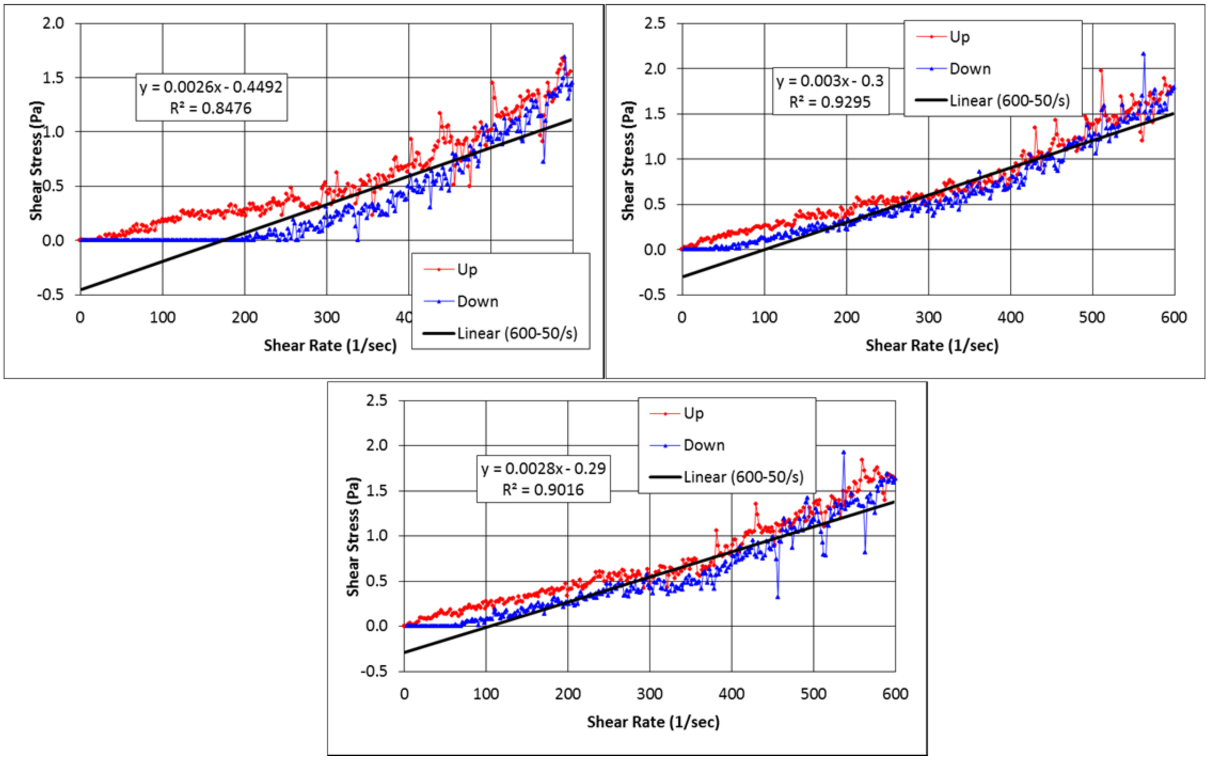


Figure 3-17: SRAT Product Flow Curves

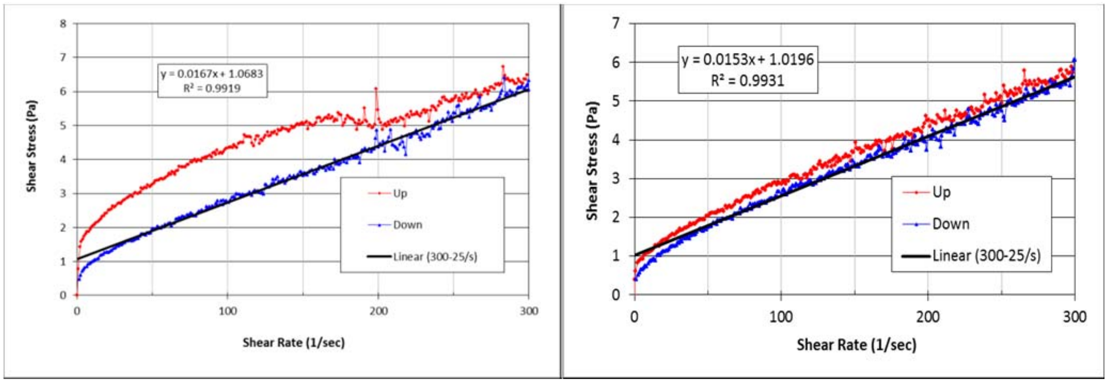


Figure 3-18: SME Product Flow Curves

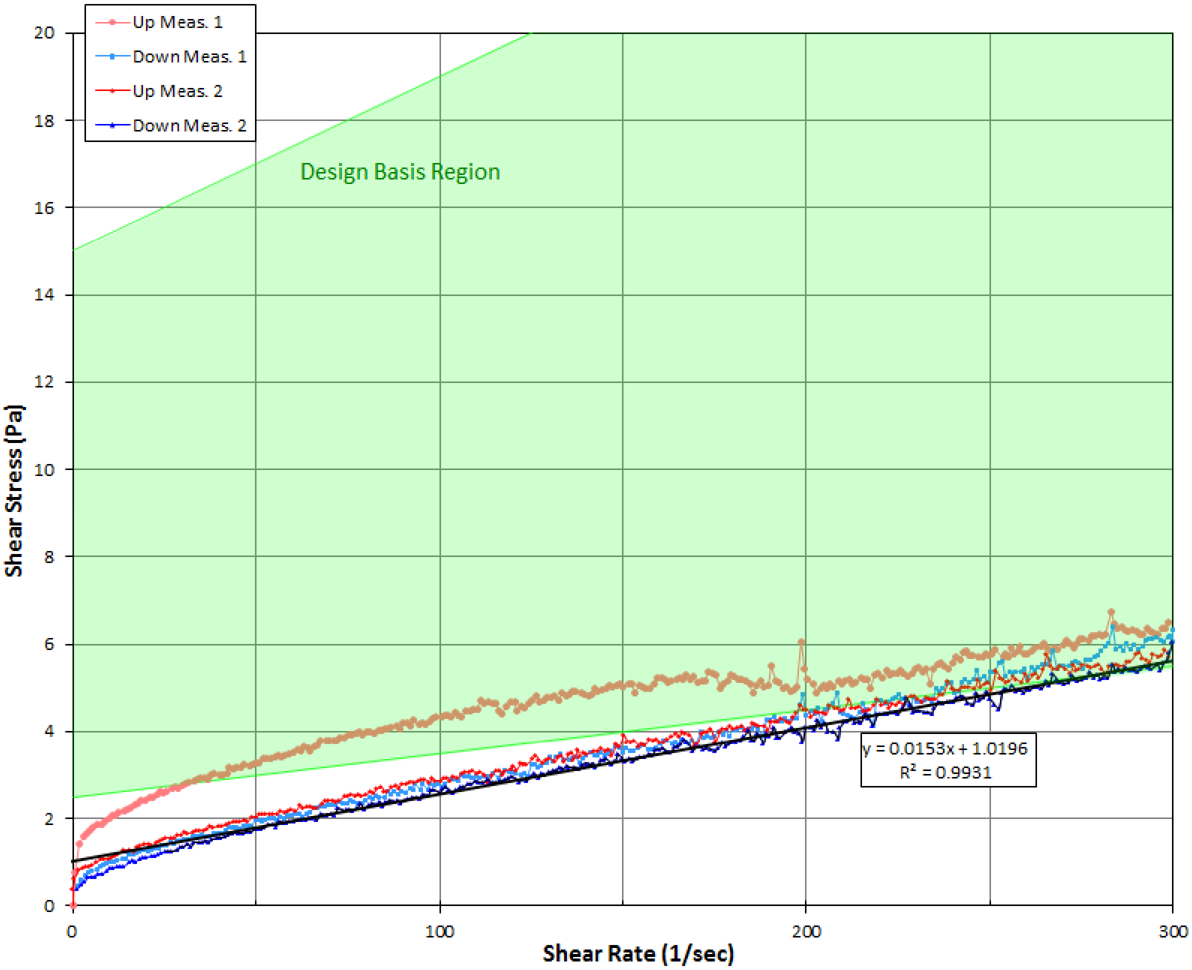


Figure 3-19: SME Product Flow Curve Compared with Design Basis

3.6 Antifoam Addition Strategy

Acid additions and boiling during CPC processing can result in significant offgas production and foaming of the sludge slurry. Foaming can result in a carryover of material from the SRAT into the offgas and condensate systems. In an effort to prevent, or minimize, a carryover event, an antifoaming agent is added. The amount of antifoam added for the SC-18 demonstration was scaled based on a 6000 gallon DWPF SRAT receipt (Table 3-24). However, approximately one tenth of the targeted amount of antifoam was added in the SRAT: ~0.077g of antifoam (0.15 gallon DWPF equivalent) and ~0.052g of antifoam (0.10 gallon DWPF equivalent). Table 3-65 reports the actual amount of antifoam initially weighed. The syringes used to deliver antifoam to the SRAT slurry were not weighed after use. Considering the SME data for antifoam addition (Table 3-66), it can be assumed that the actual amount of antifoam added during the SRAT cycle was approximately 30% less than that reported in Table 3-65. Despite adding a significantly smaller amount of antifoam, no foaming was observed throughout the demonstration.

After discussing with SRR the possibility of implementing a strategy requiring less than historical amounts of antifoam for the SME cycle, it was decided that an antifoam addition strategy that utilized smaller amounts of antifoam would be implemented. The syringes used to deliver antifoam to the slurry were weighed before and after use. Table 3-66 reports the actual amounts of antifoam added during the SME cycle. The SME cycle successfully completed without observing any significant amount of foaming.

Table 3-65: Antifoam Addition During SRAT Cycle

Time of Addition	DWPF Equivalent (gal)	Amount Added (g)
Post Nitric Acid Addition	0.15	0.0783
Post Glycolic Acid Addition	0.10	0.0525
1 hr, 26 min into boiling	0.10	0.0520

Table 3-66: Antifoam Addition During SME Cycle

Time of Addition	DWPF Equivalent (gal)	Target (g)	Amount Added (g)
Canister Decontamination #1	0.25	0.071	0.041
Canister Decontamination #2	0.25	0.071	0.048
Canister Decontamination #3	0.25	0.071	0.046
Canister Decontamination #4	0.25	0.071	0.049
Canister Decontamination #5	0.25	0.071	0.045
Canister Decontamination #6	0.25	0.071	0.056
Frit Addition #1	0.25	0.071	0.062
Frit Addition #2	0.25	0.071	0.053

3.7 Glass Fabrication and Analysis

3.7.1 Compositional Analysis

Table 3-67 shows a comparison of the published [11] and measured composition of the ARG-1 glass. The preparation method along with the percent (%) difference is also noted for each analyte. Oxides having concentrations greater than 0.25 wt% are within 10% of the published values, which demonstrates the effectiveness of the specified preparation method. The sum of oxides[b] is 98.74 wt%, which is 1.34 wt% lower than the published value of 100.08 wt%.

Table 3-68 provides a comparison of the measured multi-element standard solution results (measured with glass samples prepared by the indicated preparation method) to the reference values. All of the measured values are within 10% of the reference values, which confirms the accuracy of the ICP-AES measurements.

Table 3-67: Published [11] and Measured Oxide Values for the ARG-1 Glass

Oxide	Published (wt%)	Measured (wt%)	% Difference	Preparation Method
Al_2O_3	4.73 ± 0.022	4.69	-0.9	PF
B_2O_3	8.67 ± 0.040	8.13	-6.2	PF
BaO	0.088 ± 0.001	0.09	-1.1	MA
CaO	1.43 ± 0.009	1.44	0.8	MA
Cr_2O_3	0.093 ± 0.001	0.10	2.4	MA
CuO	0.004 ± 0.000	<0.01	133.8	MA
Fe_2O_3	14.0 ± 0.073	14.08	0.5	PF
K_2O	2.71 ± 0.016	2.56	-5.5	MA
Li_2O	3.21 ± 0.015	3.17	-1.1	PF
MgO	0.86 ± 0.005	0.86	0.4	PF
MnO_2	2.31 ± 0.012	2.31	0	PF
Na_2O	11.5 ± 0.023	10.52	-8.5	MA
NiO	1.05 ± 0.006	0.99	-5.5	MA
P_2O_5	0.22 ± 0.011	0.25	15.1	MA
SiO_2	47.9 ± 0.157	48.24	0.7	PF
SrO	0.0037 ± 0.000	0.004	1.3	MA
TiO_2	1.15 ± 0.007	1.15	-0.4	PF
ZnO	0.02 ± 0.000	0.02	-6.0	PF
ZrO_2	0.13 ± 0.005	0.13	0.5	MA
Total	100.08	98.74	----	----

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[b] The totals shown in the table were calculated using more significant figures than shown and were rounded to two decimal places.

Table 3-68: Multi-Element Standard Solution Results (SM-744-013)

Element	Reference Value (mg/L)	Measured Value (mg/L)	% Difference	Glass Sample Preparation Method
Al	4	3.69	-7.9	PF
B	20	19.45	-2.7	PF
Fe	4	4.24	6.0	PF
K	10	9.56	-4.6	MA
Li	10	10.2	2.0	MA
Na	81	85.5	5.2	MA
Si	50	53.6	7.1	PF

Table 3-69 presents the measured elemental and oxide compositions of the SB9 SC-18 Glass along with the preparation method and %RSD for each analyte. The measured value for each analyte is the average of four replicates. A majority of the components have %RSD values that are less than 5%, which indicates minimal scatter amongst the four replicates. The sum of oxides[b] is 99.05 wt%, which is within the DWPF Product Composition Control System (PCCS) limit of 95-105 wt%.

Table 3-69: SB9 SC18 Glass Composition

Element	Average Measured (wt%)	Oxide	Average Measured (wt%)	% RSD	Preparation Method
Al	3.28	Al_2O_3	6.20	1.1	PF
B	1.44	B_2O_3	4.64	1.0	PF
Ba	0.04	BaO	0.04	1.5	MA
Ca	0.46	CaO	0.65	1.8	MA
Ce	0.02	Ce_2O_3	0.02	3.0	MA
Cr	0.04	Cr_2O_3	0.06	5.4	MA
Cs	0.001	Cs_2O	0.001	5.5	PF*
Cu	0.10	CuO	0.12	1.0	MA
Fe	7.91	Fe_2O_3	11.31	1.2	PF
K	<0.20	K_2O	<0.24	----	PF
La	0.02	La_2O_3	0.03	1.8	MA
Li	1.78	Li_2O	3.82	1.0	PF
Mg	<0.08	MgO	<0.14	----	PF
Mn	2.61	MnO	3.37	1.1	PF
Mo	<0.01	MoO_3	<0.01	----	MA
Na	10.00	Na_2O	13.48	5.5	MA
Nd	0.05	Nd_2O_3	0.06	----	PF*
Ni	0.55	NiO	0.71	1.7	MA
P	0.10	P_2O_5	0.22	5.7	MA
Pb	<0.03	PbO	<0.03	----	MA
S	0.09	SO_4	0.26	4.3	MA
Si	23.90	SiO_2	51.13	2.0	PF
Th	0.40	ThO_2	0.46	1.6	MA
Ti	<0.06	TiO_2	<0.10	----	PF
U	1.52	U_3O_8	1.79	1.1	MA
Y	0.01	Y_2O_3	0.01	3.9	PF*
Zn	0.02	ZnO	0.03	5.9	PF
Zr	0.09	ZrO	0.12	1.5	MA
----	----	Total	99.05	----	----

*ICP-MS

3.7.2 PCCS Model Predictions

The measured SB9 S-C18 Glass composition reported in Table 3-69 was used to predict specific properties of the glass using the DWPF PCCS models. The results are shown in Table 3-70. All of the predicted properties were compared to SME acceptability criteria [39] to evaluate whether this glass met the DWPF processing and product quality constraints. Based on the measured composition, all of the predicted properties met the PCCS Measurement Acceptability Region (MAR) criteria.

Table 3-70: PCCS Model Prediction Results for the SB9 SC18 Glass

PCCS Model	Predicted Value
ΔG_p Value (kcal/100 g glass)	-10.12
Boron Normalized Concentration (NC_B)	0.86
Lithium Normalized Concentration (NC_{Li})	0.87
Sodium Normalized Concentration (NC_{Na})	0.85
Liquidus Temperature (°C)	881
Viscosity (poise)	62.9
Al_2O_3 concentration (wt%)	6.20
Sum of Oxides (%) *	98.78
Nepheline Discriminator Ratio	0.72
All PCCS MAR Criteria met	yes

*PCCS does not include SO_4^{2-} in the sum of oxides

4.0 Conclusions

SRNL has completed SB9 qualification of the nitric/glycolic flowsheet. CPC demonstrations utilizing the alternate reductant flowsheet were performed at an acid stoichiometry of 78.0% KMA for the SRAT cycle. The total solids measurements of the SRAT and SME products were 24.6 and 49.0 wt%, respectively, which matched the targets of 25 wt% and 48 wt% acceptably. The consistency and yield stress of the SRAT receipt material fall within the DWPF design basis, while the SRAT and SME product yield stresses are below the DWPF design basis. It should be noted that similar rheological properties were encountered during SB5 processing at DWPF. DWPF was able to successfully process SB5 by implementing process control software to mitigate issues related to the melter feed. This software is still in use at DWPF.

Based on the comparison of the iron in the SRAT product and SME product analysis, the waste loading of the SME product was 37.6%. The current DWPF target for waste loading is 36%. Calculations based on several of the other major sludge components (Al, Ca, Mn, Ni, and U) indicated waste loadings ranging from 36.6% to 37.9%, in good agreement with the Fe-based result. Based on the measured composition of the SB9 SC-18 glass, all of the predicted properties met the PCCS MAR criteria. The SB9 SC-18 glass appeared dark and shiny, without the presence of a visible salt layer or crystals present.

In the SRAT and SME cycles, nitrite was destroyed to 304 mg/kg of SRAT product slurry and 380 mg/kg of SME product slurry. After the application of uncertainty, the measured peak concentrations of carbon dioxide and nitrous oxide in the SRAT were 441 lb/hr and 0.73 vol%, respectively, and 34 lb/hr and 0.20 vol% in the SME, respectively. Hydrogen was not detected above the detection limit of 0.0014 lb/hr in the SME.

The final concentration of mercury in the SRAT product was 0.71 wt% of the total solids, which is below the target for mercury removal of 0.8 wt% of the total solids. With the additional mercury stripping during the SME cycle and the dilution of total solids with frit, the mercury concentration in the SME product dropped to 0.21 wt% of the total solids. When the SME product is put back on the same basis as the SRAT product total solids, the mercury concentration of the SME product would correspond to 0.46 wt% of SRAT total solids, near the target mercury removal to 0.45 wt% of total solids. It is expected that additional boiling would further remove mercury to levels below the target.

Antifoam 747 was added at a DWPF equivalent of 0.15 gal after nitric acid addition and 0.10 gal prior to SRAT boiling. In the SME, the DWPF equivalent of 0.25 gal of antifoam was added prior to canister decontamination water additions and frit additions. No significant foaming was observed throughout the SRAT and SME cycles.

Of the nine elements that comprise at least 0.5 wt% of the alternate reductant sludge on a dry solids basis, the CC method produced statistically equivalent results to either the AR or PF methods for seven elements (Al, Ca, Fe, Mn, Ni, Th, and U). The CC Na determinations were ~7.5 % lower than the AR Na determinations. Si determinations are precluded in the CC method because the strong attack of HF on the quartz components of the SRNL ICP-AES sample introduction system leads to high Si bias.

The precision of the CC determinations was excellent with less than 5% relative standard deviation of four determinations for all elements with a concentration of at least 0.5 wt% in the sludge.

In view of the excellent precision and agreement with reference AR and PF dissolution methods for most elements, the CC method should be adequate to provide DWPF process control analyses after performing side-by-side tests of the CC and PF methods for the first 10 SRAT batches with SB9 sludge processed with the nitric-glycolic flowsheet. During these tests, the DWPF Laboratory will be able to accumulate enough data over a long time period to determine if the CC method produces biases and, if so, if these biases are acceptable. The nitric-glycolic flowsheet does not appear to have a negative impact on the use of the CC method for DWPF process control analyses.

The 8% difference in mercury determinations after sample preparations by the DWPF method and the aqua regia method should not raise a red flag of undue concern. The DWPF method should be superior for destroying the organic compounds in the DWPF samples that can have a deleterious effect on mercury analyses by atomic absorption. The DWPF should continue with its plans to use its method for sample preparation to support mercury process control analyses.

This testing confirmed with actual waste that the recommended CQ preparation is acceptable for anion measurement by IC for SRAT and SME product slurries. CQ preparation also appeared to be acceptable for use with IC analysis of SRAT receipt slurry.

CQ preparation successfully replicated the results of the acid digestion oxalate preparation, indicating on an actual waste sample that CQ preparation is valid for use of oxalate measurement in SRAT receipt and SRAT and SME product slurries.

5.0 Recommendations

- Based on the results of this actual-waste qualification and previous simulant studies, SRNL recommends implementation of the nitric-glycolic acid flowsheet in DWPF.

- Given the excellent performance of the 4-L vessel used in the Shielded Cells, it is recommended that DWPF consider using similar equipment for future sludge batch qualifications. Additional equipment development should be pursued to re-design the 1-L vessel (e.g., mixer, heating, insulation).

- Throughout the SB9 qualification testing, no foaming was observed. DWPF should consider implementing a reduced antifoam addition strategy similar to what was used during SC-18 qualification.

- Testing with actual waste confirmed that the Caustic Quench method previously developed [7] should be used for anion measurement by IC for SRAT and SME product slurries and SRAT receipt slurry.

6.0 References

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Appendix A. Supporting Statistical Exhibits

Exhibit A1. Plots of the Solution Standard Measurements in mg/L

Reference Values (mg/L) of Elements Present in the Standard:
Al(50); Fe(50); Mn(20); Na(150); Ni(10); S (10)

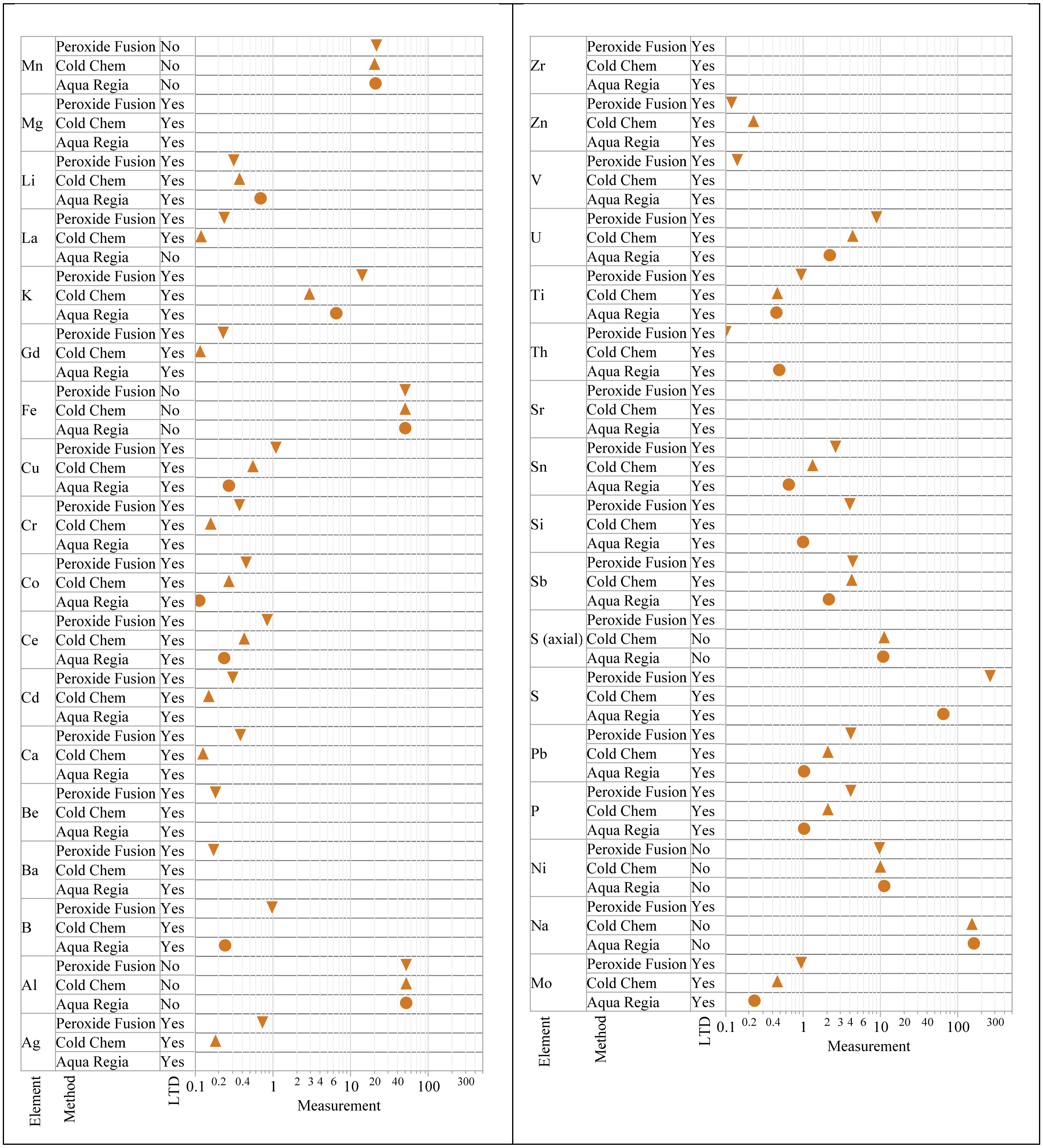
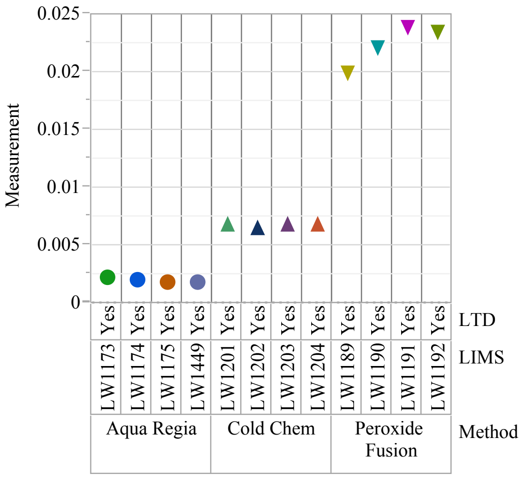


Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

Element=Ag



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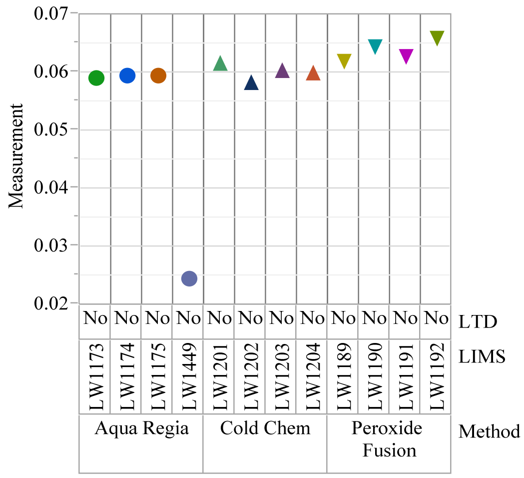

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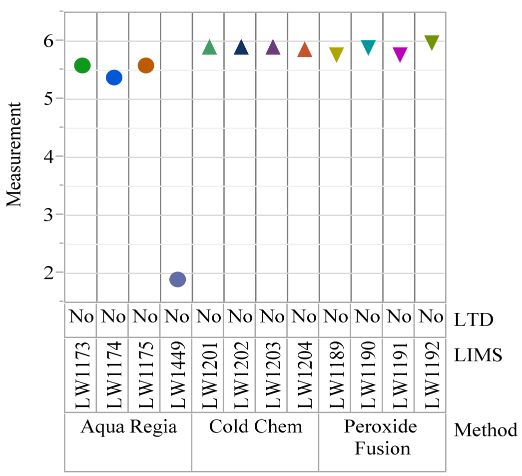

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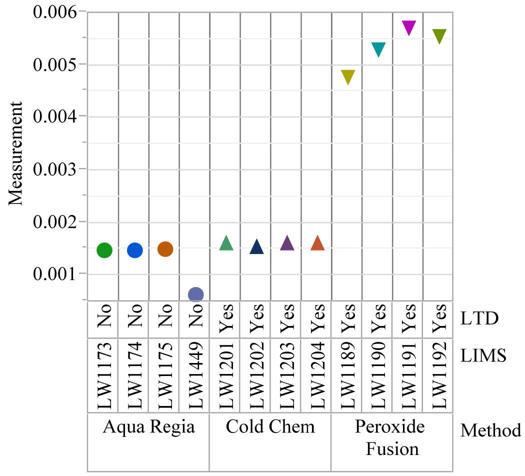

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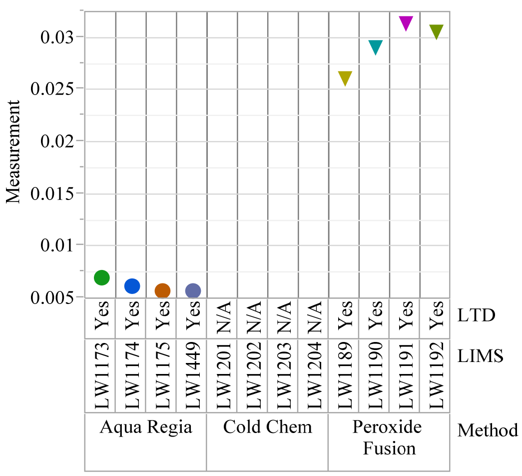

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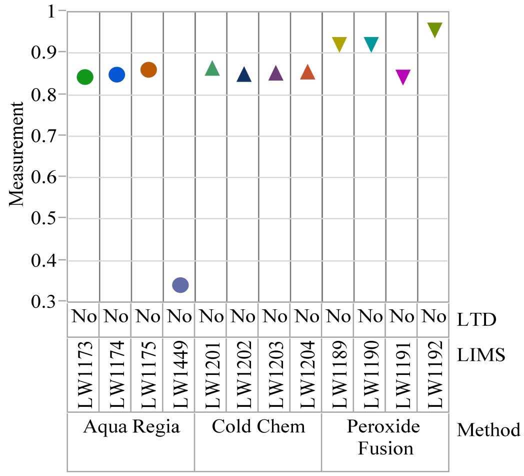
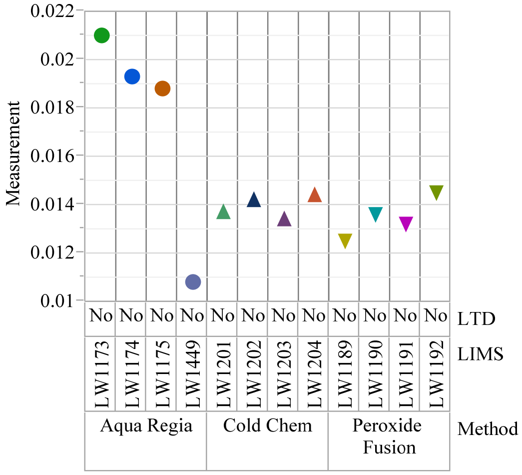


Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

Element=Cd



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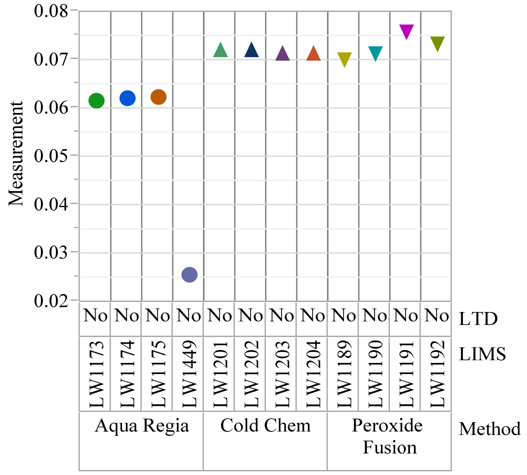
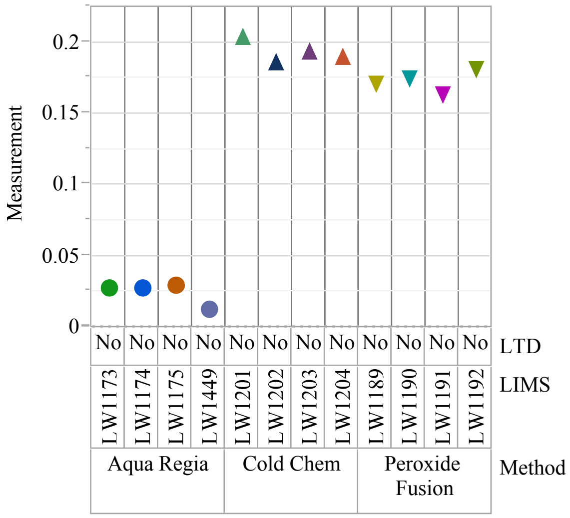
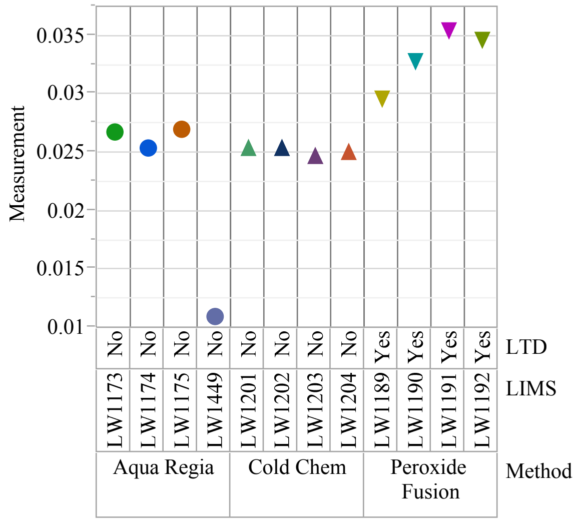


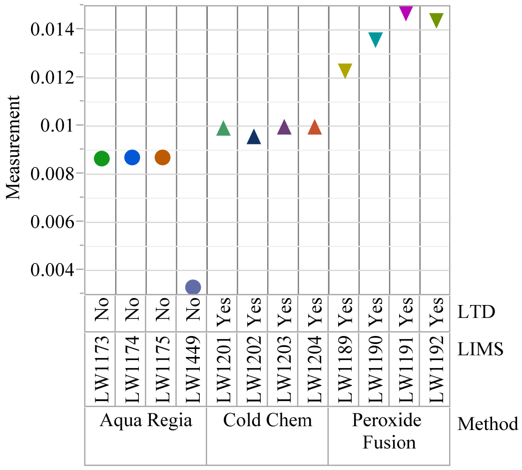
Element=Ce



Element=Cu



Element=Co



Element=Fe

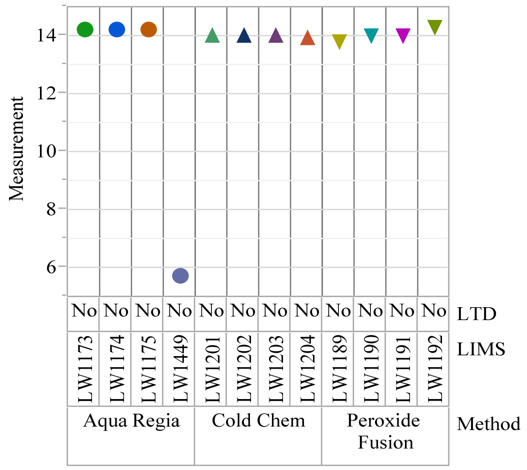
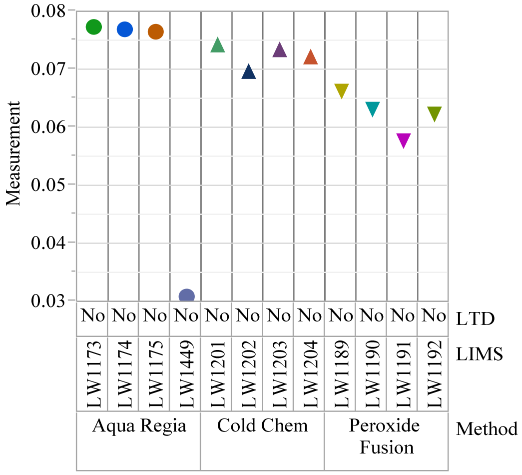


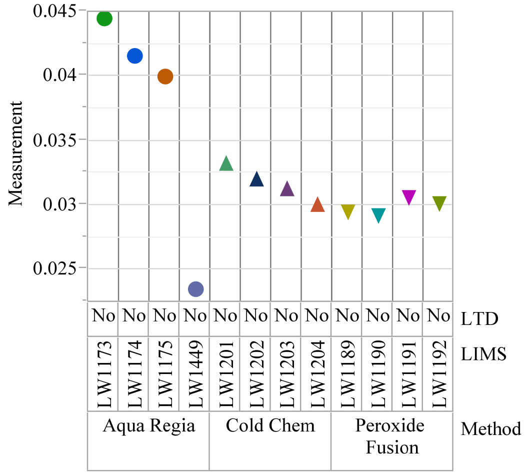
Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

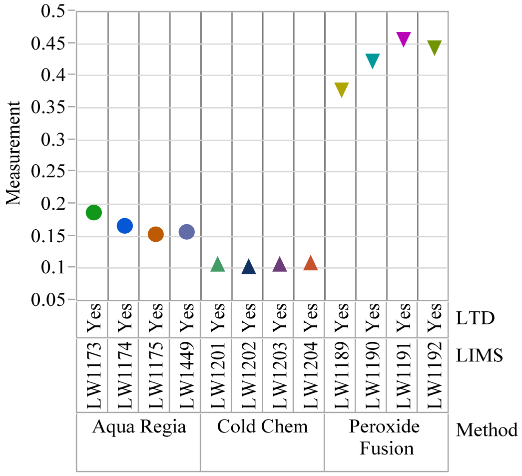
Element=Gd



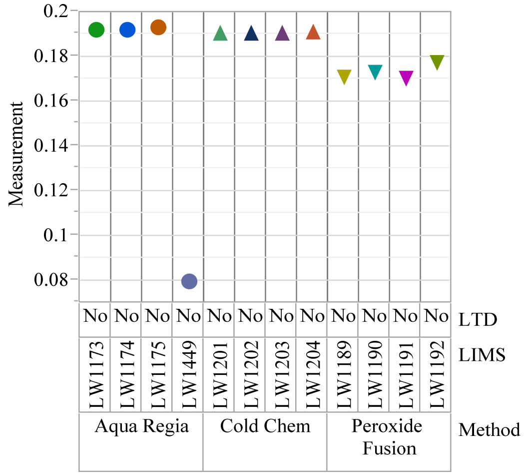
Element=Li



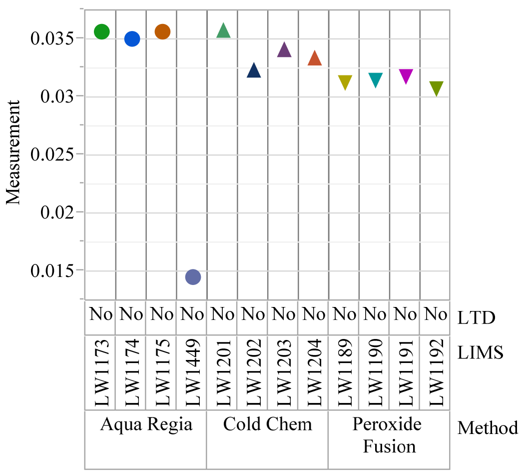
Element=K



Element=Mg



Element=La



Element=Mn

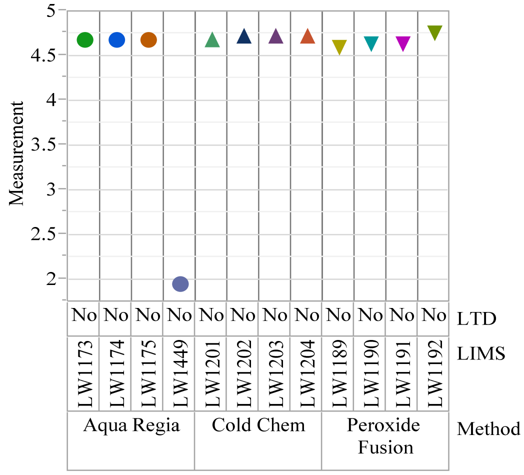
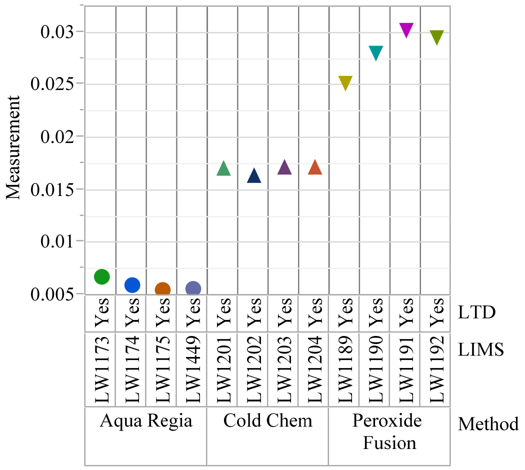


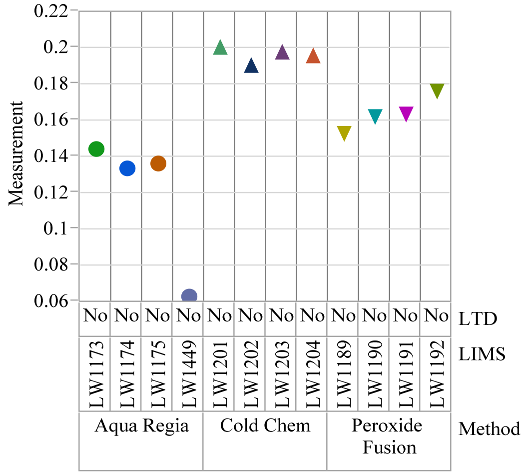
Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

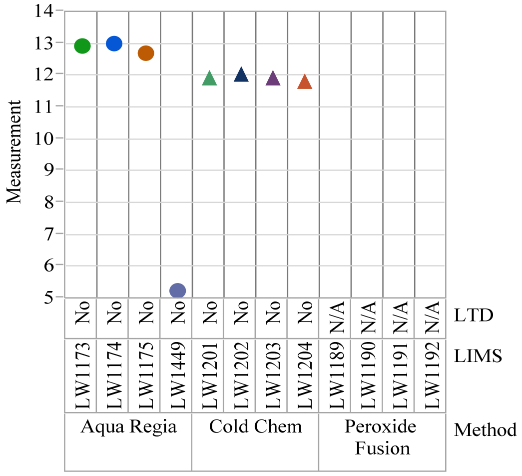
Element=Mo



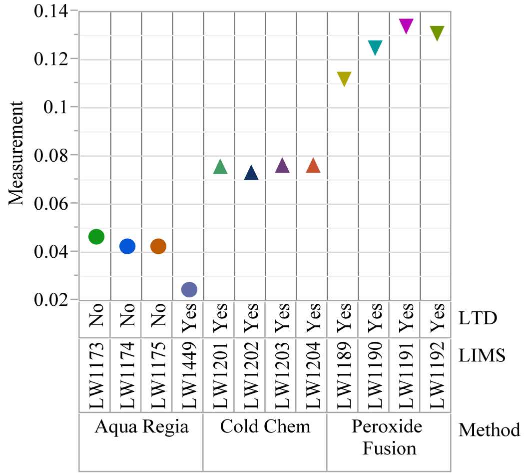
Element=P



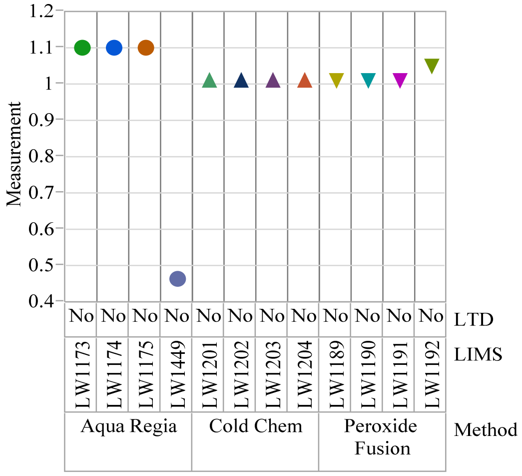
Element=Na



Element=Pb



Element=Ni



Element=S

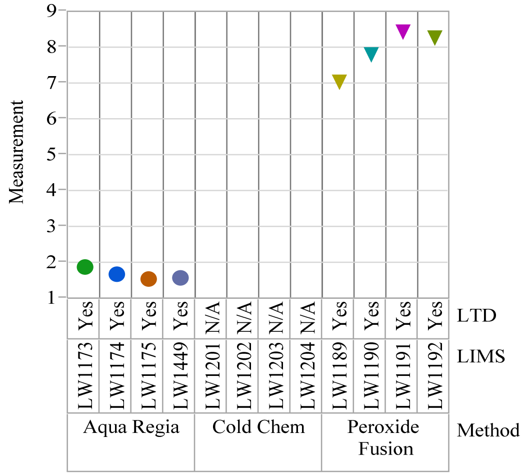
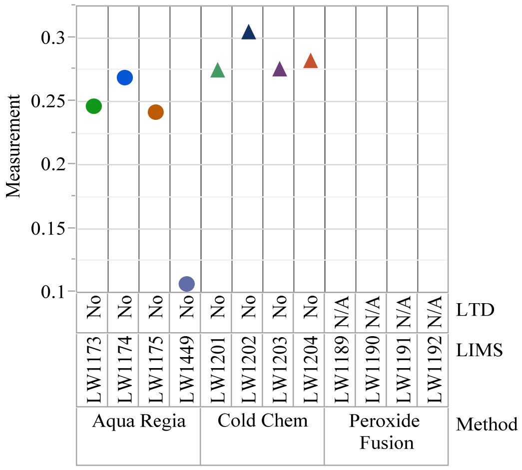


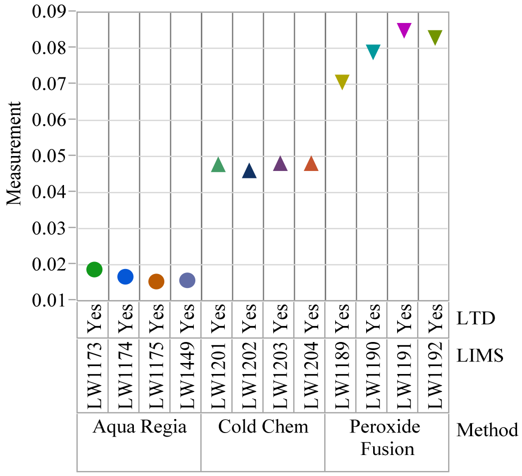
Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

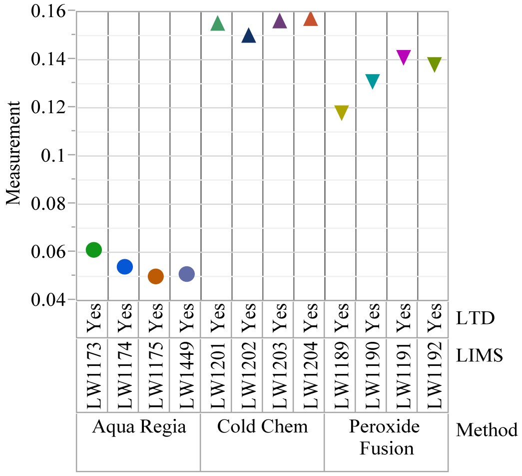
Element=S (axial)



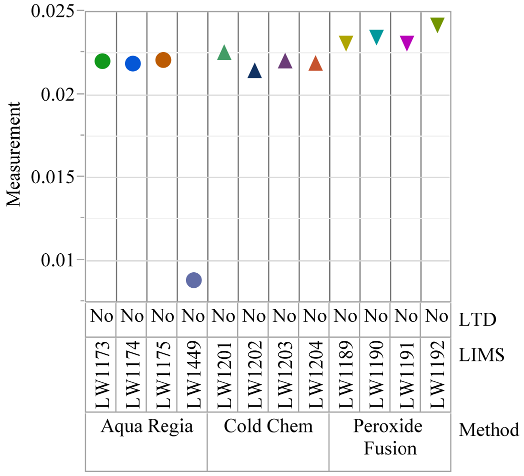
Element=Sn



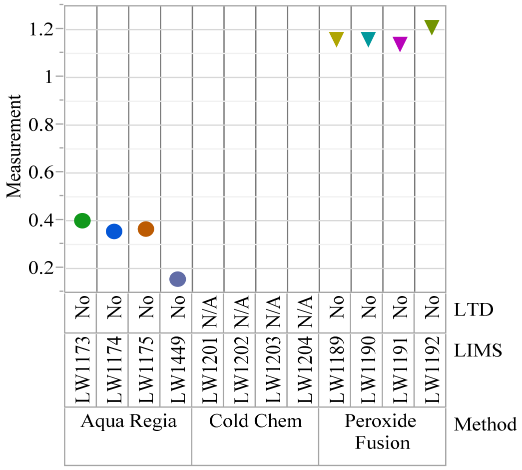
Element=Sb



Element=Sr



Element=Si



Element=Th

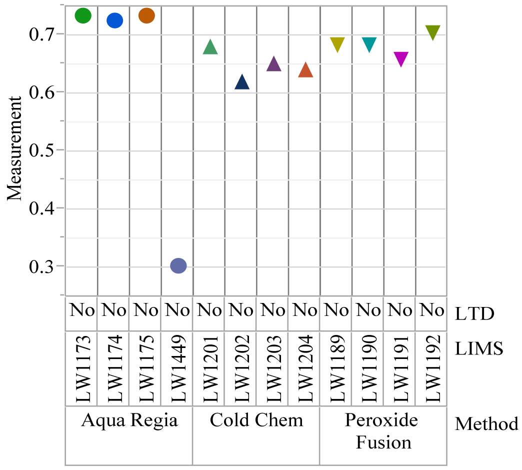
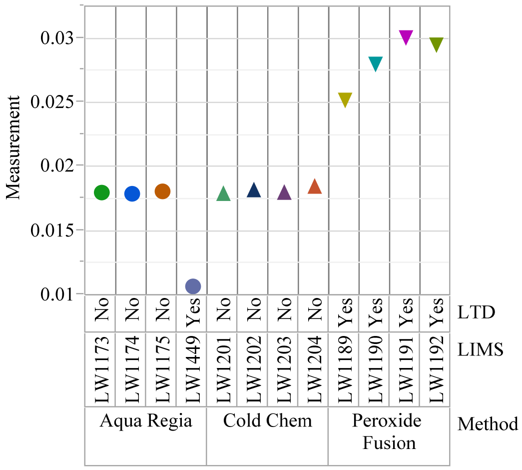


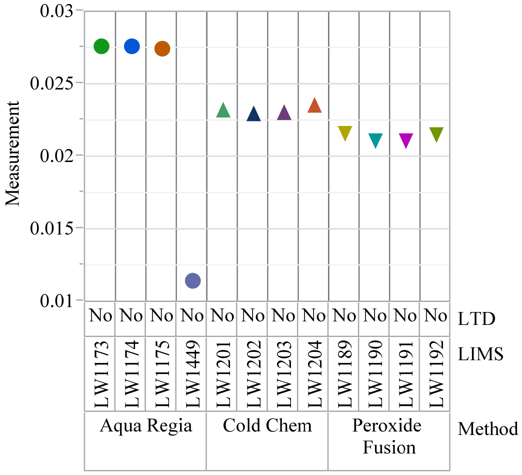
Exhibit A2. Variability Chart for SRAT Product Measurements of Elementals *(continued)*

(Measurements are in wt% of Total Solids)

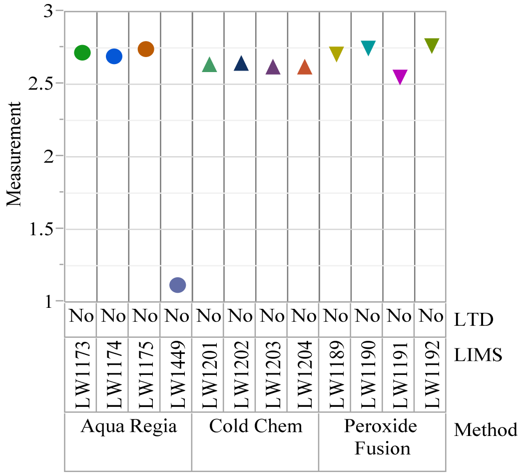
Element=Ti



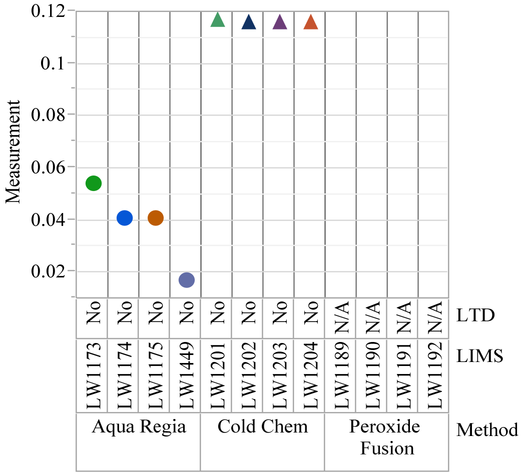
Element=Zn



Element=U



Element=Zr



Element=V

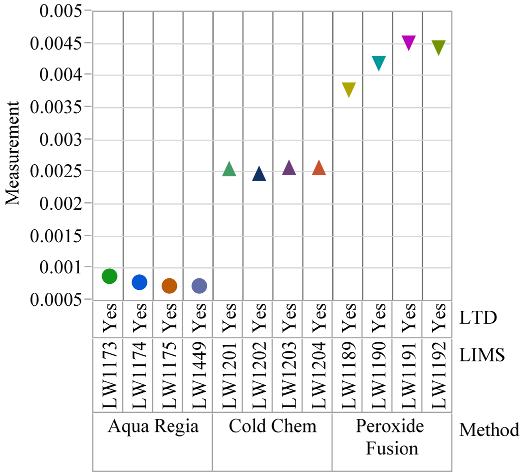
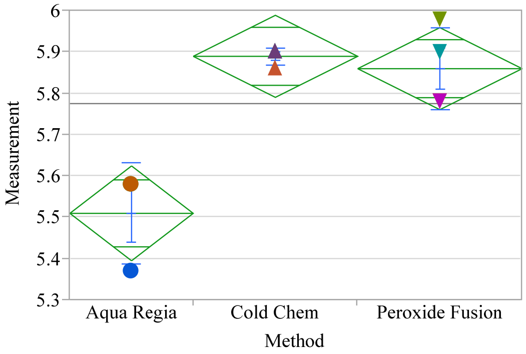


Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Al



Oneway Anova
Summary of Fit

Rsquare	0.831189
Adj Rsquare	0.788986
Root Mean Square Error	0.086168
Mean of Response	5.775455
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.29247273	0.146236	19.6951	0.0008*
Error	8	0.05940000	0.007425		
C. Total	10	0.35187273			

Means for Oneway Anova

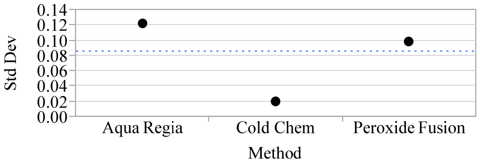
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	5.51000	0.04975	5.3953	5.6247
Cold Chem	4	5.89000	0.04308	5.7906	5.9894
Peroxide Fusion	4	5.86000	0.04308	5.7606	5.9594

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	5.51000	0.121244	0.07000	5.2088	5.8112
Cold Chem	4	5.89000	0.020000	0.01000	5.8582	5.9218
Peroxide Fusion	4	5.86000	0.097980	0.04899	5.7041	6.0159

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.1212436	0.0933333	0.0700000
Cold Chem	4	0.0200000	0.0150000	0.0100000
Peroxide Fusion	4	0.0979796	0.0800000	0.0800000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.5521	2	8	0.2694
Brown-Forsythe	1.2614	2	8	0.3341
Levene	**7.7044**	**2**	**8**	**0.0136**
Bartlett	2.8232	2	.	0.0594

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

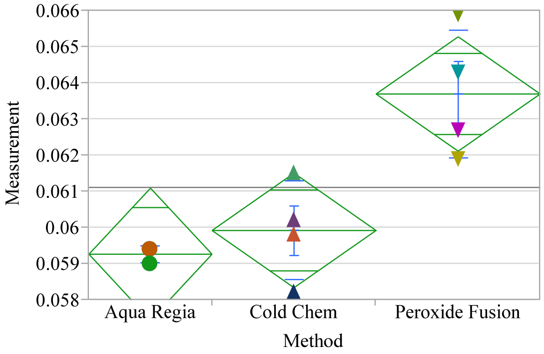
F Ratio	DFNum	DFDen	Prob > F
12.1428	2	3.3764	**0.0287***

Means Comparisons
Connecting Letters Report

Level			Mean
Cold Chem	A		5.8900000
Peroxide Fusion	A		5.8600000
Aqua Regia		B	5.5100000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Ba



Oneway Anova
Summary of Fit

Rsquare	0.738567
Adj Rsquare	0.673209
Root Mean Square Error	0.001374
Mean of Response	0.061118
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00004264	0.000021	11.3003	**0.0047***
Error	8	0.00001509	1.887e-6		
C. Total	10	0.00005774			

Means for Oneway Anova

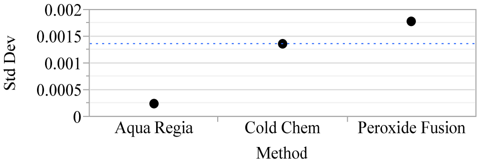
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.059267	0.00079	0.05744	0.06110
Cold Chem	4	0.059925	0.00069	0.05834	0.06151
Peroxide Fusion	4	0.063700	0.00069	0.06212	0.06528

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.059267	0.000231	0.00013	0.05869	0.05984
Cold Chem	4	0.059925	0.001360	0.00068	0.05776	0.06209
Peroxide Fusion	4	0.063700	0.001774	0.00089	0.06088	0.06652

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0002309	0.0001778	0.0001333
Cold Chem	4	0.0013598	0.0009250	0.0009250
Peroxide Fusion	4	0.0017739	0.0014000	0.0014000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1754	2	8	0.3568
Brown-Forsythe	2.7524	2	8	0.1231
Levene	**2.7477**	**2**	**8**	**0.1235**
Bartlett	2.4105	2	.	0.0898

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
10.8354	2	4.2291	0.0217*

Means Comparisons
Connecting Letters Report

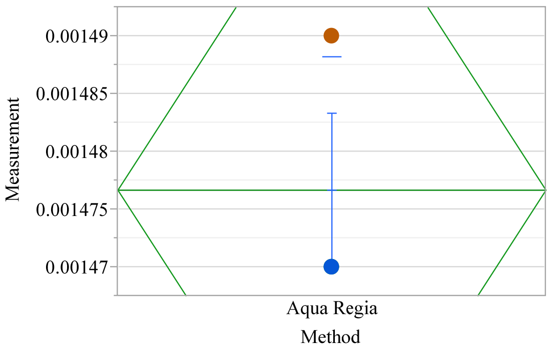
Level			Mean
Peroxide Fusion	A		0.06370000
Cold Chem		B	0.05992500
Aqua Regia		B	0.05926667

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Be



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	1.155e-5
Mean of Response	0.001477
Observations (or Sum Wgts)	3

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0	.	.	.
Error	2	2.6667e-10	1.333e-10		
C. Total	2	2.6667e-10			

Means for Oneway Anova

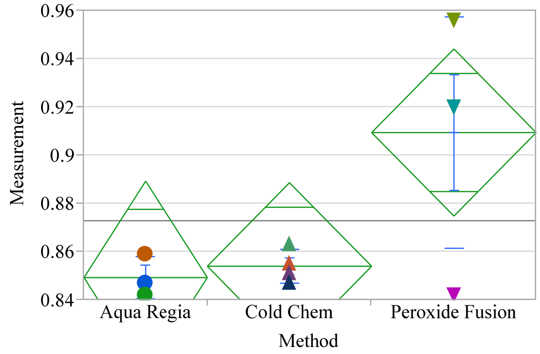
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.001477	6.6667e-6	0.00145	0.00151

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.001477	0.000012	6.6667e-6	0.00145	0.00151

Oneway Analysis of Measurement By Method Element=Ca



Oneway Anova
Summary of Fit

Rsquare	0.538941
Adj Rsquare	0.423676
Root Mean Square Error	0.030066
Mean of Response	0.872909
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00845324	0.004227	4.6757	**0.0452***
Error	8	0.00723167	0.000904		
C. Total	10	0.01568491			

Means for Oneway Anova

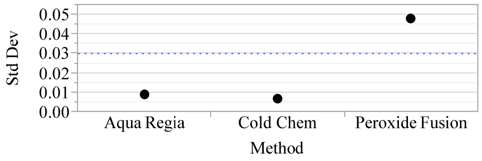
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.849333	0.01736	0.80930	0.88936
Cold Chem	4	0.854000	0.01503	0.81933	0.88867
Peroxide Fusion	4	0.909500	0.01503	0.87483	0.94417

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.849333	0.008737	0.00504	0.82763	0.87104
Cold Chem	4	0.854000	0.006831	0.00342	0.84313	0.86487
Peroxide Fusion	4	0.909500	0.048094	0.02405	0.83297	0.98603

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0087369	0.0064444	0.0056667
Cold Chem	4	0.0068313	0.0050000	0.0050000
Peroxide Fusion	4	0.0480937	0.0337500	0.0285000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3860	2	8	0.3042
Brown-Forsythe	1.2925	2	8	0.3263
Levene	**3.2935**	**2**	**8**	**0.0905**
Bartlett	4.6932	2	.	0.0092

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
2.6635	2	4.4651	0.1732

Means Comparisons
Connecting Letters Report

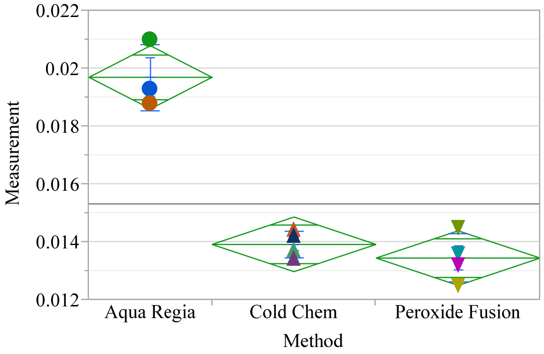
Level		Mean
Peroxide Fusion	A	0.90950000
Cold Chem	A	0.85400000
Aqua Regia	A	0.84933333

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Cd



Oneway Anova
Summary of Fit

Rsquare	0.936513
Adj Rsquare	0.920641
Root Mean Square Error	0.00082
Mean of Response	0.015327
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00007932	0.000040	59.0046	**<.0001***
Error	8	0.00000538	6.722e-7		
C. Total	10	0.00008470			

Means for Oneway Anova

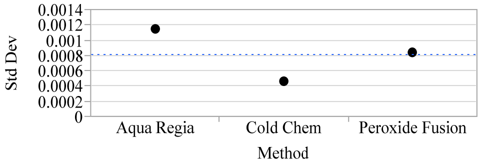
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.019700	0.00047	0.01861	0.02079
Cold Chem	4	0.013925	0.00041	0.01298	0.01487
Peroxide Fusion	4	0.013450	0.00041	0.01250	0.01440

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.019700	0.001153	0.00067	0.01684	0.02256
Cold Chem	4	0.013925	0.000457	0.00023	0.01320	0.01465
Peroxide Fusion	4	0.013450	0.000835	0.00042	0.01212	0.01478

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0011533	0.0008667	0.0007333
Cold Chem	4	0.0004573	0.0003750	0.0003750
Peroxide Fusion	4	0.0008347	0.0006000	0.0006000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.9914	2	8	0.4124
Brown-Forsythe	0.4136	2	8	0.6746
Levene	**1.4797**	**2**	**8**	**0.2839**
Bartlett	0.8649	2	.	0.4211

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

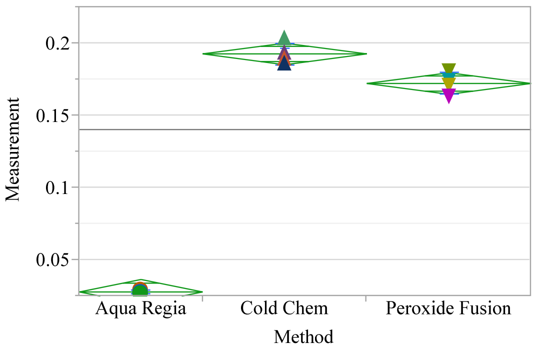
F Ratio	DFNum	DFDen	Prob > F
31.1877	2	4.0625	0.0034*

Means Comparisons
Connecting Letters Report

Level			Mean
Aqua Regia	A		0.01970000
Cold Chem		B	0.01392500
Peroxide Fusion		B	0.01345000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Ce



Oneway Anova
Summary of Fit

Rsquare	0.993709
Adj Rsquare	0.992137
Root Mean Square Error	0.006474
Mean of Response	0.140345
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.05296600	0.026483	631.8514	**<.0001***
Error	8	0.00033531	0.000042		
C. Total	10	0.05330131			

Means for Oneway Anova

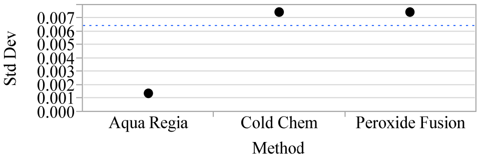
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.027933	0.00374	0.01931	0.03655
Cold Chem	4	0.192750	0.00324	0.18529	0.20021
Peroxide Fusion	4	0.172250	0.00324	0.16479	0.17971

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.027933	0.001380	0.00080	0.02451	0.03136
Cold Chem	4	0.192750	0.007411	0.00371	0.18096	0.20454
Peroxide Fusion	4	0.172250	0.007455	0.00373	0.16039	0.18411

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0013796	0.0010444	0.0008667
Cold Chem	4	0.0074106	0.0052500	0.0052500
Peroxide Fusion	4	0.0074554	0.0052500	0.0052500

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.7001	2	8	0.5246
Brown-Forsythe	1.3318	2	8	0.3168
Levene	**1.3839**	**2**	**8**	**0.3047**
Bartlett	1.9285	2	.	0.1454

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
1380.6751	2	4.334	<.0001*

Means Comparisons
Connecting Letters Report

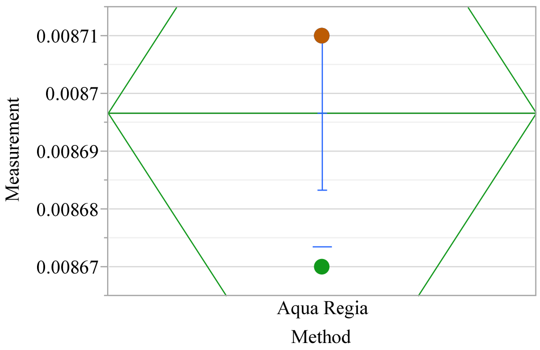
Level				Mean
Cold Chem	A			0.19275000
Peroxide Fusion		B		0.17225000
Aqua Regia			C	0.02793333

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Co



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.000023
Mean of Response	0.008697
Observations (or Sum Wgts)	3

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0	.	.	.
Error	2	1.06667e-9	5.333e-10		
C. Total	2	1.06667e-9			

Means for Oneway Anova

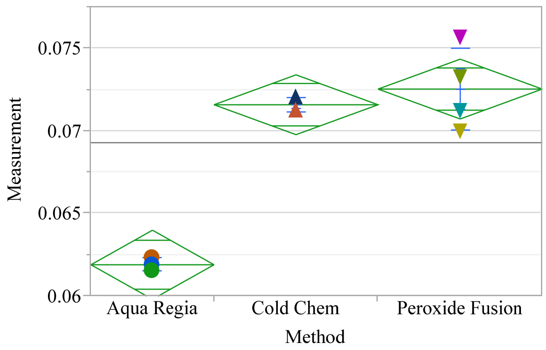
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.008697	1.33e-5	0.00864	0.00875

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.008697	0.000023	1.33e-5	0.00864	0.00875

Oneway Analysis of Measurement By Method Element=Cr



Oneway Anova
Summary of Fit

Rsquare	0.920108
Adj Rsquare	0.900135
Root Mean Square Error	0.001572
Mean of Response	0.0693
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00022769	0.000114	46.0678	<.0001*
Error	8	0.00001977	2.471e-6		
C. Total	10	0.00024746			

Means for Oneway Anova

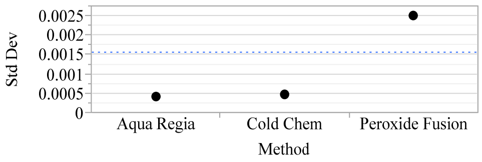
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.061900	0.00091	0.05981	0.06399
Cold Chem	4	0.071600	0.00079	0.06979	0.07341
Peroxide Fusion	4	0.072550	0.00079	0.07074	0.07436

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.061900	0.000400	0.00023	0.06091	0.06289
Cold Chem	4	0.071600	0.000462	0.00023	0.07087	0.07233
Peroxide Fusion	4	0.072550	0.002504	0.00125	0.06857	0.07653

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0004000	0.0002667	0.0002667
Cold Chem	4	0.0004619	0.0004000	0.0004000
Peroxide Fusion	4	0.0025040	0.0019500	0.0019500

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.4214	2	8	0.1506
Brown-Forsythe	6.4969	2	8	0.0211
Levene	**7.1279**	**2**	**8**	**0.0167**
Bartlett	4.2733	2	.	0.0139

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
398.0908	2	4.96	**<.0001***

Means Comparisons
Connecting Letters Report

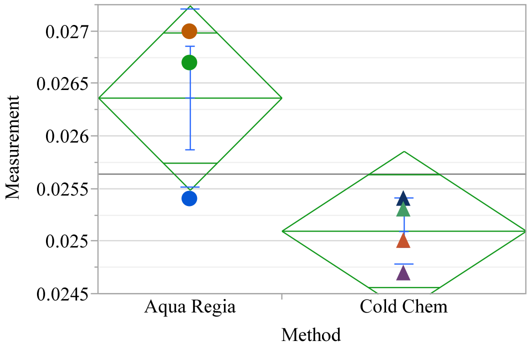
Level			Mean
Peroxide Fusion	A		0.07255000
Cold Chem	A		0.07160000
Aqua Regia		B	0.06190000

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Cu



Oneway Anova
Summary of Fit

Rsquare	0.611605
Adj Rsquare	0.533926
Root Mean Square Error	0.000591
Mean of Response	0.025643
Observations (or Sum Wgts)	7

t Test
Cold Chem-Aqua Regia
Assuming equal variances

Difference	-0.00127	t Ratio	-2.80598
Std Err Dif	0.00045	DF	5
Upper CL Dif	-0.00011	Prob > |t|	0.0377*
Lower CL Dif	-0.00243	Prob > t	0.9811
Confidence	0.95	Prob < t	0.0189*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	2.75048e-6	2.7505e-6	7.8735	**0.0377***
Error	5	1.74667e-6	3.4933e-7		
C. Total	6	4.49714e-6			

Means for Oneway Anova

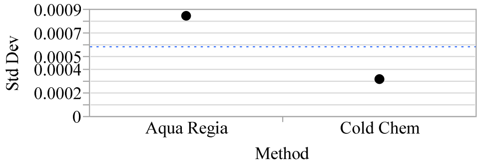
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.026367	0.00034	0.02549	0.02724
Cold Chem	4	0.025100	0.00030	0.02434	0.02586

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.026367	0.000850	0.00049	0.02425	0.02848
Cold Chem	4	0.025100	0.000316	0.00016	0.02460	0.02560

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0008505	0.0006444	0.0005333
Cold Chem	4	0.0003162	0.0002500	0.0002500

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.8485	1	5	0.2321
Brown-Forsythe	0.6974	1	5	0.4417
Levene	**5.3186**	**1**	**5**	**0.0692**
Bartlett	1.8965	1	.	0.1685
F Test 2-sided	7.2333	2	3	0.1424

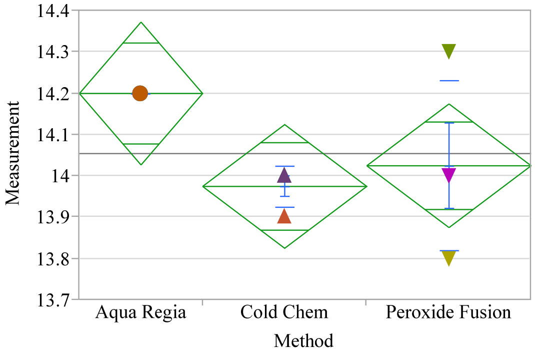
Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
6.0292	1	2.4189	0.1117

Oneway Analysis of Measurement By Method Element=Fe



Oneway Anova
Summary of Fit

Rsquare	0.406
Adj Rsquare	0.2575
Root Mean Square Error	0.129904
Mean of Response	14.05455
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.09227273	0.046136	2.7340	**0.1245**
Error	8	0.13500000	0.016875		
C. Total	10	0.22727273			

Means for Oneway Anova

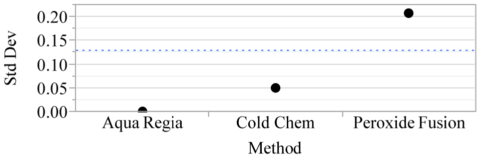
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	14.2000	0.07500	14.027	14.373
Cold Chem	4	13.9750	0.06495	13.825	14.125
Peroxide Fusion	4	14.0250	0.06495	13.875	14.175

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	14.2000	2.18e-15	1.3e-15	14.200	14.200
Cold Chem	4	13.9750	0.050000	0.02500	13.895	14.055
Peroxide Fusion	4	14.0250	0.206155	0.10308	13.697	14.353

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	2.176e-15	1.776e-15	0.0000000
Cold Chem	4	0.0500000	0.0375000	0.0250000
Peroxide Fusion	4	0.2061553	0.1375000	0.1250000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4628	2	8	0.2875
Brown-Forsythe	1.7212	2	8	0.2389
Levene	**2.7315**	**2**	**8**	**0.1247**
Bartlett	55.3163	2	.	0.0000

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
35.9496	2	4	0.0028*

Means Comparisons
Connecting Letters Report

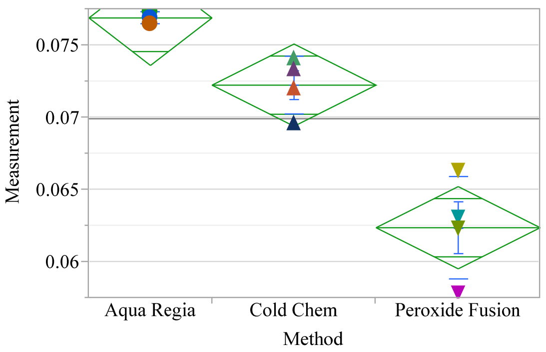
Level		Mean
Aqua Regia	A	14.200000
Peroxide Fusion	A	14.025000
Cold Chem	A	13.975000

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Gd



Oneway Anova
Summary of Fit

Rsquare	0.89019
Adj Rsquare	0.862738
Root Mean Square Error	0.00247
Mean of Response	0.069927
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00039558	0.000198	32.4266	**0.0001***
Error	8	0.00004880	6.1e-6		
C. Total	10	0.00044438			

Means for Oneway Anova

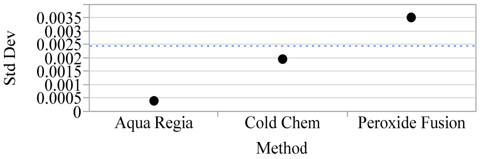
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.076900	0.00143	0.07361	0.08019
Cold Chem	4	0.072250	0.00123	0.06940	0.07510
Peroxide Fusion	4	0.062375	0.00123	0.05953	0.06522

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.076900	0.000400	0.00023	0.07591	0.07789
Cold Chem	4	0.072250	0.001967	0.00098	0.06912	0.07538
Peroxide Fusion	4	0.062375	0.003506	0.00175	0.05680	0.06795

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0004000	0.0002667	0.0002667
Cold Chem	4	0.0019672	0.0014500	0.0014500
Peroxide Fusion	4	0.0035056	0.0023250	0.0023250

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1007	2	8	0.3782
Brown-Forsythe	1.4812	2	8	0.2836
Levene	**1.5660**	**2**	**8**	**0.2667**
Bartlett	2.7885	2	.	0.0615

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

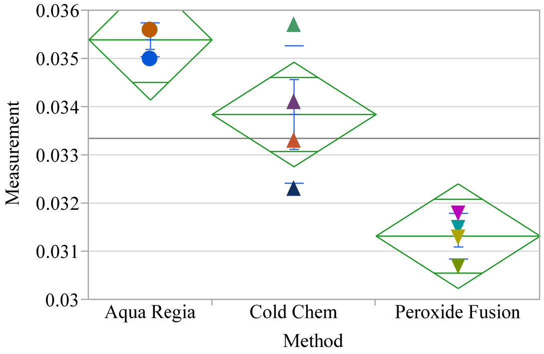
F Ratio	DFNum	DFDen	Prob > F
37.4061	2	4.2676	0.0020*

Means Comparisons
Connecting Letters Report

Level			Mean
Aqua Regia	A		0.07690000
Cold Chem	A		0.07225000
Peroxide Fusion		B	0.06237500

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=La



Oneway Anova
Summary of Fit

Rsquare	0.809166
Adj Rsquare	0.761458
Root Mean Square Error	0.000941
Mean of Response	0.033355
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00003001	0.000015	16.9607	**0.0013***
Error	8	0.00000708	8.847e-7		
C. Total	10	0.00003709			

Means for Oneway Anova

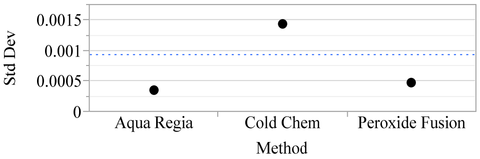
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.035400	0.00054	0.03415	0.03665
Cold Chem	4	0.033850	0.00047	0.03277	0.03493
Peroxide Fusion	4	0.031325	0.00047	0.03024	0.03241

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.035400	0.000346	0.00020	0.03454	0.03626
Cold Chem	4	0.033850	0.001436	0.00072	0.03156	0.03614
Peroxide Fusion	4	0.031325	0.000465	0.00023	0.03059	0.03206

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0003464	0.0002667	0.0002000
Cold Chem	4	0.0014364	0.0010500	0.0010500
Peroxide Fusion	4	0.0004646	0.0003250	0.0003250

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6130	2	8	0.2579
Brown-Forsythe	2.6635	2	8	0.1299
Levene	**2.8305**	**2**	**8**	**0.1176**
Bartlett	2.4229	2	.	0.0887

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
78.1623	2	5.0905	0.0002*

Means Comparisons
Connecting Letters Report

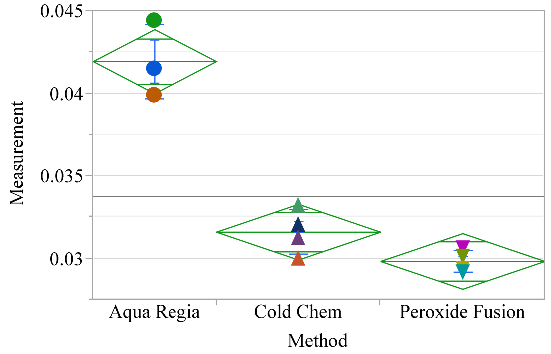
Level			Mean
Aqua Regia	A		0.03540000
Cold Chem	A		0.03385000
Peroxide Fusion		B	0.03132500

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Li



Oneway Anova
Summary of Fit

Rsquare	0.942657
Adj Rsquare	0.928321
Root Mean Square Error	0.001462
Mean of Response	0.033773
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00028101	0.000141	65.7552	**<.0001***
Error	8	0.00001709	2.137e-6		
C. Total	10	0.00029810			

Means for Oneway Anova

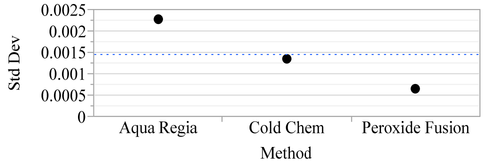
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.041933	0.00084	0.03999	0.04388
Cold Chem	4	0.031600	0.00073	0.02991	0.03329
Peroxide Fusion	4	0.029825	0.00073	0.02814	0.03151

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.041933	0.002281	0.00132	0.03627	0.04760
Cold Chem	4	0.031600	0.001347	0.00067	0.02946	0.03374
Peroxide Fusion	4	0.029825	0.000645	0.00032	0.02880	0.03085

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0022811	0.0016444	0.0015000
Cold Chem	4	0.0013466	0.0010000	0.0010000
Peroxide Fusion	4	0.0006449	0.0005250	0.0005250

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4980	2	8	0.2802
Brown-Forsythe	1.1243	2	8	0.3713
Levene	**2.2153**	**2**	**8**	**0.1716**
Bartlett	1.5434	2	.	0.2137

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

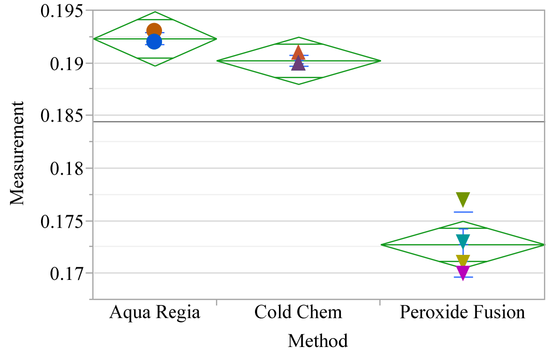
F Ratio	DFNum	DFDen	Prob > F
34.8781	2	3.8277	0.0035*

Means Comparisons
Connecting Letters Report

Level			Mean
Aqua Regia	A		0.04193333
Cold Chem		B	0.03160000
Peroxide Fusion		B	0.02982500

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Mg



Oneway Anova
Summary of Fit

Rsquare	0.966434
Adj Rsquare	0.958043
Root Mean Square Error	0.001942
Mean of Response	0.184455
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00086856	0.000434	115.1683	**<.0001***
Error	8	0.00003017	3.771e-6		
C. Total	10	0.00089873			

Means for Oneway Anova

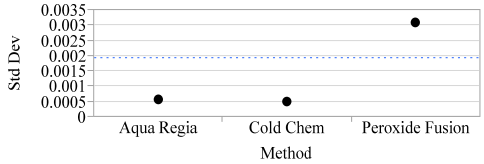
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.192333	0.00112	0.18975	0.19492
Cold Chem	4	0.190250	0.00097	0.18801	0.19249
Peroxide Fusion	4	0.172750	0.00097	0.17051	0.17499

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.192333	0.000577	0.00033	0.19090	0.19377
Cold Chem	4	0.190250	0.000500	0.00025	0.18945	0.19105
Peroxide Fusion	4	0.172750	0.003096	0.00155	0.16782	0.17768

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0005774	0.0004444	0.0003333
Cold Chem	4	0.0005000	0.0003750	0.0002500
Peroxide Fusion	4	0.0030957	0.0022500	0.0022500

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6990	2	8	0.2427
Brown-Forsythe	3.2329	2	8	0.0935
Levene	**3.9606**	**2**	**8**	**0.0637**
Bartlett	4.3432	2	.	0.0130

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
70.6753	2	4.6229	0.0003*

Means Comparisons
Connecting Letters Report

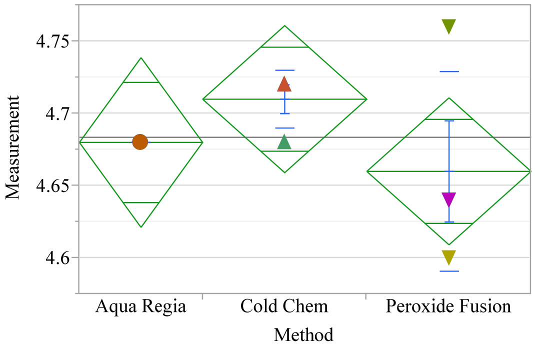
Level			Mean
Aqua Regia	A		0.19233333
Cold Chem	A		0.19025000
Peroxide Fusion		B	0.17275000

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Mn



Oneway Anova
Summary of Fit

Rsquare	0.244718
Adj Rsquare	0.055898
Root Mean Square Error	0.044159
Mean of Response	4.683636
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00505455	0.002527	1.2960	**0.3254**
Error	8	0.01560000	0.001950		
C. Total	10	0.02065455			

Means for Oneway Anova

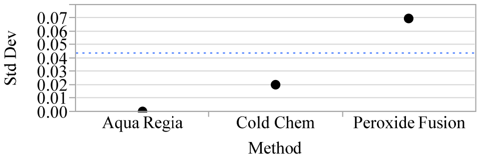
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	4.68000	0.02550	4.6212	4.7388
Cold Chem	4	4.71000	0.02208	4.6591	4.7609
Peroxide Fusion	4	4.66000	0.02208	4.6091	4.7109

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	4.68000	0.000000	0.00000	4.6800	4.6800
Cold Chem	4	4.71000	0.020000	0.01000	4.6782	4.7418
Peroxide Fusion	4	4.66000	0.069282	0.03464	4.5498	4.7702

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0000000	0.0000000	0.0000000
Cold Chem	4	0.0200000	0.0150000	0.0100000
Peroxide Fusion	4	0.0692820	0.0500000	0.0400000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2435	2	8	0.3387
Brown-Forsythe	1.1717	2	8	0.3578
Levene	**4.0464**	**2**	**8**	**0.0611**
Bartlett	.	2	.	.

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

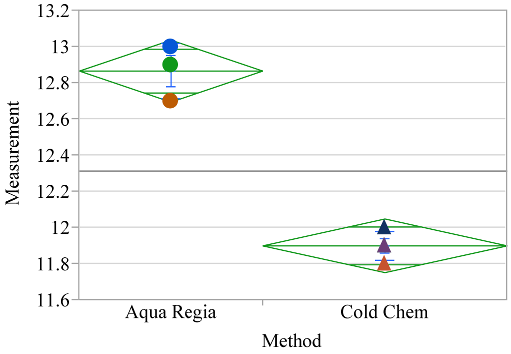
F Ratio	DFNum	DFDen	Prob > F
.	2	.	.

Means Comparisons
Connecting Letters Report

Level		Mean
Cold Chem	A	4.7100000
Aqua Regia	A	4.6800000
Peroxide Fusion	A	4.6600000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Na



Oneway Anova
Summary of Fit

Rsquare	0.960046
Adj Rsquare	0.952055
Root Mean Square Error	0.11547
Mean of Response	12.31429
Observations (or Sum Wgts)	7

t Test

Cold Chem-Aqua Regia

Assuming equal variances

Difference	-0.9667	t Ratio	-10.961
Std Err Dif	0.0882	DF	5
Upper CL Dif	-0.7400	Prob > |t|	0.0001*
Lower CL Dif	-1.1934	Prob > t	0.9999
Confidence	0.95	Prob < t	<.0001*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	1.6019048	1.60190	120.1429	**0.0001***
Error	5	0.0666667	0.01333		
C. Total	6	1.6685714			

Means for Oneway Anova

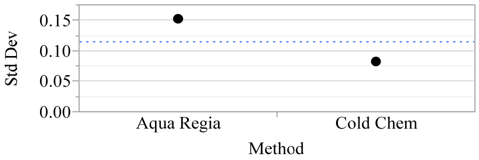
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	12.8667	0.06667	12.695	13.038
Cold Chem	4	11.9000	0.05774	11.752	12.048

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	12.8667	0.152753	0.08819	12.487	13.246
Cold Chem	4	11.9000	0.081650	0.04082	11.770	12.030

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.1527525	0.1111111	0.1000000
Cold Chem	4	0.0816497	0.0500000	0.0500000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.1256	1	5	0.3373
Brown-Forsythe	0.7143	1	5	0.4366
Levene	**1.6307**	**1**	**5**	**0.2577**
Bartlett	0.7928	1	.	0.3732
F Test 2-sided	3.5000	2	3	0.3286

Warning: Small sample sizes. Use Caution.

Welch's Test

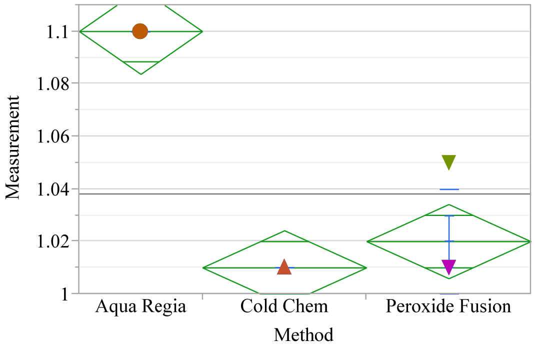
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
98.9412	1	2.8614	0.0026*

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Ni



Oneway Anova
Summary of Fit

Rsquare	0.930085
Adj Rsquare	0.912606
Root Mean Square Error	0.012247
Mean of Response	1.038182
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.01596364	0.007982	53.2121	**<.0001***
Error	8	0.00120000	0.000150		
C. Total	10	0.01716364			

Means for Oneway Anova

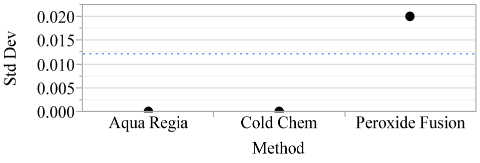
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	1.10000	0.00707	1.0837	1.1163
Cold Chem	4	1.01000	0.00612	0.9959	1.0241
Peroxide Fusion	4	1.02000	0.00612	1.0059	1.0341

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	1.10000	0.000000	0.00000	1.1000	1.1000
Cold Chem	4	1.01000	0.000000	0.00000	1.0100	1.0100
Peroxide Fusion	4	1.02000	0.020000	0.01000	0.9882	1.0518

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0000000	0.0000000	0.0000000
Cold Chem	4	0.0000000	0.0000000	0.0000000
Peroxide Fusion	4	0.0200000	0.0150000	0.0100000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.2218	2	8	0.3443
Brown-Forsythe	0.8485	2	8	0.4633
Levene	**7.6364**	**2**	**8**	**0.0140**
Bartlett	.	2	.	.

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
.	2	.	.

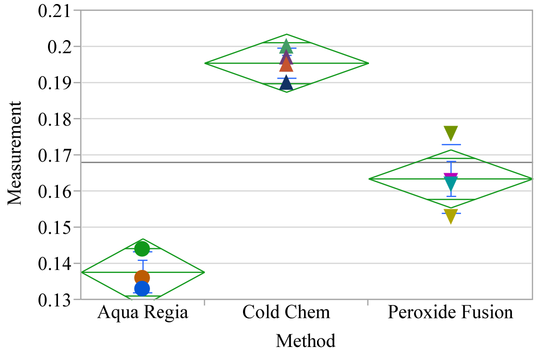
Means Comparisons
Connecting Letters Report

Level			Mean
Aqua Regia	A		1.1000000
Peroxide Fusion		B	1.0200000
Cold Chem		B	1.0100000

Levels not connected by same letter are significantly different.

Note: due to the small sample sizes and limited variation in these data, use the ANOVA results above to decide that the means are different.

Oneway Analysis of Measurement By Method Element=P



Oneway Anova
Summary of Fit

Rsquare	0.938162
Adj Rsquare	0.922703
Root Mean Square Error	0.006952
Mean of Response	0.168091
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00586624	0.002933	60.6853	**<.0001***
Error	8	0.00038667	0.000048		
C. Total	10	0.00625291			

Means for Oneway Anova

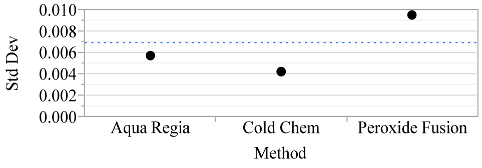
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.137667	0.00401	0.12841	0.14692
Cold Chem	4	0.195500	0.00348	0.18748	0.20352
Peroxide Fusion	4	0.163500	0.00348	0.15548	0.17152

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.137667	0.005686	0.00328	0.12354	0.15179
Cold Chem	4	0.195500	0.004203	0.00210	0.18881	0.20219
Peroxide Fusion	4	0.163500	0.009469	0.00473	0.14843	0.17857

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0056862	0.0042222	0.0036667
Cold Chem	4	0.0042032	0.0030000	0.0030000
Peroxide Fusion	4	0.0094692	0.0062500	0.0060000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8027	2	8	0.4812
Brown-Forsythe	0.4345	2	8	0.6620
Levene	**0.6096**	**2**	**8**	**0.5670**
Bartlett	0.8391	2	.	0.4321

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
100.0327	2	4.5848	0.0002*

Means Comparisons
Connecting Letters Report

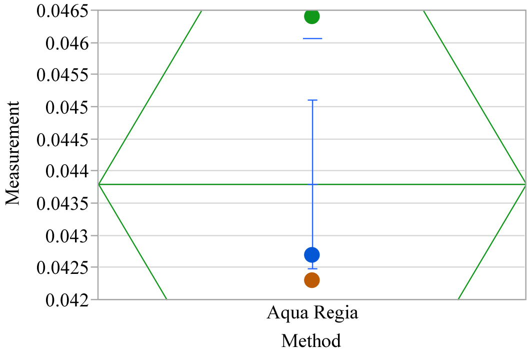
Level				Mean
Cold Chem	A			0.19550000
Peroxide Fusion		B		0.16350000
Aqua Regia			C	0.13766667

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Pb



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.002261
Mean of Response	0.0438
Observations (or Sum Wgts)	3

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.00000000	.	.	.
Error	2	0.00001022	5.11e-6		
C. Total	2	0.00001022			

Means for Oneway Anova

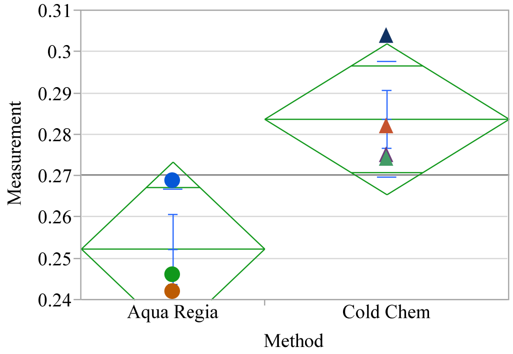
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.043800	0.00131	0.03818	0.04942

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.043800	0.002261	0.00131	0.03818	0.04942

Oneway Analysis of Measurement By Method Element=S (axial)



Oneway Anova
Summary of Fit

Rsquare	0.62634
Adj Rsquare	0.551608
Root Mean Square Error	0.014209
Mean of Response	0.270286
Observations (or Sum Wgts)	7

t Test
Cold Chem-Aqua Regia
Assuming equal variances

Difference	0.031417	t Ratio	2.895019
Std Err Dif	0.010852	DF	5
Upper CL Dif	0.059313	Prob > |t|	0.0340*
Lower CL Dif	0.003521	Prob > t	0.0170*
Confidence	0.95	Prob < t	0.9830

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	0.00169201	0.001692	8.3811	**0.0340***
Error	5	0.00100942	0.000202		
C. Total	6	0.00270143			

Means for Oneway Anova

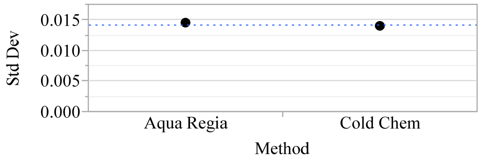
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.252333	0.00820	0.23125	0.27342
Cold Chem	4	0.283750	0.00710	0.26549	0.30201

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.252333	0.014572	0.00841	0.21614	0.28853
Cold Chem	4	0.283750	0.013961	0.00698	0.26153	0.30597

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0145717	0.0111111	0.0090000
Cold Chem	4	0.0139613	0.0101250	0.0092500

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.0061	1	5	0.9406
Brown-Forsythe	0.0008	1	5	0.9783
Levene	**0.0364**	**1**	**5**	**0.8562**
Bartlett	0.0036	1	.	0.9518
F Test 2-sided	1.0894	2	3	0.8818

Warning: Small sample sizes. Use Caution.

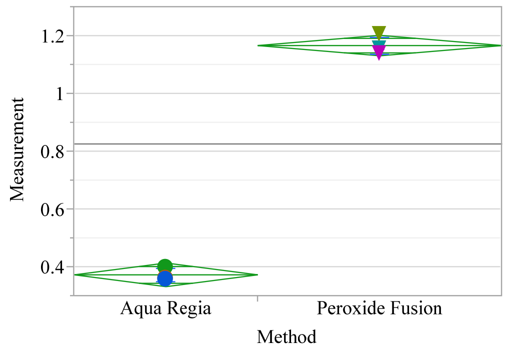
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
8.2590	1	4.3328	0.0412*

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Si



Oneway Anova
Summary of Fit

Rsquare	0.996482
Adj Rsquare	0.995779
Root Mean Square Error	0.027618
Mean of Response	0.827286
Observations (or Sum Wgts)	7

t Test
Peroxide Fusion-Aqua Regia
Assuming equal variances

Difference	0.793833	t Ratio	37.63436
Std Err Dif	0.021093	DF	5
Upper CL Dif	0.848055	Prob > |t|	<.0001*
Lower CL Dif	0.739611	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**	**Prob > F**
Method	1	1.0802938	1.08029	1416.345	**<.0001***
Error	5	0.0038137	0.00076		
C. Total	6	1.0841074			

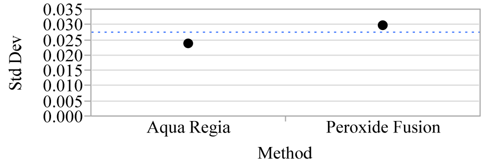
Means for Oneway Anova

Level	**Number**	**Mean**	**Std Error**	**Lower 95%**	**Upper 95%**
Aqua Regia	3	0.37367	0.01595	0.3327	0.4147
Peroxide Fusion	4	1.16750	0.01381	1.1320	1.2030

Std Error uses a pooled estimate of error variance
Means and Std Deviations

Level	**Number**	**Mean**	**Std Dev**	**Std Err Mean**	**Lower 95%**	**Upper 95%**
Aqua Regia	3	0.37367	0.023861	0.01378	0.3144	0.4329
Peroxide Fusion	4	1.16750	0.029861	0.01493	1.1200	1.2150

Tests that the Variances are Equal



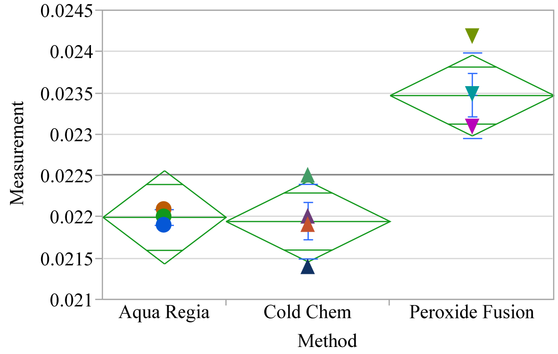
Level	**Count**	**Std Dev**	**MeanAbsDif to Mean**	**MeanAbsDif to Median**
Aqua Regia	3	0.0238607	0.0182222	0.0146667
Peroxide Fusion	4	0.0298608	0.0212500	0.0175000

Test	**F Ratio**	**DFNum**	**DFDen**	**p-Value**
O'Brien[.5]	0.1311	1	5	0.7321
Brown-Forsythe	0.0274	1	5	0.8750
Levene	**0.0777**	**1**	**5**	**0.7916**
Bartlett	0.0961	1	.	0.7566
F Test 2-sided	1.5662	3	2	0.8251

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	**DFNum**	**DFDen**	**Prob > F**
1526.9684	1	4.9265	<.0001*

Oneway Analysis of Measurement By Method Element=Sr



Oneway Anova
Summary of Fit

Rsquare	0.800246
Adj Rsquare	0.750308
Root Mean Square Error	0.000424
Mean of Response	0.022518
Observations (or Sum Wgts)	11

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**	**Prob > F**
Method	2	5.75886e-6	2.8794e-6	16.0247	**0.0016***
Error	8	1.4375e-6	1.7969e-7		
C. Total	10	7.19636e-6			

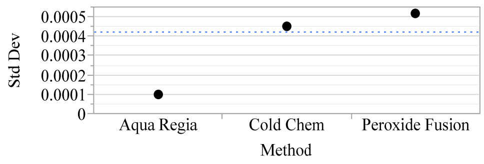
Means for Oneway Anova

Level	**Number**	**Mean**	**Std Error**	**Lower 95%**	**Upper 95%**
Aqua Regia	3	0.022000	0.00024	0.02144	0.02256
Cold Chem	4	0.021950	0.00021	0.02146	0.02244
Peroxide Fusion	4	0.023475	0.00021	0.02299	0.02396

Std Error uses a pooled estimate of error variance
Means and Std Deviations

Level	**Number**	**Mean**	**Std Dev**	**Std Err Mean**	**Lower 95%**	**Upper 95%**
Aqua Regia	3	0.022000	0.000100	5.77e-5	0.02175	0.02225
Cold Chem	4	0.021950	0.000451	0.00023	0.02123	0.02267
Peroxide Fusion	4	0.023475	0.000519	0.00026	0.02265	0.02430

Tests that the Variances are Equal



Level	**Count**	**Std Dev**	**MeanAbsDif to Mean**	**MeanAbsDif to Median**
Aqua Regia	3	0.0001000	0.0000667	0.0000667
Cold Chem	4	0.0004509	0.0003000	0.0003000
Peroxide Fusion	4	0.0005188	0.0003750	0.0003750

Test	**F Ratio**	**DFNum**	**DFDen**	**Prob > F**
O'Brien[.5]	0.7134	2	8	0.5187
Brown-Forsythe	1.0977	2	8	0.3791
Levene	**1.3658**	**2**	**8**	**0.3088**
Bartlett	1.7868	2	.	0.1675

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	**DFNum**	**DFDen**	**Prob > F**
13.4942	2	4.4078	0.0132*

Means Comparisons
Connecting Letters Report

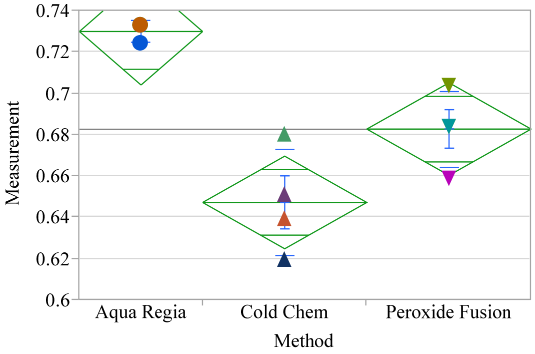
Level			**Mean**
Peroxide Fusion	A		0.02347500
Aqua Regia		B	0.02200000
Cold Chem		B	0.02195000

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Th



Oneway Anova
Summary of Fit

Rsquare	0.795078
Adj Rsquare	0.743848
Root Mean Square Error	0.019447
Mean of Response	0.682727
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.01173868	0.005869	15.5197	**0.0018***
Error	8	0.00302550	0.000378		
C. Total	10	0.01476418			

Means for Oneway Anova

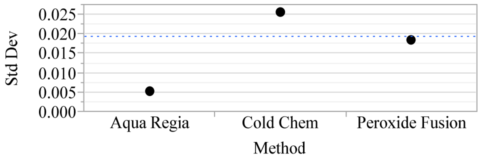
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.730000	0.01123	0.70411	0.75589
Cold Chem	4	0.647250	0.00972	0.62483	0.66967
Peroxide Fusion	4	0.682750	0.00972	0.66033	0.70517

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.730000	0.005196	0.00300	0.71709	0.74291
Cold Chem	4	0.647250	0.025513	0.01276	0.60665	0.68785
Peroxide Fusion	4	0.682750	0.018428	0.00921	0.65343	0.71207

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0051962	0.0040000	0.0030000
Cold Chem	4	0.0255131	0.0182500	0.0182500
Peroxide Fusion	4	0.0184278	0.0118750	0.0112500

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8782	2	8	0.4521
Brown-Forsythe	1.3160	2	8	0.3206
Levene	**1.2885**	**2**	**8**	**0.3273**
Bartlett	1.6927	2	.	0.1840

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
25.9789	2	4.541	0.0033*

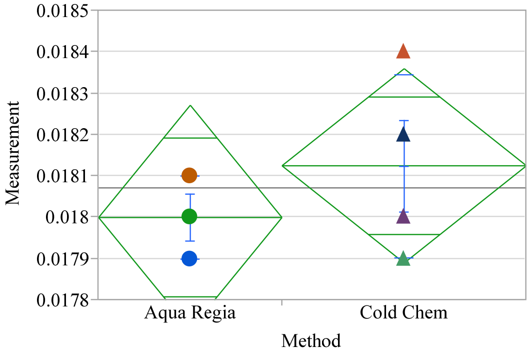
Means Comparisons

Connecting Letters Report

Level			Mean
Aqua Regia	A		0.73000000
Peroxide Fusion		B	0.68275000
Cold Chem		B	0.64725000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Ti



Oneway Anova
Summary of Fit

Rsquare	0.137868
Adj Rsquare	-0.03456
Root Mean Square Error	0.000183
Mean of Response	0.018071
Observations (or Sum Wgts)	7

t Test

Cold Chem-Aqua Regia

Assuming equal variances

Difference	0.00012	t Ratio	0.894189
Std Err Dif	0.00014	DF	5
Upper CL Dif	0.00048	Prob > |t|	0.4122
Lower CL Dif	-0.00023	Prob > t	0.2061
Confidence	0.95	Prob < t	0.7939

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	2.67857e-8	2.6786e-8	0.7996	**0.4122**
Error	5	1.675e-7	3.35e-8		
C. Total	6	1.94286e-7			

Means for Oneway Anova

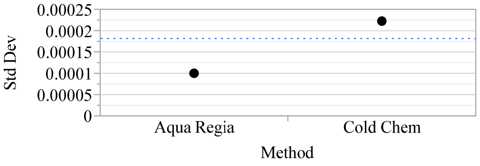
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.018000	0.00011	0.01773	0.01827
Cold Chem	4	0.018125	9.15e-5	0.01789	0.01836

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.018000	0.000100	5.77e-5	0.01775	0.01825
Cold Chem	4	0.018125	0.000222	0.00011	0.01777	0.01848

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0001000	0.0000667	0.0000667
Cold Chem	4	0.0002217	0.0001750	0.0001750

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.4609	1	5	0.2808
Brown-Forsythe	2.9443	1	5	0.1468
Levene	**3.1767**	**1**	**5**	**0.1348**
Bartlett	1.0461	1	.	0.3064
F Test 2-sided	4.9167	3	2	0.3473

Warning: Small sample sizes. Use Caution.

Welch's Test

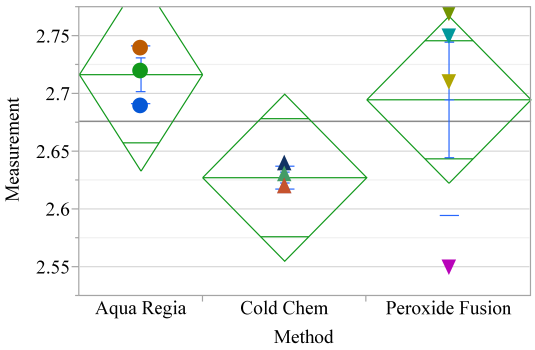
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
1.0000	1	4.3661	0.3695

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=U



Oneway Anova
Summary of Fit

Rsquare	0.334632
Adj Rsquare	0.16829
Root Mean Square Error	0.062691
Mean of Response	2.676364
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.01581288	0.007906	2.0117	**0.1960**
Error	8	0.03144167	0.003930		
C. Total	10	0.04725455			

Means for Oneway Anova

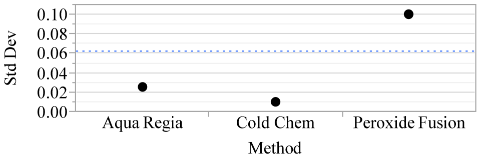
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	2.71667	0.03619	2.6332	2.8001
Cold Chem	4	2.62750	0.03135	2.5552	2.6998
Peroxide Fusion	4	2.69500	0.03135	2.6227	2.7673

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	2.71667	0.025166	0.01453	2.6542	2.7792
Cold Chem	4	2.62750	0.009574	0.00479	2.6123	2.6427
Peroxide Fusion	4	2.69500	0.099833	0.04992	2.5361	2.8539

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0251661	0.0177778	0.0166667
Cold Chem	4	0.0095743	0.0075000	0.0075000
Peroxide Fusion	4	0.0998332	0.0725000	0.0650000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3142	2	8	0.3210
Brown-Forsythe	1.6157	2	8	0.2574
Levene	**4.1229**	**2**	**8**	**0.0588**
Bartlett	5.1696	2	.	0.0057

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

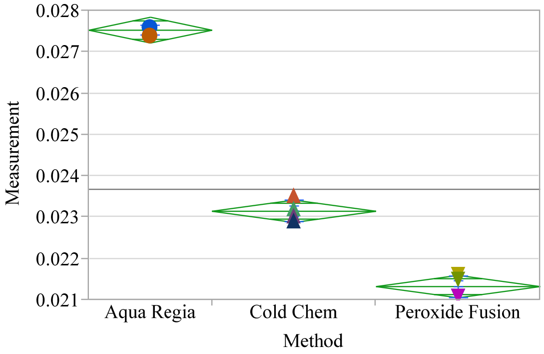
F Ratio	DFNum	DFDen	Prob > F
14.9174	2	3.6077	0.0180*

Means Comparisons
Connecting Letters Report

Level		Mean
Aqua Regia	A	2.7166667
Peroxide Fusion	A	2.6950000
Cold Chem	A	2.6275000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Element=Zn



Oneway Anova
Summary of Fit

Rsquare	0.993496
Adj Rsquare	0.991871
Root Mean Square Error	0.000236
Mean of Response	0.023682
Observations (or Sum Wgts)	11

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	0.00006785	0.000034	611.0517	**<.0001***
Error	8	0.00000044	5.552e-8		
C. Total	10	0.00006830			

Means for Oneway Anova

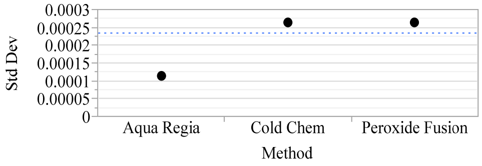
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.027533	0.00014	0.02722	0.02785
Cold Chem	4	0.023150	0.00012	0.02288	0.02342
Peroxide Fusion	4	0.021325	0.00012	0.02105	0.02160

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.027533	0.000115	6.67e-5	0.02725	0.02782
Cold Chem	4	0.023150	0.000265	0.00013	0.02273	0.02357
Peroxide Fusion	4	0.021325	0.000263	0.00013	0.02091	0.02174

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0001155	0.0000889	0.0000667
Cold Chem	4	0.0002646	0.0002000	0.0002000
Peroxide Fusion	4	0.0002630	0.0002250	0.0002250

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0320	2	8	0.3993
Brown-Forsythe	2.0241	2	8	0.1944
Levene	**2.3869**	**2**	**8**	**0.1538**
Bartlett	0.6384	2	.	0.5281

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
989.1367	2	5.1534	<.0001*

Means Comparisons
Connecting Letters Report

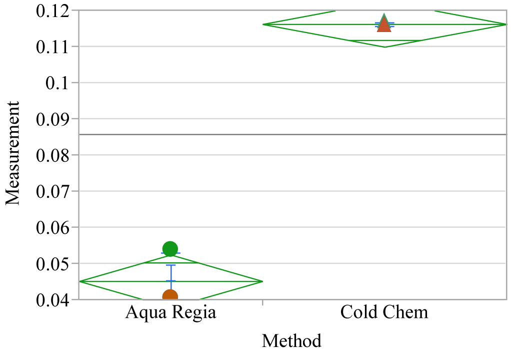
Level				Mean
Aqua Regia	A			0.02753333
Cold Chem		B		0.02315000
Peroxide Fusion			C	0.02132500

Levels not connected by same letter are significantly different.

Exhibit A3. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Methods *(continued)*

(Measurements are in wt% of Total Solids)

Oneway Analysis of Measurement By Method Element=Zr



Oneway Anova
Summary of Fit

Rsquare	0.986284
Adj Rsquare	0.983541
Root Mean Square Error	0.004908
Mean of Response	0.085786
Observations (or Sum Wgts)	7

t Test
Cold Chem-Aqua Regia
Assuming equal variances

Difference	0.071083	t Ratio	18.96177
Std Err Dif	0.003749	DF	5
Upper CL Dif	0.080720	Prob > |t|	<.0001*
Lower CL Dif	0.061447	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	0.00866201	0.008662	359.5489	<.0001*
Error	5	0.00012046	0.000024		
C. Total	6	0.00878247			

Means for Oneway Anova

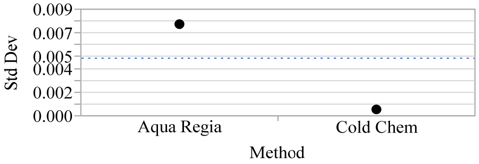
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.045167	0.00283	0.03788	0.05245
Cold Chem	4	0.116250	0.00245	0.10994	0.12256

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Aqua Regia	3	0.045167	0.007736	0.00447	0.02595	0.06439
Cold Chem	4	0.116250	0.000500	0.00025	0.11545	0.11705

Tests that the Variances are Equal



Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0077365	0.0059556	0.0044667
Cold Chem	4	0.0005000	0.0003750	0.0002500

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	2.5184	1	5	0.1734
Brown-Forsythe	1.2652	1	5	0.3117
Levene	**19.7903**	**1**	**5**	**0.0067**
Bartlett	9.8128	1	.	0.0017
F Test 2-sided	239.4133	2	3	0.0010

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
252.4702	1	2.0125	**0.0038***

Exhibit A4. Plot of the Hg Measurements in Wt% of Total Solids for the SRAT Product Samples

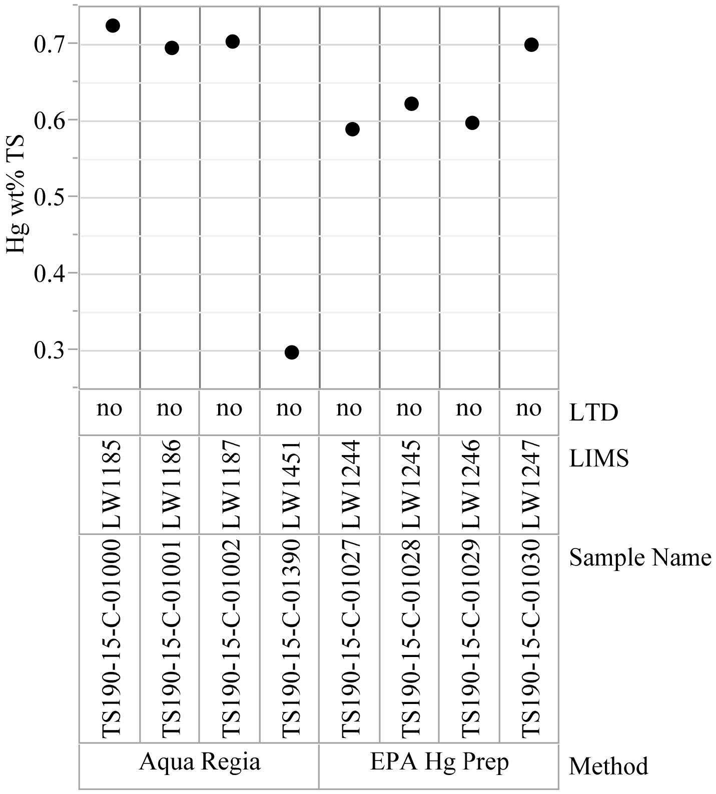
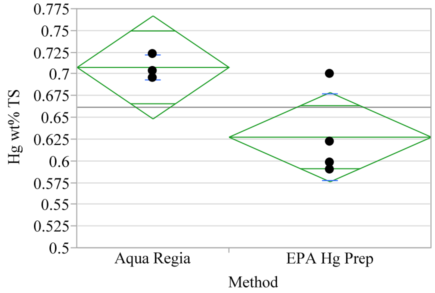


Exhibit A5. Analysis of Variance Investigating the Hg Means and Variances by Preparation Methods

Oneway Analysis of Hg wt% TS By Method



Oneway Anova
Summary of Fit

Rsquare	0.580788
Adj Rsquare	0.496945
Root Mean Square Error	0.039922
Mean of Response	0.662143
Observations (or Sum Wgts)	7

t Test
EPA Hg Prep-Aqua Regia
Assuming equal variances

Difference	-0.08025	t Ratio	-2.63194
Std Err Dif	0.03049	DF	5
Upper CL Dif	-0.00187	Prob > |t|	0.0464*
Lower CL Dif	-0.15863	Prob > t	0.9768
Confidence	0.95	Prob < t	0.0232*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	0.01104011	0.011040	6.9271	**0.0464***
Error	5	0.00796875	0.001594		
C. Total	6	0.01900886			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Aqua Regia	3	0.708000	0.02305	0.64875	0.76725
EPA Hg Prep	4	0.627750	0.01996	0.57644	0.67906

Std Error uses a pooled estimate of error variance

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Aqua Regia	3	0.0144222	0.0106667	0.0093333
EPA Hg Prep	4	0.0501755	0.0361250	0.0337500

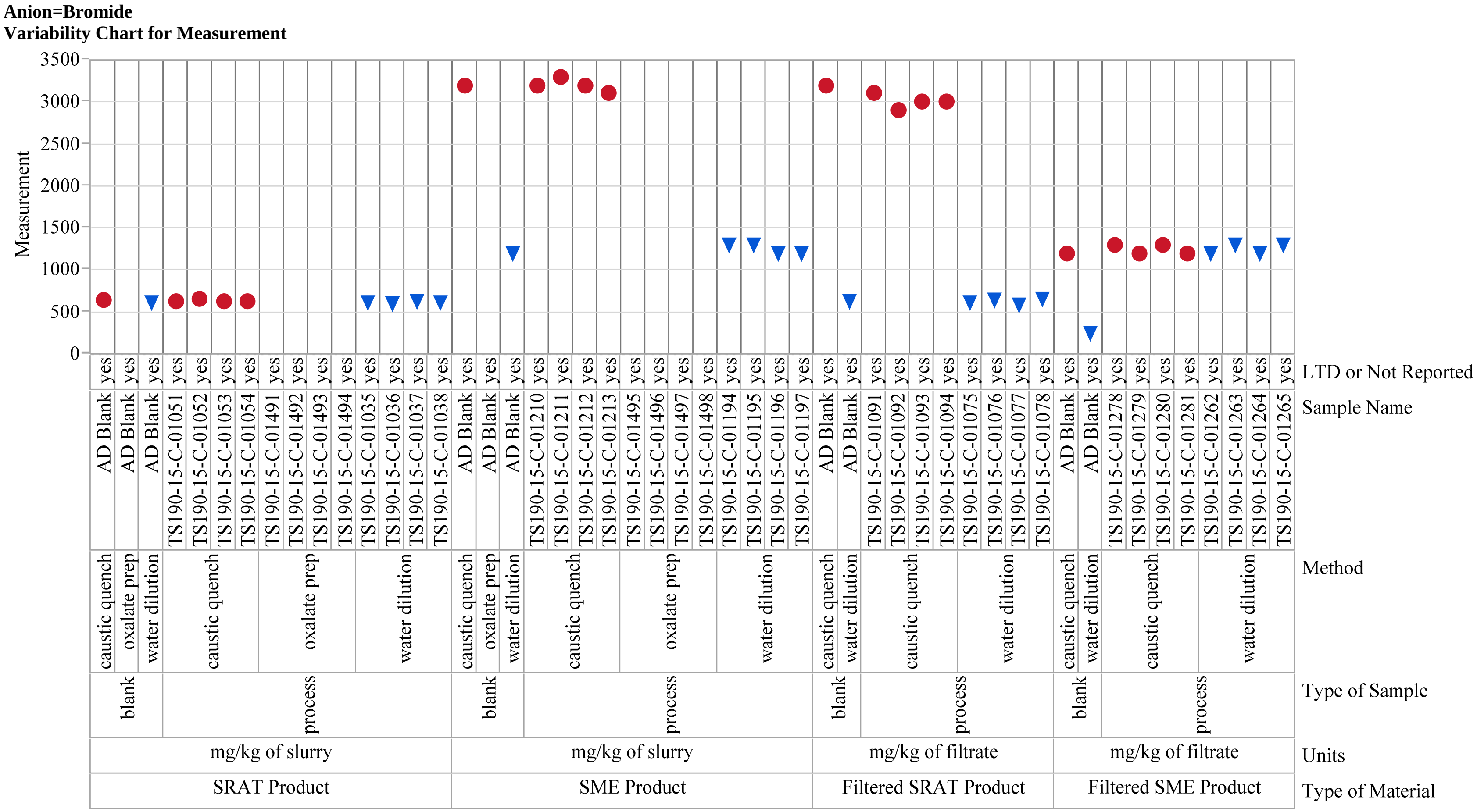
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.0381	1	5	0.3550
Brown-Forsythe	1.1642	1	5	0.3299
Levene	**2.3077**	**1**	**5**	**0.1892**
Bartlett	2.2302	1	.	0.1353
F Test 2-sided	12.1038	3	2	0.1545

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
9.2168	1	3.6313	0.0437*

Exhibit A6. Preliminary Plots of the Anion Measurements



LTD – Less Than Detectable

Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

Anion=Chloride
Variability Chart for Measurement

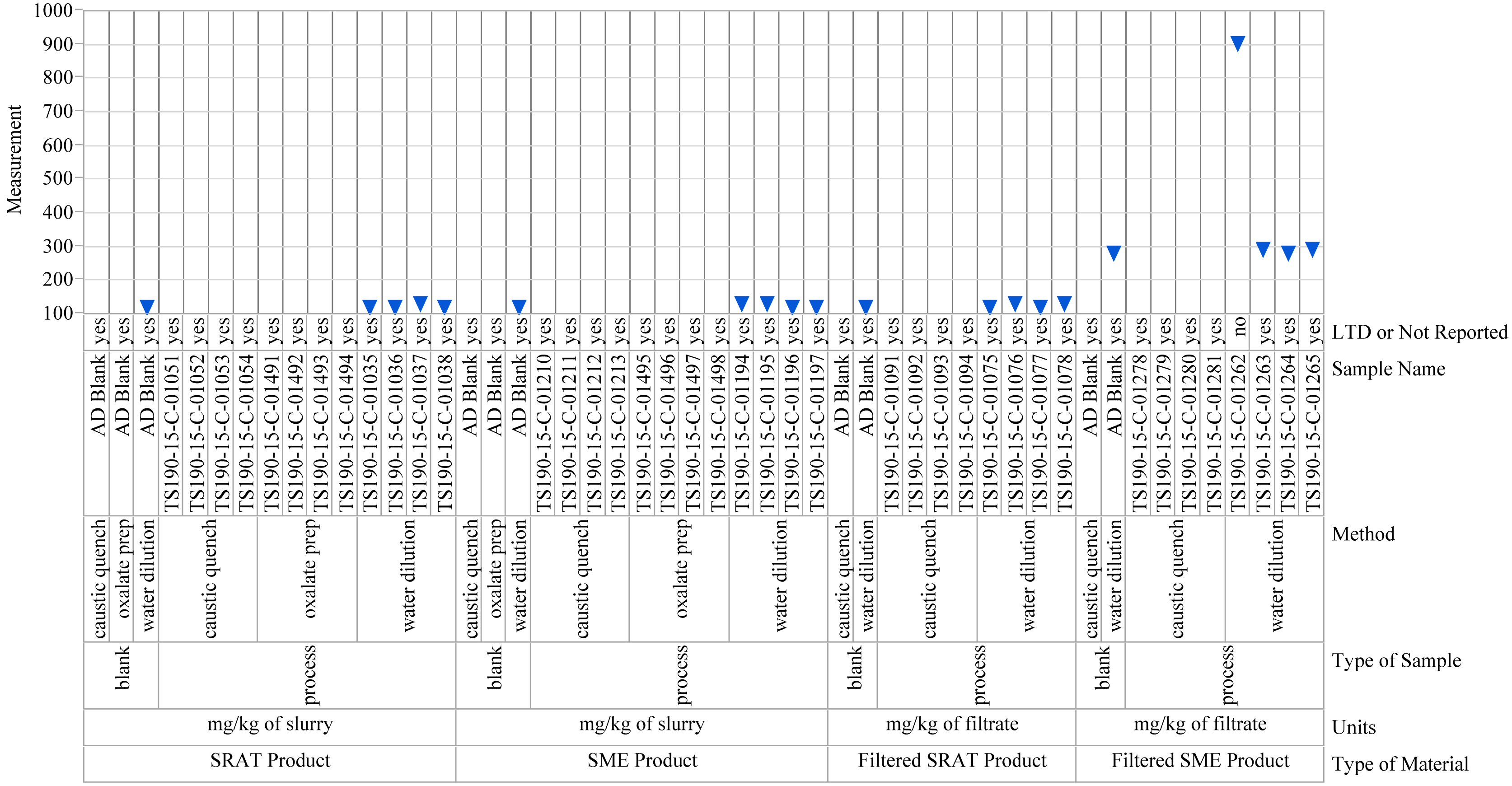
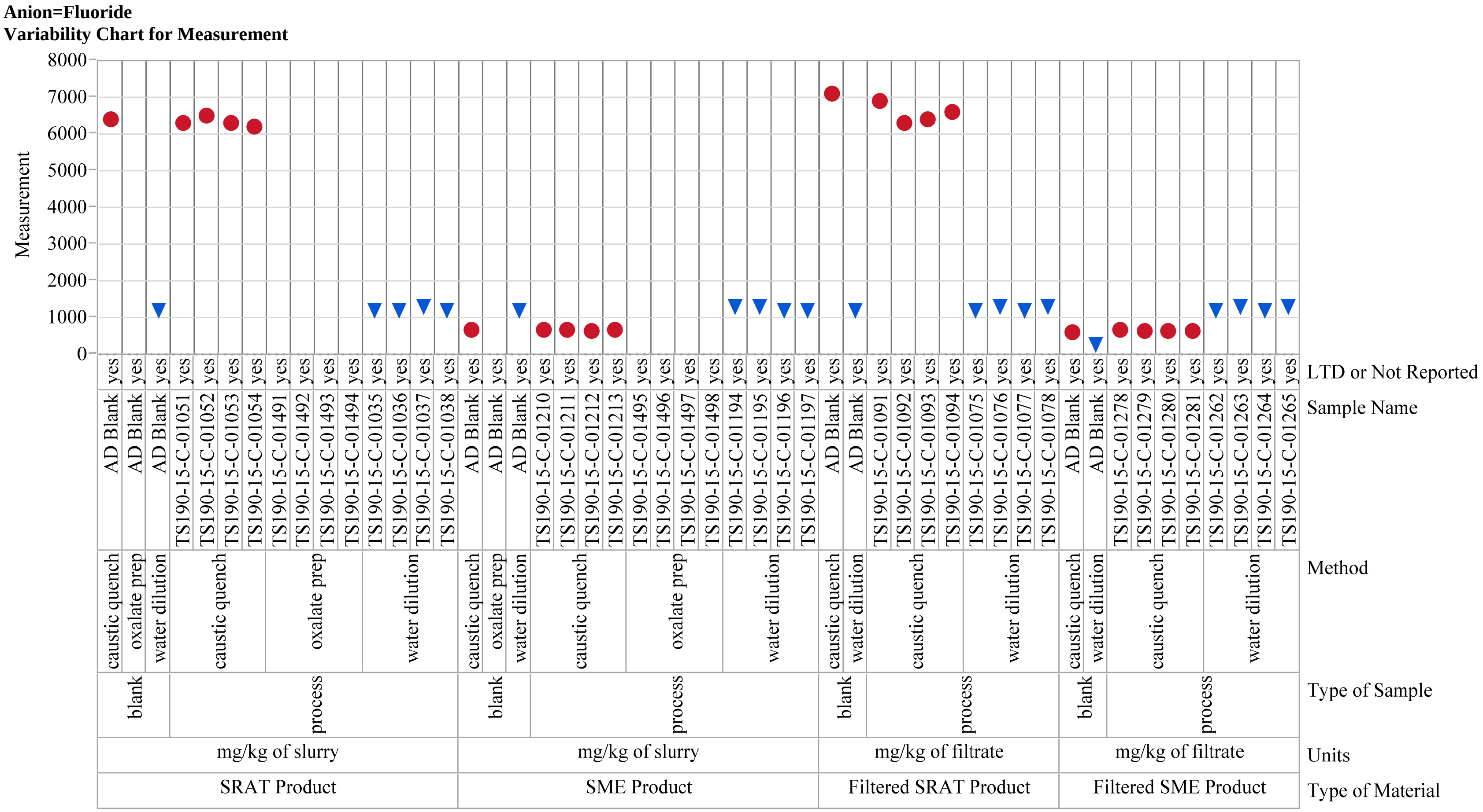


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*



LTD – Less Than Detectable

Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

Anion=Formate
Variability Chart for Measurement

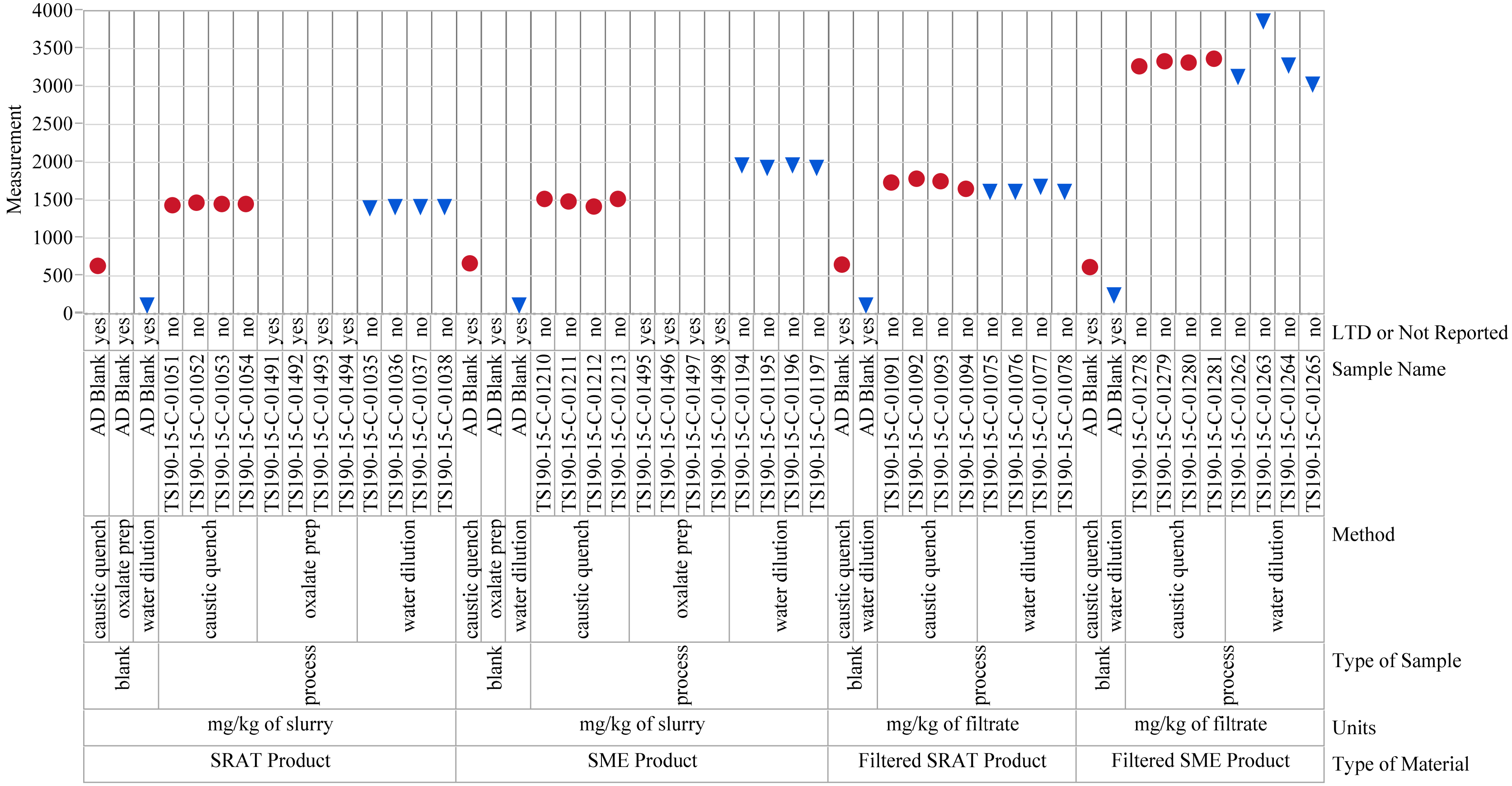


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

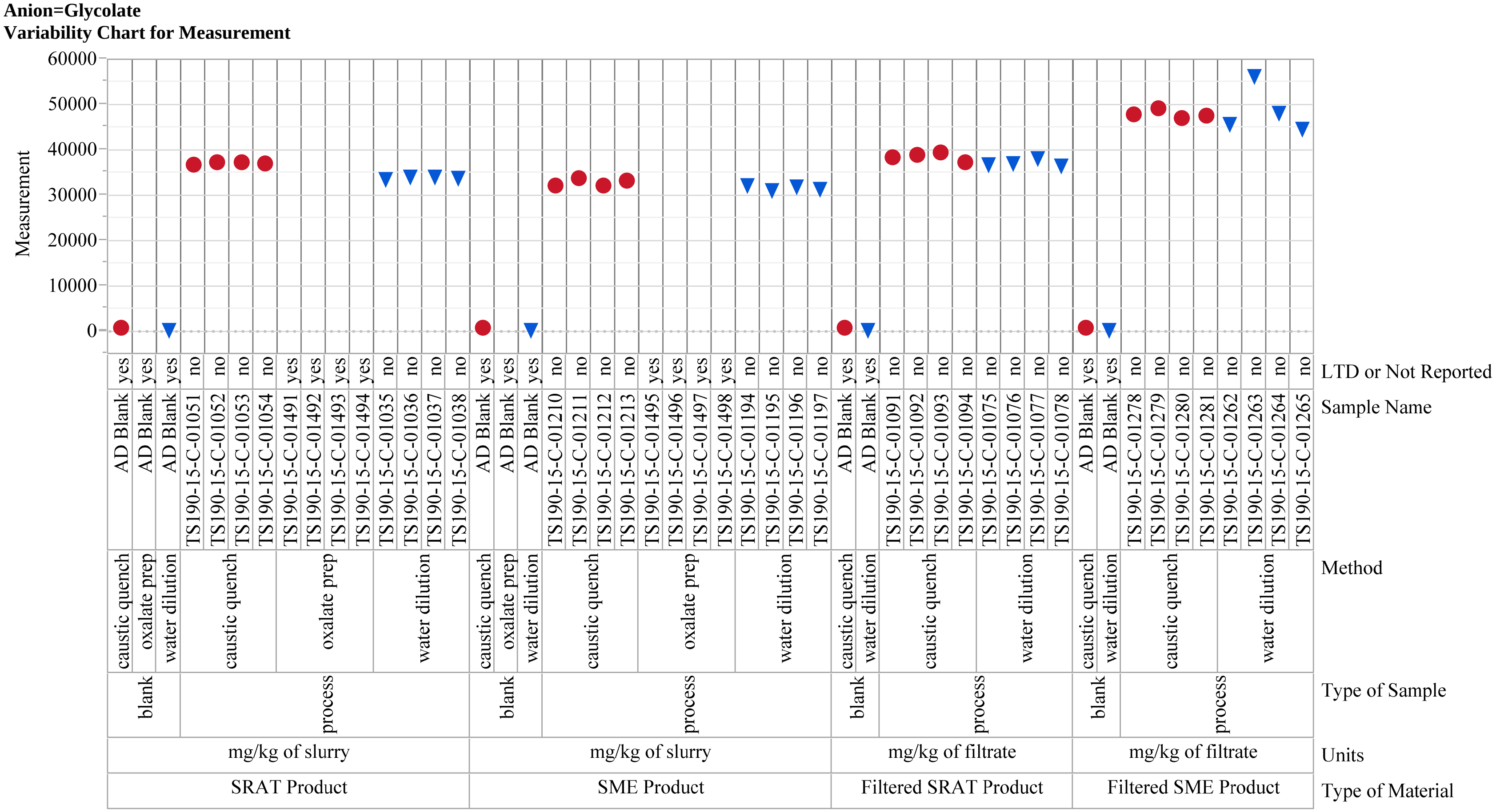

Anion=Glycolate
Variability Chart for Measurement

Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

Anion=Nitrate
Variability Chart for Measurement

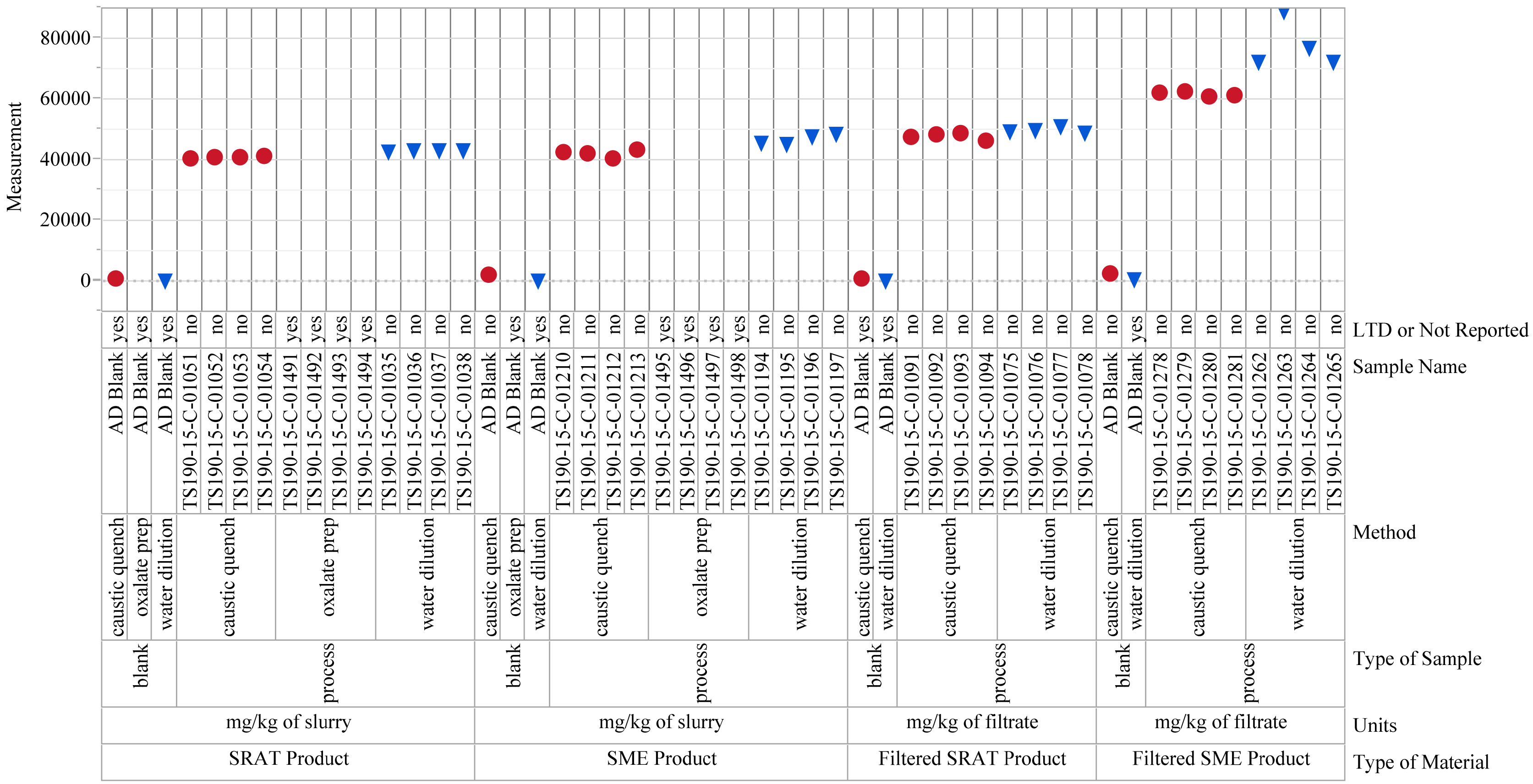


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

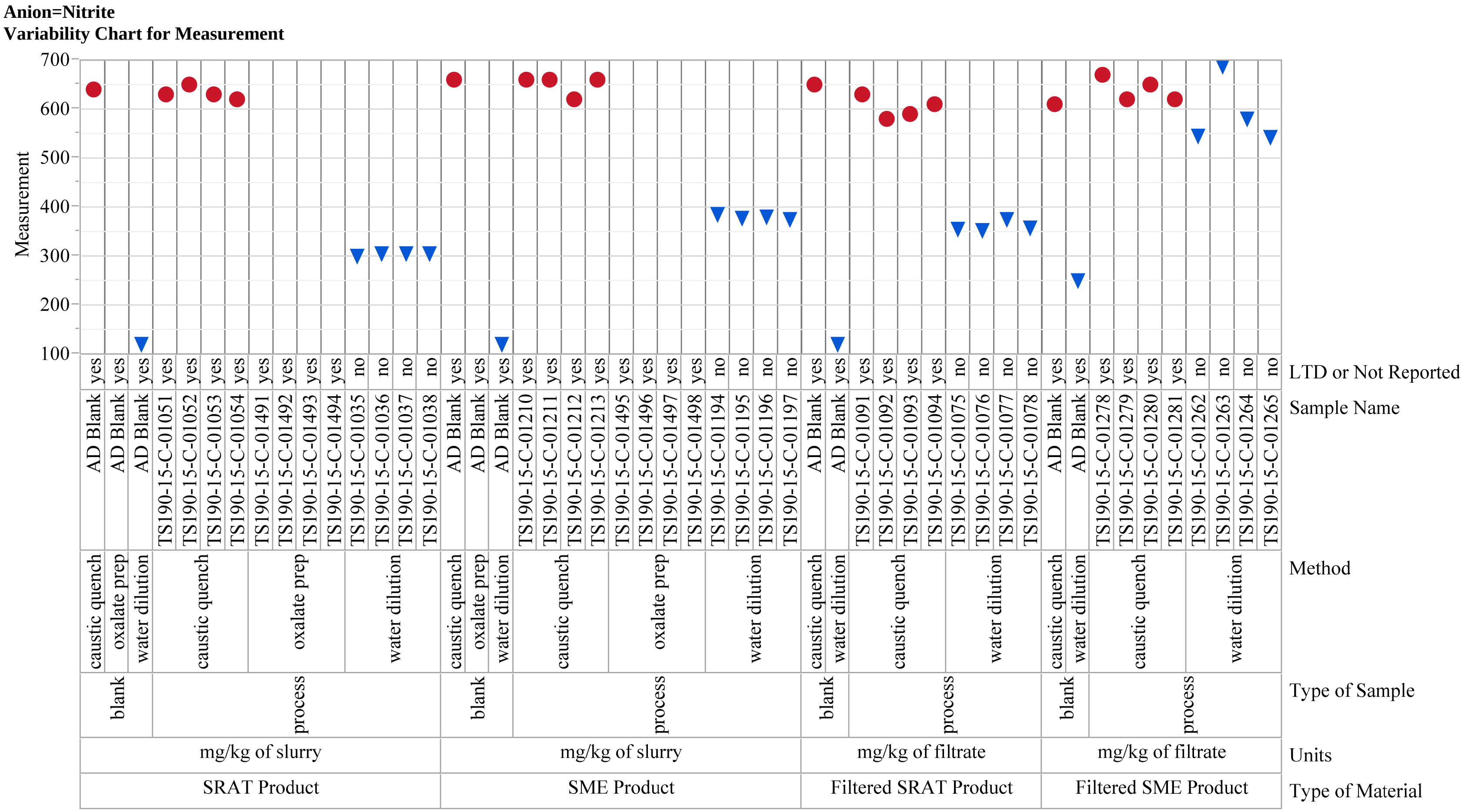


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

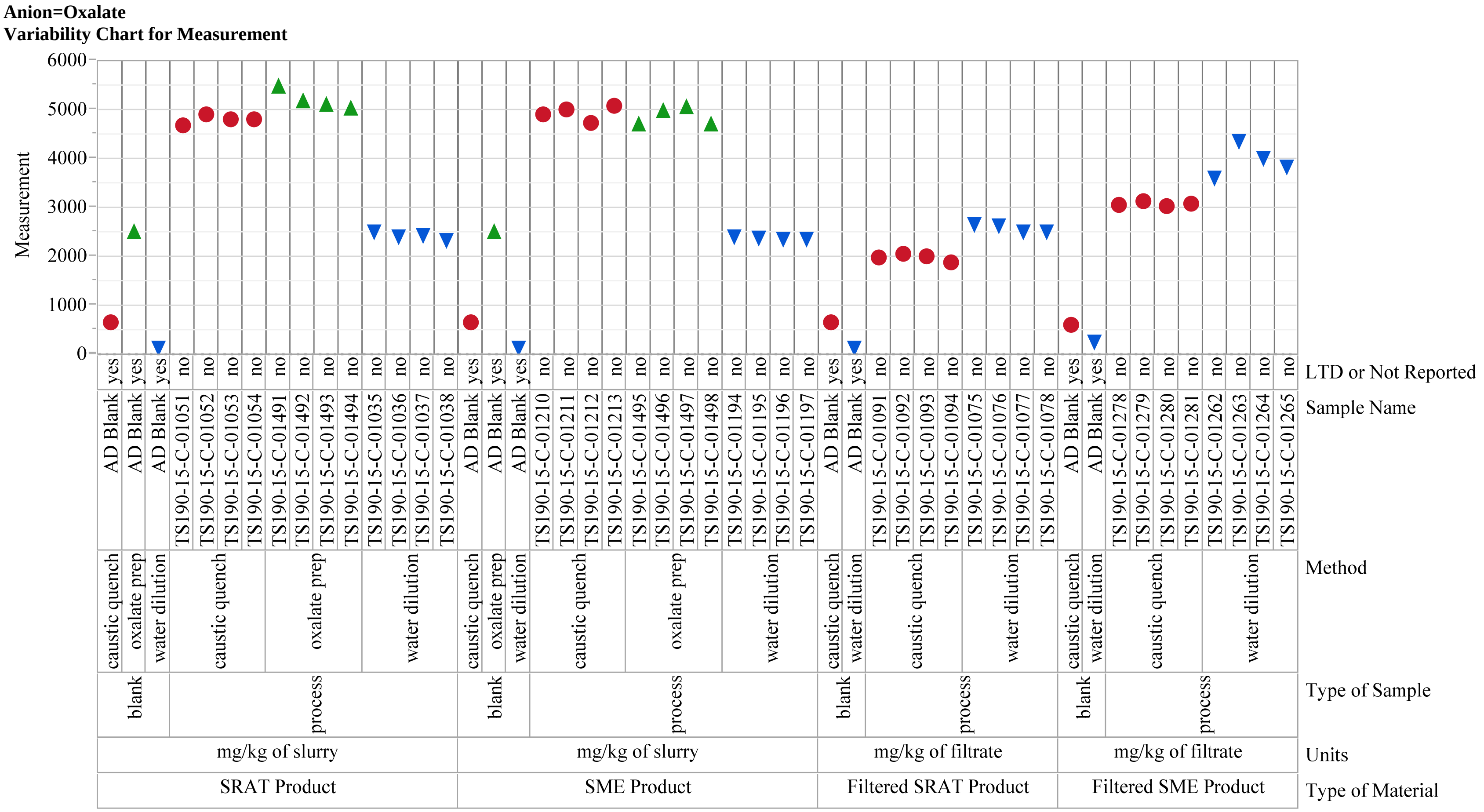


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

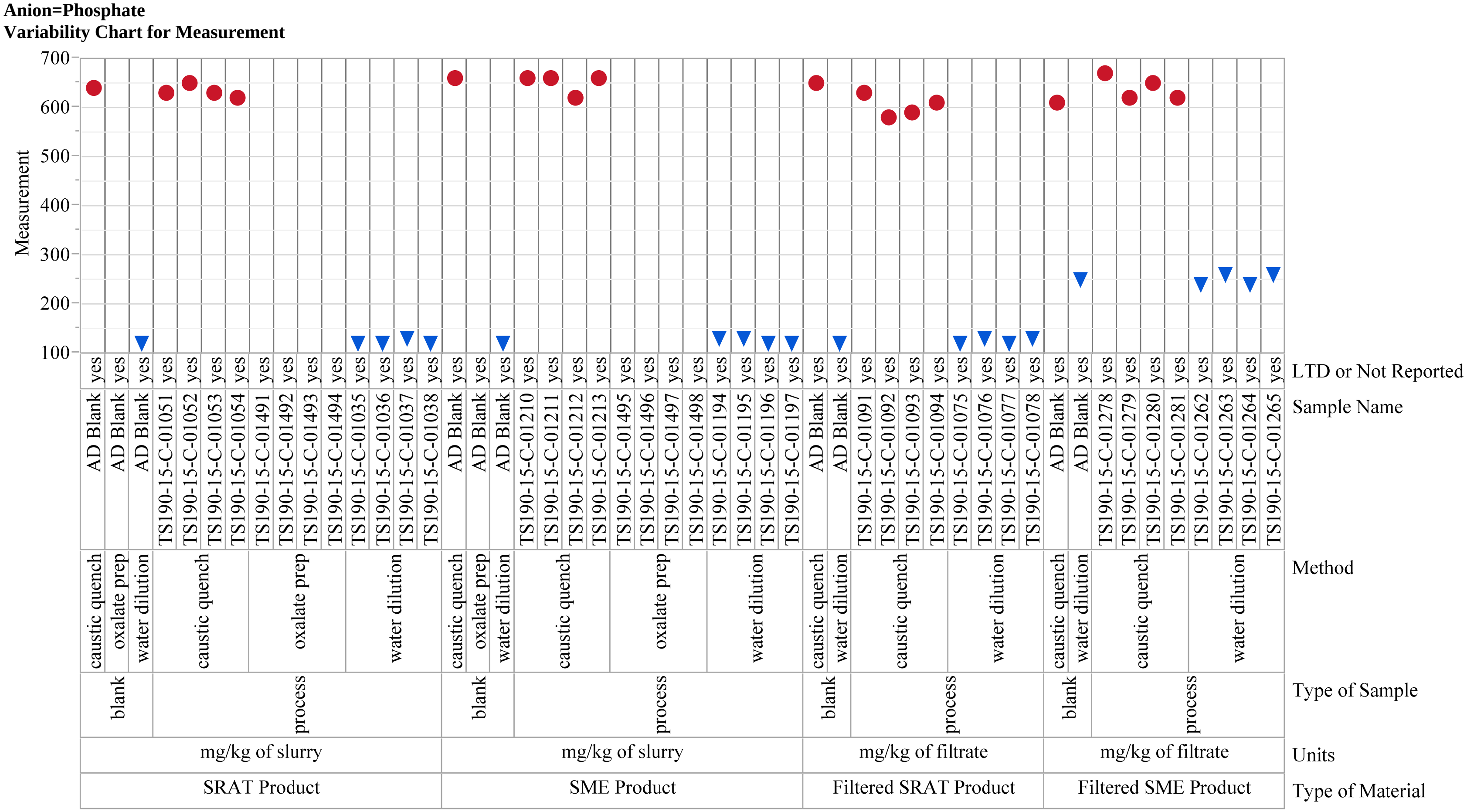


Exhibit A6. Preliminary Plots of the Anion Measurements *(continued)*

Anion=Sulfate
Variability Chart for Measurement

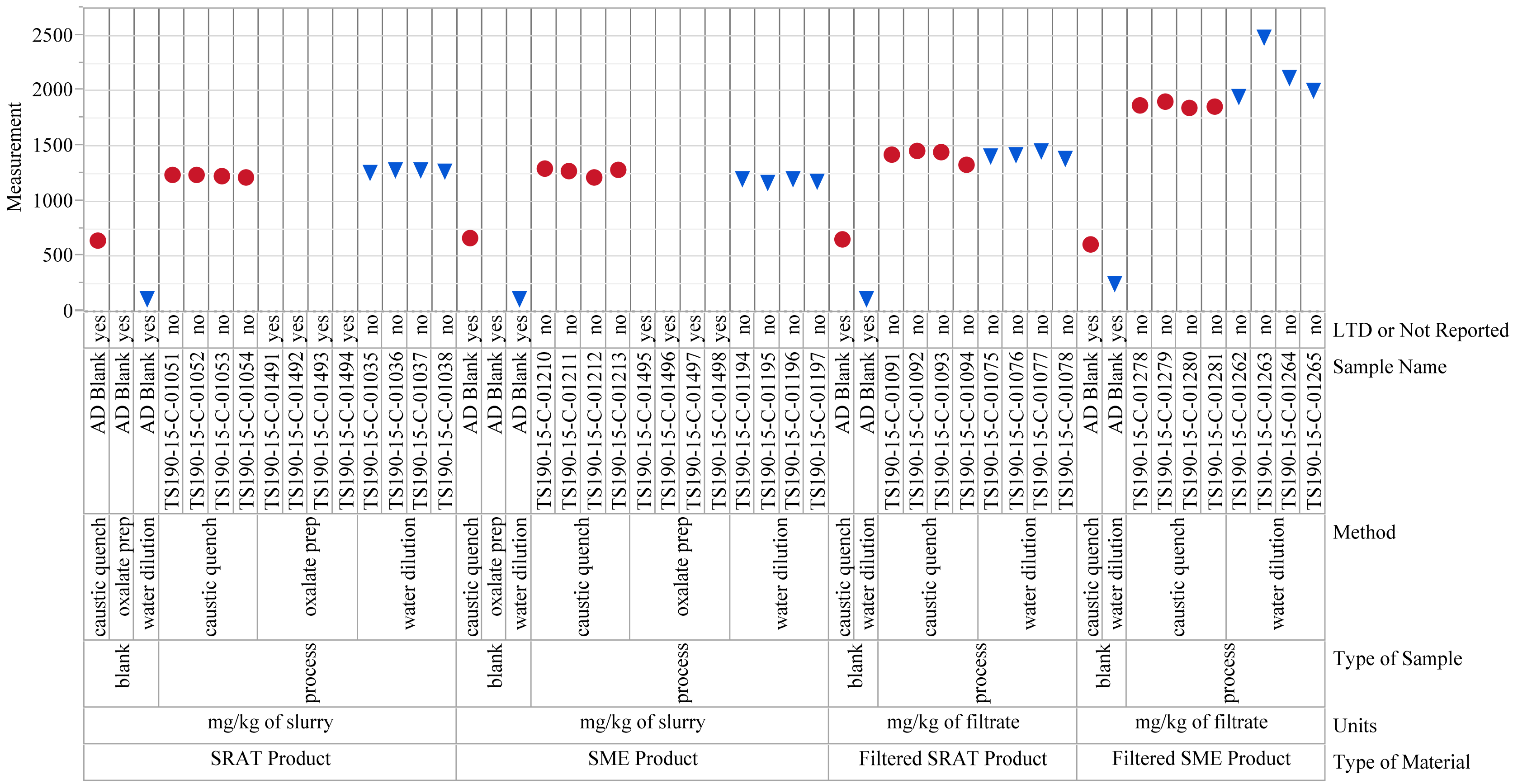
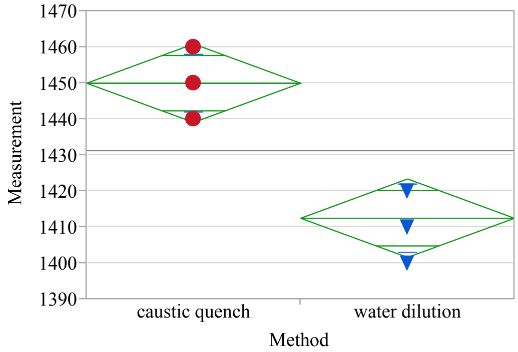


Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Formate



Oneway Anova
Summary of Fit

Rsquare	0.855513
Adj Rsquare	0.831432
Root Mean Square Error	8.897565
Mean of Response	1431.25
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-37.500	t Ratio	-5.9604
Std Err Dif	6.292	DF	6
Upper CL Dif	-22.105	Prob > |t|	0.0010*
Lower CL Dif	-52.895	Prob > t	0.9995
Confidence	0.95	Prob < t	0.0005*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	2812.5000	2812.50	35.5263	**0.0010***
Error	6	475.0000	79.17		
C. Total	7	3287.5000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1450.00	4.4488	1439.1	1460.9
water dilution	4	1412.50	4.4488	1401.6	1423.4

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-37.500	t Ratio	-5.9604
Std Err Dif	6.292	DF	5.854054
Upper CL Dif	-22.012	Prob > |t|	0.0011*
Lower CL Dif	-52.988	Prob > t	0.9995
Confidence	0.95	Prob < t	0.0005*

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	8.164966	5.000000	5.000000
water dilution	4	9.574271	7.500000	7.500000

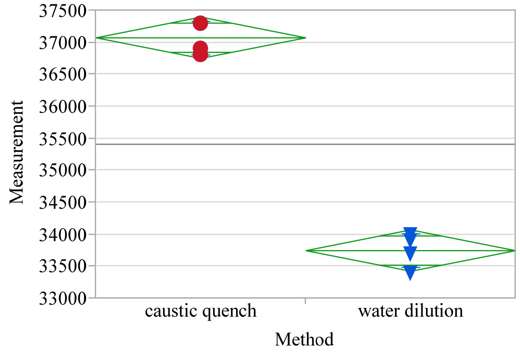
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.1234	1	6	0.7374
Brown-Forsythe	0.4286	1	6	0.5370
Levene	**0.5000**	**1**	**6**	**0.5060**
Bartlett	0.0649	1	.	0.7989
F Test 2-sided	1.3750	3	3	0.7998

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
35.5263	1	5.8541	0.0011*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Glycolate



Oneway Anova
Summary of Fit

Rsquare	0.981468
Adj Rsquare	0.978379
Root Mean Square Error	263.7865
Mean of Response	35412.5
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-3325.0	t Ratio	-17.826
Std Err Dif	186.5	DF	6
Upper CL Dif	-2868.6	Prob > |t|	<.0001*
Lower CL Dif	-3781.4	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	22111250	22111250	317.7665	**<.0001***
Error	6	417500	69583.333		
C. Total	7	22528750			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	37075.0	131.89	36752	37398
water dilution	4	33750.0	131.89	33427	34073

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-3325.0	t Ratio	-17.826
Std Err Dif	186.5	DF	5.999785
Upper CL Dif	-2868.6	Prob > |t|	<.0001*
Lower CL Dif	-3781.4	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001*

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	262.9956	225.0000	225.0000
water dilution	4	264.5751	200.0000	200.0000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.0003	1	6	0.9863
Brown-Forsythe	0.1111	1	6	0.7502
Levene	**0.1364**	**1**	**6**	**0.7246**
Bartlett	0.0001	1	.	0.9923
F Test 2-sided	1.0120	3	3	0.9924

Warning: Small sample sizes. Use Caution.

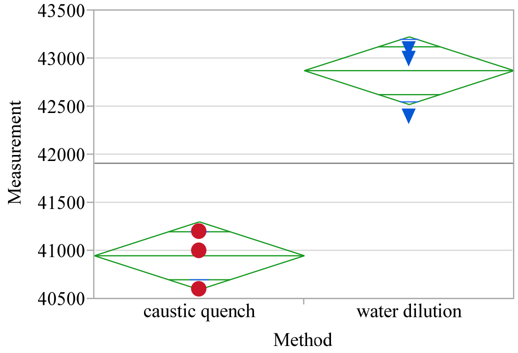
Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
317.7665	1	5.9998	<.0001*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Nitrate



Oneway Anova

Summary of Fit

Rsquare	0.937095
Adj Rsquare	0.926611
Root Mean Square Error	287.9525
Mean of Response	41912.5
Observations (or Sum Wgts)	8

t Test

water dilution-caustic quench

Assuming equal variances

Difference	1925.00	t Ratio	9.454201
Std Err Dif	203.61	DF	6
Upper CL Dif	2423.22	Prob > |t|	<.0001*
Lower CL Dif	1426.78	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	7411250.0	7411250	89.3819	**<.0001***
Error	6	497500.0	82917		
C. Total	7	7908750.0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	40950.0	143.98	40598	41302
water dilution	4	42875.0	143.98	42523	43227

Std Error uses a pooled estimate of error variance

t Test

water dilution-caustic quench

Assuming equal variances

Difference	1925.00	t Ratio	9.454201
Std Err Dif	203.61	DF	5.682994
Upper CL Dif	2430.04	Prob > |t|	0.0001*
Lower CL Dif	1419.96	Prob > t	<.0001*
Confidence	0.95	Prob < t	0.9999

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	251.6611	175.0000	150.0000
water dilution	4	320.1562	237.5000	175.0000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.1648	1	6	0.6989
Brown-Forsythe	0.0210	1	6	0.8896
Levene	**0.3138**	**1**	**6**	**0.5956**
Bartlett	0.1476	1	.	0.7008
F Test 2-sided	1.6184	3	3	0.7021

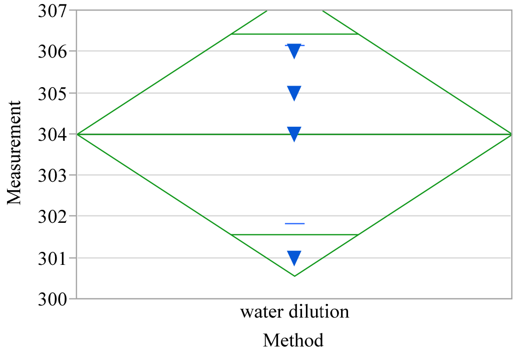
Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
89.3819	1	5.683	0.0001*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Nitrite



Oneway Anova

Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	2.160247
Mean of Response	304
Observations (or Sum Wgts)	4

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.000000	.	.	.
Error	3	14.000000	4.66667		
C. Total	3	14.000000			

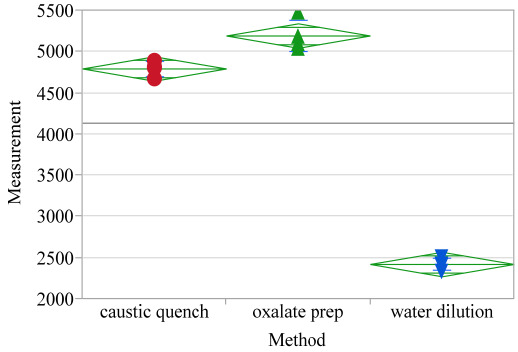
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
water dilution	4	304.000	1.0801	300.56	307.44

Std Error uses a pooled estimate of error variance

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Oxalate



Oneway Anova
Summary of Fit

Rsquare	0.991428
Adj Rsquare	0.989523
Root Mean Square Error	131.3815
Mean of Response	4135
Observations (or Sum Wgts)	12

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**	**Prob > F**
Method	2	17967350	8983675	520.4575	**<.0001***
Error	9	155350	17261		
C. Total	11	18122700			

Means for Oneway Anova

Level	**Number**	**Mean**	**Std Error**	**Lower 95%**	**Upper 95%**
caustic quench	4	4792.50	65.691	4643.9	4941.1
oxalate prep	4	5192.50	65.691	5043.9	5341.1
water dilution	4	2420.00	65.691	2271.4	2568.6

Std Error uses a pooled estimate of error variance

Tests that the Variances are Equal

Level	**Count**	**Std Dev**	**MeanAbsDif to Mean**	**MeanAbsDif to Median**
caustic quench	4	91.0586	61.2500	57.5000
oxalate prep	4	195.0000	138.7500	132.5000
water dilution	4	73.9369	50.0000	50.0000

Test	**F Ratio**	**DFNum**	**DFDen**	**Prob > F**
O'Brien[.5]	1.1367	2	9	0.3629
Brown-Forsythe	0.9891	2	9	0.4090
Levene	**1.5759**	**2**	**9**	**0.2589**
Bartlett	1.4282	2	.	0.2397

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	**DFNum**	**DFDen**	**Prob > F**
874.7682	2	5.5262	<.0001*

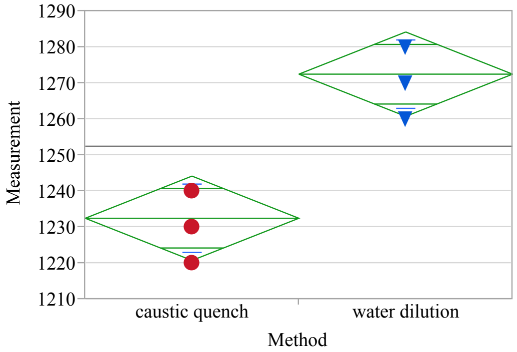
Connecting Letters Report

(this test assumes equal variances for the methods; Levene's test results support this assumption)

Level				**Mean**
oxalate prep	A			5192.5000
caustic quench		B		4792.5000
water dilution			C	2420.0000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SRAT Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Sulfate



Oneway Anova
Summary of Fit

Rsquare	0.853333
Adj Rsquare	0.828889
Root Mean Square Error	9.574271
Mean of Response	1252.5
Observations (or Sum Wgts)	8

t Test

water dilution-caustic quench

Assuming equal variances

Difference	40.0000	t Ratio	5.908392
Std Err Dif	6.7700	DF	6
Upper CL Dif	56.5657	Prob > |t|	0.0010*
Lower CL Dif	23.4343	Prob > t	0.0005*
Confidence	0.95	Prob < t	0.9995

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**	**Prob > F**
Method	1	3200.0000	3200.00	34.9091	**0.0010***
Error	6	550.0000	91.67		
C. Total	7	3750.0000			

Means for Oneway Anova

Level	**Number**	**Mean**	**Std Error**	**Lower 95%**	**Upper 95%**
caustic quench	4	1232.50	4.7871	1220.8	1244.2
water dilution	4	1272.50	4.7871	1260.8	1284.2

Std Error uses a pooled estimate of error variance

t Test

water dilution-caustic quench

Assuming equal variances

Difference	40.0000	t Ratio	5.908392
Std Err Dif	6.7700	DF	6
Upper CL Dif	56.5657	Prob > |t|	0.0010*
Lower CL Dif	23.4343	Prob > t	0.0005*
Confidence	0.95	Prob < t	0.9995

Tests that the Variances are Equal

Level	**Count**	**Std Dev**	**MeanAbsDif to Mean**	**MeanAbsDif to Median**
caustic quench	4	9.574271	7.500000	7.500000
water dilution	4	9.574271	7.500000	7.500000

Test	**F Ratio**	**DFNum**	**DFDen**	**p-Value**
O'Brien[.5]	0.0000	1	6	1.0000
Brown-Forsythe	0.0000	1	6	1.0000
Levene	**0.0000**	**1**	**6**	**1.0000**
Bartlett	0.0000	1	.	1.0000
F Test 2-sided	1.0000	3	3	1.0000

Warning: Small sample sizes. Use Caution.

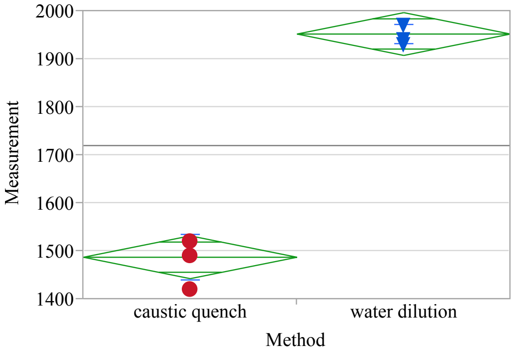
Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	**DFNum**	**DFDen**	**Prob > F**
34.9091	1	6	0.0010*

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Formate



Oneway Anova
Summary of Fit

Rsquare	0.981948
Adj Rsquare	0.97894
Root Mean Square Error	36.40055
Mean of Response	1720
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	465.000	t Ratio	18.06592
Std Err Dif	25.739	DF	6
Upper CL Dif	527.981	Prob > |t|	<.0001*
Lower CL Dif	402.019	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	432450.00	432450	326.3774	**<.0001***
Error	6	7950.00	1325		
C. Total	7	440400.00			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1487.50	18.200	1443.0	1532.0
water dilution	4	1952.50	18.200	1908.0	1997.0

Std Error uses a pooled estimate of error variance
t Test
water dilution-caustic quench
Assuming equal variances

Difference	465.000	t Ratio	18.06592
Std Err Dif	25.739	DF	4.105725
Upper CL Dif	535.743	Prob > |t|	<.0001*
Lower CL Dif	394.257	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Tests that the Variances are Equal

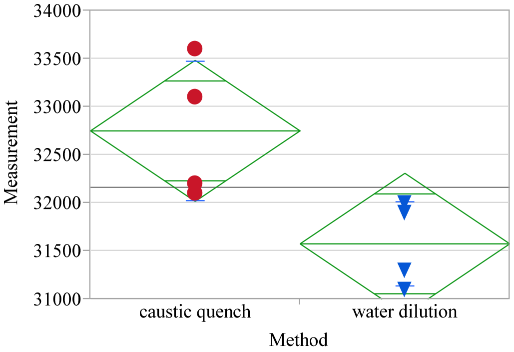
Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	47.16991	33.75000	32.50000
water dilution	4	20.61553	17.50000	17.50000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.1748	1	6	0.3200
Brown-Forsythe	0.7200	1	6	0.4287
Levene	**1.4611**	**1**	**6**	**0.2722**
Bartlett	1.5910	1	.	0.2072
F Test 2-sided	5.2353	3	3	0.2073

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
326.3774	1	4.1057	<.0001*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Glycolate



Oneway Anova
Summary of Fit

Rsquare	0.561372
Adj Rsquare	0.488268
Root Mean Square Error	599.6527
Mean of Response	32162.5
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-1175.0	t Ratio	-2.77111
Std Err Dif	424.0	DF	6
Upper CL Dif	-137.5	Prob > |t|	0.0324*
Lower CL Dif	-2212.5	Prob > t	0.9838
Confidence	0.95	Prob < t	0.0162*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	2761250.0	2761250	7.6790	**0.0324***
Error	6	2157500.0	359583		
C. Total	7	4918750.0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	32750.0	299.83	32016	33484
water dilution	4	31575.0	299.83	30841	32309

Std Error uses a pooled estimate of error variance
t Test
water dilution-caustic quench
Assuming equal variances

Difference	-1175.0	t Ratio	-2.77111
Std Err Dif	424.0	DF	4.969445
Upper CL Dif	-83.0	Prob > |t|	0.0396*
Lower CL Dif	-2267.0	Prob > t	0.9802
Confidence	0.95	Prob < t	0.0198*

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	723.4178	600.0000	600.0000
water dilution	4	442.5306	375.0000	375.0000

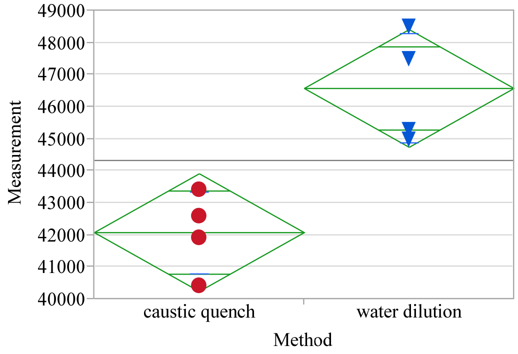
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	2.2611	1	6	0.1834
Brown-Forsythe	3.0759	1	6	0.1300
Levene	**3.9194**	**1**	**6**	**0.0951**
Bartlett	0.5976	1	.	0.4395
F Test 2-sided	2.6723	1	3	0.4409

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
7.6790	1	4.9694	0.0396*

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Nitrate



Oneway Anova
Summary of Fit

Rsquare	0.749514
Adj Rsquare	0.707767
Root Mean Square Error	1501.943
Mean of Response	44325
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	4500.00	t Ratio	4.237152
Std Err Dif	1062.03	DF	6
Upper CL Dif	7098.70	Prob > |t|	0.0055*
Lower CL Dif	1901.30	Prob > t	0.0027*
Confidence	0.95	Prob < t	0.9973

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	40500000	40500000	17.9535	**0.0055***
Error	6	13535000	2255833.3		
C. Total	7	54035000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	42075.0	750.97	40237	43913
water dilution	4	46575.0	750.97	44737	48413

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	4500.00	t Ratio	4.237152
Std Err Dif	1062.03	DF	5.561619
Upper CL Dif	7149.17	Prob > |t|	0.0065*
Lower CL Dif	1850.83	Prob > t	0.0032*
Confidence	0.95	Prob < t	0.9968

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	1273.774	925.000	925.000
water dilution	4	1699.755	1425.000	1425.000

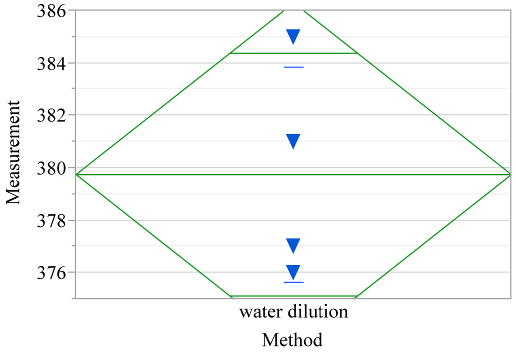
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.7231	1	6	0.4278
Brown-Forsythe	1.3423	1	6	0.2907
Levene	**1.5075**	**1**	**6**	**0.2655**
Bartlett	0.2111	1	.	0.6459
F Test 2-sided	1.7807	3	3	0.6473

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
17.9535	1	5.5616	0.0065*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Nitrite



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	4.112988
Mean of Response	379.75
Observations (or Sum Wgts)	4

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.000000	.	.	.
Error	3	50.750000	16.9167		
C. Total	3	50.750000			

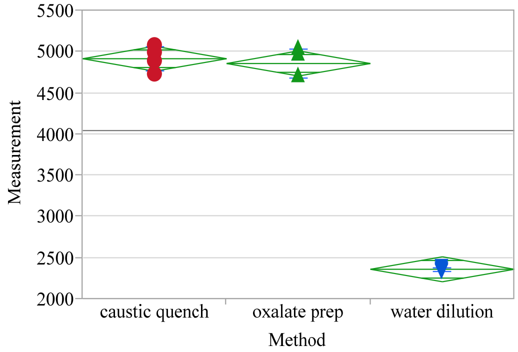
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
water dilution	4	379.750	2.0565	373.21	386.29

Std Error uses a pooled estimate of error variance

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Oxalate



Oneway Anova
Summary of Fit

Rsquare	0.99052
Adj Rsquare	0.988413
Root Mean Square Error	134.5569
Mean of Response	4046.667
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	2	17025117	8512558	470.1628	<.0001*
Error	9	162950	18106		
C. Total	11	17188067			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	4917.50	67.278	4765.3	5069.7
oxalate prep	4	4860.00	67.278	4707.8	5012.2
water dilution	4	2362.50	67.278	2210.3	2514.7

Std Error uses a pooled estimate of error variance

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	150.8587	112.5000	112.5000
oxalate prep	4	176.2574	150.0000	150.0000
water dilution	4	22.1736	17.5000	17.5000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.6638	2	9	0.1234
Brown-Forsythe	6.7848	2	9	0.0160
Levene	**7.9585**	**2**	**9**	**0.0102**
Bartlett	3.7071	2	.	0.0245

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
809.8230	2	4.1455	**<.0001***

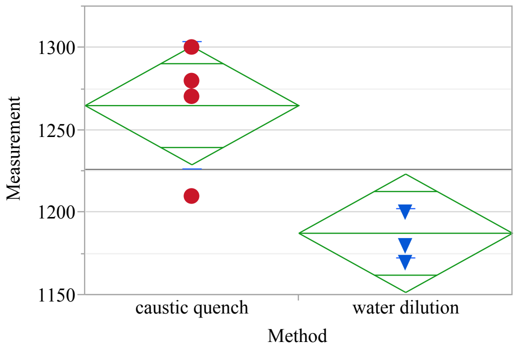
Means Comparisons
Comparisons for all pairs using Tukey-Kramer HSD
Connecting Letters Report

(this test assumes equal variances for the methods; Levene's test results **do not** support this assumption)

Level			Mean
caustic quench	A		4917.5000
oxalate prep	A		4860.0000
water dilution		B	2362.5000

Levels not connected by same letter are significantly different.

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=SME Product, Units=mg/kg of slurry, LTD or Not Reported=no, Anion=Sulfate



Oneway Anova
Summary of Fit

Rsquare	0.698909
Adj Rsquare	0.648727
Root Mean Square Error	29.36835
Mean of Response	1226.25
Observations (or Sum Wgts)	8

t Test

water dilution-caustic quench
Assuming equal variances

Difference	-77.50	t Ratio	-3.73196
Std Err Dif	20.77	DF	6
Upper CL Dif	-26.69	Prob > |t|	0.0097*
Lower CL Dif	-128.31	Prob > t	0.9951
Confidence	0.95	Prob < t	0.0049*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	12012.500	12012.5	13.9275	**0.0097***
Error	6	5175.000	862.5		
C. Total	7	17187.500			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1265.00	14.684	1229.1	1300.9
water dilution	4	1187.50	14.684	1151.6	1223.4

Std Error uses a pooled estimate of error variance

t Test

water dilution-caustic quench
Assuming equal variances

Difference	-77.50	t Ratio	-3.73196
Std Err Dif	20.77	DF	3.880196
Upper CL Dif	-19.13	Prob > |t|	0.0214*
Lower CL Dif	-135.87	Prob > t	0.9893
Confidence	0.95	Prob < t	0.0107*

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	38.72983	27.50000	25.00000
water dilution	4	15.00000	12.50000	12.50000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.2382	1	6	0.3084
Brown-Forsythe	0.7576	1	6	0.4175
Levene	**1.7705**	**1**	**6**	**0.2317**
Bartlett	2.0323	1	.	0.1540
F Test 2-sided	6.6667	3	3	0.1535

Warning: Small sample sizes. Use Caution.

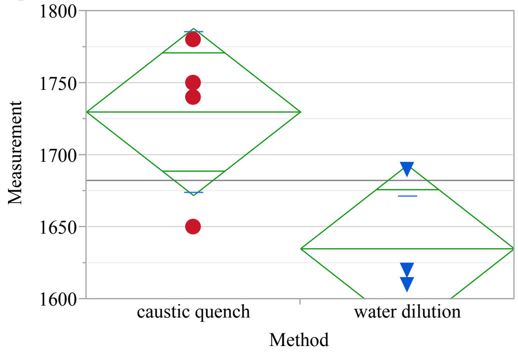
Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
13.9275	1	3.8802	0.0214*

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Formate



Oneway Anova
Summary of Fit

Rsquare	0.572108
Adj Rsquare	0.500792
Root Mean Square Error	47.43416
Mean of Response	1682.5
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-95.00	t Ratio	-2.83235
Std Err Dif	33.54	DF	6
Upper CL Dif	-12.93	Prob > |t|	0.0299*
Lower CL Dif	-177.07	Prob > t	0.9851
Confidence	0.95	Prob < t	0.0149*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	18050.000	18050.0	8.0222	**0.0299***
Error	6	13500.000	2250.0		
C. Total	7	31550.000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1730.00	23.717	1672.0	1788.0
water dilution	4	1635.00	23.717	1577.0	1693.0

Std Error uses a pooled estimate of error variance
t Test
water dilution-caustic quench
Assuming equal variances

Difference	-95.00	t Ratio	-2.83235
Std Err Dif	33.54	DF	5.198726
Upper CL Dif	-9.76	Prob > |t|	0.0350*
Lower CL Dif	-180.24	Prob > t	0.9825
Confidence	0.95	Prob < t	0.0175*

Tests that the Variances are Equal

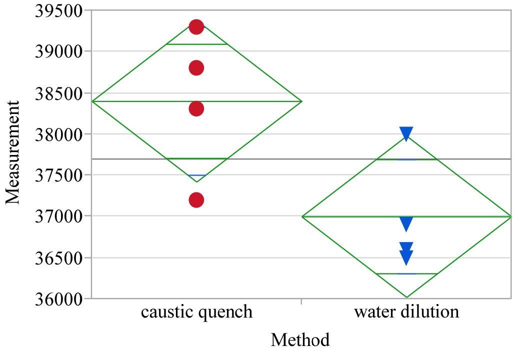
Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	55.97619	40.00000	35.00000
water dilution	4	36.96846	27.50000	20.00000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.4388	1	6	0.5323
Brown-Forsythe	0.3068	1	6	0.5997
Levene	**0.4601**	**1**	**6**	**0.5229**
Bartlett	0.4304	1	.	0.5118
F Test 2-sided	2.2927	3	3	0.5133

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
8.0222	1	5.1987	0.0350*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Glycolate



Oneway Anova
Summary of Fit

Rsquare	0.505155
Adj Rsquare	0.42268
Root Mean Square Error	800
Mean of Response	37700
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	-1400.0	t Ratio	-2.47487
Std Err Dif	565.7	DF	6
Upper CL Dif	-15.8	Prob > |t|	0.0481*
Lower CL Dif	-2784.2	Prob > t	0.9759
Confidence	0.95	Prob < t	0.0241*

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	3920000.0	3920000	6.1250	**0.0481***
Error	6	3840000.0	640000		
C. Total	7	7760000.0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	38400.0	400.00	37421	39379
water dilution	4	37000.0	400.00	36021	37979

Std Error uses a pooled estimate of error variance
t Test
water dilution-caustic quench
Assuming equal variances

Difference	-1400.0	t Ratio	-2.47487
Std Err Dif	565.7	DF	5.618941
Upper CL Dif	7.3	Prob > |t|	0.0508
Lower CL Dif	-2807.3	Prob > t	0.9746
Confidence	0.95	Prob < t	0.0254*

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	898.1462	650.0000	650.0000
water dilution	4	687.9922	500.0000	450.0000

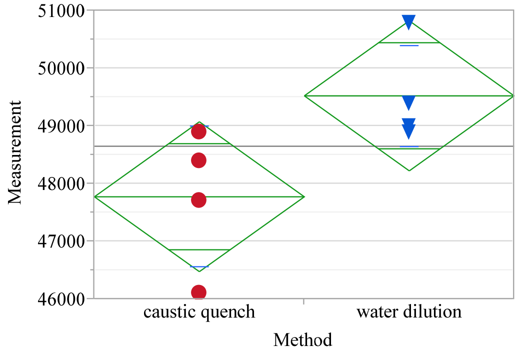
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.2559	1	6	0.6310
Brown-Forsythe	0.2857	1	6	0.6122
Levene	**0.2348**	**1**	**6**	**0.6452**
Bartlett	0.1806	1	.	0.6709
F Test 2-sided	1.7042	3	3	0.6722

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
6.1250	1	5.6189	0.0508

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Nitrate



Oneway Anova
Summary of Fit

Rsquare	0.474806
Adj Rsquare	0.387274
Root Mean Square Error	1062.623
Mean of Response	48650
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	1750.0	t Ratio	2.329024
Std Err Dif	751.4	DF	6
Upper CL Dif	3588.6	Prob > |t|	0.0587
Lower CL Dif	-88.6	Prob > t	0.0294*
Confidence	0.95	Prob < t	0.9706

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	6125000	6125000	5.4244	**0.0587**
Error	6	6775000	1129167		
C. Total	7	12900000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	47775.0	531.31	46475	49075
water dilution	4	49525.0	531.31	48225	50825

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	1750.0	t Ratio	2.329024
Std Err Dif	751.4	DF	5.446397
Upper CL Dif	3634.8	Prob > |t|	0.0631
Lower CL Dif	-134.8	Prob > t	0.0315*
Confidence	0.95	Prob < t	0.9685

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	1220.314	875.0000	875.0000
water dilution	4	877.021	637.5000	575.0000

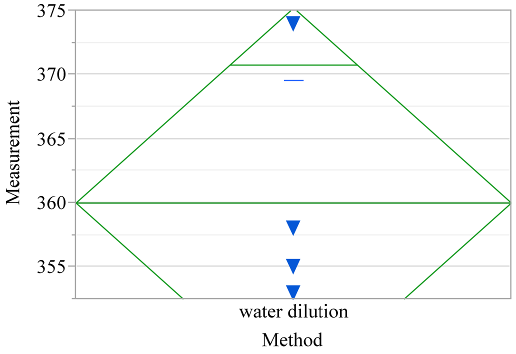
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.3626	1	6	0.5691
Brown-Forsythe	0.3467	1	6	0.5775
Levene	**0.3243**	**1**	**6**	**0.5897**
Bartlett	0.2756	1	.	0.5996
F Test 2-sided	1.9361	3	3	0.6011

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
5.4244	1	5.4464	0.0631

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Nitrite



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	9.556847
Mean of Response	360
Observations (or Sum Wgts)	4

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.00000	.	.	.
Error	3	274.00000	91.3333		
C. Total	3	274.00000			

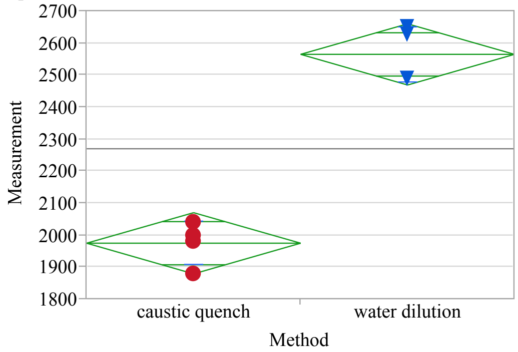
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
water dilution	4	360.000	4.7784	344.79	375.21

Std Error uses a pooled estimate of error variance

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Oxalate



Oneway Anova
Summary of Fit

Rsquare	0.950055
Adj Rsquare	0.94173
Root Mean Square Error	78.1025
Mean of Response	2270
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	590.000	t Ratio	10.68322
Std Err Dif	55.227	DF	6
Upper CL Dif	725.135	Prob > |t|	<.0001*
Lower CL Dif	454.865	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	696200.00	696200	114.1311	**<.0001***
Error	6	36600.00	6100		
C. Total	7	732800.00			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1975.00	39.051	1879.4	2070.6
water dilution	4	2565.00	39.051	2469.4	2660.6

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	590.000	t Ratio	10.68322
Std Err Dif	55.227	DF	5.672096
Upper CL Dif	727.052	Prob > |t|	<.0001*
Lower CL Dif	452.948	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	68.06859	47.50000	45.00000
water dilution	4	86.98659	75.00000	75.00000

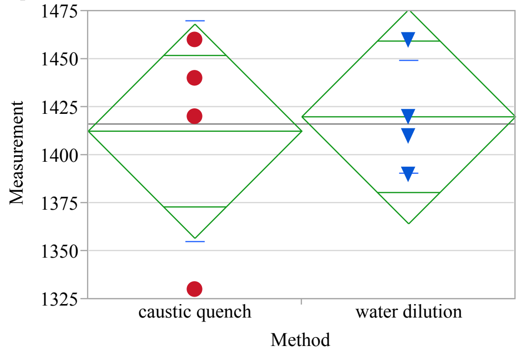
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.6650	1	6	0.4460
Brown-Forsythe	1.5429	1	6	0.2605
Levene	**1.7882**	**1**	**6**	**0.2296**
Bartlett	0.1531	1	.	0.6956
F Test 2-sided	1.6331	3	3	0.6968

Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
114.1311	1	5.6721	<.0001*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SRAT Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Sulfate



Oneway Anova
Summary of Fit

Rsquare	0.008937
Adj Rsquare	-0.15624
Root Mean Square Error	45.59788
Mean of Response	1416.25
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	7.500	t Ratio	0.232612
Std Err Dif	32.243	DF	6
Upper CL Dif	86.395	Prob > |t|	0.8238
Lower CL Dif	-71.395	Prob > t	0.4119
Confidence	0.95	Prob < t	0.5881

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	112.500	112.50	0.0541	**0.8238**
Error	6	12475.000	2079.17		
C. Total	7	12587.500			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1412.50	22.799	1356.7	1468.3
water dilution	4	1420.00	22.799	1364.2	1475.8

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	7.500	t Ratio	0.232612
Std Err Dif	32.243	DF	4.477335
Upper CL Dif	93.379	Prob > |t|	0.8263
Lower CL Dif	-78.379	Prob > t	0.4132
Confidence	0.95	Prob < t	0.5868

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	57.37305	41.25000	37.50000
water dilution	4	29.43920	20.00000	20.00000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.8670	1	6	0.3877
Brown-Forsythe	0.5589	1	6	0.4830
Levene	**1.3318**	**1**	**6**	**0.2924**
Bartlett	1.0688	1	.	0.3012
F Test 2-sided	3.7981	3	3	0.3020

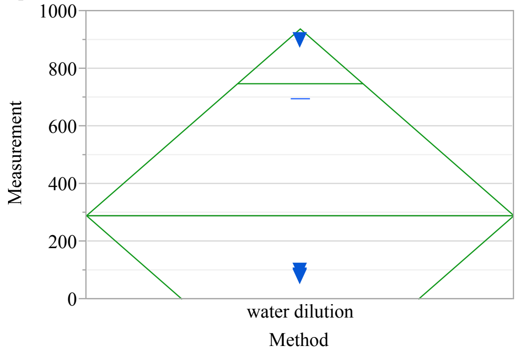
Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
0.0541	1	4.4773	0.8263

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Chloride



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	407.5205
Mean of Response	289.875
Observations (or Sum Wgts)	4

Analysis of Variance

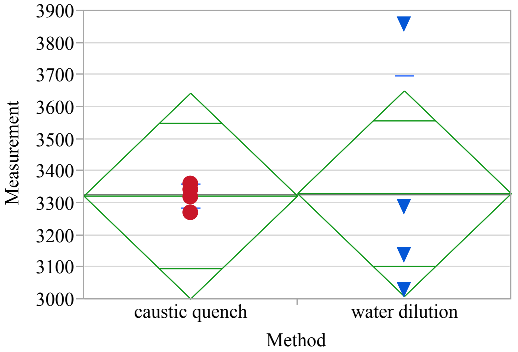
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.00	.	.	.
Error	3	498218.81	166073		
C. Total	3	498218.81			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
water dilution	4	289.875	203.76	-358.6	938.33

Std Error uses a pooled estimate of error variance

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Formate



Oneway Anova
Summary of Fit

Rsquare	0.000272
Adj Rsquare	-0.16635
Root Mean Square Error	262.3849
Mean of Response	3326.25
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	7.50	t Ratio	0.040424
Std Err Dif	185.53	DF	6
Upper CL Dif	461.49	Prob > |t|	0.9691
Lower CL Dif	-446.49	Prob > t	0.4845
Confidence	0.95	Prob < t	0.5155

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	112.50	112.5	0.0016	**0.9691**
Error	6	413075.00	68845.8		
C. Total	7	413187.50			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	3322.50	131.19	3001.5	3643.5
water dilution	4	3330.00	131.19	3009.0	3651.0

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	7.50	t Ratio	0.040424
Std Err Dif	185.53	DF	3.065704
Upper CL Dif	590.86	Prob > |t|	0.9702
Lower CL Dif	-575.86	Prob > t	0.4851
Confidence	0.95	Prob < t	0.5149

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	38.6221	27.5000	27.5000
water dilution	4	369.0528	265.0000	245.0000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.6831	1	6	0.2422
Brown-Forsythe	2.5448	1	6	0.1618
Levene	**5.2410**	**1**	**6**	**0.0620**
Bartlett	8.0993	1	.	0.0044
F Test 2-sided	91.3073	3	3	0.0038

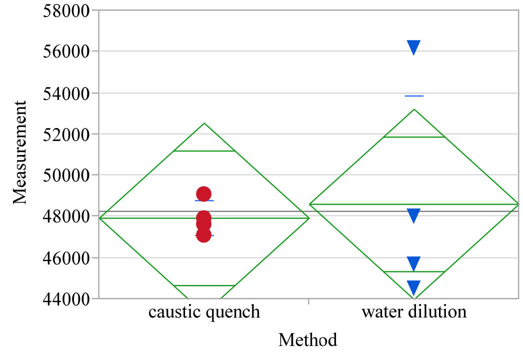
Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
0.0016	1	3.0657	0.9702

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Glycolate



Oneway Anova
Summary of Fit

Rsquare	0.010545
Adj Rsquare	-0.15436
Root Mean Square Error	3775.083
Mean of Response	48262.5
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	675.0	t Ratio	0.252867
Std Err Dif	2669.4	DF	6
Upper CL Dif	7206.8	Prob > |t|	0.8088
Lower CL Dif	-5856.8	Prob > t	0.4044
Confidence	0.95	Prob < t	0.5956

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	911250	911250	0.0639	**0.8088**
Error	6	85507500	14251250		
C. Total	7	86418750			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	47925.0	1887.5	43306	52544
water dilution	4	48600.0	1887.5	43981	53219

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	675.0	t Ratio	0.252867
Std Err Dif	2669.4	DF	3.155942
Upper CL Dif	8937.6	Prob > |t|	0.8160
Lower CL Dif	-7587.6	Prob > t	0.4080
Confidence	0.95	Prob < t	0.5920

Tests that the Variances are Equal

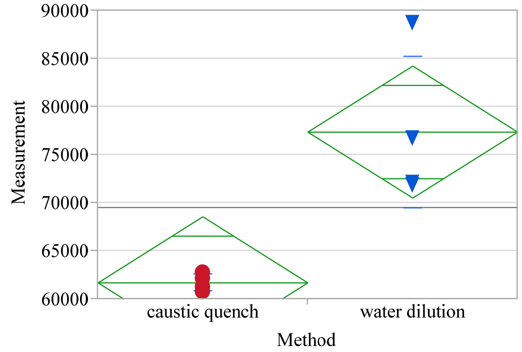
Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	850.000	587.500	575.000
water dilution	4	5270.674	3800.000	3500.000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.6182	1	6	0.2504
Brown-Forsythe	2.1588	1	6	0.1921
Levene	**4.6969**	**1**	**6**	**0.0733**
Bartlett	5.9513	1	.	0.0147
F Test 2-sided	38.4498	1	3	0.0136

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
0.0639	1	3.1559	0.8160

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Nitrate



Oneway Anova
Summary of Fit

Rsquare	0.722537
Adj Rsquare	0.676294
Root Mean Square Error	5608.141
Mean of Response	69537.5
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	15675.0	t Ratio	3.952789
Std Err Dif	3965.6	DF	6
Upper CL Dif	25378.4	Prob > |t|	0.0075*
Lower CL Dif	5971.6	Prob > t	0.0038*
Confidence	0.95	Prob < t	0.9962

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	491411250	491411250	15.6245	**0.0075***
Error	6	188707500	31451250		
C. Total	7	680118750			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	61700.0	2804.1	54839	68561
water dilution	4	77375.0	2804.1	70514	84236

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	15675.0	t Ratio	3.952789
Std Err Dif	3965.6	DF	3.074672
Upper CL Dif	28123.5	Prob > |t|	0.0276*
Lower CL Dif	3226.5	Prob > t	0.0138*
Confidence	0.95	Prob < t	0.9862

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	879.394	700.000	700.000
water dilution	4	7882.206	5662.500	5375.000

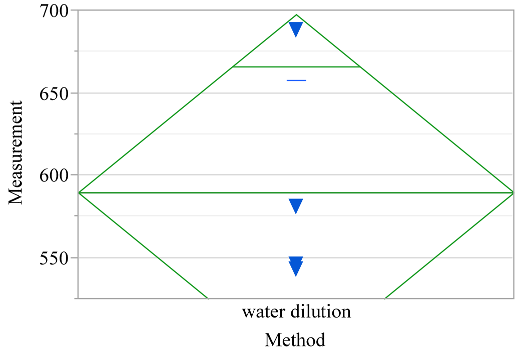
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.7231	1	6	0.2373
Brown-Forsythe	2.4881	1	6	0.1658
Levene	**5.0523**	**1**	**6**	**0.0656**
Bartlett	7.7778	1	.	0.0053
F Test 2-sided	80.3394	1	3	0.0046

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
15.6245	1	3.0747	0.0276*

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Nitrite



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	67.89453
Mean of Response	589.5
Observations (or Sum Wgts)	4

Analysis of Variance

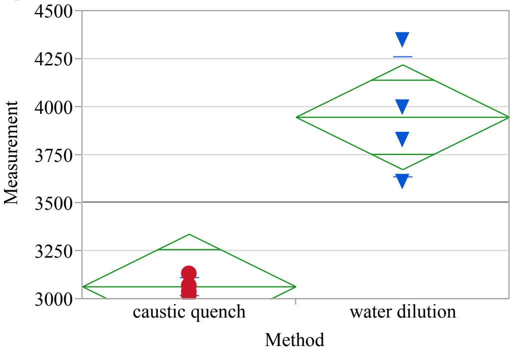
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	0	0.000	.	.	.
Error	3	13829.000	4609.67		
C. Total	3	13829.000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
water dilution	4	589.500	33.947	481.46	697.54

Std Error uses a pooled estimate of error variance

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Oxalate



Oneway Anova
Summary of Fit

Rsquare	0.838785
Adj Rsquare	0.811915
Root Mean Square Error	223.3738
Mean of Response	3506.25
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	882.50	t Ratio	5.587243
Std Err Dif	157.95	DF	6
Upper CL Dif	1268.99	Prob > |t|	0.0014*
Lower CL Dif	496.01	Prob > t	0.0007*
Confidence	0.95	Prob < t	0.9993

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	1557612.5	1557613	31.2173	**0.0014***
Error	6	299375.0	49896		
C. Total	7	1856987.5			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	3065.00	111.69	2791.7	3338.3
water dilution	4	3947.50	111.69	3674.2	4220.8

Std Error uses a pooled estimate of error variance

t Test
water dilution-caustic quench
Assuming equal variances

Difference	882.50	t Ratio	5.587243
Std Err Dif	157.95	DF	3.141472
Upper CL Dif	1372.60	Prob > |t|	0.0100*
Lower CL Dif	392.40	Prob > t	0.0050*
Confidence	0.95	Prob < t	0.9950

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	47.9583	35.0000	35.0000
water dilution	4	312.2366	227.5000	227.5000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	2.1695	1	6	0.1912
Brown-Forsythe	4.8295	1	6	0.0703
Levene	**5.0849**	**1**	**6**	**0.0650**
Bartlett	6.1899	1	.	0.0128
F Test 2-sided	42.3877	3	3	0.0118

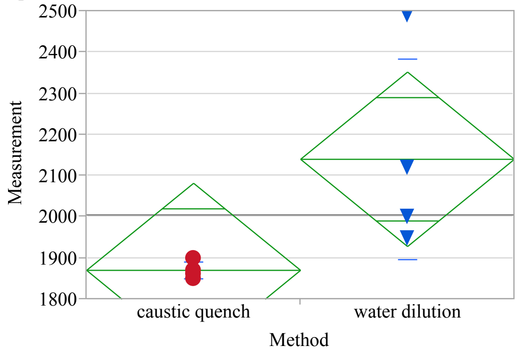
Warning: Small sample sizes. Use Caution.

Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
31.2173	1	3.1415	0.0100*

Exhibit A7. Analysis of Variance Investigating the Anion Means and Variances by Preparation Methods *(continued)*

Oneway Analysis of Measurement By Method Type of Sample=process, Type of Material=Filtered SME Product, Units=mg/kg of filtrate, LTD or Not Reported=no, Anion=Sulfate



Oneway Anova
Summary of Fit

Rsquare	0.447514
Adj Rsquare	0.355433
Root Mean Square Error	173.2051
Mean of Response	2005
Observations (or Sum Wgts)	8

t Test
water dilution-caustic quench
Assuming equal variances

Difference	270.00	t Ratio	2.204541
Std Err Dif	122.47	DF	6
Upper CL Dif	569.68	Prob > |t|	0.0697
Lower CL Dif	-29.68	Prob > t	0.0348*
Confidence	0.95	Prob < t	0.9652

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Method	1	145800.00	145800	4.8600	**0.0697**
Error	6	180000.00	30000		
C. Total	7	325800.00			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
caustic quench	4	1870.00	86.603	1658.1	2081.9
water dilution	4	2140.00	86.603	1928.1	2351.9

Std Error uses a pooled estimate of error variance
t Test
water dilution-caustic quench
Assuming equal variances

Difference	270.00	t Ratio	2.204541
Std Err Dif	122.47	DF	3.04703
Upper CL Dif	656.39	Prob > |t|	0.1133
Lower CL Dif	-116.39	Prob > t	0.0567
Confidence	0.95	Prob < t	0.9433

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
caustic quench	4	21.6025	15.0000	15.0000
water dilution	4	243.9945	175.0000	165.0000

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.7285	1	6	0.2366
Brown-Forsythe	2.8154	1	6	0.1444
Levene	**5.4276**	**1**	**6**	**0.0587**
Bartlett	8.9434	1	.	0.0028
F Test 2-sided	127.5714	3	3	0.0023

Warning: Small sample sizes. Use Caution.
Welch's Test
Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
4.8600	1	3.047	0.1133

Distribution:

T. B. Brown, 773-A
M. E. Cercy, 773-42A
D. A. Crowley, 773-43A
D. E. Dooley, 773-A
A. P. Fellinger. 773-42A
S. D. Fink, 773-A
C. C. Herman, 773-A
D. T. Hobbs, 773-A
E. N. Hoffman, 999-W
J. E. Hyatt, 773-A
K. M. Kostelnik, 773-42A
B. B. Looney, 773-42A
D. A. McGuire, 773-42A
T. O. Oliver, 773-42A
F. M. Pennebaker, 773-42A
G. N. Smoland, 773-42A
B. J. Wiedenman, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)
J. M. Bricker, 766-S
J. S. Contardi, 704-56H
T. L. Fellinger, 766-H
E. J. Freed, 704-S
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. W. Holtzscheiter, 766-H
J. F. Iaukea, 704-S
V. Jain, 766-H
C. J. Martino, 999-W
J. W. Ray, 704-27S
P. J. Ryan, 704-26S
M. A. Rios-Armstrong, 766-H
H. B. Shah, 766-H
D. C. Sherburne, 249-8H
C. B. Sudduth, 707-7E
P. R. Jackson, DOE-SR, 703-46A
J. A. Crenshaw, 703-46A
M. A. Broome, 704-29S
C. G. Sherer, 773-41A
L. C. Jamison, 704-27S
A. B. Chandler, 704-27S
A. Samadi, 704-27S

T. E. Colleran, 773-67A
A. T. Clare, 773-67A
M. C. Clark, 773-67A
S. T. Isom, 773-67A