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# **Testing of the Defense Waste Processing Facility Cold Chemical Dissolution Method in Sludge Batch 10 Qualification**

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July 2022

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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) tests the applicability of the digestion methods used by the DWPF Laboratory for elemental analysis of Sludge Receipt and Adjustment Tank (SRAT) Receipt samples and SRAT Product process control samples. DWPF SRAT samples are typically dissolved using a method referred to as the DWPF Cold Chemical or Cold Chem Method (CC), (see DWPF Procedure SW4-15.201-2.1).

This study confirmed that there is no single method that is ideal for determining all of the elements of interest in high level sludge at the Savannah River Site (SRS). However, elemental analyses performed after the CC method did not reveal extreme biases for the major elements ( $\geq 0.5$  weight percent of dry solids basis) in the sludge when compared with analyses obtained following dissolution by hot aqua regia (AR) or sodium peroxide fusion (PF) methods (even with possible residual solids and fluoride-based precipitates present for the CC method). Statistical determinations were made for a total of 19 elements. The sample digestions comprised of 13 elements with at least 0.1 weight percent (wt. %) on a dry solids basis and 8 elements (Al, Ca, Fe, Hg, Mn, Na, Th, and U) with at least 0.5 wt. % on a dry solids basis. Of the 8 elements that comprise at least 0.5 wt. % of Sludge Batch 10 (SB10) sludge on a dry solids basis, the CC method produced statistically equivalent results for Al (PF), Fe (AR), and Mn (AR) for the SRAT Receipt, and Al (PF), Ca (AR), Fe (AR), Mn (AR), and Th (PF) for the SRAT Product samples. For the same 8 elements, Ca, Hg, and Th were reported for the CC method to be biased statistically lower than the other methods when deviations between the means of the methods were observed. The CC method produced Na and U determinations that were statistically higher than those from the AR and PF method for both SRAT Receipt and Product samples. Furthermore, the Si determinations were precluded in the CC method because of HF reacting with the quartz components of the inductively coupled plasma – optical emission spectroscopy (ICP-OES) sample introduction system leading to a high Si bias.

The CC elemental analyses agreed with the AR and PF methods well enough that it should be adequate for routine process control analyses in the DWPF after much more extensive side-by-side tests of the CC method and the PF method are performed on the first 10 SRAT cycles of the SB10 campaign. The DWPF Laboratory should continue with their plans for further tests of the CC method during these 10 SRAT cycles. If a consistent difference in elemental concentrations is revealed, another type of digestion (i.e. PF) should be used to determine the concentration of the element in question.

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## LIST OF ABBREVIATIONS

ACSM	Analytical Characterization and Sample Management
ANOVA	Analysis of Variance
AR	Aqua Regia
ARG	Analytical Reference Glass
CC	Cold Chem
CV	Coefficient of Variation
DMA	Direct Mercury Analyzer
DWPF	Defense Waste Processing Facility
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
PF	Sodium Peroxide Fusion
SB9	Sludge Batch 9
SB10	Sludge Batch 10
SRAT	Sludge Receipt and Adjustment Tank
SRE	Sodium Reactor Experiment
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TS	Total Solids
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UOM	Unit of Measure
wt. %	Weight Percent
%RSD	Percent Relative Standard Deviation

## 1.0 Introduction

For each sludge batch that is processed in the Defense Waste Processing Facility (DWPF), the Savannah River National Laboratory (SRNL) tests the applicability of the digestion methods used by the DWPF lab for elemental analysis of Sludge Receipt and Adjustment Tank (SRAT) Receipt samples and SRAT Product process control samples. The DWPF Cold Chemical or Cold Chem (CC) Method has historically been shown to be effective for the routine elemental analyses of process control samples from the first 10 SRAT cycles of previous sludge batches.<sup>1-5</sup> 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, whether these biases are acceptable. The past reports have verified that the CC method presents no unacceptable biases in comparison to the aqua regia (AR) and sodium peroxide fusion (PF) digestion methods that have been used to analyze sludge over many years at SRNL. This task is governed by a Task Technical and Quality Assurance Plan (TTQAP) and Technical Task Request (TTR).<sup>6,7</sup>

Sample digestion is required to prepare samples for elemental analysis techniques such as inductively coupled plasma – optical emission spectroscopy (ICP-OES). The CC method typically involves dissolving DWPF SRAT samples with high concentrations of HF-HNO<sub>3</sub> acid at room temperature (see DWPF Procedure SW4-15.201-2.1).<sup>8</sup> The CC method was implemented as a faster and more convenient sample preparation method than that of the tandem sample preparation methods that employ heating, specifically the hot AR and PF methods performed at 115 and 675 °C, respectively.<sup>9-11</sup> However, since no heating is utilized for the CC method, it is considered to be a less rigorous method than those based on heating and should be reviewed periodically for its effectiveness in the DWPF process.

This report contains the statistical analysis and comparison of elemental analysis data of SRAT Receipt and SRAT Product samples generated during the SB10 demonstration in the SRNL Shielded Cell Facility following dissolutions by the CC, AR, and PF methods. Determinations were made for a total of 19 elements based on elemental analyses obtained from ICP-OES and a direct mercury analyzer (DMA) for the Hg results. The DWPF Laboratory does not use the CC method for Hg determinations, however, the results for the determinations of Hg from the CC method are included in this report to determine the efficacy of the CC method for Hg in SB10 sludge. This study has demonstrated that there is no single method that is perfect for determining all of the elements of interest in Savannah River Site (SRS) high level sludge. Despite incomplete dissolution of solids in the SRAT Receipt/Product sludges noted herein and in previous reports and small disparities between digestions methods, 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 Sludge Batch 10 (SB10) sludge. Furthermore, statistically equivalent results and any biases of elements are presented herein.

## 2.0 Experimental Procedure

Two three-liter qualification samples of Tank 51H sludge (samples HTF-51-19-114 and HTF-51-20-15) were evaluated to compare the DWPF CC Method against SRNL digestion methods for SB10 radioactive sludge slurries. For the SRAT Receipt samples, SRNL mixed and then washed the Tank 51H samples per the Tank Farm washing strategy.<sup>12</sup> During washing, sodium reactor experiment (SRE) solutions from Tanks 16.3 and 16.4 of H-Canyon were also added, similar to the addition performed for SB10.<sup>12</sup> More details pertaining to the preparation of the SRAT Receipt samples are found in SRNL-STI-2021-00449.<sup>12</sup> For the SRAT Product samples, the SRAT Receipt samples were run through a SRAT process in the SRNL Shielded Cell Facility (see run plan SRNL-L3120-2021-00002).<sup>13,14</sup> The SRAT process for the SRNL samples included mixing, purging, and heating to 93 °C followed by nitric acid, antifoam, and glycolic acid additions and then refluxing under nominal DWPF conditions. The sludge samples were dissolved in

quadruplicate within the SRNL Shielded Cells facility in a manner similar to the DWPF CC method, and by following the PF and AR digestion methods given below.

### 2.1 CC Method

The CC method digestion (see DWPF Procedure SW4-15.201-2.1) consisted of adding 25 mL of concentrated HF to 2.7–3.0 g of the sludge and stirring this mixture for 1 h.<sup>8</sup> Next, 25 mL of concentrated HNO<sub>3</sub> was added and the resulting mixture was stirred for an additional 30 min. Fine, dark, slow-settling solids remained in each digestion bottle after the acid additions and stirring. The dissolution mixture was diluted with de-ionized water to 250 mL in a plastic volumetric flask.

### 2.2 PF Method

The PF method (see ADS-2502, Rev. 7) consisted of weighing ~1.5 g of sludge into a Zr crucible. Water was removed from the samples by drying at 115 °C in a drying oven.<sup>11</sup> Next, 2.5 g of sodium peroxide was added to the dry sample within the crucible. The crucible was placed inside a muffle furnace that was pre-heated to 675 °C. After the temperature of the muffle furnace equilibrated to 675 °C, the crucible was heated for 15 min. The crucible was then removed from the furnace and allowed to cool for 10 min before dissolving the fusion residue with de-ionized water and concentrated HNO<sub>3</sub> (typically 20 mL HNO<sub>3</sub>). The solution was diluted to 100 mL in a plastic volumetric flask. No solids were apparent after the PF method.

### 2.3 AR Method

The AR method (see ADS-2226, Rev. 10) consisted of weighing ~1.5 g of sludge into a thick-walled Teflon pressure vessel and adding 9 mL of concentrated HCl and 3 mL of concentrated HNO<sub>3</sub>.<sup>10</sup> The Teflon pressure vessel was capped and sealed with a mechanical capping station. After all the SRAT samples, the blank, and the standard glass sample had been prepared, the vessels were placed in a drying oven pre-heated to 115 °C. The vessels were heated for 2 h after the oven temperature equilibrated to 115 °C. After cooling for 30 min, 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.

### 2.4 Blank, Standards and Analytical Information

Dissolution blanks for each of the methods were created by concurrently performing each dissolution method with only the reagents. The CC method was performed on the same day for both the SRAT Receipt and SRAT Product samples (during one dissolution evolution) and then analyzed in the instrumental laboratories on the same day. Therefore, only one blank for the CC method was submitted and utilized. In contrast, the AR and PF methods were performed on SRAT Receipt and SRAT Product samples on separate days and were then subsequently analyzed in the instrumental laboratories on separate days, necessitating the use of independent blanks.

The ARG-1 standard was dissolved concurrently with the SRAT samples and the blanks. The ARG-1 analysis provides information on the overall process of weighing samples in the cells, carrying out the dissolution, and elemental analysis by ICP-OES of a material with similar elemental composition to sludge. A multi-element standard solution to check on the accuracy of the ICP-OES instrument was not utilized for this report.

The CC digestions were suspended with a magnetic stir bar just prior to taking a 15 mL aliquot to homogenize any residual solids or precipitates in the samples (the other digestion methods are inverted 5 times before sampling). Aliquots of each digestion method (15 mL) were taken so that the samples could be removed from the Shielded Cells and characterized. The aliquots were transported to the ICP-OES and DMA instrumental laboratories to be further analyzed by the SRNL Analytical Characterization and Sample

Management (ACSM) group. For ICP-OES, each analyte was analyzed at two different dilutions using at least two different wavelengths. The DMA method was implemented at SRNL after the last CC method comparison was performed and is currently the only analytical method that SRNL uses for Hg.<sup>15</sup> Samples taken for DMA mercury determinations were collected from the CC and AR preps (the PF method was omitted because the digestion temperature is significantly above the volatilization point of Hg). It should be noted that DWPF determines mercury by cold vapor atomic absorption spectroscopy (AAS) in water and with their own digestion method (EPA Method 245.1).

## 2.5 Statistical Analysis

The statistical analysis was performed using JMP® Version 16.0.0.<sup>16</sup> JMP® produced several statistical tests and provided the outputs for this report shown in Appendix A-6. Statistical conclusions were initiated from the results of the Levene test for equality of variances. If there was no evidence that the variances among the preparations differed, the analysis of variance (ANOVA) results were used for testing equality of means for the preparations (this is equivalent to the t-test if there were only two preparations). The Welch's test for testing equality of means was used if the variances among the preparations were not considered to be equal. The p-value (given by the Prob > F value) indicates if there is a statistically significant difference in the variances or the means depending on the statistical test used. If a p-value is greater than 0.05, then the hypothesis of equality cannot be rejected at the 5% significance level. The p-values for testing the variances with the Levene's test are bolded in Appendix A-6. The JMP® output in this appendix also provides additional results for comparison of the means for those elements measured by all three preparations. This statistical evaluation assumes that the variances of the methods are equal and makes comparisons across all pairs of means and highlights those that are statistically different at the 5% significance level.

## 2.6 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 Procedure 2.60.<sup>17</sup> The TTR clarifies the tasks that are considered having a functional classification of Safety Class. Because this task is not in that category, this task is considered to have a functional classification of Production Support. Thus, this report has received a design check per Section 5.2 of Manual E7 Procedure 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.<sup>18</sup> Experimental details and the Design Checklists for this report are stored in the PerkinElmer E-Notebook system as experiments (M1511-0550-03).

## 3.0 Results and Discussion

The elemental composition of the samples prepared from the three digestion methods were reported on a weight percent (wt. %) dry solids basis. The elemental wt. % for each sample was calculated by dividing the respective wt. % total solids (TS) (15.0 and 20.1 wt. % for the SRAT Receipt and SRAT Product, respectively) from the unit of measure (UOM) for the digestion results (microgram of element per gram of SRAT slurry material).<sup>12,19,20</sup> The UOM is the actual value given from the instrument, only corrected for any dilutions performed in the Shielded Cells and by analytical. The measurements of the blanks were handled in the same manner as the SRAT samples. For the sample blanks, sample weights were supplied to the ICP-OES and DMA laboratories that were close to that of the actual samples used for the various digestion methods. This allowed for the blank concentrations to be converted from a µg/g basis to wt. % with a dry solids basis (again, 15.0 and 20.1 wt. % for the SRAT Receipt and SRAT Product, respectively). Blanks were included with each data set to aid in confirming the accuracy of the instrumental analysis for each element. The data associated with the blanks used for each set of SRAT sludge analyses are shown in Table 3-1. The blanks indicated that minimal error was introduced into the digestion analysis from the reagents. The blanks are further of use to compare the background noise of the instrument to the sample data for low wt. % elements. As such, Ca was observed in Table 3-1, indicating it as a minor contaminant that was likely introduced from a reagent used in the PF method.

**Table 3-1. Measurements of Blanks for the SB10 digestion method evaluation on a SRAT Receipt and Product**

Type - Prep	SRAT Receipt & Product - CC	SRAT Receipt - AR	SRAT Receipt - PF	SRAT Product - AR	SRAT Product - PF	SRAT Receipt & Product - CC	SRAT Receipt - AR	SRAT Receipt - PF	SRAT Receipt & Product - CC	SRAT Product - AR	SRAT Product - PF
LIMS	25488	21536	21542	24337	24170	25488	21536	21542	25488	24337	24170
UOM	µg/g slurry	µg/g slurry	µg/g slurry	µg/g slurry	µg/g slurry	wt. % TS	wt. % TS	wt. % TS	wt. % TS	wt. % TS	wt. % TS
Ag	< 16.9	< 14	< 36.6	< 15.7	< 109	< 0.011	< 0.009	< 0.024	< 0.008	< 0.008	< 0.054
Al	< 54.9	< 17.3	< 122	< 20.6	< 98.6	< 0.037	< 0.012	< 0.081	< 0.027	< 0.01	< 0.049
B	< 172	< 140	< 134	< 11.6	< 14.7	< 0.115	< 0.093	< 0.089	< 0.086	< 0.006	< 0.007
Ba	< 1.57	< 1.12	< 0.984	< 0.322	< 1.55	< 0.001	< 0.001	< 0.001	< 0.001	< 0.0002	< 0.001
Be	< 1.45	< 3.82	< 4.12	< 0.257	< 0.506	< 0.001	< 0.003	< 0.003	< 0.001	< 0.0001	< 0.0003
Ca	75	< 28.9	353	< 15.2	426	0.05	< 0.019	0.235	0.037	< 0.008	0.212
Cd	< 7.14	< 6.74	< 6.45	< 6.39	< 5.82	< 0.005	< 0.004	< 0.004	< 0.004	< 0.003	< 0.003
Ce	< 205	< 8.82	< 21.3	< 194	< 383	< 0.137	< 0.006	< 0.014	< 0.102	< 0.097	< 0.191
Co	< 4.91	< 10.1	< 7.63	< 4.27	< 3.88	< 0.003	< 0.007	< 0.005	< 0.002	< 0.002	< 0.002
Cr	< 0.851	< 17.6	< 17.6	< 0.225	< 7.41	< 0.001	< 0.012	< 0.012	< 0.0004	< 0.0001	< 0.004
Cu	< 69.7	< 11.6	< 22.1	< 70.1	< 54.8	< 0.046	< 0.008	< 0.015	< 0.035	< 0.035	< 0.027
Fe	29.5	< 7.36	< 5.55	< 6.51	< 52.6	0.02	< 0.005	< 0.004	0.015	< 0.003	< 0.026
Gd	< 122	< 3.56	< 9.69	< 0.598	< 4.23	< 0.081	< 0.002	< 0.006	< 0.061	< 0.0003	< 0.002
Hg	< 0.01	< 0.01	<i>Note 1.</i>	< 0.182	<i>Note 1.</i>	< 0.000007	< 0.000007	<i>Note 1.</i>	< 0.000005	< 0.00009	<i>Note 1.</i>
K	< 183	< 122	< 262	< 88.6	< 425	< 0.122	< 0.081	< 0.175	< 0.091	< 0.044	< 0.211
La	< 1.02	< 2.9	< 2.9	< 0.497	< 2.39	< 0.001	< 0.002	< 0.002	< 0.001	< 0.0002	< 0.001
Li	< 28.3	< 13.3	< 10.2	< 14.1	< 55.5	< 0.019	< 0.009	< 0.007	< 0.014	< 0.007	< 0.028
Mg	26.3	< 2.13	< 1.69	< 0.909	< 4.36	0.018	< 0.001	< 0.001	0.013	< 0.0005	< 0.002
Mn	< 0.556	< 3.73	< 1.33	< 0.478	< 4.41	< 0.0004	< 0.002	< 0.001	< 0.0003	< 0.0002	< 0.002
Mo	< 13.9	< 12.4	< 16.1	< 0.45	< 11.3	< 0.009	< 0.008	< 0.011	< 0.007	< 0.0002	< 0.006
Na	< 73.6	< 118	<i>Note 2.</i>	< 20.5	<i>Note 2.</i>	< 0.049	< 0.079	<i>Note 2.</i>	< 0.037	< 0.01	<i>Note 2.</i>
Ni	< 1.91	< 5.23	< 9.03	< 0.793	< 3.81	< 0.001	< 0.003	< 0.006	< 0.001	< 0.0004	< 0.002
P	< 8.32	< 193	< 151	< 2.41	< 11.6	< 0.006	< 0.129	< 0.101	< 0.004	< 0.001	< 0.006
Pb	< 44.5	< 19.6	< 9.98	< 41.1	< 33.7	< 0.03	< 0.013	< 0.007	< 0.022	< 0.02	< 0.017
S	< 26.3	< 116	< 116	< 5.87	< 28.2	< 0.018	< 0.077	< 0.077	< 0.013	< 0.003	< 0.014
Sb	< 9.19	< 28.1	< 28.1	< 4.48	< 8.7	< 0.006	< 0.019	< 0.019	< 0.005	< 0.002	< 0.004
Si	<i>Note 3.</i>	< 85.3	< 47.1	< 7.27	< 14.9	<i>Note 3.</i>	< 0.057	< 0.031	<i>Note 3.</i>	< 0.004	< 0.007
Sn	< 57.7	< 40.8	< 40.8	< 15.4	< 73.9	< 0.038	< 0.027	< 0.027	< 0.029	< 0.008	< 0.037
Sr	< 0.647	< 0.757	< 4.59	< 0.157	< 3.09	< 0.0004	< 0.001	< 0.003	< 0.0003	< 0.0001	< 0.002
Th	< 3.09	< 38.3	< 55.1	< 27.4	< 178	< 0.002	< 0.026	< 0.037	< 0.002	< 0.014	< 0.089
Ti	< 1.43	< 23	< 26.8	< 0.693	< 4.58	< 0.001	< 0.015	< 0.018	< 0.0007	< 0.0003	< 0.002
U	< 23.9	< 45.5	< 45.5	< 9.64	< 46.2	< 0.016	< 0.03	< 0.03	< 0.012	< 0.005	< 0.023
V	< 9.04	< 5.57	< 5.57	< 2.71	< 13.0	< 0.006	< 0.004	< 0.004	< 0.0045	< 0.001	< 0.006
Zn	< 0.39	< 6.89	< 7.48	< 0.643	< 1.88	< 0.0003	< 0.005	< 0.005	< 0.0002	< 0.0003	< 0.001
Zr	< 1.12	< 1.41	<i>Note 4.</i>	< 0.41	<i>Note 4.</i>	< 0.001	< 0.001	<i>Note 4.</i>	< 0.0006	< 0.0002	<i>Note 4.</i>

Note 1. Hg was not measured by DMA for the PF method because the 675 °C temperature is above the volatilization point for Hg.  
Note 2. Na is not measured by ICP-OES in PF because of Na introduction in the reagent.  
Note 3. Si is not measured by ICP-OES for the CC method due to extremely high levels observed in past analyses resulting from the high concentration of HF which attacks the quartz ICP-OES sample introduction system.  
Note 4. Zr is not measured by ICP-OES in PF because a Zr crucible is used.

Overall, a less than sign in the tabular data indicates that the analysis could not confidently report a value within 10% uncertainty (2-σ) for the analytical method used and should be recognized when reading the tables. For example, Ce in the SRAT Receipt gave an average wt. % of < 0.1297 for the CC method, which is a higher value than the other two methods but does not necessarily mean that the CC method is at a higher average wt. % than the AR and PF methods. In most cases, this is due to the concentration of the element

in the analyte being less than the detection limit for that specific element. It is not a prep or contamination issue.

Furthermore, a few elements such as Hg, Na, Si, and Zr were not submitted to analytical for specific digestion methods due to inherent issues for that element with the particular methods and are omitted throughout this report. Hg was not measured by DMA for the PF method because the digestion is performed in an open crucible at a temperature that is significantly above the volatilization point of Hg. Na was not submitted for ICP-OES analysis for the PF samples due to being introduced as a reagent during the PF method. Si was not measured by ICP-OES for the CC method due to a high concentration of HF in the analyte, which attacks the quartz in the ICP-OES sample introduction system resulting in extremely high levels being observed in past analyses.<sup>21</sup> Zr was not measured by ICP-OES for the PF method because it is a known contaminant introduced from the Zr crucible used.

In addition to the blanks and SRAT samples, ARG-1 standards were dissolved and analyzed concurrently. The ARG-1 glass samples were used to further validate the analytics, digestion procedure and nuances of performing work inside of the Shielded Cells Facility. A direct comparison of the analytics should be considered lightly since glass and sludge have different physical properties and compositions. The measurements for the ARG-1 samples associated with each set of SRAT sludge samples are shown in Table 3-2. No glass samples were submitted or prepared for Hg analysis by DMA. Reference ICP-OES data for the standard glass are also given in Table 3-2.<sup>21,22</sup> Elemental values of the reference data were calculated by dividing the oxide values of the Corning Inc. Glass Corporation glass by each respective gravimetric factor. Most elements were found to be at compositions near the expected reference values.

The data for Al differed from the reference glass for the CC and AR methods. Al may have issues during digestion by the CC and AR methods if specific crystalline structures are present. The AR method gave a significantly lower value for Si than that of the reference and PF method. The prior sludge batch reports agreed with this finding and noted that Si, from glass, was insoluble in AR.<sup>21</sup> Ti was also observed to be slightly less prevalent in the CC method and even lower for the AR method. For Zr in ARG-1, the opposite findings were true, where CC method gave good results compared to the AR method being notably lower.

**Table 3-2. Measurements (wt. %) for the ARG-1 Samples**

Type – Prep	Reference Value for ARG-1 (wt. %) <sup>21,22</sup>	SRAT Receipt & Product – CC (wt. %)	SRAT Receipt – AR (wt. %)	SRAT Product – AR (wt. %)	SRAT Receipt – PF (wt. %)	SRAT Product – PF (wt. %)
Element/LIMS		25489	21537	24338	21543	24171
Ag	0	< 0.008	< 0.008	< 0.003	< 0.021	< 0.062
Al	2.5	2.16	2.22	2.26	2.5	2.49
B	2.69	2.51	2.53	2.56	2.54	2.68
Ba	0.079	0.074	0.073	0.074	0.066	0.079
Be	0	0.002	< 0.002	< 0.003	< 0.002	< 0.003
Ca	1.02	0.831	0.994	1.02	1.08	1.36
Cd	0	< 0.004	< 0.004	< 0.001	< 0.004	< 0.003
Ce	0	< 0.102	< 0.005	< 0.038	< 0.012	< 0.218
Co	0	< 0.006	< 0.006	< 0.005	< 0.004	< 0.008
Cr	0.064	0.065	0.062	0.06	0.058	0.064
Cu	0.003	< 0.035	< 0.006	< 0.014	< 0.012	< 0.031
Fe	9.79	9.8	9.81	9.55	9.18	9.74
Gd	0	< 0.061	< 0.002	< 0.002	< 0.005	< 0.004
K	2.26	2.14	2.01	2.19	2.24	2.3
La	0	< 0.001	< 0.002	< 0.001	< 0.002	< 0.001
Li	1.49	1.44	1.45	1.52	1.41	1.52
Mg	0.52	0.494	0.503	0.504	0.486	0.516
Mn	1.46	1.45	1.44	1.47	1.38	1.46
Mo	0	< 0.007	< 0.007	< 0.0002	< 0.009	< 0.006
Na	8.52	8.35	8.16	8.26	<i>Note 1.</i>	<i>Note 1.</i>
Ni	0.827	0.816	0.818	0.825	0.739	0.799
P	0.096	0.107	< 0.106	0.109	0.113	0.105
Pb	0	< 0.025	< 0.011	< 0.008	< 0.006	< 0.019
S	0	< 0.06	< 0.063	< 0.067	< 0.065	< 0.077
Sb	0	< 0.005	< 0.015	< 0.001	< 0.016	< 0.005
Si	22.4	<i>Note 2.</i>	0.223	0.422	20	22.4
Sn	0	< 0.029	< 0.022	< 0.016	< 0.023	< 0.042
Sr	0.003	0.003	< 0.003	0.003	< 0.003	0.005
Th	0	< 0.01	< 0.021	< 0.031	< 0.031	< 0.075
Ti	0.689	0.652	0.564	0.515	0.677	0.741
U	0	< 0.012	< 0.025	< 0.003	< 0.026	< 0.026
V	0	< 0.01	< 0.003	< 0.018	< 0.003	< 0.018
Zn	0.016	0.016	0.015	0.015	0.016	0.016
Zr	0.096	0.096	0.067	0.059	<i>Note 3.</i>	<i>Note 3.</i>
<p>Note 1. Na is not measured by ICP-OES in PF because of introduction as a reagent.  Note 2. Si is not measured by ICP-OES for the CC method because the high HF concentration attacks the quartz sample introduction system of the ICP-OES instrument.  Note 3. Zr is not measured by ICP-OES from the PF method because a Zr crucible is used for the fusion.</p>						



The CC method for dissolving the SRAT Receipt and the SRAT Product samples was then compared to the AR and PF preps. Unlike for the AR and PF methods, visual observation confirmed that the CC method did not completely dissolve both the SRAT Receipt and the SRAT Product sludges. Fine, dark solids remained in solution and were found to slowly dissolve over a few days. The initial CC dissolutions that still contained solids were used for the elemental analyses. Table 3-3 to Table 3-5 provide the measurements of the various elemental concentrations reported as wt. % of TS (a dry solids basis) for the SRAT Receipt and Product samples performed in quadruplicate for each digestion method. The tables are broken up by the elements in alphabetical order. The measurements for elemental concentrations in each sample are reported in  $\mu\text{g/g}$  of slurry in Appendix A-1 through Appendix A-3. The CC and AR digestions were checked a few months after being prepped and solids were noted for both cases. For the CC method, previous reports have attributed this to  $\text{Ca}_{0.5}\text{Th}_{0.5}\text{F}$  and other fluoride precipitates.<sup>5</sup>

No issues with S were observed for the PF dissolutions of SB10 samples unlike that observed for S in the Sludge Batch 9 (SB9) report.<sup>21</sup> Appendix A-4 and A-5 provides variability plots of the elemental wt. % values for the SRAT Product and SRAT Receipt samples, respectively. Appendix A-4 and A-5 are plotted with both linear and log-scale y-axes to help show deviation between the replicates and digestion methods.

**Table 3-3. Measurements of the SB10 SRAT Receipt and Product Samples (Ag-Fe)**

Type	Prep	Sample ID	LIMS	UOM	Ag	Al	B	Ba	Be	Ca	Cd	Ce	Co	Cr	Cu	Fe
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	wt. % of TS	< 0.011	12.3	< 0.113	0.040	< 0.001	0.419	< 0.005	< 0.135	< 0.003	0.156	< 0.046	8.80
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	wt. % of TS	< 0.010	12.4	< 0.103	0.038	< 0.001	0.412	< 0.004	< 0.123	< 0.003	0.159	< 0.042	8.73
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	wt. % of TS	< 0.010	12.7	< 0.107	0.038	< 0.001	0.418	< 0.004	< 0.127	< 0.003	0.164	< 0.043	8.87
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	wt. % of TS	< 0.011	12.5	< 0.113	0.039	< 0.001	0.413	< 0.005	< 0.135	< 0.003	0.163	< 0.046	8.93
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	wt. % of TS	< 0.009	10.5	< 0.090	0.047	< 0.002	0.547	< 0.004	0.112	< 0.006	0.165	0.037	8.93
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	wt. % of TS	< 0.009	7.00	< 0.091	0.043	< 0.002	0.511	< 0.004	0.103	< 0.007	0.154	0.033	8.33
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	wt. % of TS	< 0.009	8.00	< 0.091	0.045	< 0.002	0.533	< 0.004	0.103	< 0.007	0.161	0.035	8.60
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	wt. % of TS	< 0.009	7.73	< 0.087	0.044	< 0.002	0.527	< 0.004	0.102	< 0.006	0.159	0.035	8.47
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	wt. % of TS	< 0.024	11.7	< 0.087	0.048	< 0.003	0.733	< 0.004	0.091	< 0.005	0.160	0.037	8.33
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	wt. % of TS	< 0.022	12.3	< 0.083	0.043	< 0.003	0.673	< 0.004	0.087	< 0.005	0.157	0.034	8.20
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	wt. % of TS	< 0.024	13.1	< 0.088	0.047	< 0.003	0.767	< 0.004	0.092	< 0.005	0.168	0.035	8.67
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	wt. % of TS	< 0.024	12.6	< 0.087	0.050	< 0.003	0.800	< 0.004	0.087	< 0.005	0.164	0.036	8.53
SB10 SRAT Product	CC	TS-211-20-B-263	25494	wt. % of TS	< 0.009	9.35	< 0.088	0.035	< 0.001	0.390	< 0.004	< 0.104	< 0.002	0.128	< 0.036	6.62
SB10 SRAT Product	CC	TS-211-20-B-264	25495	wt. % of TS	< 0.008	9.35	< 0.078	0.035	< 0.001	0.386	< 0.003	< 0.093	< 0.002	0.126	< 0.032	6.52
SB10 SRAT Product	CC	TS-211-20-B-265	25496	wt. % of TS	< 0.008	9.40	< 0.081	0.033	< 0.001	0.388	< 0.003	< 0.096	< 0.002	0.127	< 0.033	6.62
SB10 SRAT Product	CC	TS-211-20-B-266	25497	wt. % of TS	< 0.009	9.25	< 0.087	0.033	< 0.001	0.393	< 0.004	< 0.103	< 0.002	0.127	< 0.035	6.72
SB10 SRAT Product	AR	TS211-20-B-018	24339	wt. % of TS	< 0.007	7.76	< 0.006	0.037	< 0.0001	0.401	< 0.003	< 0.092	< 0.002	0.123	< 0.033	6.52
SB10 SRAT Product	AR	TS211-20-B-019	24340	wt. % of TS	< 0.008	7.81	< 0.006	0.037	< 0.0001	0.403	< 0.003	< 0.094	< 0.002	0.123	< 0.034	6.62
SB10 SRAT Product	AR	TS211-20-B-020	24341	wt. % of TS	< 0.008	7.91	< 0.006	0.037	< 0.0001	0.402	< 0.003	< 0.095	< 0.002	0.122	< 0.034	6.52
SB10 SRAT Product	AR	TS211-20-B-021	24342	wt. % of TS	< 0.008	7.86	< 0.006	0.037	< 0.0001	0.402	< 0.003	< 0.096	< 0.002	0.122	< 0.035	6.62
SB10 SRAT Product	PF	TS211-20-B-026	24172	wt. % of TS	< 0.054	8.81	< 0.007	0.034	< 0.0002	0.622	< 0.003	< 0.189	< 0.002	0.106	< 0.027	5.77
SB10 SRAT Product	PF	TS211-20-B-027	24173	wt. % of TS	< 0.054	8.51	< 0.007	0.035	< 0.0002	0.607	< 0.003	< 0.189	< 0.002	0.097	< 0.027	5.92
SB10 SRAT Product	PF	TS211-20-B-028	24174	wt. % of TS	< 0.053	9.60	< 0.007	0.036	< 0.0002	0.667	< 0.003	< 0.185	< 0.002	0.113	< 0.026	5.970
SB10 SRAT Product	PF	TS211-20-B-029	24175	wt. % of TS	< 0.054	8.91	< 0.007	0.035	< 0.0002	0.557	< 0.003	< 0.189	< 0.002	0.114	< 0.027	5.821

**Table 3-4. Measurements of the SB10 SRAT Receipt and Product Samples (Gd-Pb)**

Type	Prep	Sample ID	LIMS	UOM	Gd	K	La	Li	Mg	Mn	Mo	Na	Ni	P	Pb
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	wt. % of TS	< 0.080	< 0.121	0.030	< 0.019	0.219	2.77	< 0.009	17.1	0.293	0.068	< 0.029
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	wt. % of TS	< 0.073	< 0.110	0.029	< 0.017	0.226	2.79	< 0.008	17.2	0.294	0.072	< 0.027
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	wt. % of TS	< 0.075	< 0.113	0.030	< 0.018	0.226	2.89	< 0.009	17.5	0.298	0.071	< 0.028
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	wt. % of TS	< 0.080	< 0.121	0.030	< 0.019	0.223	2.82	< 0.009	17.3	0.297	0.071	< 0.029
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	wt. % of TS	0.045	< 0.079	0.035	< 0.009	0.219	2.81	< 0.008	16.9	0.296	< 0.124	< 0.013
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	wt. % of TS	0.042	< 0.079	0.032	< 0.009	0.204	2.64	< 0.008	16.5	0.276	< 0.125	< 0.013
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	wt. % of TS	0.044	< 0.080	0.034	< 0.009	0.215	2.73	< 0.008	16.5	0.287	< 0.126	< 0.013
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	wt. % of TS	0.042	< 0.076	0.034	< 0.008	0.212	2.67	< 0.008	16.4	0.279	< 0.120	< 0.012
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	wt. % of TS	0.046	< 0.169	0.022	< 0.007	0.208	2.60	< 0.010	Note 1.	0.261	< 0.097	< 0.006
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	wt. % of TS	0.043	< 0.161	0.021	< 0.006	0.198	2.53	< 0.010	Note 1.	0.259	< 0.093	< 0.006
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	wt. % of TS	0.047	< 0.172	0.022	< 0.007	0.206	2.65	< 0.011	Note 1.	0.266	< 0.099	< 0.007
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	wt. % of TS	0.046	< 0.170	0.022	< 0.007	0.207	2.64	< 0.010	Note 1.	0.266	< 0.098	< 0.006
SB10 SRAT Product	CC	TS-211-20-B-263	25494	wt. % of TS	< 0.062	< 0.094	0.027	< 0.014	0.169	2.21	< 0.007	13.1	0.223	0.054	< 0.023
SB10 SRAT Product	CC	TS-211-20-B-264	25495	wt. % of TS	< 0.055	< 0.083	0.026	< 0.013	0.167	2.20	< 0.006	13.1	0.221	0.053	< 0.020
SB10 SRAT Product	CC	TS-211-20-B-265	25496	wt. % of TS	< 0.057	< 0.086	0.026	< 0.013	0.168	2.20	< 0.007	13.1	0.221	0.054	< 0.021
SB10 SRAT Product	CC	TS-211-20-B-266	25497	wt. % of TS	< 0.062	< 0.093	0.026	< 0.014	0.167	2.22	< 0.007	13.0	0.221	0.049	< 0.023
SB10 SRAT Product	AR	TS211-20-B-018	24339	wt. % of TS	0.037	< 0.042	0.027	< 0.007	0.156	2.21	0.004	12.7	0.210	0.055	< 0.019
SB10 SRAT Product	AR	TS211-20-B-019	24340	wt. % of TS	0.037	< 0.043	0.028	< 0.007	0.157	2.24	0.004	12.8	0.211	0.055	< 0.020
SB10 SRAT Product	AR	TS211-20-B-020	24341	wt. % of TS	0.037	< 0.043	0.027	< 0.007	0.157	2.23	0.004	12.7	0.209	0.054	< 0.020
SB10 SRAT Product	AR	TS211-20-B-021	24342	wt. % of TS	0.037	< 0.044	0.027	< 0.007	0.157	2.23	0.004	12.8	0.209	0.054	< 0.020
SB10 SRAT Product	PF	TS211-20-B-026	24172	wt. % of TS	0.036	< 0.209	0.025	< 0.027	0.143	1.89	< 0.006	Note 1.	0.191	0.044	< 0.017
SB10 SRAT Product	PF	TS211-20-B-027	24173	wt. % of TS	0.036	< 0.209	0.026	< 0.027	0.141	1.96	< 0.006	Note 1.	0.197	0.050	< 0.017
SB10 SRAT Product	PF	TS211-20-B-028	24174	wt. % of TS	0.039	< 0.205	0.026	< 0.027	0.150	1.98	< 0.006	Note 1.	0.201	0.049	< 0.016
SB10 SRAT Product	PF	TS211-20-B-029	24175	wt. % of TS	0.034	< 0.210	0.024	< 0.027	0.138	1.91	< 0.006	Note 1.	0.196	0.046	< 0.017
Note 1. Na is not measured by ICP-OES in PF because of introduction as a reagent.															

**Table 3-5. Measurements of the SB10 SRAT Receipt and Product Samples (S-Zr)**

Type	Prep	Sample ID	LIMS	UOM	S	Sb	Si	Sn	Sr	Th	Ti	U	V	Zn	Zr
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	wt. % of TS	0.395	< 0.006	Note 2.	< 0.038	0.018	1.27	0.013	2.39	< 0.006	0.017	0.113
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	wt. % of TS	0.409	< 0.006	Note 2.	< 0.035	0.017	1.09	0.013	2.41	< 0.005	0.017	0.114
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	wt. % of TS	0.414	< 0.006	Note 2.	< 0.036	0.017	1.19	0.013	2.45	< 0.006	0.018	0.117
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	wt. % of TS	0.411	< 0.006	Note 2.	< 0.038	0.018	1.18	0.013	2.43	< 0.006	0.017	0.116
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	wt. % of TS	0.376	< 0.018	0.307	< 0.026	0.017	1.83	< 0.015	2.41	< 0.004	0.019	0.118
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	wt. % of TS	0.372	< 0.018	0.273	< 0.026	0.015	1.69	< 0.015	2.26	< 0.004	0.018	0.109
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	wt. % of TS	0.376	< 0.018	0.281	< 0.027	0.016	1.77	< 0.015	2.35	< 0.004	0.018	0.113
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	wt. % of TS	0.390	< 0.017	0.256	< 0.025	0.016	1.73	< 0.014	2.29	< 0.003	0.018	0.111
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	wt. % of TS	0.424	< 0.018	0.426	< 0.026	0.017	1.70	< 0.017	2.11	< 0.004	0.017	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	wt. % of TS	0.417	< 0.017	0.411	< 0.025	0.015	1.65	< 0.016	2.08	< 0.003	0.017	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	wt. % of TS	0.429	< 0.018	0.432	< 0.027	0.017	1.75	< 0.018	2.16	< 0.004	0.017	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	wt. % of TS	0.423	< 0.018	0.426	< 0.027	0.016	1.73	< 0.017	2.11	< 0.004	0.017	Note 3.
SB10 SRAT Product	CC	TS-211-20-B-263	25494	wt. % of TS	0.312	< 0.005	Note 2.	< 0.029	0.016	1.11	0.010	1.83	< 0.005	0.014	0.087
SB10 SRAT Product	CC	TS-211-20-B-264	25495	wt. % of TS	0.310	< 0.004	Note 2.	< 0.026	0.016	1.09	0.010	1.81	< 0.004	0.013	0.086
SB10 SRAT Product	CC	TS-211-20-B-265	25496	wt. % of TS	0.310	< 0.004	Note 2.	< 0.027	0.016	1.09	0.010	1.81	< 0.004	0.013	0.087
SB10 SRAT Product	CC	TS-211-20-B-266	25497	wt. % of TS	0.314	< 0.005	Note 2.	< 0.029	0.015	1.08	0.010	1.81	< 0.005	0.014	0.086
SB10 SRAT Product	AR	TS211-20-B-018	24339	wt. % of TS	0.293	< 0.002	0.205	< 0.007	0.016	1.35	0.010	1.72	< 0.001	0.014	0.085
SB10 SRAT Product	AR	TS211-20-B-019	24340	wt. % of TS	0.295	< 0.002	0.203	< 0.007	0.017	1.38	0.010	1.72	< 0.001	0.013	0.086
SB10 SRAT Product	AR	TS211-20-B-020	24341	wt. % of TS	0.292	< 0.002	0.207	< 0.008	0.016	1.32	0.010	1.71	< 0.001	0.013	0.085
SB10 SRAT Product	AR	TS211-20-B-021	24342	wt. % of TS	0.294	< 0.002	0.194	< 0.008	0.016	1.35	0.010	1.71	< 0.001	0.013	0.085
SB10 SRAT Product	PF	TS211-20-B-026	24172	wt. % of TS	0.286	< 0.004	0.306	< 0.036	0.017	1.20	0.011	1.53	< 0.006	0.013	Note 3.
SB10 SRAT Product	PF	TS211-20-B-027	24173	wt. % of TS	0.301	< 0.004	0.324	< 0.036	0.017	1.17	0.010	1.56	< 0.006	0.013	Note 3.
SB10 SRAT Product	PF	TS211-20-B-028	24174	wt. % of TS	0.282	< 0.004	0.318	< 0.036	0.018	1.22	0.012	1.57	< 0.006	0.013	Note 3.
SB10 SRAT Product	PF	TS211-20-B-029	24175	wt. % of TS	0.278	< 0.004	0.405	< 0.037	0.016	1.07	0.011	1.47	< 0.006	0.013	Note 3.
Note 2. Si is not measured by ICP-OES for the CC method because the high HF concentration attacks the quartz sample introduction system of the ICP-OES instrument.															
Note 3. Zr is not measured by ICP-OES from the PF method because a Zr crucible is used for the fusion.															

The applicability and variance between the CC and AR digestion methods for Hg were quantified through DMA. Even though DMA can analyze slurry directly, the instrument used to quantify the digestion should have minimal impact on the results from the digestion methods. If the CC method for Hg was to be implemented, the results from the DMA analysis are reported as ppm of slurry and wt. % of TS values in Table 3-6. Table 3-6 also includes the measurements of the blanks for each set of digestions.

**Table 3-6. Hg Measurements by DMA**

Type Sample	Prep	Sample ID	LIMS	Hg (µg/g)	Hg (wt. % of TS)
SRAT Receipt	CC	BLANK-CC-DMA	25512	< 0.01	0
SRAT Receipt	CC	TS-211-20-B-267	25513	3950	2.63
SRAT Receipt	CC	TS-211-20-B-268	25514	3610	2.41
SRAT Receipt	CC	TS-211-20-B-269	25515	3580	2.38
SRAT Receipt	CC	TS-211-20-B-270	25516	3840	2.56
SRAT Receipt	AR	BLANK-AR	21536	< 0.01	0
SRAT Receipt	AR	TS211-20-A-03501	21538	4580	3.06
SRAT Receipt	AR	TS211-20-A-03502	21539	4200	2.80
SRAT Receipt	AR	TS211-20-A-03503	21540	4330	2.89
SRAT Receipt	AR	TS211-20-A-03504	21541	3970	2.65
SRAT Product	CC	BLANK-CC-DMA	25512	< 0.01	0
SRAT Product	CC	TS-211-20-B-271	25517	1330	0.662
SRAT Product	CC	TS-211-20-B-272	25518	1330	0.564
SRAT Product	CC	TS-211-20-B-273	25519	1180	0.588
SRAT Product	CC	TS-211-20-B-274	25520	1200	0.599
SRAT Product	AR	BLANK-AR-Hg	24343	< 0.182	0
SRAT Product	AR	TS211-20-B-022	24344	1570	0.781
SRAT Product	AR	TS211-20-B-023	24345	1540	0.766
SRAT Product	AR	TS211-20-B-024	24346	1500	0.746
SRAT Product	AR	TS211-20-B-025	24347	1560	0.776

Elemental comparisons of the analyses were made across all of the preparation methods to provide an overview and analysis for each element analyzed. Table 3-7 summarizes these analyses and the main statistical results from Appendix A-6. Trace elements and those that did not give quantifiable results in the ICP-OES analysis for the CC digestion method of sludge were excluded from Table 3-7. The average values for the wt. % measurements of each element for the different preparation method are provided in Table 3-7, along with their respective relative standard deviation (%RSD). The relative standard deviation, also known as the coefficient of variation (CV), is defined as the ratio of the standard deviation to the average and is often expressed as a percentage.

Appendix A-6 provides a detailed statistical evaluation and review for all of the elemental measurements (wt. % on a dry solids basis) of the SRAT Receipt and Product samples. For example, the Levene test reported a p-value of 0.0733 for the Al measurement of the SRAT Receipt samples. This indicates that the hypothesis of equal variances across the three preparation methods cannot be rejected at the 5% significance level for Al across the digestion methods. If an element is rejected, it is denoted as a “yes” in the “Significant Difference in Variances” column of Table 3-7. For example, since the results of the Levene test for Al in the SRAT Receipt did not indicate that the measurement variances were different across the preparation methods, an assumption of equal variance can be made, and the ANOVA results are used for the comparison of the means. The Welch’s test would have been used for an unequal variance case.

Comparisons of the means across the preparation methods are provided for each element as part of the JMP® output of Appendix A-6 and in Table 3-7. The comparison of means is based on the pooled variances across the preparation methods and is ideal when the variances are equal among the preparation methods. For example, the results from the appendix for Ca in the SRAT Receipt samples are show as:

Level			Mean
CC		C	0.415
AR		B	0.530
PF	A		0.743

where the means for the measurements for the three preparation methods are given and labeled from the largest to the smallest average values with an “A”, “B”, and “C,” respectively. Levels denoted by a different letter are reported as being significantly different. If denoted as “AB”, it means that there is a significant difference between two of the methods while both are equivalent to one of the methods. More specifically, digestion types not having the same letter are statistically different at the 5% significance level. Thus, for this case of Ca, all of the means are statistically different from each other. The qualitative results of the statistical comparisons across the means of the measurements from the preparation methods are shown in the last three columns of Table 3-7.

**Table 3-7. SRAT Sample Results for Elements of Significance in the Dry Sludge**

SB10 SRAT Sample Type	Element	Avg wt.% CC (%RSD)	Avg wt.% AR (%RSD)	Avg wt.% PF (%RSD)	Significant Difference in Variances	Connecting Letters for Means (equal std devs)		
						CC	AR	PF
Receipt	Al	12.5 (1.38)	8.32 (18.48)	12.4 (4.95)		A	B	A
Receipt	Ba	0.0387 (2.50)	0.0449 (3.57)	0.0470 (6.19)		B	A	A
Receipt	Ca	0.416 (0.80)	0.530 (2.82)	0.743 (7.27)	yes	C	B	A
Receipt	Cr	0.161 (2.32)	0.160 (2.78)	0.162 (2.87)		A	A	A
Receipt	Fe	8.83 (0.97)	8.58 (3)	8.43 (2.46)		A	AB	B
Receipt	Hg	2.50 (4.81)	2.85 (6.03)	Note 1.		B	A	
Receipt	La	0.0299 (1.79)	0.0338 (3.42)	0.0217 (1.16)		B	A	C
Receipt	Mg	0.224 (1.43)	0.213 (3.02)	0.205 (2.21)		A	B	B
Receipt	Mn	2.82 (1.75)	2.71 (2.83)	2.61 (2.07)		A	AB	B
Receipt	Na	17.3 (1.00)	16.6 (1.44)	Note 2.		A	B	
Receipt	Ni	0.295 (0.87)	0.285 (3.14)	0.263 (1.43)	yes	A	A	B
Receipt	P	0.0707 (2.55)	< 0.124	< 0.0968	Note 5.	Note 5.		
Receipt	S	0.408 (2.05)	0.379 (2.09)	0.423 (1.16)		B	C	A
Receipt	Sr	0.0177 (2.09)	0.0163 (5.39)	0.0162 (4.21)		A	B	B
Receipt	Th	1.18 (6.00)	1.75 (3.41)	1.71 (2.56)		B	A	A
Receipt	Ti	0.0133 (1.60)	< 0.0147	< 0.0172	Note 5.	Note 5.		
Receipt	U	2.42 (1.08)	2.33 (2.76)	2.12 (1.55)		A	B	C
Receipt	Zn	0.0173 (1.62)	0.0181 (3.08)	0.0168 (1.46)		B	A	B
Receipt	Zr	0.115 (1.38)	0.113 (3.29)	Note 4.		A	A	
Note 1: Hg is not measured by DMA in the PF because the 675 °C temperature of the fusion volatilizes Hg. Note 2: Na is not measured by ICP-OES for the PF method due to an introduction of Na from the reagent. Note 3: Si is not measured by ICP-OES for the CC method because the high HF concentration attacks the quartz sample introduction system of ICP-OES. Therefore, no statistics are shown to compare the CC method of Si. Note 4: Zr is not measured by ICP-OES for the PF method because a Zr crucible is used to perform the fusion. Note 5: Statistics were not reported for these elements due to one or more values being reported as the detection limit for the instrumentation method.								

**Table 3-7. (cont.) SRAT Sample Results for Elements of Significance in the Dry Sludge**

SB10 SRAT Sample Type	Element	Avg wt.% CC (%RSD CV)	Avg wt.% AR (%RSD CV)	Avg wt.% PF (%RSD CV)	Significant Difference in Variances	Connecting Letters for Means (equal std devs)		
						CC	AR	PF
Product	Al	9.34 (0.67)	7.84 (0.82)	8.96 (5.17)		A	B	A
Product	Ba	0.0340 (3.06)	0.0367 (0.36)	0.0349 (1.93)	yes	B	A	B
Product	Ca	0.389 (0.76)	0.402 (0.21)	0.613 (7.36)	yes	B	B	A
Product	Cr	0.127 (0.49)	0.122 (0.47)	0.108 (7.49)	yes	A	A	B
Product	Fe	6.62 (1.23)	6.57 (0.87)	5.87 (1.55)		A	A	B
Product	Hg	0.603 (6.91)	0.767 (2.01)	Note 1.		B	A	
Product	La	0.0263 (1.67)	0.0274 (0.48)	0.0252 (3.81)		AB	A	B
Product	Mg	0.168 (0.61)	0.157 (0.30)	0.143 (3.55)		A	B	C
Product	Mn	2.21 (0.46)	2.23 (0.48)	1.93 (2.17)	yes	A	A	B
Product	Na	13.1 (0.54)	12.8 (0.50)	Note 2.		A	B	
Product	Ni	0.222 (0.53)	0.210 (0.45)	0.196 (2.18)		A	B	C
Product	P	0.0525 (4.56)	0.0545 (1.18)	0.0472 (5.76)		A	A	B
Product	S	0.312 (0.66)	0.293 (0.35)	0.287 (3.62)	yes	A	B	B
Product	Sr	0.0156 (0.95)	0.0164 (0.63)	0.0170 (4.86)		B	AB	A
Product	Th	1.09 (1.11)	1.35 (1.96)	1.17 (5.77)		B	A	B
Product	Ti	0.0101 (1.74)	0.00983 (0.50)	0.0110 (4.89)	yes	B	B	A
Product	U	1.81 (0.52)	1.71 (0.28)	1.53 (3.01)		A	B	C
Product	Zn	0.0135 (1.55)	0.0135 (0.60)	0.0128 (1.66)		A	A	B
Product	Zr	0.0862 (0.87)	0.0848 (0.59)	Note 4.		A	B	
Note 1: Hg is not measured by DMA in the PF because the 675 °C temperature of the fusion volatilizes Hg. Note 2: Na is not measured by ICP-OES for the PF method due to an introduction of Na from the reagent. Note 3: Si is not measured by ICP-OES for the CC method because the high HF concentration attacks the quartz sample introduction system of ICP-OES. Therefore, no statistics are showed to compare the CC method of Si. Note 4: Zr is not measured by ICP-OES for the PF method because a Zr crucible is used to perform the fusion.								

It is noted that DWPF routinely analyzes for elements such as Al, B, Ca, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr using the CC method. Of which, B, Cu, K, and Li were not of significant quantities to be quantified by ICP-OES with the CC method in this case. Additionally, Si was not measured with the CC method due to known issues of HF reacting with the ICP-OES instrument and biasing the values for Si. Differences in variances for Ca and Ni elements of the SRAT Receipt as well as Ba, Ca, Cr, Mn, S, and Ti determinations of the SRAT Product were reported. Further statistical analyses found that differences in the means were observed for all elements of interest across the three preparation methods, except for Cr and Zr of the SRAT Receipt samples. However, some elements yielded slight differences in means (i.e. Fe and Mn for the SRAT Receipt, and La and Sr for the SRAT Product) where the means of two sample digestion methods statistically agreed but differed across all three. Table 3-8 quantifies the differences of the means for the elemental analyses determined as quantifiable from this work. Of the 8 elements (Al, Ca, Fe, Hg, Mn, Na, Th, and U) that comprise at least 0.5 wt. % of SB10 sludge on a dry solids basis, Ca (SRAT Receipt), Hg, and Th were reported to be biased lower for the CC method than the other methods when deviations were observed. Furthermore, the CC method produced Na and U determinations that were higher than those from the AR and PF method for both SRAT Receipt and Product samples.

**Table 3-8. Differences in Means of the SRAT Samples Results for Elements of Significance in the Dry Sludge Comparing the Digestion Methods**

<i>Elements</i>	SRAT Receipt		SRAT Product	
	(CC-AR) / AR	(CC-PF) / PF	(CC-AR) / AR	(CC-PF) / PF
Al	50%	0%	19%	4%
Ba	-14%	-18%	-7%	-2%
Ca	-22%	-44%	-3%	-37%
Cr	1%	-1%	4%	18%
Fe	3%	5%	1%	13%
Hg	-12%		-21%	
La	-12%	38%	-4%	4%
Mg	5%	9%	7%	17%
Mn	4%	8%	-1%	14%
Na	4%		3%	
Ni	4%	12%	6%	13%
P	<i>Note 1.</i>	<i>Note 1.</i>	-4%	11%
S	8%	-4%	6%	9%
Sr	9%	10%	-5%	-8%
Th	-33%	-31%	-19%	-6%
Ti	<i>Note 1.</i>	<i>Note 1.</i>	3%	-8%
U	4%	14%	6%	18%
Zn	-5%	3%	0%	6%
Zr	2%		2%	
Note 1. The lightly shaded values are associated with <i>Note 5</i> of Table 3-7 and only gave quantifiable values for the CC method.				

Statistical determinations were made for 19 of the total 35 elements submitted to analytical. The sample digestions comprised of 13 elements (Al, Ca, Cr, Fe, Hg, Mg, Mn, Na, Ni, S, Th, U, and Zr) with at least 0.1 wt. % on a dry solids basis and 8 elements (Al, Ca, Fe, Hg, Mn, Na, Th, and U) with at least 0.5 wt. % on a dry solids basis. For B, Ce, Cu, Gd, K, and Li, the values were similar to the detection limits observed for the blank samples. For P and Ti, it is arguable if ICP-OES indicates that they are present in the sludge in a minute quantity or indicated from error with the background noise. Of the 8 elements that comprise at least 0.5 wt. % of SB10 sludge on a dry solids basis, the CC method produced statistically equivalent results for Al (PF), Fe (AR), and Mn (AR) for the SRAT Receipt, and Al (PF), Ca (AR), Fe (AR), Mn (AR), and Th (PF) for the SRAT Product samples. A summary of the results and statistical analysis for the elements of significance from the SRAT Receipt and SRAT Product sludge digestions using the CC, AR, and PF methods are provided below (all other elements submitted to analytical were less than the detection limits of ICP-OES and did not warrant a CC method discussion):

### 3.1 SRAT Receipt<sup>f</sup>

- ❖ Al: Differences in the means were observed for Al determinations. The CC Al determinations were ~50% higher than from the AR method and ~0% lower than from the PF method (no significant difference). The average Al concentration for the CC method is 12.5 wt. % of TS for the SRAT Receipt.

<sup>f</sup> The discussions of the elemental wt. % SRAT Receipt measurements are on a dry solids basis. All of the statistically significant results are at the 5% significance level. See Table 3-7 for this information in tabular form.



- ❖ Ba: Differences in the means were observed for Ba determinations with the CC Ba determinations ~14% lower than from the AR method and ~18% lower than PF Ba determinations. The average Ba concentration for the CC method is 0.0387 wt. % of TS for the SRAT Receipt.
- ❖ Ca: Differences in both the variances and the means were observed for Ca determinations. The mean of the CC Ca determinations was ~22% lower than from the AR method and ~44% lower than PF Ca determinations. This discrepancy is due to Ca contamination in sodium peroxide used in the PF method as shown in the blank analysis. However, Ca was still low for the SRAT Receipt and the ARG-1 sample with the CC method despite Ca being present in the blank. Therefore, the CC method may not provide reliable Ca determinations. The average Ca concentration is 0.416 wt. % of TS in the SRAT Receipt for the CC method.
- ❖ Cr: No differences in both the variances and the means were observed for the Cr determinations. The CC Cr determinations were ~1% higher than the AR Cr determinations and ~1% lower than the PF Cr determinations. The average Cr concentration is 0.161 wt. % of TS in the SRAT Receipt for the CC method.
- ❖ Fe: Slight differences in the mean were observed for Fe determinations. The CC Fe determinations were ~3% higher than AR Fe determinations (no significant difference) and ~5% higher than the PF Fe determinations. The average Fe concentration is 8.83 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Hg: Differences in the means were observed for Hg determinations. The CC Hg determinations were ~12% lower than AR Hg determinations. This may show that more Hg may have been lost in the CC methods. The average Hg concentration is 2.5 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ La: Differences in the means were observed for La determinations. The CC La determinations were ~12% lower than AR La determinations and ~38% higher than the PF La determinations. The average La concentration is 0.0299 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Mg: Differences in the means were observed for Mg determinations. The CC Mg determinations were ~5% higher than AR Mg determinations and ~9% higher than PF Mg determinations. This could be attributed to the presence of Mg in the blank. The average Mg concentration is 0.224 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Mn: Differences in the means were observed for Mn determinations. The CC Mn determinations were ~4% higher than AR Mn determinations (no significant difference) and ~8% higher than the PF Mn determinations. The average Mn determination for the CC dissolution method is 2.82 wt. % of TS in the SRAT Receipt.
- ❖ Na: Differences in the means were observed for Na determinations. The CC Na determinations were ~4% higher than AR Na determinations. The average Na concentration is 17.3 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Ni: Differences in the variances and slight differences in the means were observed for Ni determinations. The CC Ni determinations were ~4% higher than AR Ni determinations (no significant difference) and ~12% higher than PF Ni determinations. The average Ni concentration is 0.296 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ P: The average P concentration is 0.0707 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions. However, the P concentrations reported here closely resemble the concentrations of the blank and ARG-1 samples.

- ❖ S: Differences in the means were observed for S determinations. The CC S determinations were ~8% higher than AR S determinations and ~4% lower than the PF S determinations. The average S concentration is 0.408 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Si: Si determinations are only considered reliable from the PF method. The AR method may not dissolve all forms of Si in SRS sludge. The CC method is generally effective for Si compounds, but the high concentration of HF reacts with the quartz ICP-OES sample introduction system resulting in a very high Si bias. The average Si concentration is 0.424 wt. % of TS in the SRAT Receipt as determined from the PF dissolutions.
- ❖ Sr: Differences in the means were observed for Sr determinations. The CC Sr determinations were ~9% higher than AR Sr determinations and ~10% higher than the PF Sr determinations. The average Sr concentration is 0.0178 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Th: Differences in the means were observed for Th determinations. The CC Th determinations were ~33% lower than AR Th determinations and ~31% lower than the PF Th determinations. Th determinations should be considered using the PF method as a fluoride precipitate may occur with the CC method. The average Th concentration is 1.18 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Ti: The average Ti concentration is 0.0133 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions. However, the Ti concentrations reported here closely resemble the concentrations of the blanks.
- ❖ U: Differences in the means were observed for U determinations. The CC U determinations were ~4% higher than AR U determinations and ~14% higher than the PF Th determinations. The average U concentration is 2.42 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Zn: Slight differences in the means were observed for Zn determinations. The CC Zn determinations were ~5% lower than AR Zn determinations and ~3% higher than the PF Zn determinations. The average Zn concentration is 0.0173 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Zr: No differences in both the variances and the means were observed for the Zr determinations. The CC Zr determinations were ~2% higher than the AR Zr determinations (no significant difference). Since HF is an effective dissolution reagent for Zr compounds, CC determinations for Zr are considered to be more accurate and conservative for the SRS sludge. The average Zr concentration is 0.115 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.

### 3.2 SRAT Product<sup>f</sup>

- ❖ Al: Differences in the means of Al determinations were observed. The CC Al determinations were ~19% higher than AR Al determinations and ~4% higher than PF Al determinations. The average Al concentration is 9.34 wt. % of TS in the SRAT Product as measured using the CC dissolution method.
- ❖ Ba: Differences in both the variances and the means were observed for Ba determinations with the CC Ba determinations ~7% lower than from the AR method and ~2% lower than PF Ba determinations (no significant difference). The average Ba concentration is 0.0340 wt. % of TS in the SRAT Product as measured using the CC dissolution method.

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<sup>f</sup> Note that the discussions of the elemental wt. % SRAT Product measurements are on a dry solids basis. All of the statistically significant results are at the 5% significance level. See Table 3-7 for this information presented in tabular form.

- ❖ Ca: Differences in the variances and the means of Ca determinations were observed. There was a difference in the means with the CC Ca determinations ~3% lower than the AR Ca determinations (no significant difference) and ~37% lower than the PF Ca determinations. The CC method was slightly lower than the AR despite a small presence of Ca in the blank. The discrepancy for the PF method is due to Ca contamination in sodium peroxide used and shown in the blank analysis. The average Ca concentration is 0.389 wt. % of TS in the SRAT Product as measured using the CC dissolution method.
- ❖ Cr: Differences in the variances and the means of Cr determinations were observed. The CC Cr determinations were ~4% higher than the AR Cr determinations (no significant difference) and ~18% higher than the PF Cr determinations. The average Cr concentration is 0.127 wt. % of TS in the SRAT Product as measured using the CC dissolution method.
- ❖ Fe: Slight differences in the mean were observed for Fe determinations. The CC Fe determinations were ~1% higher than AR Fe determinations (no significant difference) and ~13% higher than the PF Fe determinations. The average Fe concentration is 6.62 wt. % of TS in the SRAT Receipt as determined from the CC dissolutions.
- ❖ Hg: Differences in the means of Hg determinations were observed. The CC Hg determinations were ~21% lower than AR Hg determinations. This may show that more Hg may have been lost in the CC methods. The average Hg concentration is 0.603 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ La: Slight differences in the means were observed for La determinations. The CC La determinations were ~4% lower than AR La determinations (no significant difference) and ~4% higher than the PF La determinations (no significant difference). The average La concentration is 0.0263 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Mg: Differences in the means were observed for Mg determinations. The CC Mg determinations were ~7% higher than AR Mg determinations and ~17% higher than PF Mg determinations. This could be attributed to the presence of Mg in the blank. The average Mg is 0.168 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Mn: Differences in both the variances and the means of Mn determinations were observed. The CC Mn determinations were ~1% lower than AR Mn determinations (no significant difference) and ~14% higher than the PF Mn determinations. The average Mn concentration is 2.21 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Na: Slight differences in the means were observed for Na determinations. The CC Na determinations were ~3% higher than AR Na determinations. Na concentration is 13.1 wt. % in the SRAT Product as determined from the CC dissolutions.
- ❖ Ni: Differences in the means were observed for Ni determinations. The CC Ni determinations were ~6% higher than AR Ni determinations and ~13% higher than PF Ni determinations. The average Ni concentration is 0.222 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ P: Differences in the means were observed for P determinations. The CC P determinations were ~4% lower than AR P determinations (no significant difference) and ~11% higher than PF P determinations. The average P concentration is 0.0525 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ S: Differences in the means were observed for S determinations. The CC S determinations were ~6% higher than AR S determinations and ~9% higher than the PF S determinations. The average S concentration is 0.312 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Si: Si determinations are only considered reliable from the PF method at SRNL. The AR method may not dissolve all forms of Si in SRS sludge. The CC method is generally effective for Si

compounds, but the high concentration of HF reacts with the quartz ICP-OES sample introduction system resulting in a very high Si bias. The average Si concentration is 0.338 wt. % of TS in the SRAT Product as determined from the PF dissolutions.

- ❖ Sr: Slight differences in the means were observed for Sr determinations. The CC Sr determinations were ~5% lower than AR Sr determinations and ~8% higher than the PF Sr determinations. The average Sr concentration is 0.0156 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Th: Differences in the means were observed for Th determinations. The CC Th determinations were ~19% lower than AR Th determinations and ~6% lower than the PF Th determinations (no significant difference). The average Th concentration is 1.09 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Ti: Differences in the variances and slight differences in the means were observed for Ti determinations. The CC Ti determinations were ~3% higher than AR Ti determinations (no significant difference) and ~8% lower than the PF Ti determinations. The average Ti concentration is 0.0101 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ U: Differences in the means were observed for U determinations. The CC U determinations were ~6% higher than AR U determinations and ~18% higher than the PF Th determinations. The average U concentration is 1.81 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Zn: Slight differences in the means were observed for Zn determinations. The CC Zn determinations were ~0% different than AR Zn determinations (no significant difference) and ~6% higher than the PF Zn determinations. The average Zn concentration is 0.01 wt. % of TS in the SRAT Product as determined from the CC dissolutions.
- ❖ Zr: Slight differences in the means were observed for the Zr determinations. The CC Zr determinations were ~2% higher than the AR Zr determinations. Since HF is an effective dissolution reagent for Zr compounds, CC determinations for Zr are considered to be more accurate and conservative for SRS sludge. The average Zr concentration is 0.0862 wt. % of TS in the SRAT Product as determined from the CC dissolutions.

#### 4.0 Conclusions

DWPF routinely uses the CC method to analyze for ~16 elements (i.e. Al, B, Ca, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr) from each sludge batch, where SRNL is involved with testing the applicability of this digestion method for sludge. The CC method did not result in complete dissolution of either the SRAT Receipt or SRAT Product with some fine, dark solids remaining. The SRAT Receipt and SRAT Product sample digestions comprised of 13 elements (Al, Ca, Cr, Fe, Hg, Mg, Mn, Na, Ni, S, Th, U, and Zr) with at least 0.1 wt. % on a dry solids basis and 8 elements (Al, Ca, Fe, Hg, Mn, Na, Th, and U) with at least 0.5 wt. % on a dry solids basis. Statistical determinations were made for 19 elements where quantifiable data was reported. The elements of B, Ce, Cu, Gd, K, and Li are often of interest but were reported with values similar to that of the detection limits observed for the blanks. Despite the incomplete dissolution for the CC method, the four replicate dissolutions and elemental analysis of the SRAT Receipt and SRAT Product of the DWPF CC method did not reveal extreme biases for determining the major elements in the sludge through the CC method versus the AR and PF methods. Of the 8 elements that were greater than 0.5 wt. % on a dry solids basis, statistically equivalent results for the CC digestion method were observed for Al (PF), Fe (AR, ~PF), and Mn (AR, ~PF) elements in the SRAT Receipt, and Al (PF), Ca (AR), Fe (AR), Mn (AR), and Th (PF) elements in the SRAT Product samples. Random errors and variances of both the dissolutions and analytical methods (ICP-OES and DMA) resulted in elemental

determinations matching for some elements as a function of dissolution method but not for others. Such errors and variances are typical for a sample set of this size, as shown by the previous sludge batch reports.

## **5.0 Recommendations, Path Forward and Future Work**

Elemental analyses of SB10 SRAT Receipt and SRAT Product samples digested by the CC method suggest that the method should be adequate for routine process control analyses in the DWPF after side-by-side tests of the CC method and PF method are performed on the first 10 SRAT cycles of the SB10 campaign. The additional data sets produced in the DWPF Laboratory will provide more opportunities to assess whether the CC method meets the data quality objectives needed for DWPF process control. The DWPF Laboratory should continue with their plans for further tests, including statistical analysis, of the CC method during these 10 SRAT cycles.

If it is of interest, SRNL can look into the filtering and characterization of any undissolved solids present from the digestion methods when evaluating SB11 sludge. Also, it might be of interest to determine if a correction factor for SB11 can be elucidated for major elements that were biased using the CC method when compared to the PF method.

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**Appendix A-1. Measurements of the SB10 SRAT Receipt and Product Samples (Ag-Fe)**

Type	Prep	Sample ID	LIMS	UOM	Ag	Al	B	Ba	Be	Ca	Cd	Ce	Co	Cr	Cu	Fe
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	µg/g	< 16.7	18400	< 170	60.0	< 1.44	628	< 7.06	< 202	< 4.85	234	< 68.9	13200
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	µg/g	< 15.2	18600	< 155	56.9	< 1.31	618	< 6.42	< 184	< 4.41	239	< 62.7	13100
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	µg/g	< 15.7	19000	< 160	57.0	< 1.35	627	< 6.64	< 190	< 4.56	246	< 64.8	13300
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	µg/g	< 16.7	18800	< 170	58.4	< 1.43	620	< 7.04	< 202	< 4.84	245	< 68.8	13400
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	µg/g	< 13.4	15800	< 135	70.5	< 3.67	821	< 6.48	168	< 9.73	247	55.0	13400
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	µg/g	< 13.6	10500	< 136	64.8	< 3.71	767	< 6.55	154	< 9.83	231	50.1	12500
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	µg/g	< 13.7	12000	< 137	67.4	< 3.74	800	< 6.60	155	< 9.91	241	52.3	12900
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	µg/g	< 13.0	11600	< 131	66.4	< 3.55	790	< 6.28	153	< 9.43	238	52.1	12700
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	µg/g	< 35.5	17500	< 130	71.5	< 4.00	1100	< 6.27	136	< 7.41	240	55.6	12500
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	µg/g	< 33.7	18400	< 124	64.6	< 3.79	1010	< 5.94	131	< 7.03	236	51.2	12300
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	µg/g	< 36.1	19700	< 132	70.8	< 4.06	1150	< 6.37	138	< 7.53	252	52.6	13000
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	µg/g	< 35.6	18900	< 131	75.1	< 4.01	1200	< 6.29	131	< 7.44	246	53.5	12800
SB10 SRAT Product	CC	TS-211-20-B-263	25494	µg/g	< 17.3	18800	< 176	69.9	< 1.49	783	< 7.31	< 210	< 5.02	257	< 71.4	13300
SB10 SRAT Product	CC	TS-211-20-B-264	25495	µg/g	< 15.4	18800	< 157	70.5	< 1.33	776	< 6.52	< 187	< 4.48	254	< 63.6	13100
SB10 SRAT Product	CC	TS-211-20-B-265	25496	µg/g	< 15.9	18900	< 162	66.7	< 1.36	780	< 6.71	< 192	< 4.61	255	< 65.5	13300
SB10 SRAT Product	CC	TS-211-20-B-266	25497	µg/g	< 17.2	18600	< 175	66.5	< 1.48	790	< 7.26	< 208	< 4.99	255	< 70.9	13500
SB10 SRAT Product	AR	TS211-20-B-018	24339	µg/g	< 14.9	15600	< 11.1	73.6	< 0.244	807	< 6.07	< 184	< 4.05	247	< 66.5	13100
SB10 SRAT Product	AR	TS211-20-B-019	24340	µg/g	< 15.3	15700	< 11.3	74.2	< 0.251	811	< 6.24	< 189	< 4.16	247	< 68.4	13300
SB10 SRAT Product	AR	TS211-20-B-020	24341	µg/g	< 15.5	15900	< 11.4	73.7	< 0.254	808	< 6.31	< 191	< 4.21	245	< 69.1	13100
SB10 SRAT Product	AR	TS211-20-B-021	24342	µg/g	< 15.6	15800	< 11.5	73.9	< 0.255	809	< 6.33	< 192	< 4.23	245	< 69.4	13300
SB10 SRAT Product	PF	TS211-20-B-026	24172	µg/g	< 108	17700	< 14.5	68.5	< 0.501	1250	< 5.75	< 379	< 3.83	213	< 54.2	11600
SB10 SRAT Product	PF	TS211-20-B-027	24173	µg/g	< 108	17100	< 14.5	70.8	< 0.501	1220	< 5.74	< 379	< 3.83	195	< 54.1	11900
SB10 SRAT Product	PF	TS211-20-B-028	24174	µg/g	< 106	19300	< 14.2	71.5	< 0.491	1340	< 5.65	< 372	< 3.77	228	< 53.2	12000
SB10 SRAT Product	PF	TS211-20-B-029	24175	µg/g	< 109	17900	< 14.6	69.4	< 0.502	1120	< 5.77	< 380	< 3.85	230	< 54.4	11700



## Appendix A-2. Measurements of the SB10 SRAT Receipt and Product Samples (Gd-Pb)

Type	Prep	Sample ID	LIMS	UOM	Gd	K	La	Li	Mg	Mn	Mo	Na	Ni	P	Pb
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	µg/g	< 120	< 181	44.6	< 28.1	329	4160	< 13.8	25600	439	102	< 44.1
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	µg/g	< 110	< 165	43.8	< 25.4	339	4190	< 12.5	25800	441	108	< 40.1
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	µg/g	< 113	< 170	45.1	< 26.3	339	4330	< 12.9	26200	447	107	< 41.4
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	µg/g	< 120	< 181	45.7	< 27.9	334	4230	< 13.7	26000	446	107	< 43.9
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	µg/g	67.1	< 118	52.9	< 12.8	329	4220	< 11.9	25400	444	< 186	< 18.8
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	µg/g	62.9	< 119	48.7	< 12.9	306	3960	< 12.0	24800	414	< 187	< 19.0
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	µg/g	65.5	< 120	50.8	< 13.0	322	4090	< 12.1	24700	431	< 189	< 19.2
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	µg/g	63.1	< 114	50.3	< 12.4	318	4000	< 11.6	24600	419	< 180	< 18.3
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	µg/g	69.1	< 254	32.4	< 9.90	312	3900	< 15.7	Note 1.	391	< 146	< 9.70
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	µg/g	64.6	< 241	32.1	< 9.38	297	3800	< 14.9	Note 1.	388	< 139	< 9.19
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	µg/g	71.2	< 258	33.0	< 10.1	309	3980	< 15.9	Note 1.	399	< 149	< 9.85
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	µg/g	69.4	< 255	32.6	< 9.93	310	3960	< 15.7	Note 1.	399	< 147	< 9.73
SB10 SRAT Product	CC	TS-211-20-B-263	25494	µg/g	< 125	< 188	53.7	< 28.9	339	4450	< 14.2	26400	449	109	< 45.6
SB10 SRAT Product	CC	TS-211-20-B-264	25495	µg/g	< 111	< 167	53.1	< 25.8	335	4420	< 12.7	26300	444	107	< 40.6
SB10 SRAT Product	CC	TS-211-20-B-265	25496	µg/g	< 114	< 172	52.8	< 26.6	338	4420	< 13.1	26400	444	108	< 41.8
SB10 SRAT Product	CC	TS-211-20-B-266	25497	µg/g	< 124	< 186	51.6	< 28.8	335	4460	< 14.2	26100	445	98.5	< 45.3
SB10 SRAT Product	AR	TS211-20-B-018	24339	µg/g	74.2	< 84.1	55.1	< 13.4	314	4450	7.41	25500	422	110	< 39.1
SB10 SRAT Product	AR	TS211-20-B-019	24340	µg/g	74.5	< 86.5	55.5	< 13.7	316	4500	7.71	25800	425	111	< 40.1
SB10 SRAT Product	AR	TS211-20-B-020	24341	µg/g	74.2	< 87.4	54.9	< 13.9	316	4480	7.63	25600	421	108	< 40.5
SB10 SRAT Product	AR	TS211-20-B-021	24342	µg/g	74.0	< 87.8	55.0	< 13.9	315	4490	7.44	25700	421	109	< 40.7
SB10 SRAT Product	PF	TS211-20-B-026	24172	µg/g	72.9	< 420	50.8	< 54.9	287	3790	< 11.2	Note 1.	383	88.3	< 33.3
SB10 SRAT Product	PF	TS211-20-B-027	24173	µg/g	71.7	< 420	51.7	< 54.8	284	3930	< 11.1	Note 1.	395	100	< 33.2
SB10 SRAT Product	PF	TS211-20-B-028	24174	µg/g	79.1	< 413	52.4	< 53.9	302	3970	< 11.1	Note 1.	404	98.8	< 32.7
SB10 SRAT Product	PF	TS211-20-B-029	24175	µg/g	69.1	< 422	48.0	< 55.1	278	3830	< 11.2	Note 1.	394	92.7	< 33.4
Note 1. Na is not measured by ICP-OES in PF because of introduction as a reagent.															

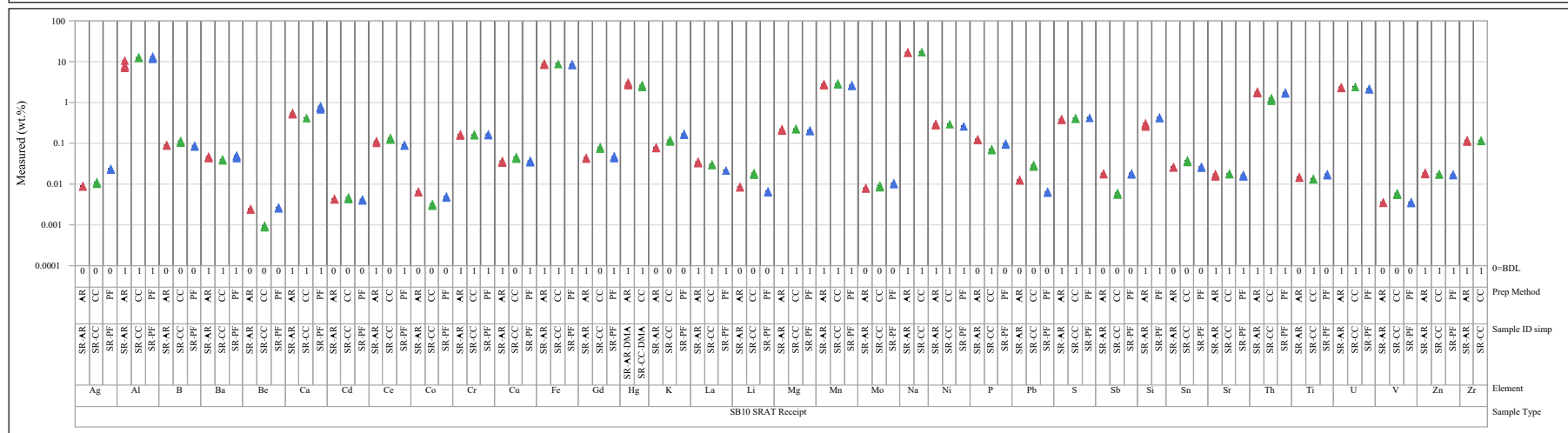
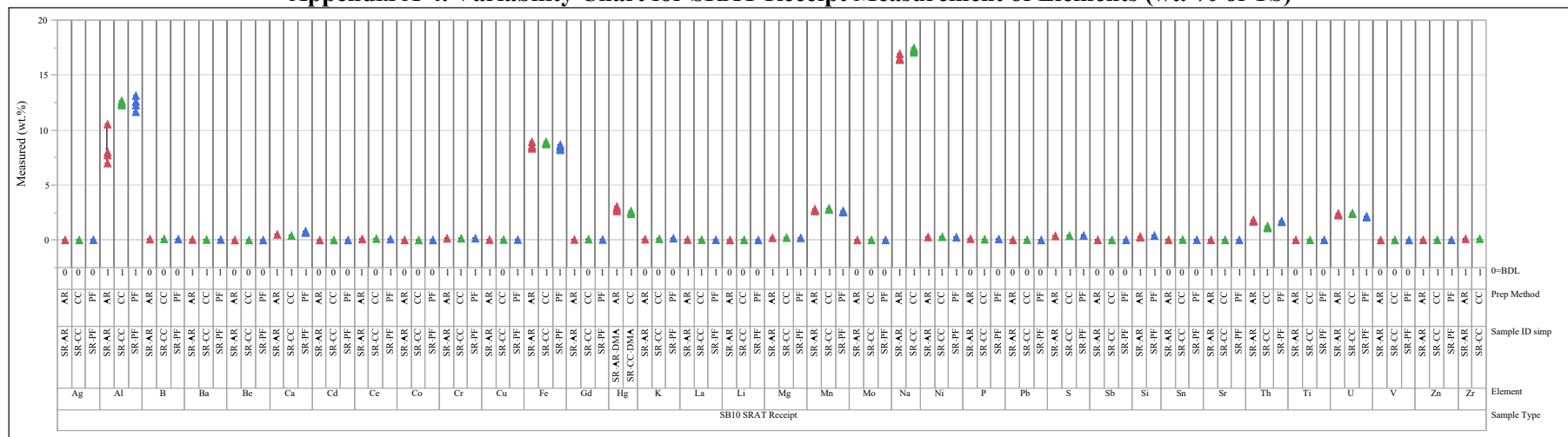
## Appendix A-3. Measurements of the SB10 SRAT Receipt and Product Samples (S-Zr)

Type	Prep	Sample ID	LIMS	UOM	S	Sb	Si	Sn	Sr	Th	Ti	U	V	Zn	Zr
SB10 SRAT Receipt	CC	TS-211-20-B-259	25490	μg/g	593	< 9.09	Note 2.	< 57.1	27.2	1900	19.5	3580	< 8.95	25.5	170
SB10 SRAT Receipt	CC	TS-211-20-B-260	25491	μg/g	614	< 8.26	Note 2.	< 51.9	26.2	1640	20.2	3620	< 8.14	25.9	171
SB10 SRAT Receipt	CC	TS-211-20-B-261	25492	μg/g	621	< 8.54	Note 2.	< 53.7	26.1	1780	20.1	3670	< 8.41	26.5	175
SB10 SRAT Receipt	CC	TS-211-20-B-262	25493	μg/g	617	< 9.06	Note 2.	< 56.9	27.0	1770	20.1	3650	< 8.92	25.8	174
SB10 SRAT Receipt	AR	TS211-20-A-03501	21538	μg/g	564	< 27.0	461	< 39.2	26.2	2740	< 22.1	3610	< 5.35	28.3	177
SB10 SRAT Receipt	AR	TS211-20-A-03502	21539	μg/g	558	< 27.2	410	< 39.6	23.0	2530	< 22.3	3390	< 5.40	26.3	164
SB10 SRAT Receipt	AR	TS211-20-A-03503	21540	μg/g	564	< 27.5	422	< 39.9	24.6	2650	< 22.5	3520	< 5.45	27.2	169
SB10 SRAT Receipt	AR	TS211-20-A-03504	21541	μg/g	585	< 26.1	384	< 38.0	24.2	2590	< 21.4	3440	< 5.18	26.9	167
SB10 SRAT Receipt	PF	TS211-20-A-03497	21544	μg/g	636	< 27.3	639	< 39.6	25.3	2550	< 26	3170	< 5.41	24.8	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03498	21545	μg/g	626	< 25.8	617	< 37.6	23.0	2480	< 24.6	3120	< 5.13	25.2	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03499	21546	μg/g	644	< 27.7	648	< 40.3	24.9	2630	< 26.4	3240	< 5.49	25.3	Note 3.
SB10 SRAT Receipt	PF	TS211-20-A-03500	21547	μg/g	634	< 27.4	639	< 39.8	24.0	2600	< 26.1	3170	< 5.43	25.7	Note 3.
SB10 SRAT Product	CC	TS-211-20-B-263	25494	μg/g	628	< 9.41	Note 2.	< 59.1	31.7	2230	20.6	3670	< 9.26	27.4	175
SB10 SRAT Product	CC	TS-211-20-B-264	25495	μg/g	624	< 8.38	Note 2.	< 52.7	31.5	2200	20.1	3630	< 8.25	26.7	172
SB10 SRAT Product	CC	TS-211-20-B-265	25496	μg/g	623	< 8.63	Note 2.	< 54.2	31.3	2200	20.8	3640	< 8.50	26.9	174
SB10 SRAT Product	CC	TS-211-20-B-266	25497	μg/g	632	< 9.35	Note 2.	< 58.7	31.0	2170	20.1	3630	< 9.20	27.6	172
SB10 SRAT Product	AR	TS211-20-B-018	24339	μg/g	589	< 4.25	413	< 14.6	32.8	2710	19.7	3450	< 2.57	27.3	170
SB10 SRAT Product	AR	TS211-20-B-019	24340	μg/g	592	< 4.37	409	< 15.0	33.3	2780	19.9	3450	< 2.64	27.1	172
SB10 SRAT Product	AR	TS211-20-B-020	24341	μg/g	587	< 4.42	417	< 15.2	33.1	2650	19.7	3440	< 2.67	26.9	170
SB10 SRAT Product	AR	TS211-20-B-021	24342	μg/g	590	< 4.44	389	< 15.3	33.0	2720	19.7	3430	< 2.68	27.1	170
SB10 SRAT Product	PF	TS211-20-B-026	24172	μg/g	574	< 8.61	615	< 73.1	34.2	2420	21.4	3080	< 12.8	26.2	Note 3.
SB10 SRAT Product	PF	TS211-20-B-027	24173	μg/g	606	< 8.60	651	< 73.0	34.5	2360	21.0	3130	< 12.8	25.2	Note 3.
SB10 SRAT Product	PF	TS211-20-B-028	24174	μg/g	567	< 8.45	640	< 71.8	35.9	2450	23.3	3160	< 12.6	25.8	Note 3.
SB10 SRAT Product	PF	TS211-20-B-029	24175	μg/g	558	< 8.64	815	< 73.4	31.9	2150	22.7	2950	< 12.9	25.5	Note 3.

Note 2. Si is not measured by ICP-OES for the CC method because the high HF concentration attacks the quartz sample introduction system of the ICP-OES instrument.

Note 3. Zr is not measured by ICP-OES from the PF method because a Zr crucible is used for the fusion.

## Appendix A-4. Variability Chart for SRAT Receipt Measurement of Elements (wt. % of TS)



Note: The elements that were below detect are given by their proxy value from Tables A-4 to Tables A-5.

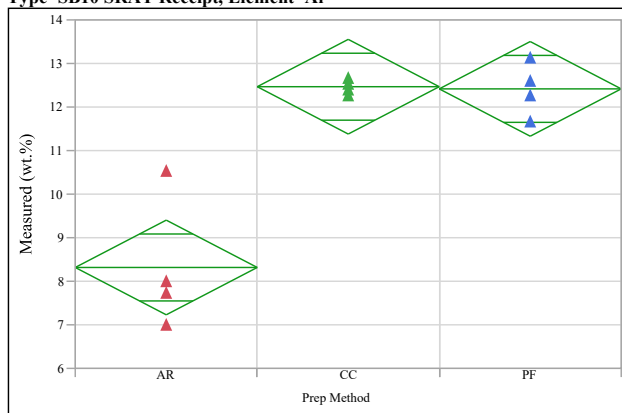
Sample Type	Element	Sample ID simp	Prep Method	0-BDL
SB10 SRAT Product	Ag	SP-AR	AR	0
		SP-CC	CC	0
	Al	SP-PF	PF	0
		SP-AR	AR	8
		SP-CC	CC	9
		SP-PF	PF	8.5
	B	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Ba	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Be	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Ca	SP-AR	AR	0.5
		SP-CC	CC	1
		SP-PF	PF	0.5
		SP-AR	AR	0
	Cd	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Ce	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Co	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Cr	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Cu	SP-AR	AR	0
		SP-CC	CC	0
		SP-PF	PF	0
		SP-AR	AR	0
	Fe	SP-AR	AR	6.5
		SP-CC	CC	6.5
		SP-PF	PF	5.5
		SP-AR	AR	0
	Gd	SP-AR	AR	0
		SP-CC	CC	0
SP-PF		PF	0	
SP-AR		AR	0	
Hg	SP-AR-DNA	AR	0.5	
	SP-CC-DNA	CC	0.5	
	SP-AR	AR	0	
	SP-CC	CC	0	
K	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
La	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Li	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Mg	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Mn	SP-AR	AR	2	
	SP-CC	CC	2	
	SP-PF	PF	1.5	
	SP-AR	AR	0	
Mo	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Na	SP-AR	AR	12	
	SP-CC	CC	12	
	SP-PF	PF	0	
	SP-AR	AR	0	
Ni	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
P	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Pb	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
S	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Sb	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Si	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Sn	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Sr	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
Th	SP-AR	AR	1.5	
	SP-CC	CC	1.5	
	SP-PF	PF	0	
	SP-AR	AR	0	
Ti	SP-AR	AR	0	
	SP-CC	CC	0	
	SP-PF	PF	0	
	SP-AR	AR	0	
U	SP-AR			

Sample Type	Element	Sample ID	simp	Prep Method	0=BDL
Ag	Ag	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	1	1
		SP-CC	CC	1	1
		SP-PF	PF	1	1
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Al	Al	SP-AR	AR	1	1
		SP-CC	CC	1	1
		SP-PF	PF	1	1
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
B	B	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Ba	Ba	SP-AR	AR	1	1
		SP-CC	CC	1	1
		SP-PF	PF	1	1
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Be	Be	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Ca	Ca	SP-AR	AR	1	1
		SP-CC	CC	1	1
		SP-PF	PF	1	1
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Cd	Cd	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Ce	Ce	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Co	Co	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Cr	Cr	SP-AR	AR	1	1
		SP-CC	CC	1	1
		SP-PF	PF	1	1
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF	PF	0	0
		SP-AR	AR	0	0
Cu	Cu	SP-AR	AR	0	0
		SP-CC	CC	0	0
		SP-PF			

A-5

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Al



Oneway Anova  
Summary of Fit

Rsquare 0.84524  
Adj Rsquare 0.810849  
Root Mean Square Error 0.960838  
Mean of Response 11.06666  
Observations (or Sum Wgts) 12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	45.379929	22.6900	24.5773	0.0002*
Error	9	8.308881	0.9232		
C. Total	11	53.688810			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	8.3167	0.48042	7.230	9.403
CC	4	12.4667	0.48042	11.380	13.553
PF	4	12.4167	0.48042	11.330	13.503

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	8.3166658	1.5370712	0.7685356	5.8708425	10.762489
CC	4	12.466663	0.1721322	0.0860661	12.192762	12.740563
PF	4	12.416663	0.6143369	0.3071684	11.439115	13.39421

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	1.537071	1.108332	0.9499993
CC	4	0.172132	0.133332	0.1333325
PF	4	0.614337	0.450003	0.4500025

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3706	2	9	0.3023
Brown-Forsythe	1.3185	2	9	0.3146
Levene	3.5423	2	9	0.0733
Bartlett	4.4340	2	.	0.0119*

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
12.4796	2	4.3352	0.0159*

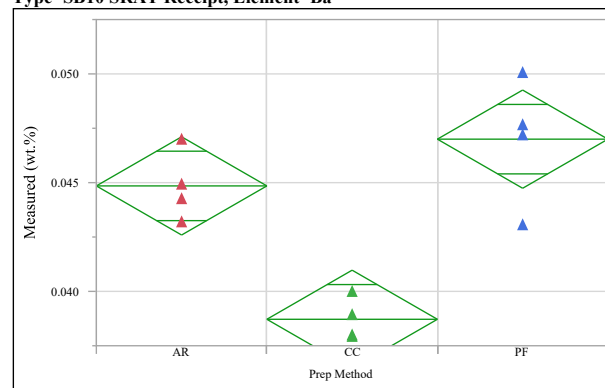
Means Comparisons for all pairs using Tukey-Kramer HSD

Connecting Letters Report

Level	Mean
CC	A
PF	A
AR	B

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Ba



Oneway Anova  
Summary of Fit

Rsquare 0.804678  
Adj Rsquare 0.761273  
Root Mean Square Error 0.001997  
Mean of Response 0.043522  
Observations (or Sum Wgts) 12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00014780	0.000074	18.5389	0.0006*
Error	9	0.00003587	3.986e-6		
C. Total	11	0.00018367			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.044850	0.00100	0.04259	0.04711
CC	4	0.038717	0.00100	0.03646	0.04097
PF	4	0.047000	0.00100	0.04474	0.04926

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0448498	0.0016013	0.0008007	0.0423016	0.0473979
CC	4	0.0387165	0.0009698	0.0004849	0.0371733	0.0402597
PF	4	0.0469995	0.0029075	0.0014537	0.0423731	0.0516259

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0016013	0.0011167	0.0011167
CC	4	0.0009698	0.0007500	0.0007500
PF	4	0.0029075	0.0019668	0.0018665

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1085	2	9	0.3712
Brown-Forsythe	0.7308	2	9	0.5080
Levene	1.0654	2	9	0.3843
Bartlett	1.4809	2	.	0.2274

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
27.5965	2	5.2459	0.0016*

Means Comparisons for all pairs using Tukey-Kramer HSD

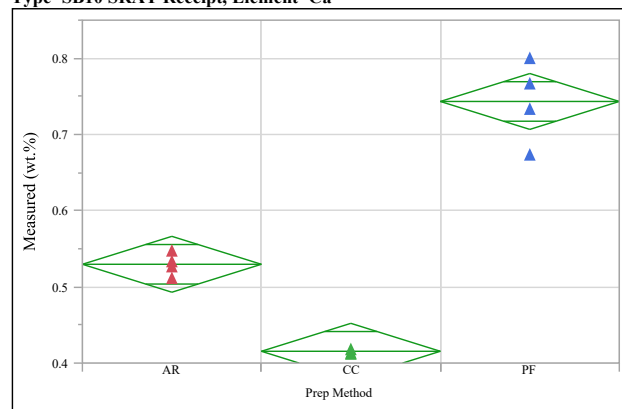
Connecting Letters Report

Level	Mean
PF	A
AR	A
CC	B

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Ca



### Oneway Anova

#### Summary of Fit

Rsquare	0.959052
Adj Rsquare	0.949952
Root Mean Square Error	0.03242
Mean of Response	0.562833
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.22154948	0.110775	105.3942	<.0001*
Error	9	0.00945947	0.001051		
C. Total	11	0.23100895			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.529666	0.01621	0.49300	0.56634
CC	4	0.415500	0.01621	0.37883	0.45217
PF	4	0.743333	0.01621	0.70666	0.78000

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.5296663	0.0149518	0.0074759	0.5058746	0.5534579
CC	4	0.4154998	0.0033276	0.0016638	0.4102047	0.4207948
PF	4	0.7433333	0.0540234	0.0270117	0.6573697	0.8292963

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0149518	0.0106667	0.0106667
CC	4	0.0033276	0.0028332	0.0028332
PF	4	0.0540234	0.0400000	0.0400000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.1878	2	9	0.1681
Brown-Forsythe	5.0241	2	9	0.0343*
Levene	5.3724	2	9	0.0291*
Bartlett	6.6363	2	.	0.0013*

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
157.1644	2	4.2027	0.0001*

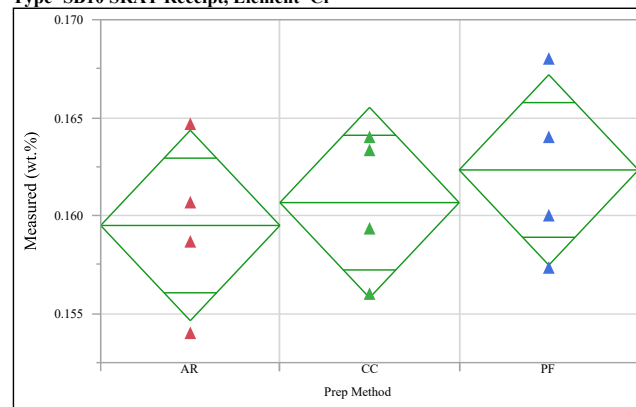
#### Means Comparisons for all pairs using Tukey-Kramer HSD

##### Connecting Letters Report

Level	Mean
PF A	0.74333300
AR B	0.52966625
CC C	0.41549975

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Cr



### Oneway Anova

#### Summary of Fit

Rsquare	0.088995
Adj Rsquare	-0.11345
Root Mean Square Error	0.004296
Mean of Response	0.160833
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00001623	8.113e-6	0.4396	0.6574
Error	9	0.00016611	0.000018		
C. Total	11	0.00018233			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.159500	0.00215	0.15464	0.16436
CC	4	0.160667	0.00215	0.15581	0.16553
PF	4	0.162333	0.00215	0.15747	0.16719

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.1594995	0.0044344	0.0022172	0.1524433	0.1665557
CC	4	0.1606665	0.0037317	0.0018659	0.1547285	0.1666045
PF	4	0.1623333	0.0046668	0.0023334	0.1549074	0.1697591

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0044344	0.0031665	0.0031665
CC	4	0.0037317	0.0030000	0.0030000
PF	4	0.0046668	0.0036668	0.0036668

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.1337	2	9	0.8766
Brown-Forsythe	0.1125	2	9	0.8949
Levene	0.1197	2	9	0.8886
Bartlett	0.0689	2	.	0.9334

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
0.3508	2	5.9407	0.7178

#### Means Comparisons for all pairs using Tukey-Kramer HSD

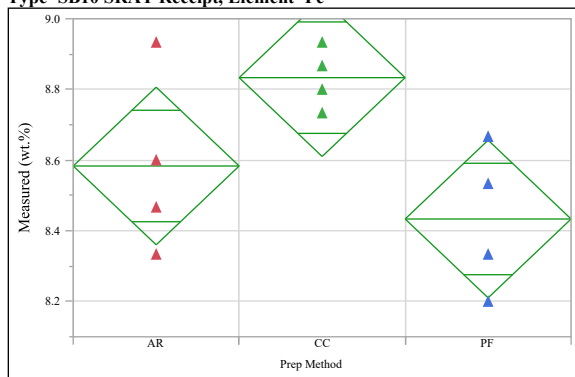
##### Connecting Letters Report

Level	Mean
PF A	0.16233325
CC A	0.16066650
AR A	0.15949950

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Fe



### Oneway Anova

#### Summary of Fit

Rsquare	0.482759
Adj Rsquare	0.367816
Root Mean Square Error	0.197203
Mean of Response	8.616666
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.32666667	0.163333	4.2000	<b>0.0515</b>
Error	9	0.34999973	0.038889		
C. Total	11	0.67666640			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	8.58333	0.09860	8.3603	8.8064
CC	4	8.83333	0.09860	8.6103	9.0564
PF	4	8.43333	0.09860	8.2103	8.6564

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	8.583333	0.2574807	0.1287404	8.1736237	8.9930423
CC	4	8.833333	0.0860662	0.0430331	8.6963825	8.9702835
PF	4	8.433333	0.2072748	0.1036374	8.1035125	8.7631535

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.2574807	0.1833335	0.1833335
CC	4	0.0860662	0.0666665	0.0666665
PF	4	0.2072748	0.1666665	0.1666665

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0119	2	9	0.4014
Brown-Forsythe	1.4828	2	9	0.2776
Levene	1.6539	2	9	<b>0.2445</b>
Bartlett	1.3393	2	.	0.2620

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

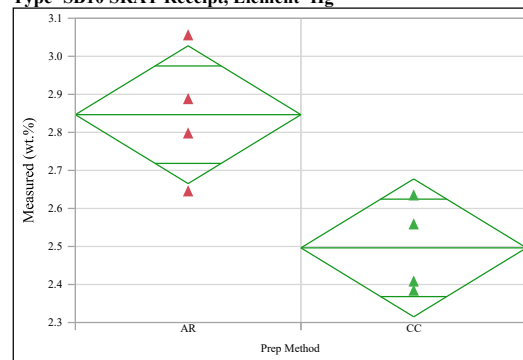
F Ratio	DFNum	DFDen	Prob > F
6.4785	2	4.9019	<b>0.0421*</b>

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

Level	Mean
CC	A
AR	A B
PF	B

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Receipt, Element=Hg



### Oneway Anova

#### Summary of Fit

Rsquare	0.650808
Adj Rsquare	0.592609
Root Mean Square Error	0.148095
Mean of Response	2.671425
Observations (or Sum Wgts)	8

#### Pooled t Test, CC-AR, Assuming equal variances

Difference	-0.35018	t Ratio	-3.34403
Std Err Dif	0.10472	DF	6
Upper CL Dif	-0.09394	Prob >  t	<b>0.0155*</b>
Lower CL Dif	-0.60642	Prob > t	0.9922
Confidence	0.95	Prob < t	<b>0.0078*</b>

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.24525627	0.245256	11.1825	<b>0.0155*</b>
Error	6	0.13159273	0.021932		
C. Total	7	0.37684899			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	2.84652	0.07405	2.6653	3.0277
CC	4	2.49633	0.07405	2.3151	2.6775

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	2.8465163	0.1715614	0.0857807	2.5735238	3.1195087
CC	4	2.4963333	0.1201288	0.0600644	2.3051815	2.687485

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.1715614	0.1251503	0.1251503
CC	4	0.1201288	0.1001332	0.1001332

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.5594	1	6	0.4828
Brown-Forsythe	0.2539	1	6	0.6323
Levene	0.2604	1	6	<b>0.6280</b>
Bartlett	0.3199	1	.	0.5717
F Test 2-sided	2.0396	3	3	0.5732

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
11.1825	1	5.3716	<b>0.0184*</b>

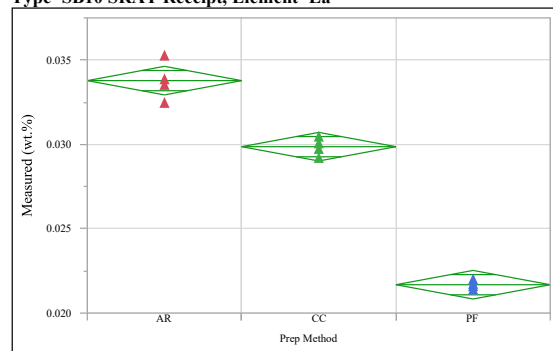
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Level	Mean
AR	A
CC	B

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=La



### Oneway Anova Summary of Fit

Rsquare	0.983695
Adj Rsquare	0.980071
Root Mean Square Error	0.000749
Mean of Response	0.028444
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00030493	0.000152	271.4855	<.0001*
Error	9	0.00000505	5.616e-7		
C. Total	11	0.00030999			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.033783	0.00037	0.03294	0.03463
CC	4	0.029866	0.00037	0.02902	0.03071
PF	4	0.021683	0.00037	0.02084	0.02253

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0337828	0.0011552	0.0005776	0.0319446	0.0356209
CC	4	0.0298663	0.0005358	0.0002679	0.0290137	0.0307188
PF	4	0.0216833	0.0002516	0.0001258	0.0212828	0.0220837

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0011552	0.0007832	0.0007832
CC	4	0.0005358	0.0003997	0.0003997
PF	4	0.0002516	0.0001832	0.0001832

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4939	2	9	0.2753
Brown-Forsythe	1.7867	2	9	0.2221
Levene	1.8193	2	9	<b>0.2170</b>
Bartlett	2.5974	2	.	0.0745

Warning: Small sample sizes. Use Caution.

### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

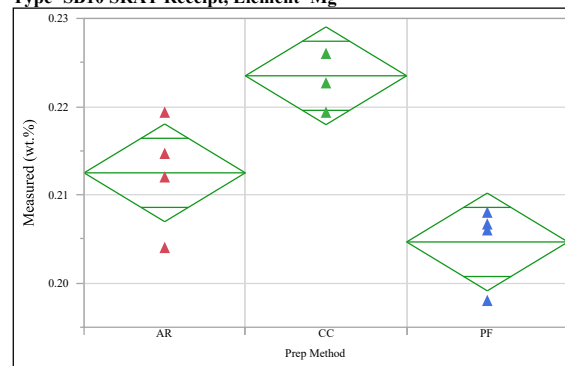
F Ratio	DFNum	DFDen	Prob > F
478.9378	2	4.838	<.0001*

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

Connecting Letters Report		
Level		Mean
AR	A	0.03378275
CC	B	0.02986625
PF	C	0.02168325

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Mg



### Oneway Anova Summary of Fit

Rsquare	0.768446
Adj Rsquare	0.716989
Root Mean Square Error	0.004896
Mean of Response	0.213555
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00071607	0.000358	14.9339	<b>0.0014*</b>
Error	9	0.00021577	0.000024		
C. Total	11	0.00093184			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.212500	0.00245	0.20696	0.21804
CC	4	0.223500	0.00245	0.21796	0.22904
PF	4	0.204667	0.00245	0.19913	0.21020

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.2124998	0.006426	0.003213	0.2022745	0.222725
CC	4	0.2234998	0.0031916	0.0015958	0.2184212	0.2285783
PF	4	0.2046665	0.0045215	0.0022607	0.1974719	0.2118611

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0064260	0.0044998	0.0044998
CC	4	0.0031916	0.0025003	0.0025003
PF	4	0.0045215	0.0033332	0.0026665

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.6633	2	9	0.5386
Brown-Forsythe	0.4522	2	9	0.6499
Levene	0.5559	2	9	<b>0.5921</b>
Bartlett	0.6161	2	.	0.5401

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
21.5275	2	5.5946	<b>0.0024*</b>

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

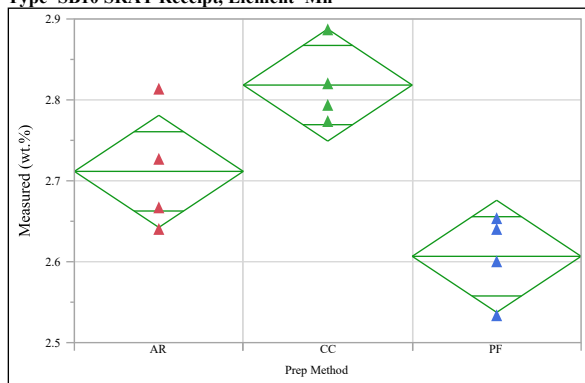
Connecting Letters Report		
Level		Mean
CC	A	0.22349975
AR	B	0.21249975
PF	B	0.20466650

Levels not connected by same letter are significantly different.



## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Mn



### Oneway Anova Summary of Fit

Rsquare	0.726372
Adj Rsquare	0.665566
Root Mean Square Error	0.061242
Mean of Response	2.712222
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.08960727	0.044804	11.9457	<b>0.0029*</b>
Error	9	0.03375550	0.003751		
C. Total	11	0.12336277			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	2.71167	0.03062	2.6424	2.7809
CC	4	2.81833	0.03062	2.7491	2.8876
PF	4	2.60667	0.03062	2.5374	2.6759

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	2.7116663	0.0768596	0.0384298	2.5893655	2.833967
CC	4	2.818333	0.0494037	0.0247019	2.7397207	2.8969453
PF	4	2.6066665	0.0538861	0.026943	2.5209217	2.6924113

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0768596	0.0583332	0.0583332
CC	4	0.0494037	0.0350000	0.0350000
PF	4	0.0538861	0.0400000	0.0400000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.5237	2	9	0.6093
Brown-Forsythe	0.4939	2	9	0.6258
Levene	0.6143	2	9	<b>0.5622</b>
Bartlett	0.3021	2	.	0.7392

Warning: Small sample sizes. Use Caution.

### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

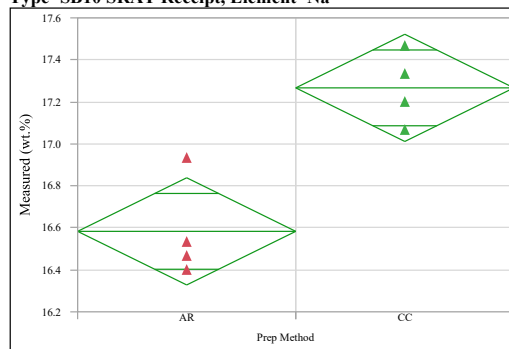
F Ratio	DFNum	DFDen	Prob > F
15.0734	2	5.8412	<b>0.0049*</b>

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

Connecting Letters Report		
Level	Mean	
CC	A	2.8183330
AR	A B	2.7116663
PF	B	2.6066665

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Na



### Oneway Anova Summary of Fit

Rsquare	0.781497
Adj Rsquare	0.74508
Root Mean Square Error	0.208611
Mean of Response	16.925
Observations (or Sum Wgts)	8

### Pooled t Test, CC-AR, Assuming equal variances

Difference	0.68333	t Ratio	4.63245
Std Err Dif	0.14751	DF	6
Upper CL Dif	1.04428	Prob >  t	<b>0.0036*</b>
Lower CL Dif	0.32239	Prob > t	<b>0.0018*</b>
Confidence	0.95	Prob < t	0.9982

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.9338866	0.933887	21.4596	<b>0.0036*</b>
Error	6	0.2611102	0.043518		
C. Total	7	1.1949968			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	16.5833	0.10431	16.328	16.839
CC	4	17.2667	0.10431	17.011	17.522

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	16.58333	0.2395981	0.1197991	16.202076	16.964584
CC	4	17.266663	0.1721322	0.0860661	16.992762	17.540563

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.2395981	0.1750000	0.1500000
CC	4	0.1721322	0.1333325	0.1333325

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.3276	1	6	0.5879
Brown-Forsythe	0.0261	1	6	0.8770
Levene	0.3087	1	6	<b>0.5986</b>
Bartlett	0.2762	1	.	0.5992
F Test 2-sided	1.9375	3	3	0.6007

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
21.4596	1	5.4454	<b>0.0046*</b>

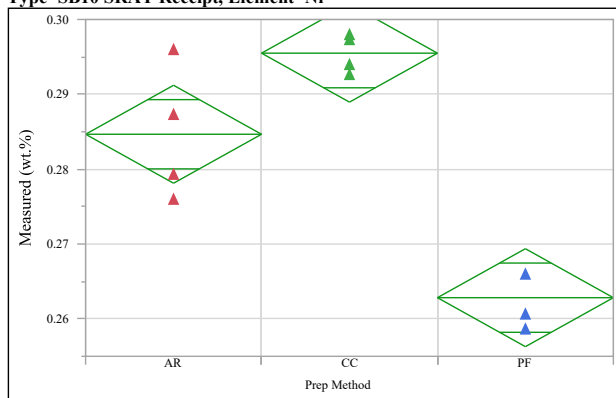
### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Connecting Letters Report		
Level		Mean
CC	A	17.266663
AR	B	16.583330

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Ni



### Oneway Anova Summary of Fit

Rsquare	0.880318
Adj Rsquare	0.853722
Root Mean Square Error	0.005784
Mean of Response	0.281
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00221490	0.001107	33.0996	<.0001*
Error	9	0.00030112	0.000033		
C. Total	11	0.00251603			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.284667	0.00289	0.27812	0.29121
CC	4	0.295500	0.00289	0.28896	0.30204
PF	4	0.262833	0.00289	0.25629	0.26938

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.2846665	0.0089277	0.0044639	0.2704605	0.2988725
CC	4	0.2954998	0.002575	0.0012875	0.2914024	0.2995971
PF	4	0.262833	0.003747	0.0018735	0.2568707	0.2687953

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0089277	0.0070000	0.0070000
CC	4	0.0025750	0.0021668	0.0021668
PF	4	0.0037470	0.0031670	0.0031670

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.3802	2	9	0.1480
Brown-Forsythe	4.3034	2	9	0.0488*
Levene	5.0690	2	9	0.0335*
Bartlett	2.1151	2	.	0.1206

Warning: Small sample sizes. Use Caution.

### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

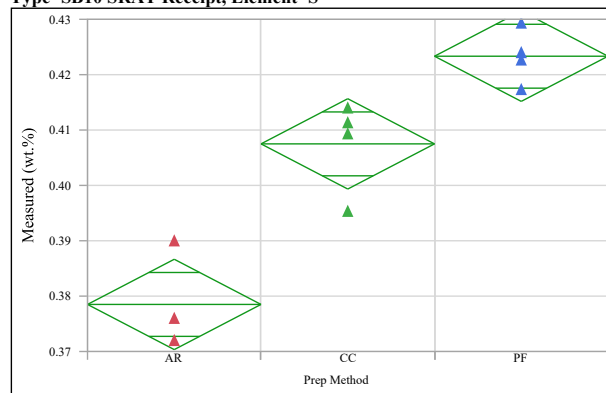
F Ratio	DFNum	DFDen	Prob > F
91.7245	2	5.3038	<.0001*

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

Level	Mean
CC A	0.29549975
AR A	0.28466650
PF B	0.26283300

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=S



### Oneway Anova Summary of Fit

Rsquare	0.898295
Adj Rsquare	0.875694
Root Mean Square Error	0.007213
Mean of Response	0.403111
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00413557	0.002068	39.7458	<.0001*
Error	9	0.00046823	0.000052		
C. Total	11	0.00460379			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.378500	0.00361	0.37034	0.38666
CC	4	0.407500	0.00361	0.39934	0.41566
PF	4	0.423333	0.00361	0.41517	0.43149

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.3785	0.0078951	0.0039476	0.3659371	0.3910629
CC	4	0.4074998	0.0083334	0.0041667	0.3942394	0.4207601
PF	4	0.423333	0.0049292	0.0024646	0.4154896	0.4311764

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0078951	0.0057500	0.0045000
CC	4	0.0083334	0.0060834	0.0051667
PF	4	0.0049292	0.0033335	0.0033335

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.3061	2	9	0.7437
Brown-Forsythe	0.1058	2	9	0.9007
Levene	0.5650	2	9	0.5873
Bartlett	0.3812	2	.	0.6830

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
41.7093	2	5.6295	0.0004*

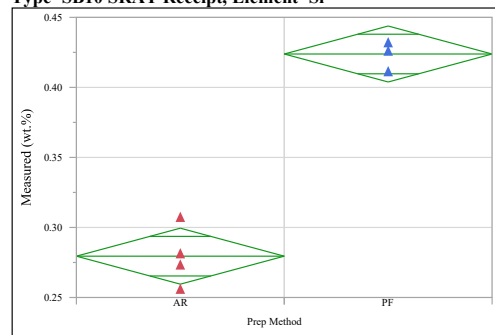
### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Level	Mean
PF A	0.42333300
CC B	0.40749975
AR C	0.37850000

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Si



### Oneway Anova

#### Summary of Fit

Rsquare	0.963003
Adj Rsquare	0.956837
Root Mean Square Error	0.016333
Mean of Response	0.351667
Observations (or Sum Wgts)	8

#### Pooled t Test, PF-AR, Assuming equal variances

Difference	0.144334	t Ratio	12.49707
Std Err Dif	0.011549	DF	6
Upper CL Dif	0.172594	Prob >  t	<.0001*
Lower CL Dif	0.116073	Prob > t	<.0001*
Confidence	0.95	Prob < t	1.0000

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.04166432	0.041664	156.1768	<.0001*
Error	6	0.00160066	0.000267		
C. Total	7	0.04326498			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.279500	0.00817	0.25952	0.29948
PF	4	0.423833	0.00817	0.40385	0.44382

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	0.2794998	0.0213566	0.0106783	0.2455166	0.3134829
PF	4	0.4238333	0.0088004	0.0044002	0.4098298	0.4378367

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0213566	0.0148333	0.0148333
PF	4	0.0088004	0.0062501	0.0051667

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.3646	1	6	0.2871
Brown-Forsythe	1.7215	1	6	0.2375
Levene	1.5666	1	6	<b>0.2573</b>
Bartlett	1.8013	1	.	0.1796
F Test 2-sided	5.8892	3	3	0.1794

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

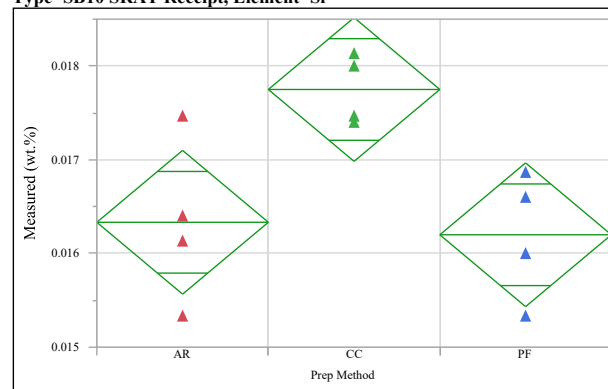
F Ratio	DFNum	DFDen	Prob > F
156.1768	1	3.9903	<b>0.0002*</b>

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

Connecting Letters Report		
Level		Mean
PF	A	0.42383325
AR	B	0.27949975

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Si



### Oneway Anova

#### Summary of Fit

Rsquare	0.588031
Adj Rsquare	0.496482
Root Mean Square Error	0.000678
Mean of Response	0.016761
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00000590	2.9516e-6	6.4231	<b>0.0185*</b>
Error	9	0.00000414	4.5953e-7		
C. Total	11	0.00001004			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.016333	0.00034	0.01557	0.01710
CC	4	0.017750	0.00034	0.01698	0.01852
PF	4	0.016200	0.00034	0.01543	0.01697

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	0.016333	0.0008809	0.0004405	0.0149312	0.0177348
CC	4	0.0177498	0.0003707	0.0001854	0.0171598	0.0183397
PF	4	0.0161998	0.000682	0.000341	0.0151146	0.0172849

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0008809	0.0006000	0.0006000
CC	4	0.0003707	0.0003168	0.0003168
PF	4	0.0006820	0.0005333	0.0005333

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.8256	2	9	0.4686
Brown-Forsythe	0.6491	2	9	0.5453
Levene	0.6822	2	9	<b>0.5298</b>
Bartlett	0.8765	2	.	0.4162

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
9.3556	2	5.2776	<b>0.0184*</b>

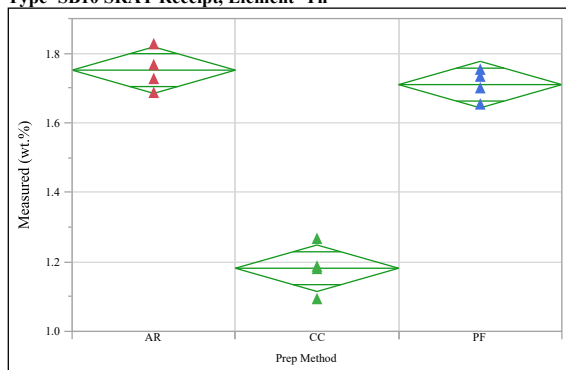
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Connecting Letters Report		
Level		Mean
CC	A	0.01774975
AR	B	0.01633300
PF	B	0.01619975

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Th



Oneway Anova  
Summary of Fit

Rsquare	0.962477
Adj Rsquare	0.954138
Root Mean Square Error	0.05915
Mean of Response	1.547777
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.80769610	0.403848	115.426	<.0001*
Error	9	0.03148882	0.003499		
C. Total	11	0.83918493			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	1.75167	0.02958	1.6848	1.8186
CC	4	1.18167	0.02958	1.1148	1.2486
PF	4	1.71000	0.02958	1.6431	1.7769

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	1.751666	0.0597216	0.0298608	1.656636	1.8466964
CC	4	1.1816663	0.0708414	0.0354207	1.068942	1.2943907
PF	4	1.7099998	0.0437162	0.0218581	1.640438	1.779562

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0597216	0.0450000	0.0450000
CC	4	0.0708414	0.0449998	0.0449998
PF	4	0.0437162	0.0333333	0.0333333

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.3745	2	9	0.6978
Brown-Forsythe	0.1467	2	9	0.8656
Levene	0.1506	2	9	<b>0.8623</b>
Bartlett	0.2937	2	.	0.7455

Warning: Small sample sizes. Use Caution.

Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

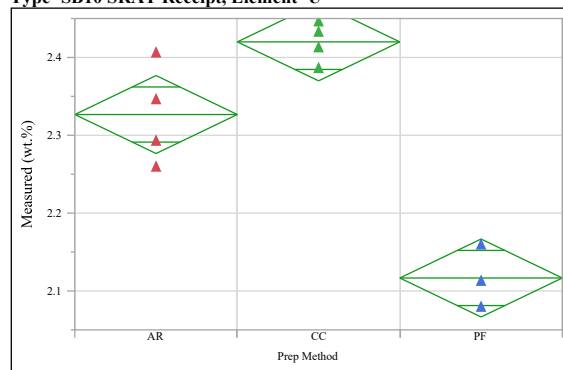
F Ratio	DFNum	DFDen	Prob > F
84.9123	2	5.756	<.0001*

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

Connecting Letters Report		
Level		Mean
AR	A	1.7516660
PF	A	1.7099998
CC	B	1.1816663

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=U



Oneway Anova  
Summary of Fit

Rsquare	0.916274
Adj Rsquare	0.897669
Root Mean Square Error	0.044277
Mean of Response	2.287777
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.19309587	0.096548	49.2470	<.0001*
Error	9	0.01764434	0.001960		
C. Total	11	0.21074021			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	2.32667	0.02214	2.2766	2.3767
CC	4	2.42000	0.02214	2.3699	2.4701
PF	4	2.11667	0.02214	2.0666	2.1667

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	2.3266663	0.064175	0.0320877	2.2245489	2.4287836
CC	4	2.4199995	0.026105	0.0130526	2.3784603	2.4615387
PF	4	2.1166665	0.032886	0.016443	2.0643377	2.1689953

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0641754	0.0499998	0.0499998
CC	4	0.0261052	0.0200000	0.0200000
PF	4	0.0328859	0.0216668	0.0200000

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6726	2	9	0.2412
Brown-Forsythe	2.2924	2	9	0.1568
Levene	2.4560	2	9	<b>0.1409</b>
Bartlett	1.1879	2	.	0.3049

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
93.4109	2	5.5486	<.0001*

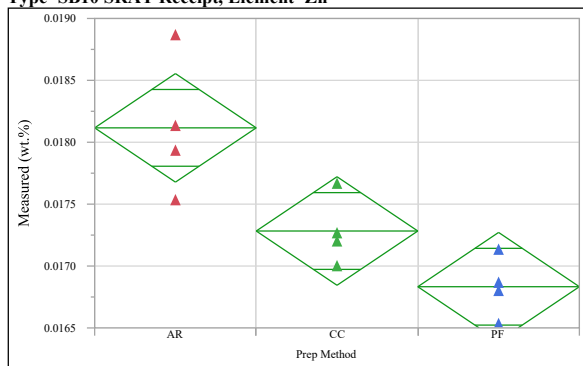
Means Comparisons for all pairs using Tukey-Kramer HSD  
Connecting Letters Report

Connecting Letters Report		
Level		Mean
CC	A	2.4199995
AR	B	2.3266663
PF	C	2.1166665

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Zn



### Oneway Anova

#### Summary of Fit

Rsquare	0.714926
Adj Rsquare	0.651576
Root Mean Square Error	0.000388
Mean of Response	0.017411
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	3.39138e-6	1.6957e-6	11.2854	<b>0.0035*</b>
Error	9	1.3523e-6	1.5026e-7		
C. Total	11	4.74368e-6			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.018116	0.00019	0.01768	0.01855
CC	4	0.017283	0.00019	0.01684	0.01772
PF	4	0.016833	0.00019	0.01639	0.01727

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0181163	0.0005586	0.0002793	0.0172274	0.0190051
CC	4	0.017283	0.0002793	0.0001396	0.0168386	0.0177274
PF	4	0.016833	0.0002464	0.0001232	0.0164409	0.0172251

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0005586	0.0003832	0.0003832
CC	4	0.0002793	0.0001915	0.0001830
PF	4	0.0002464	0.0001665	0.0001665

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.0826	2	9	0.3790
Brown-Forsythe	0.9399	2	9	0.4259
Levene	0.9981	2	9	<b>0.4060</b>
Bartlett	1.0856	2	.	0.3377

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

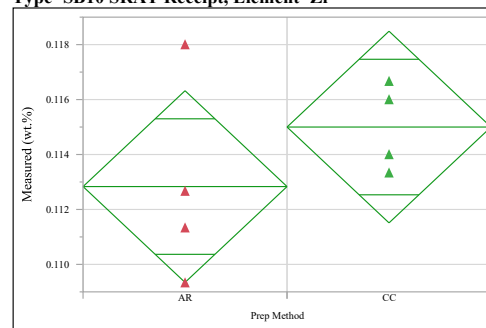
F Ratio	DFNum	DFDen	Prob > F
8.7046	2	5.6248	<b>0.0190*</b>

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

Level	Mean
AR	A
CC	B
PF	B

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Receipt, Element=Zr



### Oneway Anova

#### Summary of Fit

Rsquare	0.161417
Adj Rsquare	0.021653
Root Mean Square Error	0.002851
Mean of Response	0.113916
Observations (or Sum Wgts)	8

#### Pooled t Test, CC-AR, Assuming equal variances

Difference	0.00217	t Ratio	1.074676
Std Err Dif	0.00202	DF	6
Upper CL Dif	0.00710	Prob >  t	0.3238
Lower CL Dif	-0.00277	Prob > t	0.1619
Confidence	0.95	Prob < t	0.8381

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.00000939	9.3896e-6	1.1549	<b>0.3238</b>
Error	6	0.00004878	8.13e-6		
C. Total	7	0.00005817			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.112833	0.00143	0.10934	0.11632
CC	4	0.115000	0.00143	0.11151	0.11849

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.112833	0.003707	0.0018535	0.1069343	0.1187317
CC	4	0.1149998	0.0015869	0.0007934	0.1124747	0.1175248

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0037070	0.0025835	0.0025000
CC	4	0.0015869	0.0013333	0.0013333

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.2197	1	6	0.3117
Brown-Forsythe	0.8399	1	6	0.3948
Levene	1.2528	1	6	<b>0.3058</b>
Bartlett	1.6641	1	.	0.1970
F Test 2-sided	5.4572	3	3	0.1970

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
1.1549	1	4.0638	0.3421

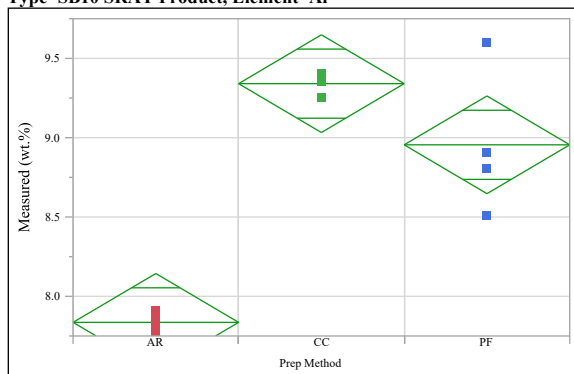
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Level	Mean
CC	A
AR	A

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=Al



### Oneway Anova

#### Summary of Fit

Rsquare	0.87984
Adj Rsquare	0.853137
Root Mean Square Error	0.272373
Mean of Response	8.710613
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	4.8889048	2.44445	32.9499	<.0001*
Error	9	0.6676820	0.07419		
C. Total	11	5.5565868			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	7.83582	0.13619	7.5277	8.1439
CC	4	9.34080	0.13619	9.0327	9.6489
PF	4	8.95522	0.13619	8.6471	9.2633

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	7.8358205	0.0642283	0.0321141	7.733619	7.938022
CC	4	9.3407955	0.0626023	0.0313011	9.2411813	9.4404097
PF	4	8.9552235	0.0631591	0.0315796	8.218234	9.692213

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0642283	0.0497510	0.0497510
CC	4	0.0626023	0.0435322	0.0373135
PF	4	0.0631591	0.0323832	0.02985075

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7059	2	9	0.2354
Brown-Forsythe	2.3260	2	9	0.1534
Levene	3.9646	2	9	<b>0.0582</b>
Bartlett	6.2297	2	.	<b>0.0020*</b>

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

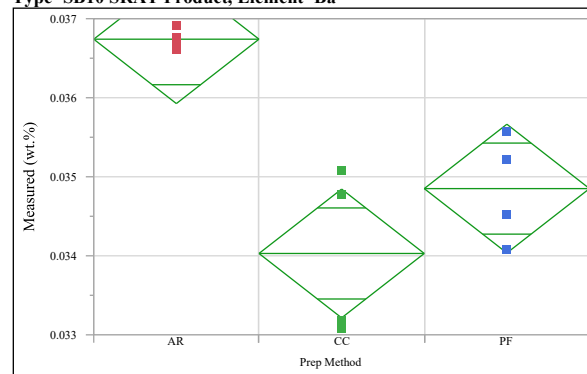
F Ratio	DFNum	DFDen	Prob > F
501.8697	2	5.3649	<.0001*

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

Connecting Letters Report		
Level		Mean
CC	A	9.3407955
PF	A	8.9552235
AR	B	7.8358205

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=Ba



### Oneway Anova

#### Summary of Fit

Rsquare	0.768122
Adj Rsquare	0.716594
Root Mean Square Error	0.00072
Mean of Response	0.035207
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00001546	7.7323e-6	14.9068	<b>0.0014*</b>
Error	9	0.00000467	5.1871e-7		
C. Total	11	0.00002013			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.036741	0.00036	0.03593	0.03756
CC	4	0.034030	0.00036	0.03321	0.03484
PF	4	0.034850	0.00036	0.03404	0.03566

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0367408	0.0001318	6.5923e-5	0.036531	0.0369505
CC	4	0.0340295	0.001042	0.000521	0.0323715	0.0356875
PF	4	0.0348503	0.0006731	0.000337	0.0337792	0.0359213

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0001318	0.0000998	0.0000998
CC	4	0.0010420	0.0008955	0.0008955
PF	4	0.0006731	0.0005472	0.0005472

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	10.5961	2	9	<b>0.0043*</b>
Brown-Forsythe	24.0991	2	9	<b>0.0002*</b>
Levene	25.6878	2	9	<b>0.0002*</b>
Bartlett	3.6483	2	.	<b>0.0260*</b>

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
24.0426	2	4.2082	<b>0.0050*</b>

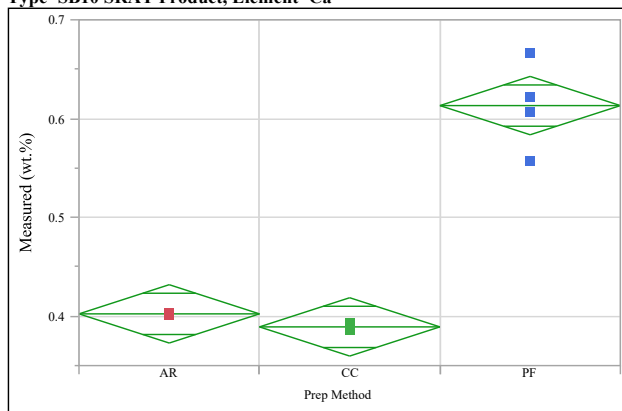
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Connecting Letters Report		
Level		Mean
AR	A	0.03674075
PF	B	0.03485025
CC	B	0.03402950

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Ca



### Oneway Anova Summary of Fit

Rsquare	0.953705
Adj Rsquare	0.943417
Root Mean Square Error	0.02611
Mean of Response	0.468242
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.12639660	0.063198	92.7018	<.0001*
Error	9	0.00613564	0.000682		
C. Total	11	0.13253224			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.402363	0.01306	0.37283	0.43190
CC	4	0.389179	0.01306	0.35965	0.41871
PF	4	0.613184	0.01306	0.58365	0.64272

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.4023628	0.0008496	0.0004248	0.4010109	0.4037146
CC	4	0.3891785	0.0029398	0.0014699	0.3845007	0.3938563
PF	4	0.6131835	0.0451204	0.0225602	0.5413869	0.6849801

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0008496	0.0006217	0.0006217
CC	4	0.0029398	0.0021145	0.0021145
PF	4	0.0451204	0.0310945	0.0310945

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.0540	2	9	0.1842
Brown-Forsythe	4.7101	2	9	0.0398*
Levene	4.7244	2	9	0.0396*
Bartlett	13.2274	2	.	<.0001*

Warning: Small sample sizes. Use Caution.

### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

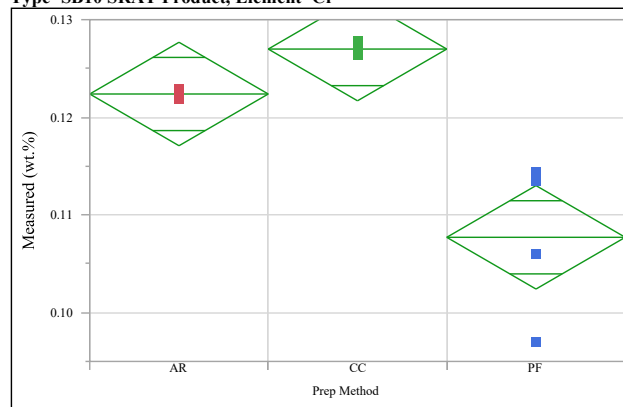
F Ratio	DFNum	DFDen	Prob > F
70.3143	2	4.3076	0.0005*

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

Level	Mean
PF	A
AR	B
CC	B

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Cr



### Oneway Anova Summary of Fit

Rsquare	0.804201
Adj Rsquare	0.76069
Root Mean Square Error	0.004684
Mean of Response	0.119029
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00081101	0.000406	18.4827	0.0007*
Error	9	0.00019746	0.000022		
C. Total	11	0.00100846			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.122388	0.00234	0.11709	0.12769
CC	4	0.126990	0.00234	0.12169	0.13229
PF	4	0.107711	0.00234	0.10241	0.11301

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.1223875	0.0005745	0.0002872	0.1214734	0.1233016
CC	4	0.1269895	0.0006258	0.0003129	0.1259936	0.1279854
PF	4	0.1077108	0.0080683	0.0040341	0.0948723	0.1205492

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0005745	0.0004975	0.0004975
CC	4	0.0006258	0.0004352	0.0003730
PF	4	0.0080683	0.0062188	0.0062188

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	2.7130	2	9	0.1197
Brown-Forsythe	7.0289	2	9	0.0145*
Levene	9.6816	2	9	0.0057*
Bartlett	9.3215	2	.	<.0001*

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
59.8357	2	5.3301	0.0002*

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

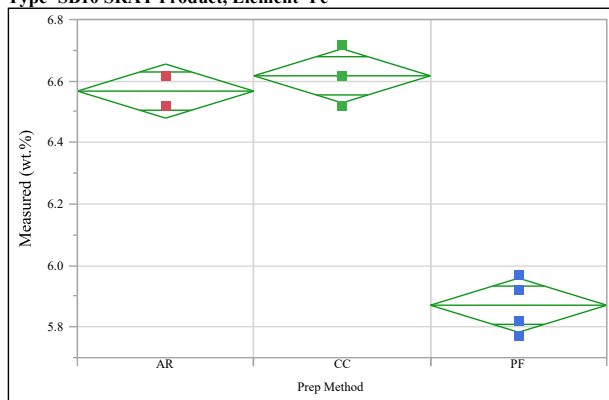
Level	Mean
CC	A
AR	A
PF	B

Levels not connected by same letter are significantly different.



## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Fe



### Oneway Anova

#### Summary of Fit

Rsquare	0.962372
Adj Rsquare	0.95401
Root Mean Square Error	0.077785
Mean of Response	6.351575
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	1.3927032	0.696352	115.0904	<.0001*
Error	9	0.0544543	0.006050		
C. Total	11	1.4471575			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	6.56716	0.03889	6.4792	6.6551
CC	4	6.61691	0.03889	6.5289	6.7049
PF	4	5.87065	0.03889	5.7827	5.9586

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	6.5671635	0.0574481	0.028724	6.4757508	6.6585762
CC	4	6.6169148	0.0812435	0.0406217	6.4876383	6.7461912
PF	4	5.8706465	0.090833	0.0454165	5.7261109	6.0151821

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0574481	0.0497515	0.0497515
CC	4	0.0812435	0.0497514	0.0497512
PF	4	0.0908330	0.0746270	0.0746270

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	0.5376	2	9	0.6018
Brown-Forsythe	0.6000	2	9	0.5694
Levene	0.6000	2	9	<b>0.5694</b>
Bartlett	0.2730	2	.	0.7611

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
87.6575	2	5.753	<.0001*

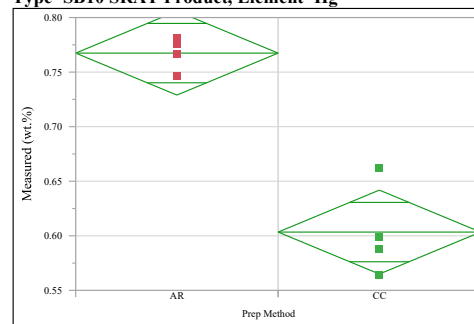
#### Means Comparisons for all pairs using Tukey-Kramer HSD

##### Connecting Letters Report

Level	Mean
CC	A
AR	A
PF	B

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Hg



### Oneway Anova

#### Summary of Fit

Rsquare	0.900716
Adj Rsquare	0.884169
Root Mean Square Error	0.031435
Mean of Response	0.685416
Observations (or Sum Wgts)	8

#### Pooled t Test, CC-AR, Assuming equal variances

Difference	-0.16399	t Ratio	-7.37785
Std Err Dif	0.02223	DF	6
Upper CL Dif	-0.10960	Prob >  t	<b>0.0003*</b>
Lower CL Dif	-0.21838	Prob > t	0.9998
Confidence	0.95	Prob < t	<b>0.0002*</b>

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.05378724	0.053787	54.4327	<b>0.0003*</b>
Error	6	0.00592886	0.000988		
C. Total	7	0.05971610			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.767413	0.01572	0.72895	0.80587
CC	4	0.603420	0.01572	0.56496	0.64188

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.7674125	0.0154015	0.0077008	0.7429052	0.7919198
CC	4	0.6034198	0.0417023	0.0208511	0.5370621	0.6697774

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0154015	0.0111940	0.0111940
CC	4	0.0417023	0.0293346	0.0271268

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.2933	1	6	0.2988
Brown-Forsythe	1.0414	1	6	0.3469
Levene	1.9889	1	6	<b>0.2081</b>
Bartlett	2.2156	1	.	0.1366
F Test 2-sided	7.3315	3	3	0.1360

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
54.4327	1	3.8034	<b>0.0022*</b>

#### Means Comparisons for all pairs using Tukey-Kramer HSD

##### Connecting Letters Report

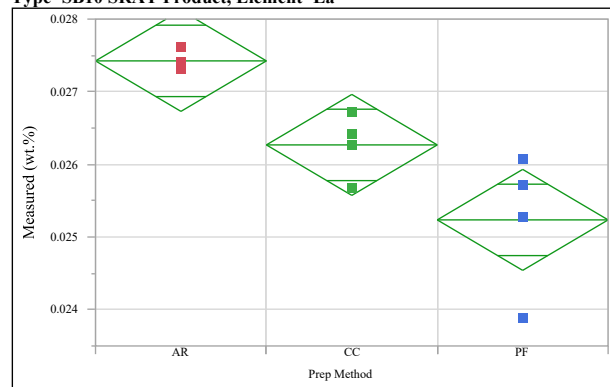
Level	Mean
AR	A
CC	B

Levels not connected by same letter are significantly different.



## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=La



### Oneway Anova

#### Summary of Fit

Rsquare	0.738354
Adj Rsquare	0.680211
Root Mean Square Error	0.000615
Mean of Response	0.02631
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.0000959	4.7969e-6	12.6988	<b>0.0024*</b>
Error	9	0.0000340	3.7774e-7		
C. Total	11	0.0001299			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.027425	0.00031	0.02673	0.02812
CC	4	0.026268	0.00031	0.02557	0.02696
PF	4	0.025236	0.00031	0.02454	0.02593

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0274248	0.0001306	6.529e-5	0.027217	0.0276325
CC	4	0.026268	0.0004394	0.0002197	0.0255688	0.0269672
PF	4	0.0252358	0.0009608	0.0004804	0.023707	0.0267645

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0001306	0.0000931	0.0000868
CC	4	0.0004394	0.0002985	0.0002985
PF	4	0.0009608	0.0006779	0.0006593

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.4356	2	9	0.2876
Brown-Forsythe	1.9146	2	9	0.2029
Levene	2.7067	2	9	<b>0.1201</b>
Bartlett	3.7567	2	.	<b>0.0234*</b>

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

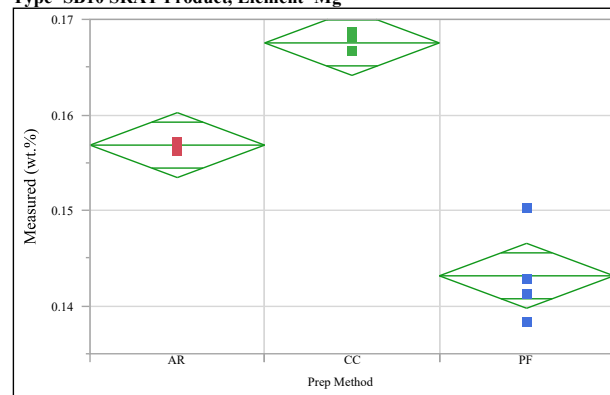
F Ratio	DFNum	DFDen	Prob > F
19.1735	2	4.3883	<b>0.0068*</b>

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

Connecting Letters Report			
Level	Mean		
AR	A	0.02742475	
CC	A	B	0.02626800
PF		B	0.02523575

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Mg



### Oneway Anova

#### Summary of Fit

Rsquare	0.936317
Adj Rsquare	0.922165
Root Mean Square Error	0.003005
Mean of Response	0.155845
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00119451	0.000597	66.1624	<b>&lt;.0001*</b>
Error	9	0.00008124	9.027e-6		
C. Total	11	0.00127576			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.156840	0.00150	0.15344	0.16024
CC	4	0.167537	0.00150	0.16414	0.17094
PF	4	0.143159	0.00150	0.13976	0.14656

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.15684	0.0004763	0.0002381	0.1560821	0.1575979
CC	4	0.1675368	0.0010257	0.0005129	0.1659046	0.1691689
PF	4	0.1431588	0.0050796	0.0025398	0.135076	0.1512415

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0004763	0.0003730	0.0003730
CC	4	0.0010257	0.0008707	0.0008707
PF	4	0.0050796	0.0035446	0.0033583

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7173	2	9	0.2335
Brown-Forsythe	2.4439	2	9	0.1420
Levene	3.8230	2	9	<b>0.0628</b>
Bartlett	6.2487	2	.	<b>0.0019*</b>

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
173.1088	2	4.7111	<b>&lt;.0001*</b>

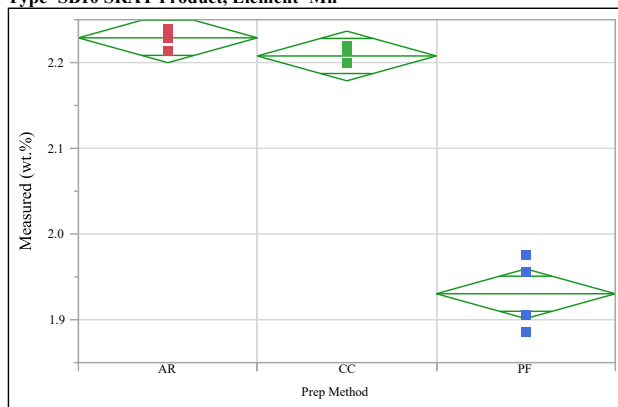
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Connecting Letters Report		
Level		Mean
CC	A	0.16753675
AR	B	0.15684000
PF	C	0.14315875

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Mn



### Oneway Anova Summary of Fit

Rsquare	0.974069
Adj Rsquare	0.968306
Root Mean Square Error	0.025624
Mean of Response	2.122305
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.22197908	0.110990	169.0350	<.0001*
Error	9	0.00590946	0.000657		
C. Total	11	0.22788854			

### Means for Oneway Anova

Level	Num	Mean	Std Error	Lower 95%	Upper 95%
AR	4	2.22886	0.01281	2.1999	2.2578
CC	4	2.20771	0.01281	2.1787	2.2367
PF	4	1.93035	0.01281	1.9014	1.9593

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	2.2288553	0.0107475	0.0053738	2.2117535	2.245957
CC	4	2.2077113	0.0102562	0.0051281	2.1913913	2.2240312
PF	4	1.9303478	0.0418225	0.0209112	1.8637988	1.9968967

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0107475	0.0074628	0.0074628
CC	4	0.0102562	0.0087062	0.0087062
PF	4	0.0418225	0.0348257	0.0348257

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	5.9343	2	9	0.0227*
Brown-Forsythe	15.2639	2	9	0.0013*
Levene	16.1512	2	9	0.0011*
Bartlett	3.3827	2	.	0.0340*

Warning: Small sample sizes. Use Caution.

### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

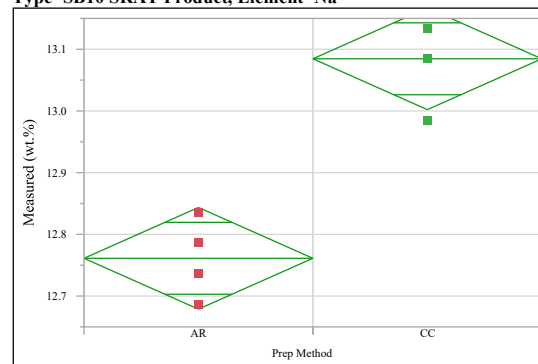
F Ratio	DFNum	DFDen	Prob > F
85.1921	2	5.4351	<.0001*

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

Level	Mean
AR	2.2288553
CC	2.2077113
PF	1.9303478

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Na



### Oneway Anova Summary of Fit

Rsquare	0.884816
Adj Rsquare	0.865619
Root Mean Square Error	0.067364
Mean of Response	12.92288
Observations (or Sum Wgts)	8

### Pooled t Test, CC-AR, Assuming equal variances

Difference	0.323383	t Ratio	6.789
Std Err Dif	0.047633	DF	6
Upper CL Dif	0.439937	Prob >  t	0.0005*
Lower CL Dif	0.206828	Prob > t	0.0002*
Confidence	0.95	Prob < t	0.9998

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	0.20915248	0.209152	46.0905	0.0005*
Error	6	0.02722718	0.004538		
C. Total	7	0.23637966			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	12.7612	0.03368	12.679	12.844
CC	4	13.0846	0.03368	13.002	13.167

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	12.761188	0.0642308	0.0321154	12.658982	12.863393
CC	4	13.08457	0.0703571	0.0351786	12.972616	13.196524

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0642308	0.0497525	0.0497525
CC	4	0.0703571	0.0497500	0.0497500

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.0369	1	6	0.8541
Brown-Forsythe	0.0000	1	6	0.9999
Levene	0.0000	1	6	0.9999
Bartlett	0.0213	1	.	0.8839
F Test 2-sided	1.1999	3	3	0.8845

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
46.0905	1	5.9509	0.0005*

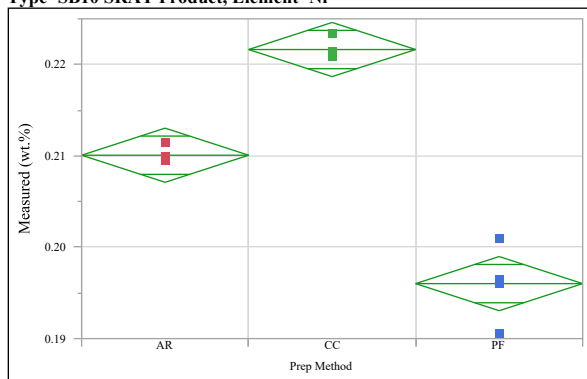
### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Level	Mean
CC	13.084570
AR	12.761188

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=Ni



### Oneway Anova

#### Summary of Fit

Rsquare	0.955167
Adj Rsquare	0.945204
Root Mean Square Error	0.002621
Mean of Response	0.209245
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00131710	0.000659	95.8725	<.0001*
Error	9	0.00006182	6.869e-6		
C. Total	11	0.00137892			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.210074	0.00131	0.20711	0.21304
CC	4	0.221642	0.00131	0.21868	0.22461
PF	4	0.196020	0.00131	0.19306	0.19898

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.210074	0.0009417	0.0004709	0.2085755	0.2115725
CC	4	0.2216415	0.0011845	0.0005922	0.2197567	0.2235263
PF	4	0.1960195	0.0042799	0.0021399	0.1892093	0.2028297

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0009417	0.0006840	0.0006220
CC	4	0.0011845	0.0008707	0.0007465
PF	4	0.0042799	0.0027365	0.0027365

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6722	2	9	0.2412
Brown-Forsythe	1.6959	2	9	0.2371
Levene	1.7198	2	9	0.2331
Bartlett	3.4682	2	.	0.0312*

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
136.2995	2	5.3462	<.0001*

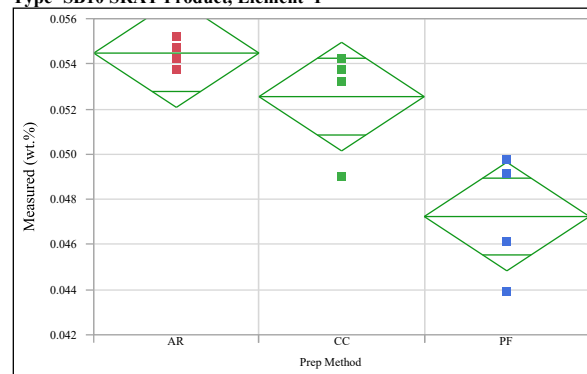
#### Means Comparisons for all pairs using Tukey-Kramer HSD

##### Connecting Letters Report

Level	Mean
CC A	0.22164150
AR B	0.21007400
PF C	0.19601950

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=P



### Oneway Anova

#### Summary of Fit

Rsquare	0.734379
Adj Rsquare	0.675352
Root Mean Square Error	0.002126
Mean of Response	0.051422
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00011242	0.000056	12.4414	0.0026*
Error	9	0.00004066	4.518e-6		
C. Total	11	0.00015308			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.054477	0.00106	0.05207	0.05688
CC	4	0.052549	0.00106	0.05015	0.05495
PF	4	0.047239	0.00106	0.04483	0.04964

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Leve	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.054477	0.0006421	0.0003211	0.0534552	0.0554988
CC	4	0.0525493	0.0023975	0.0011987	0.0487343	0.0563642
PF	4	0.0472385	0.0027191	0.0013596	0.0429117	0.0515653

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0006421	0.0004975	0.0004975
CC	4	0.0023975	0.0017721	0.0014302
PF	4	0.0027191	0.0022140	0.0022140

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.1863	2	9	0.3489
Brown-Forsythe	1.6619	2	9	0.2431
Levene	3.8113	2	9	0.0632
Bartlett	2.1695	2	.	0.1142

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
12.3561	2	4.4615	0.0152*

#### Means Comparisons for all pairs using Tukey-Kramer HSD

##### Connecting Letters Report

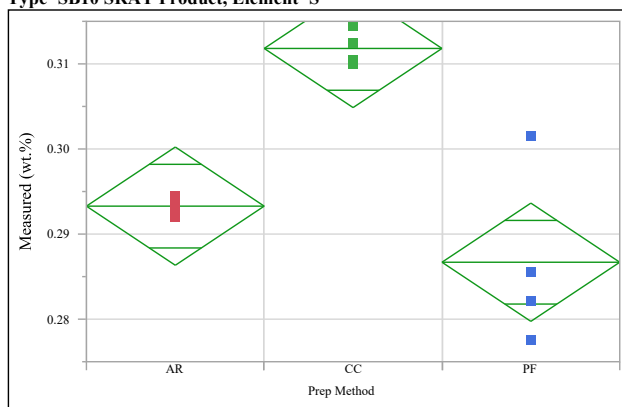
Level	Mean
AR A	0.05447700
CC A	0.05254925
PF B	0.04723850

Levels not connected by same letter are significantly different.

A

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=S



### Oneway Anova Summary of Fit

Rsquare	0.799835
Adj Rsquare	0.755354
Root Mean Square Error	0.006144
Mean of Response	0.297263
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.00135750	0.000679	17.9815	0.0007*
Error	9	0.00033972	0.000038		
C. Total	11	0.00169723			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.293283	0.00307	0.28633	0.30023
CC	4	0.311815	0.00307	0.30487	0.31876
PF	4	0.286691	0.00307	0.27974	0.29364

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	0.293283	0.0010359	0.0005179	0.2916347	0.2949313
CC	4	0.3118153	0.0020461	0.001023	0.3085595	0.315071
PF	4	0.286691	0.0103914	0.0051957	0.2701559	0.3032261

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0010359	0.0007465	0.0007465
CC	4	0.0020461	0.0016168	0.0016168
PF	4	0.0103914	0.0074005	0.0068410

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.6528	2	9	0.2447
Brown-Forsythe	2.2593	2	9	0.1603
Levene	4.3615	2	9	0.0474*
Bartlett	6.1514	2	.	0.0021*

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
116.4281	2	4.8056	<.0001*

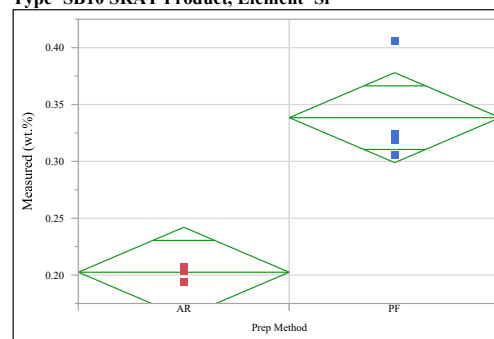
### Means Comparisons for all pairs using Tukey-Kramer HSD

#### Connecting Letters Report

Level	Mean
CC A	0.31181525
AR B	0.29328300
PF B	0.28669100

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Si



### Oneway Anova

#### Summary of Fit

Rsquare	0.854857
Adj Rsquare	0.830666
Root Mean Square Error	0.032341
Mean of Response	0.27046
Observations (or Sum Wgts)	8

### Pooled t Test, PF-AR, Assuming equal variances

Difference	0.135946	t Ratio	5.94462
Std Err Dif	0.022869	DF	6
Upper CL Dif	0.191903	Prob >  t	0.0010*
Lower CL Dif	0.079988	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
Prep Method	1	0.03696236	0.036962	35.3385	0.0010*
Error	6	0.00627571	0.001046		
C. Total	7	0.04323807			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.202487	0.01617	0.16292	0.24205
PF	4	0.338433	0.01617	0.29886	0.37800

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err	Lower 95%	Upper 95%
AR	4	0.202487	0.0061872	0.0030936	0.1926418	0.2123322
PF	4	0.3384325	0.0453169	0.0226585	0.2663232	0.4105418

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0061872	0.0044775	0.0039800
PF	4	0.0453169	0.0335198	0.0262435

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.4586	1	6	0.2726
Brown-Forsythe	1.2740	1	6	0.3021
Levene	5.9495	1	6	0.0505
Bartlett	6.7707	1	.	0.0093*
F Test 2-sided	53.6460	3	3	0.0084*

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
35.3385	1	3.1118	0.0086*

### Means Comparisons for all pairs using Tukey-Kramer HSD

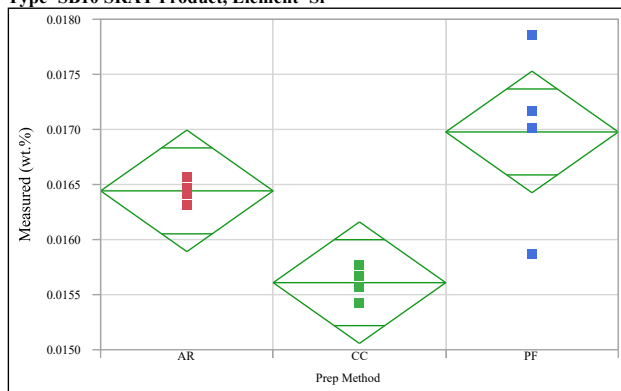
#### Connecting Letters Report

Level	Mean
PF A	0.33843250
AR B	0.20248700

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Sr



### Oneway Anova

#### Summary of Fit

Rsquare	0.639851
Adj Rsquare	0.559817
Root Mean Square Error	0.000488
Mean of Response	0.016343
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	3.80225e-6	1.9011e-6	7.9948	<b>0.0101*</b>
Error	9	2.14015e-6	2.3779e-7		
C. Total	11	5.9424e-6			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.016442	0.00024	0.01589	0.01699
CC	4	0.015609	0.00024	0.01506	0.01616
PF	4	0.016977	0.00024	0.01643	0.01753

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0164423	0.0001037	5.1842e-5	0.0162773	0.0166072
CC	4	0.015609	0.0001488	0.0000744	0.0153722	0.0158458
PF	4	0.016977	0.0008249	0.0004125	0.0156644	0.0182896

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0001037	0.0000747	0.0000747
CC	4	0.0001488	0.0001120	0.0001120
PF	4	0.0008249	0.0005535	0.0005350

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.7807	2	9	0.2231
Brown-Forsythe	2.4169	2	9	0.1445
Levene	3.0315	2	9	<b>0.0985</b>
Bartlett	5.7732	2	.	<b>0.0031*</b>

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
39.0701	2	5.1668	<b>0.0008*</b>

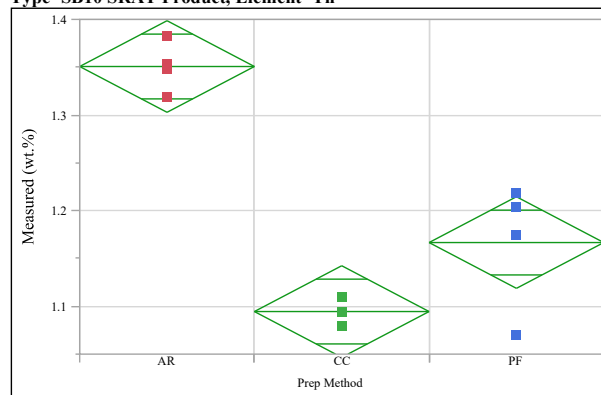
Means Comparisons for all pairs using Tukey-Kramer HSD

#### Connecting Letters Report

Level	Mean
PF A	0.01697700
AR A B	0.01644225
CC B	0.01560900

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Th



### Oneway Anova

#### Summary of Fit

Rsquare	0.896409
Adj Rsquare	0.873389
Root Mean Square Error	0.042345
Mean of Response	1.20398
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.13964984	0.069825	38.9401	<b>&lt;.0001*</b>
Error	9	0.01613823	0.001793		
C. Total	11	0.15578808			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	1.35075	0.02117	1.3028	1.3986
CC	4	1.09453	0.02117	1.0466	1.1424
PF	4	1.16667	0.02117	1.1188	1.2146

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	1.3507458	0.0264819	0.0132409	1.3086072	1.3928843
CC	4	1.094527	0.0121862	0.0060931	1.075136	1.113918
PF	4	1.1666663	0.0673024	0.0336512	1.0595731	1.2737594

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0264819	0.0174127	0.0174127
CC	4	0.0121862	0.0074625	0.0074625
PF	4	0.0673024	0.0485076	0.0447763

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3936	2	9	0.2970
Brown-Forsythe	1.5470	2	9	0.2646
Levene	3.1194	2	9	<b>0.0935</b>
Bartlett	3.2704	2	.	<b>0.0380*</b>

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
135.8589	2	4.7754	<b>&lt;.0001*</b>

Means Comparisons for all pairs using Tukey-Kramer HSD

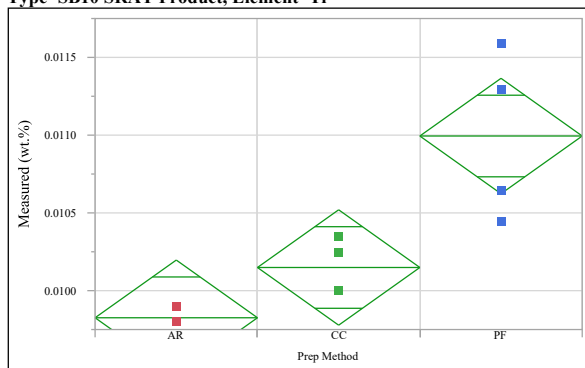
#### Connecting Letters Report

Level	Mean
AR A	1.3507458
PF B	1.1666663
CC B	1.0945270

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=Ti



### Oneway Anova

#### Summary of Fit

Rsquare	0.750539
Adj Rsquare	0.695103
Root Mean Square Error	0.000328
Mean of Response	0.010323
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	2.91378e-6	1.4569e-6	13.5389	0.0019*
Error	9	9.68472e-7	1.0761e-7		
C. Total	11	3.88225e-6			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.009826	0.00016	0.00945	0.01020
CC	4	0.010149	0.00016	0.00978	0.01052
PF	4	0.010995	0.00016	0.01062	0.01137

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0098258	0.0000495	2.475e-5	0.009747	0.0099045
CC	4	0.010149	0.0001768	8.8414e-5	0.0098676	0.0104304
PF	4	0.0109945	0.0005377	0.0002688	0.0101389	0.0118501

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0000495	0.0000371	0.0000248
CC	4	0.0001768	0.0001490	0.0001490
PF	4	0.0005377	0.0004480	0.0004480

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	6.1612	2	9	0.0206*
Brown-Forsythe	20.8166	2	9	0.0004*
Levene	22.7704	2	9	0.0003*
Bartlett	5.2649	2	.	0.0052*

Warning: Small sample sizes. Use Caution.

#### Welch's Test

Welch Anova testing Means Equal, allowing Std Devs Not Equal

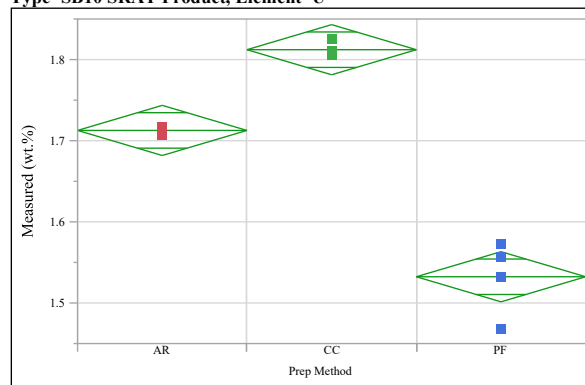
F Ratio	DFNum	DFDen	Prob > F
13.1686	2	4.32	0.0145*

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

Connecting Letters Report		
Level		Mean
PF	A	0.01099450
CC	B	0.01014900
AR	B	0.00982575

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample  
Type=SB10 SRAT Product, Element=U



### Oneway Anova

#### Summary of Fit

Rsquare	0.95993
Adj Rsquare	0.951026
Root Mean Square Error	0.027325
Mean of Response	1.685737
Observations (or Sum Wgts)	12

#### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	0.16099044	0.080495	107.8040	<.0001*
Error	9	0.00672013	0.000747		
C. Total	11	0.16771057			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	1.71269	0.01366	1.6818	1.7436
CC	4	1.81219	0.01366	1.7813	1.8431
PF	4	1.53234	0.01366	1.5014	1.5632

Std Error uses a pooled estimate of error variance

#### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	1.7126858	0.0047632	0.0023816	1.7051064	1.7202651
CC	4	1.8121888	0.0094175	0.0047088	1.7972034	1.8271741
PF	4	1.5323378	0.0461375	0.0230687	1.4589227	1.6057528

#### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0047632	0.0037313	0.0037313
CC	4	0.0094175	0.0068406	0.0062187
PF	4	0.0461375	0.0323384	0.0323382

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.8185	2	9	0.2171
Brown-Forsythe	3.0062	2	9	0.1000
Levene	3.8659	2	9	0.0614
Bartlett	5.9792	2	.	0.0025*

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
189.0628	2	4.8067	<.0001*

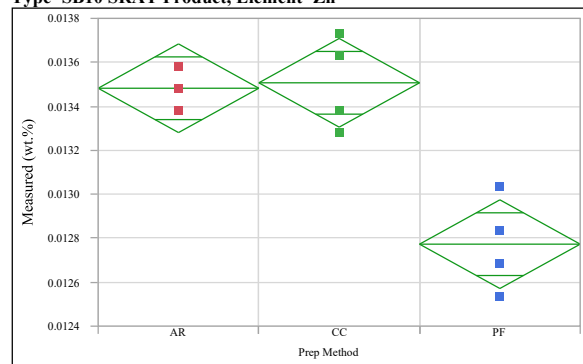
#### Means Comparisons for all pairs using Tukey-Kramer HSD Connecting Letters Report

Connecting Letters Report		
Level		Mean
CC	A	1.8121888
AR	B	1.7126858
PF	C	1.5323378

Levels not connected by same letter are significantly different.

## Appendix A-6. Analysis of Variance Investigating the Elemental Means and Variances of the Preparation Method

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=Zn



### Oneway Anova Summary of Fit

Rsquare	0.829276
Adj Rsquare	0.791337
Root Mean Square Error	0.000178
Mean of Response	0.013254
Observations (or Sum Wgts)	12

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	2	1.38987e-6	6.9494e-7	21.8583	<b>0.0004*</b>
Error	9	2.86135e-7	3.1793e-8		
C. Total	11	1.67601e-6			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.013482	0.00009	0.01328	0.01368
CC	4	0.013507	0.00009	0.01331	0.01371
PF	4	0.012773	0.00009	0.01257	0.01297

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0134823	8.1242e-5	4.0621e-5	0.013353	0.0136115
CC	4	0.013507	0.000209	0.0001045	0.0131744	0.0138396
PF	4	0.012773	0.0002123	0.0001062	0.0124352	0.0131108

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0000812	0.0000499	0.0000497
CC	4	0.0002090	0.0001740	0.0001740
PF	4	0.0002123	0.0001615	0.0001615

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	1.3566	2	9	0.3055
Brown-Forsythe	3.2764	2	9	0.0853
Levene	3.3130	2	9	<b>0.0835</b>
Bartlett	1.1822	2	.	0.3066

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
17.6264	2	4.9373	<b>0.0056*</b>

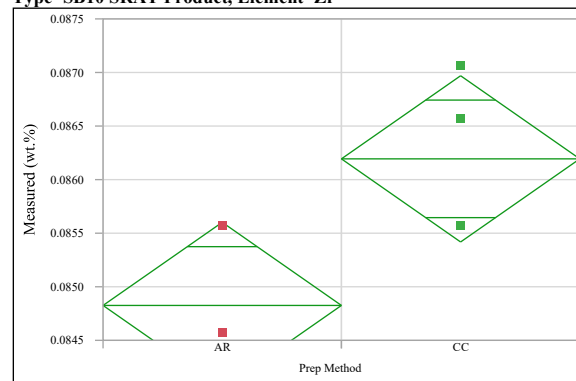
Means Comparisons for all pairs using Tukey-Kramer HSD

### Connecting Letters Report

Level	Mean
CC A	0.01350700
AR A	0.01348225
PF B	0.01277300

Levels not connected by same letter are significantly different.

Oneway Analysis of Measured (wt.%) By Prep Method Sample Type=SB10 SRAT Product, Element=Zr



### Oneway Anova Summary of Fit

Rsquare	0.608083
Adj Rsquare	0.542763
Root Mean Square Error	0.000634
Mean of Response	0.08551
Observations (or Sum Wgts)	8

### Pooled t Test, CC-AR, Assuming equal variances

Difference	0.001368	t Ratio	3.051123
Std Err Dif	0.000448	DF	6
Upper CL Dif	0.002465	Prob >  t	<b>0.0225*</b>
Lower CL Dif	0.000271	Prob > t	<b>0.0112*</b>
Confidence	0.95	Prob < t	0.9888

### Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep Method	1	3.74285e-6	3.7428e-6	9.3093	<b>0.0225*</b>
Error	6	2.41232e-7	4.0205e-7		
C. Total	7	6.15516e-6			

### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.084826	0.00032	0.08405	0.08560
CC	4	0.086194	0.00032	0.08542	0.08697

Std Error uses a pooled estimate of error variance

### Means and Std Deviations

Level	Num	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AR	4	0.0848258	0.0004975	0.0002487	0.0840341	0.0856174
CC	4	0.0861938	0.0007461	0.000373	0.0850066	0.0873809

### Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
AR	4	0.0004975	0.0003731	0.0002487
CC	4	0.0007461	0.0006218	0.0006218

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	1.0902	1	6	0.3367
Brown-Forsythe	1.7995	1	6	0.2283
Levene	2.3995	1	6	<b>0.1723</b>
Bartlett	0.4111	1	.	0.5214
F Test 2-sided	2.2488	3	3	0.5229

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
9.3093	1	5.2276	<b>0.0268*</b>

Means Comparisons for all pairs using Tukey-Kramer HSD

### Connecting Letters Report

Level	Mean
CC A	0.08619375
AR B	0.08482575

Levels not connected by same letter are significantly different.



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