DEFENSE WASTE PROCESSING FACILITY (DWPF) ANALYTICAL METHOD VERIFICATION FOR THE SLUDGE BATCH 5 QUALIFICATION SAMPLE

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July 25, 2008

Analytical Development
Savannah River National Laboratory
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DE-AC09-08SR22470

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TABLE OF CONTENTS

LIST OF AND FIGURES AND TABLES VIII
LIST OF ACRONYMS X
1.0 SUMMARY 12
2.0 EXPERIMENTAL 14
3.0 INTRODUCTION AND RESULTS 16
4.0 CONCLUSIONS 26
5.0 RECOMMENDATIONS 28
6.0 REFERENCES 30
7.0 ACKNOWLEDGEMENTS 32
8.0 APPENDIX A. SUPPORTING INFORMATION 34

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LIST OF AND FIGURES AND TABLES

Figure 3-1. Angle of Intersection and Significance for Compariosn Circles.....17
Table 3-1. Elemental concentrations of SB5 SRAT Receipt radioactive sludge slurry obtained from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions. Values are presented on a weight percent (Wt%) total solids basis. 18
Table 3-2. Statistical comparison of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions of SB5 SRAT Receipt sludge. Digestions not having the same letter are statistically different at the 5% significance level.....19
Table 3-3. Elemental concentrations of SB5 SRAT Product radioactive sludge slurry obtained from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions. Values are presented on a weight percent (Wt%) total solids basis. 20
Table 3-4. Statistical comparison of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions of SB5 SRAT Product sludge. Digestions not having the same letter are statistically different at the 5% significance level.....21
Table 3-5. Elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Receipt. Values are presented on a weight percent (Wt%) total solids basis. 22
Table 3-6. Continuation of elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Receipt. Values are presented on a weight percent (Wt%) total solids basis. 23
Table 3-7. Elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Product. Values are presented on a weight percent (Wt%) total solids basis.....24
Table 3-8. Continuation of elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Product. Values are presented on a weight percent (Wt%) total solids basis.....25

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LIST OF ACRONYMS

AD	Analytical Development
AR	Aqua Regia
ARG	Analytical Reference Glass
CC	Cold Chem Method
DI	De-Ionized
DOE	Department of Energy
DWPF	Defense Waste Processing Facility
ICP-ES	Inductively Coupled Plasma Emission Spectroscopy
PF	Sodium Peroxide/Hydroxide Fusion
PS&E	Process Science and Engineering
SB	Sludge Batch
SRAT	Slurry Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory

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1.0 SUMMARY

For each sludge batch that is processed in the Defense Waste Processing Facility (DWPF), the Savannah River National Laboratory (SRNL) performs confirmation of the applicability of the digestion method to be used by the DWPF lab 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 room temperature HF-HNO_3 acid dissolution (i.e., DWPF Cold Chem Method, see Procedure SW4-15.201) and then analyzed by inductively coupled plasma – atomic emission spectroscopy (ICP-AES).

This report contains the results and comparison of data generated from performing the Aqua Regia (AR), Sodium Peroxide/Hydroxide Fusion (PF) and DWPF Cold Chem (CC) method digestion of Sludge Batch 5 (SB5) SRAT Receipt and SB5 SRAT Product samples. The SB5 SRAT Receipt and SB5 SRAT Product samples were prepared in the SRNL Shielded Cells, and the SRAT Receipt material is representative of the sludge that constitutes the SB5 Batch composition. This is the sludge in Tank 51 that is to be transferred into Tank 40, which will contain the heel of Sludge Batch 4 (SB4), to form the SB5 Blend composition. The results for any one particular element should not be used in any way to identify the form or speciation of a particular element in the sludge or used to estimate ratios of compounds in the sludge.

A statistical comparison of the data validates the use of the DWPF CC method for SB5 Batch composition. However, the difficulty that was encountered in using the CC method for SB4 brings into question the adequacy of CC for the SB5 Blend. Also, it should be noted that visible solids remained in the final diluted solutions of all samples digested by this method at SRNL (8 samples total), which is typical for the DWPF CC method but not seen in the other methods.

Recommendations to the DWPF for application to SB5 based on studies to date:

- A dissolution study should be performed on the WAPS sample by SRNL which consists of the final composition of the sludge (the SB5 Blend).

- Given the heel of SB4 in Tank 40, the DWPF lab should monitor the aluminum concentration in the first 10 SRAT Receipt batches of SB5 using both CC and sodium peroxide/hydroxide fusion to evaluate the adequacy of aluminum recovery by the CC method for this sludge batch.

- SRNL and the DWPF lab should investigate if comparisons between the elemental concentrations of the SME product glass (adjusted for frit addition) obtained by the mixed acid and peroxide fusion digestion and the SRAT Receipt and SRAT Product elemental concentrations obtained *via* the DWPF CC method provide insight into the adequacy of the CC method for analysis of the SRAT Product. The DWPF lab would need to calcine the SRAT product at 1050 ºC for the best comparison. If a consistent difference in elemental concentrations is revealed, another type of digestion (i.e. sodium peroxide/hydroxide fusion) should be used to determine the concentration of the element in question. Particular emphasis should be placed on monitoring the aluminum concentration in SB5.

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2.0 EXPERIMENTAL

The radioactive sludge slurry used in this study for verification of the DWPF CC method is from the three liter sample of Tank 51 sludge slurry taken on March 21, 2008. The sample was delivered to SRNL, characterized and then modified by a series of wash/decant cycles to match the LWO planned preparation strategy. The sludge used in this testing corresponded to Wash F, identified by Bannochie et. al. in SRNL-PSE-2008-1126, which is the SB5 qualification sample.

The sludge samples were dissolved in quadruplicate in the SRNL Shielded Cells facility in a manner similar to the DWPF CC method, and by PF and AR digestion. For detailed steps of the PF digestion, see ADS procedure 2227.[1] For detailed steps of the AR digestion, see ADS procedure 2502.[2] Three replicate dissolutions of the analytical reference glass (ARG) standard were performed concurrently with each set of digestions above. Additional quality control measures included ICP-AES analyses of a multi-element standard as a check for ICP-AES accuracy independent of digestions.

The CC method digestion involved adding 25 mL of concentrated HF to radioactive sludge slurry (~6.0 g for the SRAT Receipt at 17.1 wt% total solids and ~4.0 g for the SRAT Product at 26.52 wt% total solids) and stirring for 1 hr. Then, 25 mL of concentrated HNO_3 was added and the mixture was stirred for an additional 30 minutes prior to diluting with de-ionized (DI) water to 250 mL in a pre-weighed volumetric flask. The density of the solution was obtained from the weight of the 250 mL of solution. Approximately 5 mL was taken from the 250 mL volumetric flask and added to a pre-weighed 100 mL volumetric flask. The exact volume of the solution diluted was obtained from the weight of the solution and its density. The effective dilution was ~5000 fold. Visible solids remained in each radioactive sample bottle. During digestion of the SRAT product, the weight of the solution transferred to the 100 mL volumetric flasks inadvertently was not recorded, and, therefore, the exact volume of solution transferred could not be calculated. Therefore, the average mass of solution transferred to the 100 mL volumetric flask during digestion of the SRAT Receipt was used as the mass transferred for the SRAT Product solution. These values were subsequently used to calculate the total digestion factor for the SRAT Product samples. Approximately 0.25 g of ARG powdered glass were dissolved in triplicate and serial diluted ~5000 fold with de-ionized water with each set of digestions.

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3.0 INTRODUCTION AND RESULTS

The DWPF is currently processing and immobilizing radioactive sludge slurry into a durable borosilicate glass. The DWPF has already processed four sludge batches (Sludge Batch 1A, Sludge Batch 1B, Sludge Batch 2 and Sludge Batch 3) and is currently processing a fifth (Sludge Batch 4). A sludge batch is defined as a single tank of sludge or a combination of sludges from different tanks that has been or will be qualified before being transferred to DWPF. Thus, following the sludge batch preparation plan of the Liquid Waste Organization (LWO), the qualified sludge in Tank 51 is to be blended with the heel of the previous sludge batch in Tank 40. The sludge being qualified at the SRNL (referred to as a "batch" composition in sludge batch planning) is to be combined with the heel of the previous sludge batch in DWPF to yield the "blend" composition. The next batch of radioactive sludge slurry to be processed by the DWPF is SB5. The subject of this report is the SB5 material from Tank 51 that was qualified at SRNL, which is the SB5 Batch sludge slurry, while the SB5 Blend will consist of the following:

- Sludge Batch 4 heel
- Sludge slurry from Tank 7
- Pu/Np solution from H-canyon

LWO performed a caustic wash of the Sludge Batch 4 slurry remaining in Tank 51 to reduce the aluminum concentration and the total mass of sludge solids being fed to the DWPF before combining with the other materials.[3]

The radioactive sludge slurry used in this study for verification of the DWPF CC method is from the three liter sample of Tank 51 sludge slurry taken on March 21, 2008. The sample was delivered to SRNL, characterized and then modified by a series of wash/decant cycles to match the LWO planned preparation strategy. The sludge used in this testing corresponded to Wash F, identified by Bannochie et. al. in SRNL-PSE-2008-1126, which is the SB5 qualification sample.

The SRAT receipt and SRAT product analytical sub-samples were digested in quadruplicate using the AR, PF and the DWPF CC method. Three ARG samples were digested concurrently with each set of digestions and two multi-element ICP-AES standards were submitted along with each sample batch for analysis containing known concentrations of Al, B, Fe, Li, Na, and Si.

The measurements of the SB5 Batch samples generated from this study are provided in Table A1 of Appendix A. The results from each type of digestion are summarized in Table 3-1 for the SRAT Receipt and Table 3-3 for the SRAT Product samples. The ICP-AES results of the sixteen elements that are analyzed by the DWPF lab are presented on a weight percent (Wt%) of total solids basis. Sodium (Na) and zirconium (Zr) are not reported for the PF digestions, which are performed in a Zr crucible using Na containing reagents. Silicon (Si) is not reported for the DWPF CC method because HF in the solution leaches Si out the nebulizer of the ICP-AES instrument at SRNL.

Statistical comparisons of the data from the three digestion methods (for Na there are only two digestion methods) are provided in Exhibit A1 in Appendix A. The results were generated using JMP Version 6.0.3.[4] Note that Zr is shown for the PF digestions and Si is shown for the CC digestions. The plots of this exhibit show a 95% confidence interval for the mean (a mean diamond) of each set of measurements as well as comparison circles for the digestion means.

Comparisons of each pair of digestion means for each element except Na can be made visually by examining how the comparison circles intersect. The outside angle of intersection conveys whether the digestion means are significantly different (Figure 3-1). Circles for means that are significantly different

either do not intersect or intersect slightly so that the outside angle of intersection is less than 90 degrees. If the circles intersect by an angle of more than 90 degrees or if they are nested, the means are not significantly different (at an overall 5% level of significance).[4]

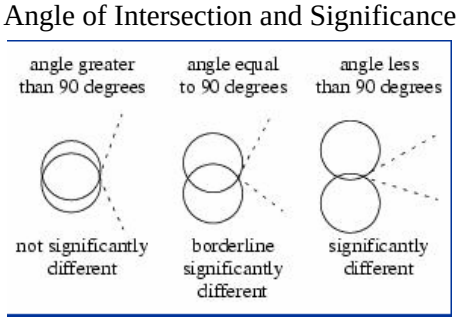

Figure 3-1. Angle of Intersection and Significance for Compariosn Circles.

While there may be difficulty in interpreting the comparison circles for some of the elements, the exhibit also contains some tabulated results to help in drawing conclusions from these results. For each element, the mean concentration of the samples by each digestion method is provided, and means that are not connected by the same letter in the listing of the exhibit are significantly different. For example consider the SRAT Product Al measurements. The results of the exhibit indicate that the mean of the AR results differs from the mean of the PF and CC results.

For the Na, Si, and Zr comparisons, the JMP output from an analysis of variance of the measurements for two digestions is provided, and only the 95% confidence mean diamond of each digestion is shown. Overlap marks show for each diamond, and overlap marks in one diamond that are closer to the mean of another diamond than that diamond's overlap marks indicate that those two groups are not different at the 95% confidence level. The visual comparisons are supported by an F test that compares the means of the AR and CC digestions for Na and Zr and the means of the AR and PF digestions for Si. If the p value is less than 0.05, then the means are statistically different at the 5% level. From Exhibit A1, there is an indication of a difference in the AR and CC means for the SRAT Product for Na but not the SRAT Receipt Na.

Summaries of the statistical comparisons of Exhibit A1 are shown in Table 3-2 and Table 3-4. Following the format used in the exhibit, digestions not having the same letter are statistically different at the 5% significance level. Consider the SRAT Receipt Al results, the average mean of Al measured in the PF and DWPF CC method digestions are statistically the same (and both columns have the letter A), but these means are statistically different from the mean average obtained from the AR digestion (which has the letter B in the column). ARG results are presented and compared for each digestion type for the SRAT Receipt and SRAT Product samples in Tables 5 and 6, respectively.

Undissolved solids remained in the DWPF Cold Chem digestate solutions in each case. The identity of the undissolved solids has **not** been determined for these samples. However, during verification of the DWPF CC method for previous sludge batches, boehmite (AlO(OH)), muscovite (K,Na)(Al, Mg, $Fe)_2(Si_{3.1}Al_{0.9})O_{10}(OH)_2$, silicon dioxide ($SiO_2$), potassium sodium aluminum fluoride ($K_2NaAl_3F_{12}$), potassium aluminum fluoride (K_2AlF_5), aluminum fluoride (AlF_3), chiolite ($Na_5Al_3F_{14}$), cryolite (Na_3AlF_6), sodium magnesium aluminum hexafluoride ($NaMgAlF_6$), $FeZrF_6$ and Na_2FeAlF_7 have been found.[5] No undissolved solids were noticed in the PF or AR solutions by visual inspection.

Table 3-1. Elemental concentrations of SB5 SRAT Receipt radioactive sludge slurry obtained from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions. Values are presented on a weight percent (Wt%) total solids basis.

Element	Aqua Regia Digestion Avg Wt%*	%RSD#	Na_2O_2/ NaOH Fusion Digestion Avg Wt%*	%RSD	DWPF Cold Chem Digestion Avg Wt%*	%RSD#
Al	7.62E+00	6.4E+00	8.91E+00	1.9E+00	8.87E+00	4.9E+00
B	<2.63E-02	NA	<2.62E-02	NA	<1.33E-01	NA
Ca	1.31E+00	1.9E+00	1.34E+00	2.9E+00	1.35E+00	1.9E+00
Cr	4.67E-02	3.1E+00	1.41E-01	1.1E+02	4.24E-02	5.9E-01
Cu	6.45E-02	3.3E+00	6.02E-02	2.9E+00	5.75E-02	1.3E+00
Fe	1.60E+01	2.1E+00	1.65E+01	4.6E+00	1.53E+01	2.0E+00
Li	3.54E-02	5.6E+00	3.62E-02	6.4E+00	3.11E-02	1.1E+01
K	<1.18E-01	NA	7.85E-01	7.1E+00	<6.00E-01	NA
Mg	6.81E-01	1.9E+00	6.04E-01	6.4E-01	6.09E-01	1.6E+00
Mn	3.74E+00	1.9E+00	3.58E+00	1.2E+00	3.61E+00	1.3E+00
Na	1.52E+01	1.7E+00	NA	NA	1.54E+01	1.4E+00
Ni	2.32E+00	1.2E+00	2.37E+00	2.4E+00	2.24E+00	1.7E+00
Si	3.97E-01	3.4E+01	9.23E-01	1.6E+00	NA	NA
Ti	1.96E-02	2.7E+00	2.03E-02	1.5E+00	1.99E-02	7.1E+00
U	5.58E+00	1.7E+00	5.57E+00	8.2E-01	5.67E+00	1.5E+00
Zr	9.16E-02	6.2E+01	NA	NA	2.59E-01	1.3E+00

*All averages are based upon four replicate dissolutions and ICP-AES determinations.
NA = Not Applicable. #%RSD is the percent relative standard deviation for the measurements.

Table 3-2. Statistical comparison of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions of SB5 SRAT Receipt sludge. Digestions not having the same letter are statistically different at the 5% significance level.

Element	Aqua Regia Digestion*	Na_2O_2/ NaOH Fusion Digestion*	DWPF Cold Chem Method Digestion*
Al	B	A	A
B	<MDL	<MDL	<MDL
Ca	A	A	A
Cr	A	A	A
Cu	A	B	B
Fe	AB	A	B
K	<MDL	A	<MDL
Li	A	A	A
Mg	A	B	B
Mn	A	B	B
Na	A	NA	B
Ni	AB	A	B
Si	B	B	NA
Ti	A	A	A
U	A	A	A
Zr	B	NA	A

*All averages are based upon four replicate dissolutions and ICP-AES determinations.
NA = Not Applicable. <MDL = Less than minimum detection limit.

The SRAT Receipt sample digested by the PF and DWPF CC methods have a statistical difference in the means for Fe and Ni out of the major elements (>1.0 wt% total solids basis - Al, Ca, Fe, Mn, Ni and U). The relative difference between the mean concentration for Fe in the DWPF CC digestions and the PF digestions is 7.6%. The relative difference for Ni is 5.2%.

The SRAT Receipt sample digested by the AR and DWPF CC methods have a statistical difference in the means for Al and Mn out of the major elements (>1.0 wt% total solids basis - Al, Ca, Fe, Mn, Ni and U). The relative difference between the mean results for Al in the DWPF CC method digestions and the AR digestions is 15%. The relative difference for Mn is 3.4%.

A statistical difference in the mean result for Al and Mn is noted for the SRAT Receipt sample digested by the AR and PF methods. The relative difference between the mean results for Al in the AR digestions and the PF digestions is 16%. The relative difference for Mn is 4.2%.

The statistical difference noted for elements having a relative small difference (Fe, Ni and Mn) indicate a tight precision in the ICP-AES measurements. It is unclear why the aluminum value is so low in the AR digestion. As noted above, there were no undissolved solids in the final diluted solutions. The Al concentration obtained by AR digestion of the ARG was high by ~1% relative to the standard value for this reference glass. The Al value obtained by the DWPF CC method for the ARG glass was ~5% high relative to the standard value and the Al value was high by ~1% for the ARG digested by the PF method. Note that PF and CC Al concentrations are statistically equivalent for both the SB5 SRAT Receipt and SRAT Product sample given the disparity for SB4.[6]

Table 3-3. Elemental concentrations of SB5 SRAT Product radioactive sludge slurry obtained from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions. Values are presented on a weight percent (Wt%) total solids basis.

Element	Aqua Regia Digestion Avg Wt%*	%RSD#	Na_2O_2/ NaOH Fusion Digestion Avg Wt%*	%RSD	DWPF Cold Chem Digestion Avg Wt%*	%RSD#
Al	5.37E+00	3.6E+00	7.14E+00	2.9E+00	6.70E+00	4.0E+00
B	<2.41E-02	NA	<2.34E-02	NA	1.99E-01	1.6E+01
Ca	1.13E+00	3.2E+00	1.07E+00	3.6E+00	1.07E+00	2.3E+00
Cr	3.95E-02	3.2E+00	3.99E-02	7.0E+00	3.97E-02	2.9E+00
Cu	5.24E-02	4.9E+00	4.64E-02	4.5E+00	4.76E-02	4.5E+00
Fe	1.35E+01	3.5E+00	1.24E+01	3.2E+00	1.30E+01	2.0E+00
Li	2.86E-02	3.4E+00	3.18E-02	7.3E+00	2.89E-02	2.6E+00
K	<1.08E-01	NA	<5.27E-01	NA	<5.65E-01	NA
Mg	6.04E-01	3.0E+00	5.25E-01	3.3E+00	5.51E-01	1.5E+00
Mn	3.15E+00	3.5E+00	3.02E+00	3.4E+00	3.21E+00	1.4E+00
Na	1.28E+01	3.4E+00	NA	NA	1.36E+01	2.4E+00
Ni	2.05E+00	3.4E+00	1.94E+00	3.1E+00	2.01E+00	1.9E+00
Si	3.44E-01	1.9E+01	7.57E-01	5.5E+00	NA	NA
Ti	1.38E-02	3.3E+00	1.71E-02	4.0E+00	1.58E-02	2.2E+00
U	4.82E+00	3.9E+00	4.94E+00	2.8E+00	4.87E+00	2.9E-01
Zr	9.85E-02	9.6E+00	NA	NA	2.19E-01	1.1E+00

*All averages are based upon four replicate dissolutions and ICP-AES determinations.
NA = Not Applicable. #%RSD is the percent relative standard deviation for the measurements.

Table 3-4. Statistical comparison of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions of SB5 SRAT Product sludge. Digestions not having the same letter are statistically different at the 5% significance level.

Element	Aqua Regia Digestion*	Na_2O_2/ NaOH Fusion Digestion*	DWPF Cold Chem Method Digestion*
Al	B	A	A
B	<MDL	<MDL	A
Ca	A	A	A
Cr	A	A	A
Cu	A	B	B
Fe	A	B	AB
K	<MDL	<MDL	<MDL
Li	B	A	AB
Mg	A	B	B
Mn	AB	B	A
Na	A	NA	A
Ni	A	A	A
Si	B	B	NA
Ti	C	A	B
U	A	A	A
Zr	B	NA	A

*All averages are based upon four replicate dissolutions and ICP-AES determinations.
NA = Not Applicable. <MDL = Less than minimum detection limit.

The SRAT Product sample digested by the PF and DWPF CC methods have a statistical difference in the means for Mn out of the major elements (>1.0 wt% total solids basis - Al, Ca, Fe, Mn, Ni and U). The relative difference between the mean concentration for Mn in the DWPF CC digestions and the PF digestions is 6.3%.

The SRAT Product sample digested by the AR and DWPF CC methods have a statistical difference in the means for Al and Na out of the major elements (>1.0 wt% total solids basis - Al, Ca, Fe, Mn, Ni and U). The relative difference between the mean results for Al in the DWPF CC method digestions and the AR digestions is 22%, and the relative difference between the mean results for Na in the DWPF CC method digestions and the AR digestions is 6.1%.

A statistical difference in the mean result for Al and Fe is noted for the SRAT Product sample digested by the AR and PF methods. The relative difference between the mean results for Al in the AR digestions and the PF digestions is 28%. The relative difference for Fe is 8.5%.

Table 3-5, Table 3-6, Table 3-7, and Table 3-8 contain the ICP-AES measured weight percent elemental results from triplicate digestions of the ARG standard performed concurrently with the SB5 radioactive sludge to determine if the dissolutions were complete and the resulting analyses accurate. Comparison to the known elemental weight percent in the ARG standard is also given in Tables 5-8. The experimentally measured values agree well with the ARG standards for all three digestions where expected.

Table 3-5. Elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Receipt. Values are presented on a weight percent (Wt%) total solids basis.

Aqua Regia*				
Element	**Average**	**%RSD**	**Standard Value**	**%Difference (Measured *vs* Standard Value)**
Al	2.52E+00	2.6E+00	2.50E+00	0.6
B	2.77E+00	3.3E+00	2.69E+00	3.0
Ca	1.12E+00	2.3E+00	1.02E+00	9.3
Cr	7.02E-02	2.9E+00	6.40E-02	9.7
Cu	<1.00E-02	NA	3.00E-03	NA
Fe	1.04E+01	2.7E+00	9.79E+00	5.7
K	2.34E+00	1.5E+00	2.26E+00	3.5
Li	1.55E+00	2.6E+00	1.49E+00	4.0
Mg	6.07E-01	2.7E+00	5.20E-01	16.6
Mn	1.50E+00	3.0E+00	1.46E+00	2.4
Na	9.04E+00	2.7E+00	8.52E+00	6.0
Ni	8.42E-01	3.6E+00	8.27E-01	1.8
Si	4.15E-01	4.8E+01	2.24E+01	NA
Ti	5.97E-01	5.0E+00	6.90E-01	-13.6
U	NA	NA	NA	NA
Zr	5.16E-02	4.3E+01	9.60E-02	-46.3
Sodium Peroxide/Hydroxide Fusion*				
Al	2.49E+00	2.06E+00	2.50E+00	-0.5
B	2.57E+00	2.37E+00	2.69E+00	-4.5
Ca	1.21E+00	3.43E+00	1.02E+00	19.0
Cr	6.90E-02	3.23E+00	6.40E-02	7.8
Cu	<4.00E-02	NA	3.00E-03	NA
Fe	9.71E+00	2.10E+00	9.79E+00	-0.8
K	2.85E+00	6.38E+00	2.26E+00	26.0
Li	1.49E+00	2.93E+00	1.49E+00	0.0
Mg	5.34E-01	1.87E+00	5.20E-01	2.8
Mn	1.33E+00	2.17E+00	1.46E+00	-8.7
Na	NA	NA	8.52E+00	NA
Ni	7.83E-01	3.41E+00	8.27E-01	-5.3
Si	2.04E+01	2.14E+00	2.24E+01	-8.9
Ti	6.62E-01	2.48E+00	6.90E-01	-4.1
U	NA	NA	NA	NA
Zr	NA	NA	9.60E-02	NA

*All averages are based upon three replicate dissolutions and ICP-AES determinations NA = Not applicable.

Table 3-6. Continuation of elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Receipt. Values are presented on a weight percent (Wt%) total solids basis.

DWPF Cold Chem Method*				
Element	**Average**	**%RSD**	**Known Value**	**%Difference (Measured *vs* Known Value)**
Al	2.63E+00	1.7E+00	2.50E+00	5.2
B	2.80E+00	2.3E+00	2.69E+00	4.2
Ca	1.15E+00	2.4E+00	1.02E+00	12.3
Cr	6.87E-02	4.8E+00	6.40E-02	7.3
Cu	<4.19E-02	1.1E+00	3.00E-03	NA
Fe	1.02E+01	2.0E+00	9.79E+00	4.1
K	2.38E+00	6.3E-01	2.26E+00	5.3
Li	1.62E+00	1.3E+00	1.49E+00	8.6
Mg	5.56E-01	2.1E+00	5.20E-01	6.9
Mn	1.47E+00	1.9E+00	1.46E+00	0.7
Na	9.11E+00	1.5E+00	8.52E+00	6.9
Ni	8.07E-01	1.1E+00	8.27E-01	-2.4
Si	NA	NA	2.24E+01	NA
Ti	7.26E-01	1.7E+00	6.90E-01	5.2
U	NA	NA	NA	NA
Zr	1.08E-01	1.6E+00	9.60E-02	12.5

*All averages are based upon three replicate dissolutions and ICP-AES determinations NA = Not applicable. <MDL = less than minimum detection limit.

Table 3-7. Elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Product. Values are presented on a weight percent (Wt%) total solids basis.

Aqua Regia*				
Element	**Average**	**%RSD**	**Standard Value**	**%Difference (Measured *vs* Standard Value)**
Al	2.35E+00	2.50E+00	2.50E+00	-6.0
B	2.63E+00	2.69E+00	2.69E+00	-2.4
Ca	1.05E+00	1.02E+00	1.02E+00	3.0
Cr	6.37E-02	6.40E-02	6.40E-02	-0.5
Cu	<4.00E-02	NA	3.00E-03	NA
Fe	9.64E+00	9.79E+00	9.79E+00	-1.6
K	2.11E+00	2.26E+00	2.26E+00	-6.8
Li	1.48E+00	1.49E+00	1.49E+00	-0.7
Mg	5.89E-01	5.20E-01	5.20E-01	13.2
Mn	1.41E+00	1.46E+00	1.46E+00	-3.4
Na	8.14E+00	8.52E+00	8.52E+00	-4.5
Ni	8.27E-01	8.27E-01	8.27E-01	0.0
Si	6.68E-01	2.24E+01	2.24E+01	-97.0
Ti	5.92E-01	6.90E-01	6.90E-01	-14.2
U	NA	NA	NA	NA
Zr	6.03E-02	9.60E-02	9.60E-02	-37.2
Sodium Peroxide/Hydroxide Fusion*				
Al	2.32E+00	3.4E+00	2.50E+00	-7.2
B	2.62E+00	3.0E+00	2.69E+00	-2.6
Ca	1.06E+00	2.5E+00	1.02E+00	3.9
Cr	6.34E-02	3.9E+00	6.40E-02	-1.0
Cu	<4.00E-02	NA	3.00E-03	NA
Fe	8.96E+00	3.2E+00	9.79E+00	-8.5
K	2.17E+00	1.6E+00	2.26E+00	-4.0
Li	1.48E+00	2.4E+00	1.49E+00	-0.7
Mg	5.50E-01	5.2E+00	5.20E-01	5.7
Mn	1.32E+00	3.5E+00	1.46E+00	-9.4
Ni	7.90E-01	4.2E+00	8.27E-01	-4.4
Si	2.04E+01	1.3E+01	2.24E+01	-8.8
Ti	6.63E-01	2.9E+00	6.90E-01	-3.9
U	NA	NA	NA	NA
Zr	NA	NA	9.60E-02	NA

*All averages are based upon three replicate dissolutions and ICP-ES determinations NA = Not applicable.

Table 3-8. Continuation of elemental concentrations of ARG standard from ICP-AES analysis of Aqua Regia, DWPF Cold Chem method and Sodium Peroxide/Hydroxide Fusion digestions performed concurrently with SB5 SRAT Product. Values are presented on a weight percent (Wt%) total solids basis.

DWPF Cold Chem Method*				
Element	**Average**	**%RSD**	**Known Value**	**%Difference (Measured *vs* Known Value)**
Al	2.58E+00	1.7E+00	2.50E+00	3.2
B	2.92E+00	4.2E+00	2.69E+00	8.6
Ca	1.03E+00	1.2E+00	1.02E+00	1.3
Cr	6.84E-02	8.1E-01	6.40E-02	6.9
Cu	<1.10E-02	NA	3.00E-03	NA
Fe	1.00E+01	6.2E-01	9.79E+00	2.4
K	2.24E+00	2.1E+00	2.26E+00	-0.8
Li	1.51E+00	3.2E+00	1.49E+00	1.2
Mg	5.55E-01	5.2E-01	5.20E-01	6.7
Mn	1.47E+00	8.6E-01	1.46E+00	0.7
Na	8.89E+00	1.1E+00	8.52E+00	4.4
Ni	8.41E-01	2.6E-01	8.27E-01	1.7
Si	NA	NA	2.24E+01	NA
Ti	7.13E-01	1.6E+00	6.90E-01	3.4
U	NA	NA	NA	NA
Zr	1.05E-01	2.0E+00	9.60E-02	9.9

*All averages are based upon three replicate dissolutions and ICP-AES determinations NA = Not applicable. <MDL = less than minimum detection limit.

4.0 CONCLUSIONS

The results presented in the memo validate the use of the DWPF CC method for use with the SB5 material. The relative small difference observed between the three digestions (AR, PF and DWPF CC) for elements composing greater than 1 wt% of the solids (except for Al) indicate the DWPF CC digestion method is sufficient for digesting SB5 process samples. The reason for the discrepancy in the aluminum concentration obtained from the AR digestion compared to the DWPF CC method and PF method is not known. The peroxide fusion digestion method is the best method for digesting aluminosilicates and aluminum hydroxides that otherwise might be insoluble in acid digestions. Given that the measured aluminum concentration in samples digested using the DWPC CC method is statistically equivalent to the measured aluminum concentration in PF digested samples, the DWPF CC method appears to be adequately dissolving aluminum containing species in the SB5 Batch sludge.

However, the SB5 Batch material is to be mixed with the heel of SB4 to form the SB5 Blend that is to be processed at DWPF, and a difficulty was encountered in using the CC method for SB4.[6] This difficulty brings into question the adequacy of CC for the SB5 Blend (see recommendations below).

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5.0 RECOMMENDATIONS

The following recommendations are based upon results in this memo:

- A dissolution study should be performed on the WAPS sample by SRNL which consists of the final composition of the sludge (the SB5 Blend).

- Given the heel of SB4 in Tank 40, the DWPF lab should monitor the aluminum concentration in the first 10 SRAT Receipt batches of SB5 using both CC and sodium peroxide/hydroxide fusion to evaluate the adequacy of aluminum recovery by the CC method for this sludge batch.

- SRNL and the DWPF lab should investigate if comparisons between the elemental concentrations of the SME product glass (adjusted for frit addition) obtained by the mixed acid and peroxide fusion digestion and the SRAT Receipt and SRAT Product elemental concentrations obtained *via* the DWPF CC method provide insight into the adequacy of the CC method for analysis of the SRAT Product. The DWPF lab would need to calcine the SRAT product at 1050 ºC for the best comparison. If a consistent difference in elemental concentrations is revealed, another type of digestion (i.e. sodium peroxide/hydroxide fusion) should be used to determine the concentration of the element in question. Particular emphasis should be placed on monitoring the aluminum concentration in SB5.

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6.0 REFERENCES

1. C.J. Coleman, "Alkali Fusion Dissolutions of Sludge and Glass for Elemental and Anion Analysis", ADS Procedure ADS-2502, Rev. 6.
2. C.J. Coleman, "Aqua Regia Dissolution of Sludge for Elemental Analysis", ADS Procedure ADS-2226, Rev. 7.
3. M.S. Hay, J.M. Pareizs, C.J. Bannochie, M.E. Stone, D.R. Click and D.J. McCabe, "Characterization and Aluminum Dissolution Demonstration With A 3 Liter Tank 51H Sample," WSRC-STI-2007-00697, Rev. 0; J.A. Pike, "Preliminary Results for Low Temperature Aluminum Removal from Sludge Batch 5," LWO-LWE-2008-00067 Rev. 0; J.A. Pike and J.M. Gillam, "Flowsheet for Aluminum Removal from Sludge Batch 5," LWO-PIT-2007-00042 Rev. 2.
4. JMP Statistical Discovery Software *v* 6.0; SAS Institute Inc., Cary, NC, 2002.
5. C.J. Coleman, F.M. Pennebaker, B.H. Burch and D.R. Click, "Evaluation of the DWPF Cold Chem Dissolution Method with DWPF Sludge Batch 3 Simulant", WSRC-TR-02-00496, Rev. 0. See also D.R. Click, "Evaluation of the DWPF Cold Chem Dissolution Method with Tank 7 and Tank 51 Radioactive Sludges", WSRC-TR-2003-00580. D.R. Click, C.J. Coleman, K.E. Zeigler and T.B. Edwards, "Sludge Batch Four (4) Defense Waste Processing Facility (DWPF) Process Analytical Method Verification", WSRC-STI-2006-00025 Rev. 0.
6. D.R. Click, C.J. Coleman, K.E. Zeigler and T.B. Edwards, "Sludge Batch Four (4) Defense Waste Processing Facility (DWPF) Process Analytical Method Verification", WSRC-STI-2006-00025 Rev. 0.

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7.0 ACKNOWLEDGEMENTS

The authors would like to acknowledge Ramona Galloway and Monica Jenkins.

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8.0 APPENDIX A. SUPPORTING INFORMATION

Table A1. Measurements Generated by this Study

Type	Digestion	Sample ID	Element	Wt% Measurement		Type	Digestion	Sample ID	Element	Wt% Measurement
SRAT Product	CC	300250343	Al	6.3845		SRAT Receipt	CC	300249668	Al	9.5096
SRAT Product	CC	300250343	B	0.155		SRAT Receipt	CC	300249668	B	0.1347
SRAT Product	CC	300250343	Ca	1.0381		SRAT Receipt	CC	300249668	Ca	1.3828
SRAT Product	CC	300250343	Cr	0.0388		SRAT Receipt	CC	300249668	Cr	0.0421
SRAT Product	CC	300250343	Cu	0.0453		SRAT Receipt	CC	300249668	Cu	0.0586
SRAT Product	CC	300250343	Fe	12.6715		SRAT Receipt	CC	300249668	Fe	15.678
SRAT Product	CC	300250343	K	0.5751		SRAT Receipt	CC	300249668	K	0.6066
SRAT Product	CC	300250343	Li	0.0279		SRAT Receipt	CC	300249668	Li	0.0356
SRAT Product	CC	300250343	Mg	0.541		SRAT Receipt	CC	300249668	Mg	0.622
SRAT Product	CC	300250343	Mn	3.1581		SRAT Receipt	CC	300249668	Mn	3.6702
SRAT Product	CC	300250343	Na	13.1589		SRAT Receipt	CC	300249668	Na	15.678
SRAT Product	CC	300250343	Ni	1.9543		SRAT Receipt	CC	300249668	Ni	2.2926
SRAT Product	CC	300250343	Si	.		SRAT Receipt	CC	300249668	Si	.
SRAT Product	CC	300250343	Ti	0.0163		SRAT Receipt	CC	300249668	Ti	0.0219
SRAT Product	CC	300250343	U	4.8493		SRAT Receipt	CC	300249668	U	5.7058
SRAT Product	CC	300250343	Zr	0.2179		SRAT Receipt	CC	300249668	Zr	0.2632
SRAT Product	CC	300250345	Al	6.9276		SRAT Receipt	CC	300249670	Al	8.6577
SRAT Product	CC	300250345	B	0.2016		SRAT Receipt	CC	300249670	B	0.1319
SRAT Product	CC	300250345	Ca	1.0941		SRAT Receipt	CC	300249670	Ca	1.3238
SRAT Product	CC	300250345	Cr	0.0414		SRAT Receipt	CC	300249670	Cr	0.0424
SRAT Product	CC	300250345	Cu	0.0466		SRAT Receipt	CC	300249670	Cu	0.0569
SRAT Product	CC	300250345	Fe	13.1863		SRAT Receipt	CC	300249670	Fe	14.9496
SRAT Product	CC	300250345	K	0.5638		SRAT Receipt	CC	300249670	K	0.594
SRAT Product	CC	300250345	Li	0.0288		SRAT Receipt	CC	300249670	Li	0.0295
SRAT Product	CC	300250345	Mg	0.559		SRAT Receipt	CC	300249670	Mg	0.599
SRAT Product	CC	300250345	Mn	3.2583		SRAT Receipt	CC	300249670	Mn	3.5637
SRAT Product	CC	300250345	Na	13.4729		SRAT Receipt	CC	300249670	Na	15.151
SRAT Product	CC	300250345	Ni	2.0209		SRAT Receipt	CC	300249670	Ni	2.2047
SRAT Product	CC	300250345	Si	.		SRAT Receipt	CC	300249670	Si	.
SRAT Product	CC	300250345	Ti	0.0154		SRAT Receipt	CC	300249670	Ti	0.0191
SRAT Product	CC	300250345	U	4.8732		SRAT Receipt	CC	300249670	U	5.5872
SRAT Product	CC	300250345	Zr	0.2198		SRAT Receipt	CC	300249670	Zr	0.2552
SRAT Product	CC	300250346	Al	6.5737		SRAT Receipt	CC	300249671	Al	8.7637
SRAT Product	CC	300250346	B	0.2069		SRAT Receipt	CC	300249671	B	0.1327
SRAT Product	CC	300250346	Ca	1.0601		SRAT Receipt	CC	300249671	Ca	1.3373
SRAT Product	CC	300250346	Cr	0.039		SRAT Receipt	CC	300249671	Cr	0.0425
SRAT Product	CC	300250346	Cu	0.0481		SRAT Receipt	CC	300249671	Cu	0.0572
SRAT Product	CC	300250346	Fe	12.9159		SRAT Receipt	CC	300249671	Fe	15.1972
SRAT Product	CC	300250346	K	0.5463		SRAT Receipt	CC	300249671	K	0.5978
SRAT Product	CC	300250346	Li	0.0298		SRAT Receipt	CC	300249671	Li	0.0317
SRAT Product	CC	300250346	Mg	0.5463		SRAT Receipt	CC	300249671	Mg	0.6079
SRAT Product	CC	300250346	Mn	3.1943		SRAT Receipt	CC	300249671	Mn	3.5865
SRAT Product	CC	300250346	Na	13.8881		SRAT Receipt	CC	300249671	Na	15.3998
SRAT Product	CC	300250346	Ni	1.9999		SRAT Receipt	CC	300249671	Ni	2.2289
SRAT Product	CC	300250346	Si	.		SRAT Receipt	CC	300249671	Si	.
SRAT Product	CC	300250346	Ti	0.0157		SRAT Receipt	CC	300249671	Ti	0.0189
SRAT Product	CC	300250346	U	4.8608		SRAT Receipt	CC	300249671	U	5.6229
SRAT Product	CC	300250346	Zr	0.2171		SRAT Receipt	CC	300249671	Zr	0.2589
SRAT Product	CC	300250348	Al	6.9311		SRAT Receipt	CC	300249673	Al	8.5403
SRAT Product	CC	300250348	B	0.2314		SRAT Receipt	CC	300249673	B	0.134
SRAT Product	CC	300250348	Ca	1.0836		SRAT Receipt	CC	300249673	Ca	1.3399
SRAT Product	CC	300250348	Cr	0.0397		SRAT Receipt	CC	300249673	Cr	0.0427
SRAT Product	CC	300250348	Cu	0.0503		SRAT Receipt	CC	300249673	Cu	0.0573
SRAT Product	CC	300250348	Fe	13.2278		SRAT Receipt	CC	300249673	Fe	15.2396
SRAT Product	CC	300250348	K	0.576		SRAT Receipt	CC	300249673	K	0.6034
SRAT Product	CC	300250348	Li	0.029		SRAT Receipt	CC	300249673	Li	0.0278
SRAT Product	CC	300250348	Mg	0.5564		SRAT Receipt	CC	300249673	Mg	0.6086
SRAT Product	CC	300250348	Mn	3.2459		SRAT Receipt	CC	300249673	Mn	3.6207
SRAT Product	CC	300250348	Na	13.7647		SRAT Receipt	CC	300249673	Na	15.4953
SRAT Product	CC	300250348	Ni	2.0452		SRAT Receipt	CC	300249673	Ni	2.2501
SRAT Product	CC	300250348	Si	.		SRAT Receipt	CC	300249673	Si	.
SRAT Product	CC	300250348	Ti	0.0158		SRAT Receipt	CC	300249673	Ti	0.0194

Table A1. Measurements Generated by this Study

Type	Digestion	Sample ID	Element	Wt% Measurement		Type	Digestion	Sample ID	Element	Wt% Measurement
SRAT Product	CC	300250348	U	4.8811		SRAT Receipt	CC	300249673	U	5.7788
SRAT Product	CC	300250348	Zr	0.2226		SRAT Receipt	CC	300249673	Zr	0.2598
SRAT Product	PF	300250136	Al	7.07		SRAT Receipt	PF	300249476	Al	8.98
SRAT Product	PF	300250136	B	0.0227		SRAT Receipt	PF	300249476	B	0.0261
SRAT Product	PF	300250136	Ca	1.07		SRAT Receipt	PF	300249476	Ca	1.35
SRAT Product	PF	300250136	Cr	0.0372		SRAT Receipt	PF	300249476	Cr	0.365
SRAT Product	PF	300250136	Cu	0.0449		SRAT Receipt	PF	300249476	Cu	0.0624
SRAT Product	PF	300250136	Fe	12.2		SRAT Receipt	PF	300249476	Fe	17.5
SRAT Product	PF	300250136	K	0.51		SRAT Receipt	PF	300249476	K	0.846
SRAT Product	PF	300250136	Li	0.0305		SRAT Receipt	PF	300249476	Li	0.035
SRAT Product	PF	300250136	Mg	0.512		SRAT Receipt	PF	300249476	Mg	0.598
SRAT Product	PF	300250136	Mn	2.98		SRAT Receipt	PF	300249476	Mn	3.59
SRAT Product	PF	300250136	Na	.		SRAT Receipt	PF	300249476	Na	.
SRAT Product	PF	300250136	Ni	1.92		SRAT Receipt	PF	300249476	Ni	2.43
SRAT Product	PF	300250136	Si	0.765		SRAT Receipt	PF	300249476	Si	0.922
SRAT Product	PF	300250136	Ti	0.0172		SRAT Receipt	PF	300249476	Ti	0.0203
SRAT Product	PF	300250136	U	4.87		SRAT Receipt	PF	300249476	U	5.56
SRAT Product	PF	300250136	Zr	.		SRAT Receipt	PF	300249476	Zr	.
SRAT Product	PF	300250138	Al	7.22		SRAT Receipt	PF	300249478	Al	9.12
SRAT Product	PF	300250138	B	0.024		SRAT Receipt	PF	300249478	B	0.0263
SRAT Product	PF	300250138	Ca	1.09		SRAT Receipt	PF	300249478	Ca	1.28
SRAT Product	PF	300250138	Cr	0.038		SRAT Receipt	PF	300249478	Cr	0.105
SRAT Product	PF	300250138	Cu	0.0472		SRAT Receipt	PF	300249478	Cu	0.0607
SRAT Product	PF	300250138	Fe	12.5		SRAT Receipt	PF	300249478	Fe	16.6
SRAT Product	PF	300250138	K	0.539		SRAT Receipt	PF	300249478	K	0.802
SRAT Product	PF	300250138	Li	0.0352		SRAT Receipt	PF	300249478	Li	0.0393
SRAT Product	PF	300250138	Mg	0.542		SRAT Receipt	PF	300249478	Mg	0.605
SRAT Product	PF	300250138	Mn	3.05		SRAT Receipt	PF	300249478	Mn	3.63
SRAT Product	PF	300250138	Na	.		SRAT Receipt	PF	300249478	Na	.
SRAT Product	PF	300250138	Ni	1.96		SRAT Receipt	PF	300249478	Ni	2.39
SRAT Product	PF	300250138	Si	0.811		SRAT Receipt	PF	300249478	Si	0.943
SRAT Product	PF	300250138	Ti	0.0173		SRAT Receipt	PF	300249478	Ti	0.0207
SRAT Product	PF	300250138	U	4.98		SRAT Receipt	PF	300249478	U	5.52
SRAT Product	PF	300250138	Zr	.		SRAT Receipt	PF	300249478	Zr	.
SRAT Product	PF	300250139	Al	6.88		SRAT Receipt	PF	300249479	Al	8.8
SRAT Product	PF	300250139	B	0.0227		SRAT Receipt	PF	300249479	B	0.0264
SRAT Product	PF	300250139	Ca	1.02		SRAT Receipt	PF	300249479	Ca	1.36
SRAT Product	PF	300250139	Cr	0.0431		SRAT Receipt	PF	300249479	Cr	0.049
SRAT Product	PF	300250139	Cu	0.0445		SRAT Receipt	PF	300249479	Cu	0.0584
SRAT Product	PF	300250139	Fe	12		SRAT Receipt	PF	300249479	Fe	16
SRAT Product	PF	300250139	K	0.512		SRAT Receipt	PF	300249479	K	0.779
SRAT Product	PF	300250139	Li	0.0301		SRAT Receipt	PF	300249479	Li	0.034
SRAT Product	PF	300250139	Mg	0.509		SRAT Receipt	PF	300249479	Mg	0.607
SRAT Product	PF	300250139	Mn	2.9		SRAT Receipt	PF	300249479	Mn	3.57
SRAT Product	PF	300250139	Na	.		SRAT Receipt	PF	300249479	Na	.
SRAT Product	PF	300250139	Ni	1.87		SRAT Receipt	PF	300249479	Ni	2.34
SRAT Product	PF	300250139	Si	0.714		SRAT Receipt	PF	300249479	Si	0.908
SRAT Product	PF	300250139	Ti	0.0161		SRAT Receipt	PF	300249479	Ti	0.02
SRAT Product	PF	300250139	U	4.8		SRAT Receipt	PF	300249479	U	5.57
SRAT Product	PF	300250139	Zr	.		SRAT Receipt	PF	300249479	Zr	.
SRAT Product	PF	300250141	Al	7.37		SRAT Receipt	PF	300249481	Al	8.75
SRAT Product	PF	300250141	B	0.0243		SRAT Receipt	PF	300249481	B	0.026
SRAT Product	PF	300250141	Ca	1.11		SRAT Receipt	PF	300249481	Ca	1.36
SRAT Product	PF	300250141	Cr	0.0414		SRAT Receipt	PF	300249481	Cr	0.0461
SRAT Product	PF	300250141	Cu	0.049		SRAT Receipt	PF	300249481	Cu	0.0594
SRAT Product	PF	300250141	Fe	12.9		SRAT Receipt	PF	300249481	Fe	15.8
SRAT Product	PF	300250141	K	0.548		SRAT Receipt	PF	300249481	K	0.712
SRAT Product	PF	300250141	Li	0.0315		SRAT Receipt	PF	300249481	Li	0.0366
SRAT Product	PF	300250141	Mg	0.538		SRAT Receipt	PF	300249481	Mg	0.604
SRAT Product	PF	300250141	Mn	3.14		SRAT Receipt	PF	300249481	Mn	3.53
SRAT Product	PF	300250141	Na	.		SRAT Receipt	PF	300249481	Na	.
SRAT Product	PF	300250141	Ni	2.01		SRAT Receipt	PF	300249481	Ni	2.3
SRAT Product	PF	300250141	Si	0.738		SRAT Receipt	PF	300249481	Si	0.917

Table A1. Measurements Generated by this Study

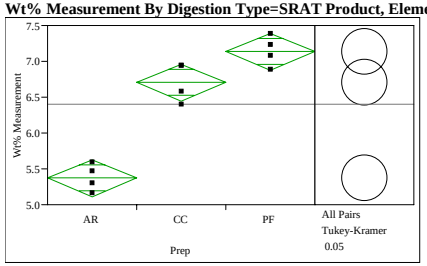
Type	Digestion	Sample ID	Element	Wt% Measurement		Type	Digestion	Sample ID	Element	Wt% Measurement
SRAT Product	PF	300250141	Ti	0.0177		SRAT Receipt	PF	300249481	Ti	0.0202
SRAT Product	PF	300250141	U	5.12		SRAT Receipt	PF	300249481	U	5.63
SRAT Product	PF	300250141	Zr	.		SRAT Receipt	PF	300249481	Zr	.
SRAT Product	AR	300250117	Al	5.46		SRAT Receipt	AR	300249460	Al	6.98
SRAT Product	AR	300250117	B	0.0244		SRAT Receipt	AR	300249460	B	0.0261
SRAT Product	AR	300250117	Ca	1.15		SRAT Receipt	AR	300249460	Ca	1.31
SRAT Product	AR	300250117	Cr	0.0398		SRAT Receipt	AR	300249460	Cr	0.0461
SRAT Product	AR	300250117	Cu	0.0536		SRAT Receipt	AR	300249460	Cu	0.0658
SRAT Product	AR	300250117	Fe	13.7		SRAT Receipt	AR	300249460	Fe	16
SRAT Product	AR	300250117	K	0.11		SRAT Receipt	AR	300249460	K	0.117
SRAT Product	AR	300250117	Li	0.0293		SRAT Receipt	AR	300249460	Li	0.0331
SRAT Product	AR	300250117	Mg	0.616		SRAT Receipt	AR	300249460	Mg	0.676
SRAT Product	AR	300250117	Mn	3.2		SRAT Receipt	AR	300249460	Mn	3.74
SRAT Product	AR	300250117	Na	13		SRAT Receipt	AR	300249460	Na	15.2
SRAT Product	AR	300250117	Ni	2.08		SRAT Receipt	AR	300249460	Ni	2.32
SRAT Product	AR	300250117	Si	0.367		SRAT Receipt	AR	300249460	Si	0.481
SRAT Product	AR	300250117	Ti	0.0138		SRAT Receipt	AR	300249460	Ti	0.0197
SRAT Product	AR	300250117	U	4.89		SRAT Receipt	AR	300249460	U	5.59
SRAT Product	AR	300250117	Zr	0.0934		SRAT Receipt	AR	300249460	Zr	0.144
SRAT Product	AR	300250119	Al	5.15		SRAT Receipt	AR	300249462	Al	7.53
SRAT Product	AR	300250119	B	0.0241		SRAT Receipt	AR	300249462	B	0.0269
SRAT Product	AR	300250119	Ca	1.16		SRAT Receipt	AR	300249462	Ca	1.34
SRAT Product	AR	300250119	Cr	0.0393		SRAT Receipt	AR	300249462	Cr	0.0488
SRAT Product	AR	300250119	Cu	0.0554		SRAT Receipt	AR	300249462	Cu	0.0668
SRAT Product	AR	300250119	Fe	13.8		SRAT Receipt	AR	300249462	Fe	16.5
SRAT Product	AR	300250119	K	0.108		SRAT Receipt	AR	300249462	K	0.121
SRAT Product	AR	300250119	Li	0.029		SRAT Receipt	AR	300249462	Li	0.0371
SRAT Product	AR	300250119	Mg	0.616		SRAT Receipt	AR	300249462	Mg	0.697
SRAT Product	AR	300250119	Mn	3.21		SRAT Receipt	AR	300249462	Mn	3.83
SRAT Product	AR	300250119	Na	13		SRAT Receipt	AR	300249462	Na	15.6
SRAT Product	AR	300250119	Ni	2.1		SRAT Receipt	AR	300249462	Ni	2.35
SRAT Product	AR	300250119	Si	0.401		SRAT Receipt	AR	300249462	Si	0.514
SRAT Product	AR	300250119	Ti	0.0144		SRAT Receipt	AR	300249462	Ti	0.0197
SRAT Product	AR	300250119	U	4.97		SRAT Receipt	AR	300249462	U	5.7
SRAT Product	AR	300250119	Zr	0.111		SRAT Receipt	AR	300249462	Zr	0.0925
SRAT Product	AR	300250120	Al	5.59		SRAT Receipt	AR	300249463	Al	8.09
SRAT Product	AR	300250120	B	0.0241		SRAT Receipt	AR	300249463	B	0.0258
SRAT Product	AR	300250120	Ca	1.08		SRAT Receipt	AR	300249463	Ca	1.28
SRAT Product	AR	300250120	Cr	0.0379		SRAT Receipt	AR	300249463	Cr	0.0457
SRAT Product	AR	300250120	Cu	0.0502		SRAT Receipt	AR	300249463	Cu	0.0634
SRAT Product	AR	300250120	Fe	12.8		SRAT Receipt	AR	300249463	Fe	15.7
SRAT Product	AR	300250120	K	0.109		SRAT Receipt	AR	300249463	K	0.116
SRAT Product	AR	300250120	Li	0.0272		SRAT Receipt	AR	300249463	Li	0.0369
SRAT Product	AR	300250120	Mg	0.578		SRAT Receipt	AR	300249463	Mg	0.667
SRAT Product	AR	300250120	Mn	2.98		SRAT Receipt	AR	300249463	Mn	3.67
SRAT Product	AR	300250120	Na	12.1		SRAT Receipt	AR	300249463	Na	15
SRAT Product	AR	300250120	Ni	1.95		SRAT Receipt	AR	300249463	Ni	2.28
SRAT Product	AR	300250120	Si	0.247		SRAT Receipt	AR	300249463	Si	0.217
SRAT Product	AR	300250120	Ti	0.0135		SRAT Receipt	AR	300249463	Ti	0.0202
SRAT Product	AR	300250120	U	4.55		SRAT Receipt	AR	300249463	U	5.49
SRAT Product	AR	300250120	Zr	0.1		SRAT Receipt	AR	300249463	Zr	0.117
SRAT Product	AR	300250122	Al	5.29		SRAT Receipt	AR	300249465	Al	7.88
SRAT Product	AR	300250122	B	0.0236		SRAT Receipt	AR	300249465	B	0.0262
SRAT Product	AR	300250122	Ca	1.14		SRAT Receipt	AR	300249465	Ca	1.3
SRAT Product	AR	300250122	Cr	0.041		SRAT Receipt	AR	300249465	Cr	0.046
SRAT Product	AR	300250122	Cu	0.0502		SRAT Receipt	AR	300249465	Cu	0.0621
SRAT Product	AR	300250122	Fe	13.7		SRAT Receipt	AR	300249465	Fe	15.9
SRAT Product	AR	300250122	K	0.106		SRAT Receipt	AR	300249465	K	0.118
SRAT Product	AR	300250122	Li	0.029		SRAT Receipt	AR	300249465	Li	0.0343
SRAT Product	AR	300250122	Mg	0.607		SRAT Receipt	AR	300249465	Mg	0.683
SRAT Product	AR	300250122	Mn	3.19		SRAT Receipt	AR	300249465	Mn	3.7
SRAT Product	AR	300250122	Na	12.9		SRAT Receipt	AR	300249465	Na	15.1
SRAT Product	AR	300250122	Ni	2.08		SRAT Receipt	AR	300249465	Ni	2.32

Table A1. Measurements Generated by this Study

Type	Digestion	Sample ID	Element	Wt% Measurement		Type	Digestion	Sample ID	Element	Wt% Measurement
SRAT Product	AR	300250122	Si	0.359		SRAT Receipt	AR	300249465	Si	0.375
SRAT Product	AR	300250122	Ti	0.0134		SRAT Receipt	AR	300249465	Ti	0.0189
SRAT Product	AR	300250122	U	4.88		SRAT Receipt	AR	300249465	U	5.52
SRAT Product	AR	300250122	Zr	0.0894		SRAT Receipt	AR	300249465	Zr	0.0129

Exhibit A1. Statistical Comparisons of Digestion Methods

Wt% Measurement By Digestion Type=SRAT Product, Element=Al

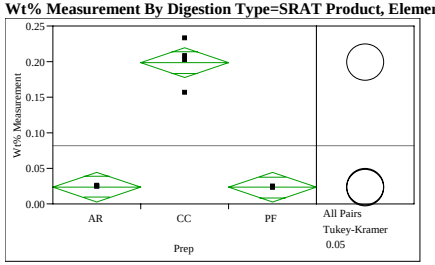


Means Comparisons

Level		Mean
PF	A	7.1350000
CC	A	6.7042332
AR	B	5.3725000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=B

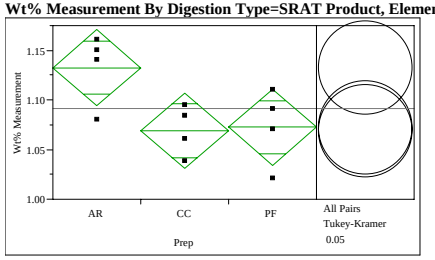


Means Comparisons

Level		Mean
CC	A	0.19872387
AR	B	0.02405000
PF	B	0.02342500

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Ca

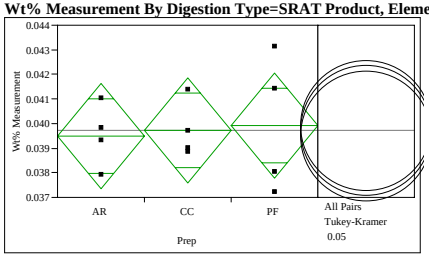


Means Comparisons

Level		Mean
AR	A	1.1325000
PF	A	1.0725000
CC	A	1.0689745

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Cr

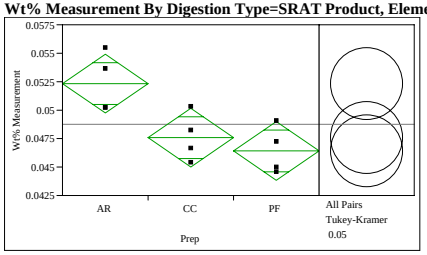


Means Comparisons

Level		Mean
PF	A	0.03992500
CC	A	0.03972001
AR	A	0.03950000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Cu

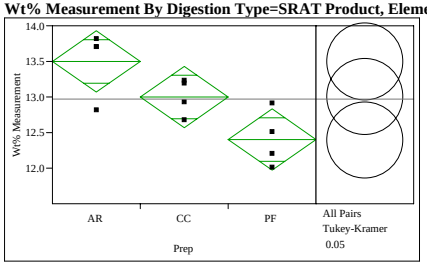


Means Comparisons

Level		Mean
AR	A	0.05235000
CC	B	0.04758194
PF	B	0.04640000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Fe

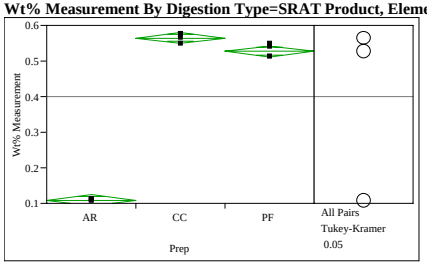


Means Comparisons

Level			Mean
AR	A		13.500000
CC	A	B	13.000378
PF		B	12.400000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=K

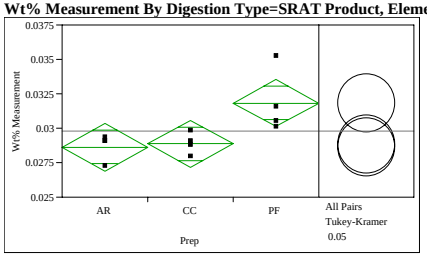


Means Comparisons

Level			Mean
CC	A		0.56527208
PF		B	0.52725000
AR		C	0.10825000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Li

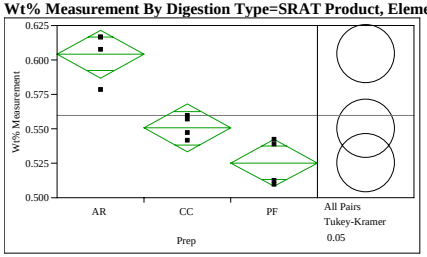


Means Comparisons

Level			Mean
PF	A		0.03182500
CC	A	B	0.02886200
AR		B	0.02862500

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=Mg



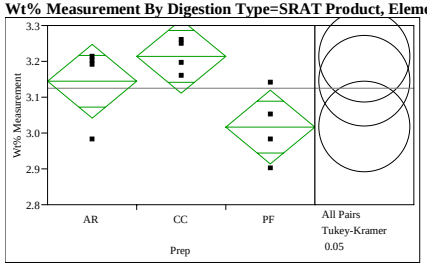
Means Comparisons

Level			Mean
AR	A		0.60425000
CC		B	0.55066766
PF		B	0.52525000

Levels not connected by same letter are significantly different.

Exhibit A1. Statistical Comparisons of Digestion Methods

Wt% Measurement By Digestion Type=SRAT Product, Element=Mn

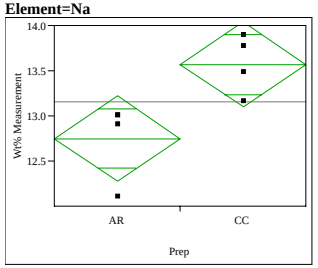


Means Comparisons

Level		Mean
CC	A	3.2141689
AR	A B	3.1450000
PF	B	3.0175000

Levels not connected by same letter are significantly different.

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Product, Element=Na



Missing Rows 4

Oneway Anova
Summary of Fit

Rsquare	0.603093
Adj Rsquare	0.536942
Root Mean Square Error	0.384604
Mean of Response	13.16058
Observations (or Sum Wgts)	8

Analysis of Variance

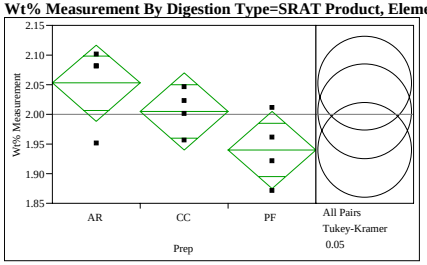
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	1.3485746	1.34857	9.1169	0.0234
Error	6	0.8875220	0.14792		
C. Total	7	2.2360967			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	12.7500	0.19230	12.279	13.221
CC	4	13.5712	0.19230	13.101	14.042

Std Error uses a pooled estimate of error variance

Wt% Measurement By Digestion Type=SRAT Product, Element=Ni

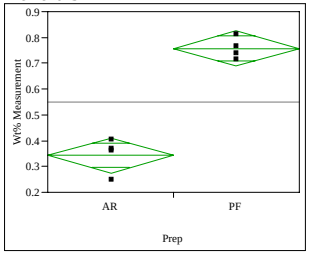


Means Comparisons

Level		Mean
AR	A	2.0525000
CC	A	2.0050865
PF	A	1.9400000

Levels not connected by same letter are significantly different.

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Product, Element=Si



Missing Rows 4
Oneway Anova
Summary of Fit

Rsquare	0.948412
Adj Rsquare	0.939814
Root Mean Square Error	0.055679
Mean of Response	0.55025
Observations (or Sum Wgts)	8

Analysis of Variance

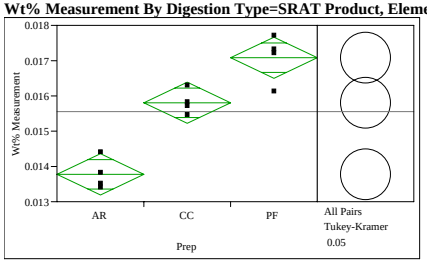
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	0.34196450	0.341965	110.3052	<.0001
Error	6	0.01860100	0.003100		
C. Total	7	0.36056550			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.343500	0.02784	0.27538	0.41162
PF	4	0.757000	0.02784	0.68888	0.82512

Std Error uses a pooled estimate of error variance

Wt% Measurement By Digestion Type=SRAT Product, Element=Ti

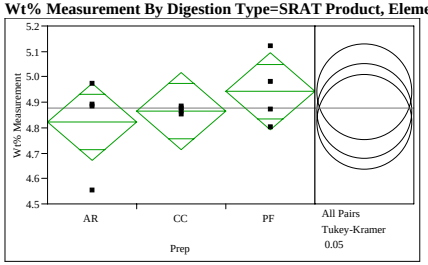


Means Comparisons

Level		Mean
PF	A	0.01707500
CC	B	0.01580453
AR	C	0.01377500

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Product, Element=U



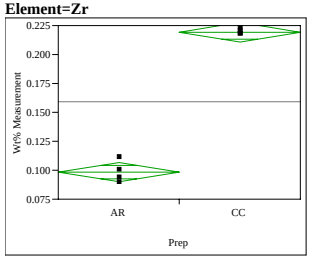
Means Comparisons

Level		Mean
PF	A	4.9425000
CC	A	4.8661046
AR	A	4.8225000

Levels not connected by same letter are significantly different.

Exhibit A1. Statistical Comparisons of Digestion Methods

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Product, Element=Zr



Missing Rows 4

Oneway Anova

Summary of Fit

Rsquare	0.990337
Adj Rsquare	0.988727
Root Mean Square Error	0.006895
Mean of Response	0.1589
Observations (or Sum Wgts)	8

Analysis of Variance

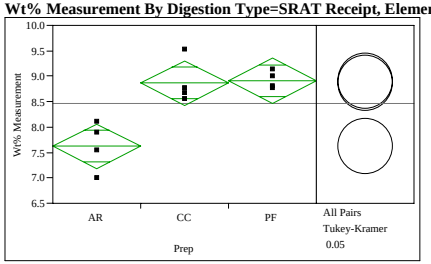
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	0.02923362	0.029234	614.9268	<.0001
Error	6	0.00028524	0.000048		
C. Total	7	0.02951886			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.098450	0.00345	0.09001	0.10689
CC	4	0.219350	0.00345	0.21091	0.22779

Std Error uses a pooled estimate of error variance

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Al

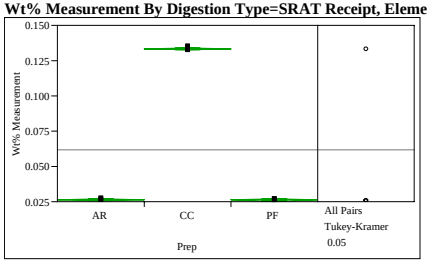


Means Comparisons

Level		Mean
PF	A	8.9125000
CC	A	8.8678370
AR	B	7.6200000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=B

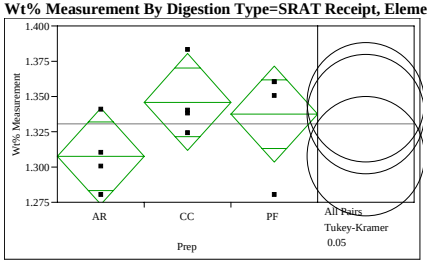


Means Comparisons

Level		Mean
CC	A	0.13331590
AR	B	0.02625000
PF	B	0.02620000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Ca

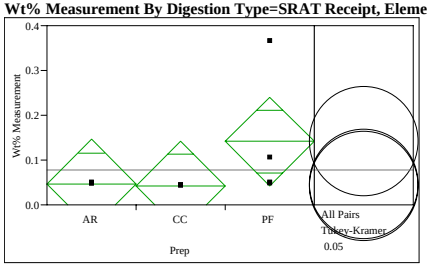


Means Comparisons

Level		Mean
CC	A	1.3459458
PF	A	1.3375000
AR	A	1.3075000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Cr

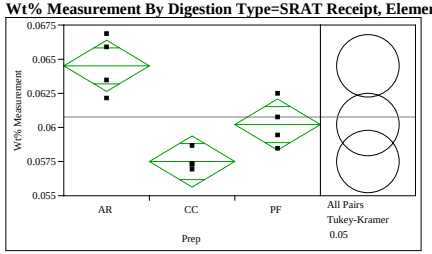


Means Comparisons

Level		Mean
PF	A	0.14127500
AR	A	0.04665000
CC	A	0.04242121

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Cu

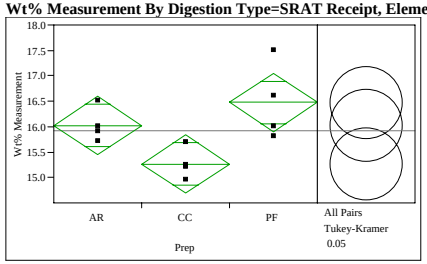


Means Comparisons

Level		Mean
AR	A	0.06452500
PF	B	0.06022500
CC	B	0.05749950

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Fe

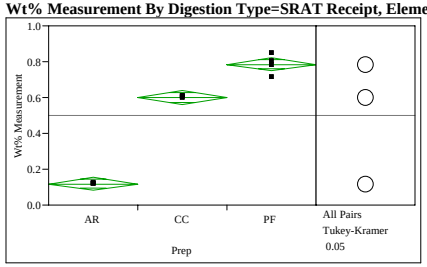


Means Comparisons

Level			Mean
PF	A		16.475000
AR	A	B	16.025000
CC		B	15.266112

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=K



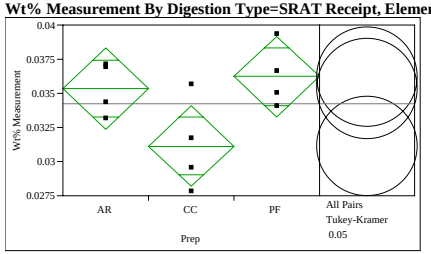
Means Comparisons

Level			Mean
PF	A		0.78475000
CC		B	0.60043039
AR		C	0.11800000

Levels not connected by same letter are significantly different.

Exhibit A1. Statistical Comparisons of Digestion Methods

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Li

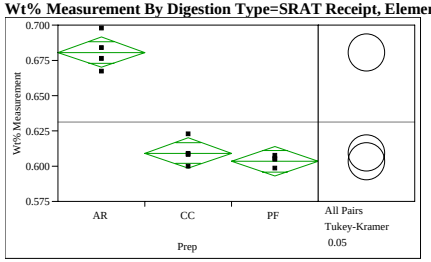


Means Comparisons

Level		Mean
PF	A	0.03622500
AR	A	0.03535000
CC	A	0.03113718

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Mg

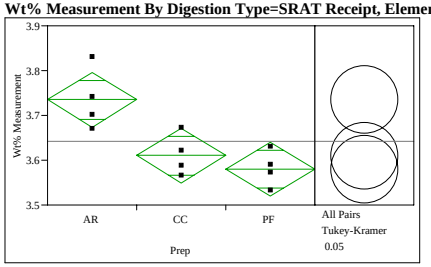


Means Comparisons

Level		Mean
AR	A	0.68075000
CC	B	0.60935539
PF	B	0.60350000

Levels not connected by same letter are significantly different.

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Mn

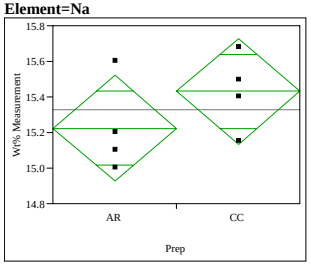


Means Comparisons

Level		Mean
AR	A	3.7350000
CC	B	3.6102929
PF	B	3.5800000

Levels not connected by same letter are significantly different.

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Receipt, Element=Na



Missing Rows 4

Oneway Anova

Summary of Fit

Rsquare	0.194302
Adj Rsquare	0.06002
Root Mean Square Error	0.242218
Mean of Response	15.32801
Observations (or Sum Wgts)	8

Analysis of Variance

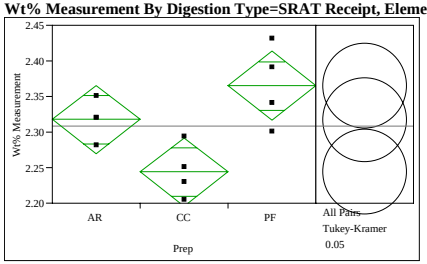
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	0.08489260	0.084893	1.4470	0.2743
Error	6	0.35201693	0.058669		
C. Total	7	0.43690953			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	15.2250	0.12111	14.929	15.521
CC	4	15.4310	0.12111	15.135	15.727

Std Error uses a pooled estimate of error variance

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Ni

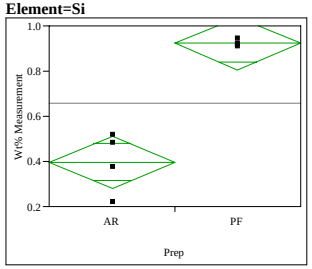


Means Comparisons

Level			Mean
PF	A		2.3650000
AR	A	B	2.3175000
CC		B	2.2440867

Levels not connected by same letter are significantly different.

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Receipt, Element=Si



Missing Rows 4

Oneway Anova

Summary of Fit

Rsquare	0.910578
Adj Rsquare	0.895674
Root Mean Square Error	0.095123
Mean of Response	0.659625
Observations (or Sum Wgts)	8

Analysis of Variance

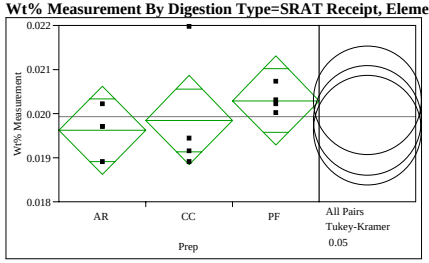
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	0.55282613	0.552826	61.0973	0.0002
Error	6	0.05428975	0.009048		
C. Total	7	0.60711588			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.396750	0.04756	0.28037	0.5131
PF	4	0.922500	0.04756	0.80612	1.0389

Std Error uses a pooled estimate of error variance

Wt% Measurement By Digestion Type=SRAT Receipt, Element=Ti



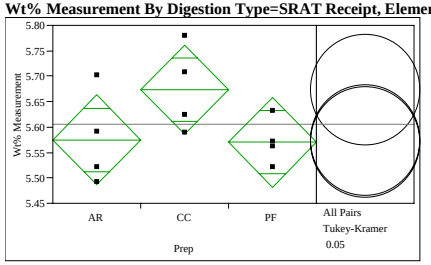
Means Comparisons

Level		Mean
PF	A	0.02030000
CC	A	0.01985123
AR	A	0.01962500

Levels not connected by same letter are significantly different.

Exhibit A1. Statistical Comparisons of Digestion Methods

Wt% Measurement By Digestion Type=SRAT Receipt, Element=U

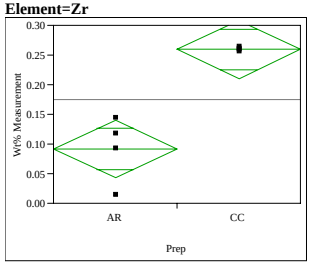


Means Comparisons

Level		Mean
CC	A	5.6736862
AR	A	5.5750000
PF	A	5.5700000

Levels not connected by same letter are significantly different.

Oneway Analysis of Wt% Measurement By Prep Type=SRAT Receipt, Element=Zr



Missing Rows 4

Oneway Anova

Summary of Fit

Rsquare	0.853938
Adj Rsquare	0.829594
Root Mean Square Error	0.040037
Mean of Response	0.175438
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Prep	1	0.05622981	0.056230	35.0784	0.0010
Error	6	0.00961785	0.001603		
C. Total	7	0.06584766			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
AR	4	0.091600	0.02002	0.04262	0.14058
CC	4	0.259275	0.02002	0.21029	0.30826

Std Error uses a pooled estimate of error variance

Distribution
C.J. Bannochie, 773-42A
M.J. Barnes, 773-A
N.E. Bibler, 773-A
J.M. Bricker, 704-27S
L.M. Chandler, 773-A
C.J. Coleman, 773-A
B.A. Davis, 773-A
T.B. Edwards, 773-42A
M.T. Feller, 704-28S
T.L. Fellinger, 704-26S
J.C. Griffin, 773-A
C.C. Herman, 999-W
J.F. Iaukea, 704-30S
R.T. McNew, 704-27S
R.N. Mahannah, 704-28S
T.A. Nance, 773-42A
A.B. Osteen, 704-28S
J.E. Occhipinti, 704-S
D.K. Peeler, 999-W
F.M. Pennebaker, 773-A
J.W. Ray, 704-S
M.E. Stone, 999-W

ADS Files