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ALTERNATIVE ANALYTICAL DIGESTION SCHEME FOR THE DEFENSE WASTE PROCESSING FACILITY (DWPF) SLURRY RECEIPT AND ADJUSTMENT TANK (SRAT) ANALYSES

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September 17, 2007

Analytical Development
Savannah River National Laboratory
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500

SRNL
SAVANNAH RIVER NATIONAL LABORATORY

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EXECUTIVE SUMMARY

As part of the radioactive sludge batch qualification, Savannah River National Laboratory (SRNL) performs a verification of the digestion methods to be used by the Defense Waste Processing Facility (DWPF) Lab for elemental analysis of Sludge Receipt and Adjustment Tank (SRAT) receipt process control samples and SRAT product process control samples.[1] Verification of these methods on Sludge Batch 4 (SB4) radioactive sludge slurry indicated SB4 contains a higher concentration of aluminum (Al) than previous sludge batches.[2] Aluminum plays a direct role in vitrification chemistry. At moderate levels, Al assists in glass forming, but at elevated levels Al can increase the viscosity of the molten glass which can adversely impact glass production rate and the volume of glass produced via limiting waste loading.[3] Most of the Al present in SB4 is in the form of Al hydroxide as a mixture of *gibbsite* [α-aluminum trihydroxide, α-$Al(OH)_3$] and *boehmite* (α-aluminum oxyhydroxide, α-AlOOH) in an unknown ratio. Testing done at SRNL indicates *Gibbsite* is soluble at low pH but *boehmite* has limited solubility in the acid mixture (DWPF Cold Chem Method (CC), 25 mL nitric acid (HNO_3) and 25 mL hydrofluoric acid (HF)) used by DWPF to digest process control samples.[2] Because Al plays such an important part in vitrification chemistry, it is necessary to have a robust digestion method that will dissolve all forms of Al present in the radioactive sludge while not increasing the analytical lab turnaround time.

SRNL initially suggested that the DWPF lab use the sodium peroxide/hydroxide fusion (PF) digestion method[4] to digest SRAT receipt and SRAT product radioactive sludge as an alternative to the acid digestion method to ensure complete digestion based on results obtained from digesting a SB4 radioactive sample.[2] However, this change may have a significant impact on the DWPF lab analytical turnaround time due to the inefficiency in drying the radioactive sludge contained in a peanut-vial (~12-16 hrs) prior to performing the PF. Therefore, a modified digestion scheme was tested using simulant sludge that takes advantage of both digestion methods (CC and PF). The experimental work involved 1) performing the CC method on simulant sludge containing both *boehmite* and *gibbsite*, 2) filtering the digestate to collect undissolved solids, 3) drying the filter and the solids collected (2 hr step versus ~12-16 hr step for drying peanut vial full of sludge), 4) heating the solids and filter to 675 ºC (causing complete oxidation of the filter), and 5) performing a PF digestion of the solids. The solutions from each type of digestion were analyzed by Inductively Coupled Plasma Emission Spectroscopy (ICP-ES) and the results were combined. The measured Al concentration from the PF digestion on a dried solids basis was 10.7% with a relative standard deviation of 0.92% and 10.1% with a relative standard deviation of 0.43% from the CC+PF digestion. The Al concentration measured in the digestion solutions from the CC method before performing the PF was ~8.8 wt% of the solubilized solids on a total dried solids basis. The Al hydroxide dissolution results are discussed in this report. Also discussed are the experimental results obtained for all elements DWPF measures and a statistical comparison of that data.

The following conclusions and recommendations are based upon spectroscopic and statistical analysis of results from experimental digestion tests conducted with SB4 simulant sludge slurry:

- The CC + PF digestion method will result in reduced DWPF Lab analytical turnaround time over the PF only digestion method.
- The CC + PF digestion resulted in complete digestion of all forms of Al and the measured combined concentration of Al was nearly equal to that of the PF only method.
- Pursue a side-by-side development and comparison of the combined digestion method (DWPF CC plus PF of undissolved solids) using radioactive sludge.
- Perform periodic analyses (X-ray diffraction) on solids that may be in the DWPF CC analytical process digestion samples at DWPF to help further refine the digestion method.

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LIST OF ACRONYMS

AD	Analytical Development
ARG	Analytical Reference Glass
CC	Cold Chem Method
CXRD	Contained X-ray Diffraction
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
SRM	Standard Reference Material
SRNL	Savannah River National Laboratory

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1.0 INTRODUCTION AND BACKGROUND

Heavy-water reactors at the Savannah River Site were operated for approximately 30 years to produce nuclear materials for the nation's defense. Low temperature reactor operation allowed the use of Al-clad, Al-uranium (U) alloy fuel. At the end of the reactor cycle, and upon cooling, dissolution and separation of the components in this fuel produced an aqueous waste stream with a high concentration of Al. The aqueous waste was sent to H-Area tank farm where it was adjusted to prevent waste tank corrosion. The sludge from one of these tanks (Tank 11) was blended with the heel from Tank 7 (F-Area Tank) to produce the latest sludge batch feed Sludge Batch 4 (SB4) for the DWPF.[5]

Aluminum plays a direct role in vitrification chemistry. At moderate levels, Al assists in glass forming. At elevated levels, Al can increase the viscosity of the molten glass which can adversely impact the glass production rate and the volume of glass produced via limiting waste loading.[3] In addition, an increased Al concentration may contribute to nepheline ($NaAlSiO_4$) formation as a devitrification product during canister cooling.[5] To meet the Tank Farm needs for tank space, a caustic leaching step to reduce the Al levels for SB4 was not pursued but may be pursued for future DWPF sludge batches. Therefore, SB4 contains a much higher weight percent Al concentration than previous sludge batches. The aluminum present in SB4 is, in part, present as a mixture of *gibbsite* [α-aluminum trihydroxide, α-$Al(OH)_3$] and *boehmite* (α-aluminum oxyhydroxide, α-AlOOH) in an unknown ratio. The Al hydroxide content in Tank 11, before blending with Tank 7, was estimated to be an 80:20 *boehmite*/*gibbsite* mixture. An additional complication arises as *gibbsite* to *boehmite* transformation may occur in the self-heating alkaline environment encountered in the high-level waste storage tanks.[3]

Experimental studies involving digestion of high-level waste at SRNL indicate *boehmite* has limited solubility in the acid mixture (DWPF Cold Chem Method (CC), 25 mL HNO_3 and 25 mL HF) used by the DWPF Lab to digest process control samples; *gibbsite* is soluble in the CC mixture.[2] ICP-ES data from digested as-received Tank 51 SB4 radioactive sludge slurry and washed Tank 51 SB4 radioactive sludge slurry samples which were previously characterized indicate the difference in the aluminum concentration between the two digestions ~was 6.% (10.5 wt% Al on a dried solids basis measured in the Na_2O_2/NaOH fusion *vs* 9.85 wt% Al on a dried solids measured in the DWPF CC method) and ~27% (16.4 wt% Al on a dried solids basis *vs* 12.5 wt% Al on a dried solids basis measured by the DWPF Cold Chem method) for the washed Tank 51 radioactive sludge slurry sample. Undissolved solids remained in the DWPF CC digestate solutions in each case. The undissolved solids in the as-received Tank 51 SB4 digestate solutions were determined to be boehmite (AlO(OH)), muscovite $(K,Na)(Al, Mg, Fe)_2(Si_{3.1}Al_{0.9})O_{10}(OH)_2$, and silicon dioxide ($SiO_2$). The undissolved solids in the washed Tank 51 SB4 digestate solutions were determined to be potassium sodium aluminum fluoride ($K_2NaAl_3F_{12}$), potassium aluminum fluoride (K_2AlF_5), aluminum fluoride (AlF_3), and chiolite ($Na_5Al_3F_{14}$). No undissolved solids remained in the Na_2O_2/NaOH fusion digestate solutions.[2] The difference in the observed solubility is a kinetic phenomenon. Because Al plays such an important part in vitrification chemistry it is necessary to have a robust digestion method that will dissolve all forms of Al present in the radioactive sludge and dissolve precipitated species while not increasing the DWPF analytical lab turn-around-time. This report details experimental digestion studies performed on simulant sludge without *boehmite* and spiked with *boehmite*. Statistical comparisons of data generated from Inductively Coupled Plasma Emission Spectroscopy (ICP-ES) analysis of sample solutions from two different digestion schemes are presented for Al and the other elements that are analyzed by the DWPF Lab (16 elements in all), except U.

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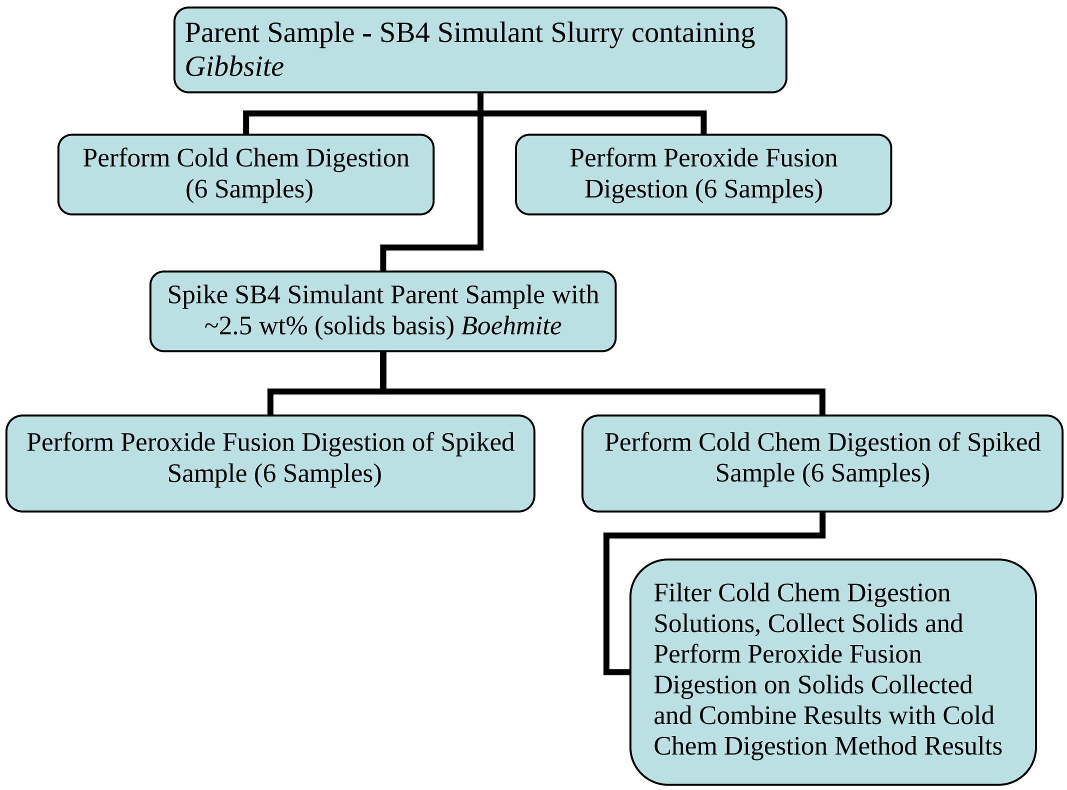
2.0 EXPERIMENTAL

Experimental Outline

M. E. Stone of Process Science and Engineering (PS&E) provided SB4 simulant sludge for this study which contained Al hydroxide in the form of *gibbsite*. The experimental outline is pictorially represented below and a detailed description follows.



A portion of the parent SB4 slurry sample was dissolved using two digestion techniques (PF and CC) and the resulting solutions were analyzed by ICP-ES. The Al results from the PF digestion were used to represent the 'true' concentration of Al in the parent sample and used to calculate how much *boehmite* to add to the sample. *Boehmite* (0.9069 g) was spiked into 74.1318 g of SB4 simulant slurry (21.7 ± 0.2 wt % solids) to increase the Al concentration of the sample by ca. 2.5%. The calculated weight percent total solids in the SB4 spiked sample was 22.65 and the measured weight percent total solids in the sample was 22.55. This corresponds to 0.0534 g of *boehmite*/g of solids. *Boehmite* is 44.98% wt % Al. A portion of the spiked sample was digested using two digestions techniques (PF and CC). The PF digestion completely dissolved the entire sample. However, solids remained in the CC digestion solutions and were isolated by filtration, dried and subjected to a PF digestion. All sets of samples were analyzed by ICP-ES. The ICP-ES results of the PF digestion of the spiked samples were compared to the sum of the ICP-ES results of the CC digestion and the extra PF digestion which was necessary to dissolve the solids remaining in the CC digestion solutions.

Digestion of SB4 Sludge Slurry Parent Sample

The initial PF digestion of SB4 simulant sludge slurry containing Al in the form of *gibbsite* was performed according to ADS Procedure 2502 by adding ~1.1 g of SB4 simulant sludge (21.7 ± 0.2 wt % solids) to six separate zirconium (Zr) crucibles. The slurry in the crucibles was dried until two consecutive weighings varied by ± 0.01 g or less. The crucibles were cooled, and ~1.5 g of sodium peroxide (Na_2O_2) and ~1.0 gram of sodium hydroxide (NaOH) were added to each. The mixtures were heated to 675 ºC and maintained at the temperature for 10 min. The resulting flux in each crucible was allowed to cool and then dissolved by the addition of deionized (DI) water and 25 mL of HNO_3. Upon dilution the solutions were cloudy with a faint reddish-brown color. The addition of 3 drops of 30% hydrogen peroxide (H_2O_2) clarified the solutions. The samples were diluted to 250 mL. For quality control, three Analytical Reference Glass (ARG) standards (~0.25 g) were digested in a manner similar to that for the SB4 slurry. The ARG samples and one multi-element ICP standard were submitted for analysis by ICP-ES along with the digested SB4 slurry samples.

The DWPF CC digestions of SB4 simulant sludge slurry were performed by adding ~4.1 g of simulant sludge (21.7 ± 0.2 wt % solids, 6 samples were prepared) to a 125 mL plastic bottle followed by 25 mL of concentrated HF. The solution was stirred for 1 hr, 25 mL of concentrated HNO_3 was added, and the solution was further stirred for 30 min. The resulting solutions were serially diluted to an effective volume of 5000 mL. For quality control, three ARG standards (~1 g) were digested in a manner similar to that of the SB4 slurry. The ARG samples and one multi-element ICP standard were submitted for analysis by ICP-ES along with the digested SB4 slurry samples.

Digestion of SB4 Sludge Slurry Spiked Sample

PF digestion of the spiked SB4 simulant sludge was performed in a manner similar to that previously noted. The resulting solutions were submitted for analysis by ICP-ES.

DWPF CC digestions of *boehmite* spiked SB4 simulant sludge were performed in a manner similar to that above. Approximately 4.2 g of simulant sludge (22.55 ± 0.05 wt % solids) was added to each of six 125 mL plastic bottles followed by 25 mL of concentrated HF. Each solution was stirred for 1 hr, then 25 mL of HNO_3 was added, and the solution was stirred for 30 min. The samples were then diluted to 250 mL with DI water in a volumetric flask. Every diluted solution had solids remaining at the bottom of the volumetric flask. The solutions were filtered through separate 0.2-micron nylon filters with pre-determined weights and the solids that were collected were dried in an oven at 110 ºC for 2 hr. Another weight of each filter and the solids collected was obtained, and the weight of the solids on each filter determined. Approximately 5 mL of the filtered solution was further diluted to 100 mL in a volumetric flask using DI water. The solids collected on the filter and the filter were heated to 675 ºC for 10 min (to oxidize the nylon filter) in a Zr crucible. The remaining solids were subjected to a PF digestion. Each sample solution was analyzed by ICP-ES separately. It is conceivable to combine the DWPF CC digestion solutions and the digestion solutions resulting from the filtration with PF digestion of the solids collected from the former digestion solutions for analysis.

Digestion of Nylon Filters, Soda Feldspar and Boehmite

Three nylon filters were subjected to 675 ºC in Zr crucibles for 10 min in order to determine the impact of the nylon filters on the experiments with the SB4 simulant. The experiments were performed in the following manner. Three 0.2-micron nylon filters were placed into separate weighed Zr crucibles, and they were heated to 675 ºC for 10 minutes. The filters oxidized completely and difference between the starting weight of the Zr crucible and the final weight of the Zr crucible + filter after heating was negligible.

Three soda feldspar samples (~0.3 g) containing ~20.5 wt% Al and three *boehmite* samples (~0.05 g) were heated to 675 ºC in a Zr crucible with a nylon filter. The filter underwent complete combustion. The remaining white powder in each crucible was subjected to a PF digestion in the manner previously described. The average recovery of Al from PF digestion of *boehmite* was ~96%. The average recovery of Al from PF digestion of feldspar was also ~96%.

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3.0 RESULTS

The experimental results (shown for the 16 elements for which DWPF analyzes) from the initial digestion and ICP-ES analysis of the SB4 simulant parent sample are shown in Table 3.1. Six sub-samples of the parent sample were digested by each method (CC and PF) along with 3 ARG samples. The ICP-ES analysis results of the ARG standards are shown in Table 3.1.

Table 3.1 ICP-ES analysis results from digested (DWPF CC and PF) SB4 simulant sludge slurry and statistical comparison of the data. Values are presented on a wt % dried solids basis.

Element	Elemental Wt % from DWPF CC Digestion*	% RSD#	Element	Elemental Wt % from PF Digestion *	% RSD#	Statistically Different Means (at a 5% significance level)
Al	8.52E+00	1.5E+00	Al	8.21E+00	1.4E+00	Yes
B	<7.12E-02	NA	B	4.01E-02	1.2E+01	NA
Ca	1.09E+00	1.5E+01	Ca	1.25E+00	1.9E+00	Yes
Cr	1.52E-02	1.9E+01	Cr	1.22E-01	1.1E+00	Yes
Cu	5.53E-02	1.6E+01	Cu	4.99E-02	2.4E+00	No
Fe	1.60E+01	1.1E+00	Fe	1.54E+01	1.2E+00	Yes
Li	<2.05E-02	NA	Li	<4.16E-03	NA	NA
K	1.26E+00	2.8E+01	K	1.14E+00	4.5E+00	No
Mg	6.56E-01	8.2E+00	Mg	6.29E-01	1.8E+00	No
Mn	4.08E+00	1.2E+00	Mn	3.90E+00	1.3E+00	Yes
Na	1.12E+01	1.2E+00	Na	NA	NA	NA
Ni	2.23E+00	1.2E+00	Ni	2.18E+00	1.3E+00	Yes
Si	NA	NA	Si	9.14E-01	1.4E+00	NA
Ti	1.64E-02	7.2E+00	Ti	1.63E-02	4.1E+00	No
U^	NA	NA	NA	NA	NA	NA
Zr	1.81E-01	2.9E+00	Zr	NA	NA	NA

*Six samples were digested by each method and analyzed by ICP-ES. #% RSD = percent relative standard deviation.
^Uranium is not present in the simulant sludge.

A statistical comparison of the ICP-ES data was performed by Tommy Edwards (Statistical Consulting) using JMP software[6] (see appendix B). The JMP software compares sets of data and reports a numerical value. Comparison of the reported value versus a known threshold value determines if the means are statistically different. In this report, for ease of reading, 'yes' and 'no' are used to designate if data have statistically different means at the 5% significance level. The average concentrations of nearly every element are lower in the PF digestion as compared to the DWPF CC digestion, except Ca. The results of the statistical comparison between the means of the PF digestion and the CC digestion indicate statistically different means (at the 5% confidence level) for Al, Ca, Cr, Fe, Mn, and Ni. The reason for the lower concentrations observed in the PF results is unclear. It should be noted that a small absolute difference between two average values may be statistically different (in this case at the 5% significance level) if there is a small variation between each of their measured values as is the case for Al. However, even if a statistical difference exists between two numbers that have a small absolute difference, there may not be a practical difference in the way the results are evaluated in a process lab. Calcium is a contaminant in the PF reagents and so the higher average concentration measured by ICP-ES in the PF samples was expected. Chromium is present but at a very low concentration, which may help to explain the statistical difference. Boron (B), lithium (Li), sodium (Na), silicon (Si), U, and zirconium (Zr) were not evaluated during the statistical analysis. The B concentration in the solutions resulting from digestion of the simulant sludge using the DWPC CC method was below the detection limit of the ICP-ES. The Li concentration was below the ICP-ES detection limit in both digestion solutions submitted. Sodium is part of the fusion reagents and Si is etched from the glassware of the ICP-ES instrument during analysis. Uranium is not present in the simulant sludge and the fusion is performed in a Zr crucible, a source of Zr contamination.

A statistical comparison of the ICP-ES data from tandem digestions (CC and PF) of ARG standards revealed fewer statistical differences overall (see Appendix A). However, statistical differences in the measured ICP-ES concentrations were seen for Al, Ca, Cr, and Mn.

Two multi-element ICP standards were submitted and analyzed by ICP-ES along with the samples and ARG standards. The multi-element standard contains Al, B, Fe, Li, K, Si and Na. One standard is analyzed within the first few samples of the sample batch and the other is analyzed at the end of the sample batch. The results from an analysis of the multi-element standards are summarized in Table 3.3.

Table 3.2 ICP-ES analysis results from digested (DWPF CC and PF) ARG samples and statistical comparison of the data. Values are presented on a wt % dried solids basis.

Element	Elemental Wt % from DWPF CC Digestion *	% RSD#	Elemental Wt % from PF Digestion *	% RSD#	Statistically Different Means (at a 5% significance level)	DWPF CC Digestion Result - Ratio to True Value	PF Digestion Result - Ratio to True Value
Al	2.27E+00	2.1E+00	2.38E+00	1.6E+00	Yes	0.91	0.95
B	2.80E+00	5.3E+00	2.67E+00	1.7E+00	No	1.04	0.99
Ca	9.96E-01	2.7E+00	1.13E+00	2.5E+00	Yes	0.98	1.11
Cr	6.97E-02	2.7E+00	6.43E-02	2.8E+00	Yes	1.09	1.00
Cu	<4.73E-02	NA	<8.64E-03	NA	NA	NA	NA
Fe	9.85E+00	1.6E+00	9.61E+00	1.6E+00	No	1.01	0.98
Li	1.41E+00	1.8E+00	1.37E+00	8.4E-01	No	0.95	0.92
K	1.80E+00	1.9E+01	2.26E+00	3.3E+00	No	0.80	1.00
Mg	4.68E-01	2.8E+00	4.80E-01	3.5E+00	No	0.90	0.92
Mn	1.43E+00	1.5E+00	1.37E+00	1.5E+00	Yes	0.98	0.94
Na	9.86E+00	1.3E+00	NA	NA	NA	1.16	NA
Ni	8.21E-01	8.8E-01	7.97E-01	1.9E+00	No	0.99	0.96
Si	NA	NA	2.30E+01	1.3E+00	NA	NA	1.03
Ti	6.83E-01	1.3E+00	6.82E-01	1.0E+00	No	0.99	0.99
U^	NA	NA	NA	NA	NA	NA	NA
Zr	8.83E-02	6.1E+00	2.65E-02	2.3E+01	NA	0.92	NA

*Three samples were digested by each method and analyzed by ICP-ES. #% RSD = percent relative standard deviation.
^Uranium is not present in the simulant sludge.

Table 3.3 Multi-element ICP-ES standard results. Standards were analyzed as part of the sample batch that included the samples in Table 3.1 and Table 3.2.

		DWPF CC				PF			
Element in ICP-ES Std	Reference Value	Result (mg/L) Run 1*	Delta	Result (mg/L) Run 2^	Delta	Result (mg/L) Run 1*	Delta	Result (mg/L) Run 1^	Delta
Al	4.0	3.9	-2.5	3.9	-3.8	3.81	-4.8	3.82	-4.5
B	20.0	20.0	0	20.0	0	20.1	0.5	20.1	0.5
Fe	4.0	4.1	1.5	3.97	-0.8	4.03	0.8	4.00	0
Li	10.0	9.6	-3.7	9.39	-6.1	9.54	-4.6	9.43	-5.7
K	10.0	8.7	-13.2	7.77	-22.3	8.39	-16.1	10.5	5
Si	50.0	NA	NA	NA	NA	51.8	3.6	51.7	3.4
Na	81.0	95.6	18.0	97.3	20.1	NA	NA	NA	NA

*Multi-element standard analyzed within the first few samples of the batch. ^Multi-element standard analyzed at the end of the sample batch.

The next step in the experimental scheme was to add ~2.5 wt% Al, in the form of *boehmite*, to the parent sample. Although gibbsite is sufficiently soluble in acid media and the average Al concentration in the solutions generated by the DWPF CC method was higher than the Al concentration in the PF solutions, the average PF Al value was used as the baseline Al concentration. The parent sample was then spiked with *boehmite* and dissolved by performing a PF digestion and the DWPF CC method. The solutions resulting from the PF digestion were clear and did not contain visible solids. However, the solutions resulting from the initial dilution of the acid mixture used in the DWPF CC method contained visible solids in all six cases. The solutions were filtered using a 0.2-micron nylon filter prior to serial dilution of the sample. The solids that were collected on the nylon filter and the filter itself were dried in the oven at 110 ºC for 2 hr. The filters along with the solids were transferred to Zr crucibles, heated to 675 ºC and maintained at that temperature for ten minutes. The Zr crucibles were removed from the oven and cooled to room temperature. The residues left in the Zr crucibles were subjected to a PF digestion as described in the experimental section. The experimentally measured concentrations from the DWPF CC method plus the PF digestion of the resulting solids were combined to yield the final result. The results from ICP-ES analysis of samples generated from digestion of *boehmite* spiked simulant sludge are presented in Table 3.4

Table 3.4 ICP-ES analysis results from digested (DWPF Cold Chem Method + PF digestion of the undissolved solids in the DWPF Cold Chem Method solutions *versus* PF) SB4 simulant sludge slurry spiked with *boehmite* and statistical comparison of the data. Values are presented on a wt% dried solids basis.

Element	Elemental Wt % from DWPF CC Digestion + PF Digestion*	% RSD#	Elemental Wt % from PF Digestion *	% RSD#	Statistically Different Means (at a 5% significance level)	Ratio (PF + CC Average Concentration to PF Average Concentration)
Al	1.01E+01	4.3E-01	1.07E+01	9.2E-01	Yes	0.94
B	7.50E-02	1.1E+02	4.60E-02	1.8E+01	No	1.95
Ca	9.95E-01	1.9E+01	1.29E+00	3.0E+00	Yes	0.77
Cr	1.22E-01	2.0E+01	1.25E-01	1.3E+00	No	0.98
Cu	4.39E-02	8.4E+01	4.30E-02	4.3E-02	No	1.22
Fe	1.51E+01	1.9E-01	1.53E+01	3.6E-01	No	0.99
Li	8.12E-03	2.4E+02	<9.00E-03	NA	NA	NA
K	1.97E-01	2.0E+02	1.15E+00	1.1E+01	Yes	0.26
Mg	5.92E-01	6.6E+00	6.47E-01	9.4E+01	Yes	0.92
Mn	3.91E+00	2.6E+00	3.95E+00	4.1E+01	No	0.99
Na	8.33E+00	1.4E+00	NA	NA	NA	NA
Ni	2.16E+00	1.8E+00	2.15E+00	3.8E-01	No	1.00
Si	NA	NA	8.35E-01	8.9E-01	NA	NA
Ti	2.9E-02	1.9E+02	1.73E-02	6.3E+01	No	1.68
U^	NA	NA	NA	NA	NA	NA
Zr	1.85E-01	1.1E+01	NA	NA	NA	NA

*Six samples were digested by each method and analyzed by ICP-ES. #% RSD = percent relative standard deviation.
^Uranium is not present in the simulant sludge.

As seen in Table 3.4 the difference in the ICP-ES measured and averaged values for Al, Ca, K and Mg are statistically different at the 5% significance level. There does not appear to be a low bias between data from each digestion method compared to the results in Table 3.1. Lithium was below the detection limit in one case, Na is part of the fusion reagents, and Si is etched from the glassware of the ICP-ES instrument; therefore these elements were not evaluated in the statistical comparison. As noted in Table 3.4 fewer elements had a large enough difference in the mean values to result in a statistically significant difference. However, a larger spread in the data was seen for the experimentally obtained concentrations from the CC+PF digestion, see Appendix C.

The present set of experiments provided us the opportunity to examine the elemental make-up of the undissolved and/or precipitated solids from the DWPF CC digestion of the simulant sludge. Several elements were detected upon ICP-ES analysis of the filter that was digested by PF (containing undissolved and/or precipitated solids from the DWPF CC digestions) and are shown in Table 3.5.

The elements present in the filtered and undissolved solids include Al, Ca, Cr, Fe, K, Mg, Mn, Ni and Ti. Many of these metals are most likely counterions of a fluoride salt formed by precipitation with such a large amount of fluoride available in the digestions. Alternately some species may be like *boehmite* in the sense that they are kinetically slow to dissolve in acid media. Previous work involving CC digestions of both radioactive and non-radioactive sludge found Al, Fe, K, Mg, Na, Si and Zr in undissolved or precipitated solids when analyzed using x-ray. The species previously identified were potassium sodium aluminum fluoride ($K_2NaAl_3F_{12}$), potassium aluminum fluoride (K_2AlF_5), aluminum fluoride (AlF_3), chiolite ($Na_5Al_3F_{14}$), muscovite $(K,Na)(Al, Mg, Fe)_2(Si_{3.1}Al_{0.9})$, and other mixed metal aluminum fluorides such as $FeZrF_6$ and Na_2FeAlF_7.

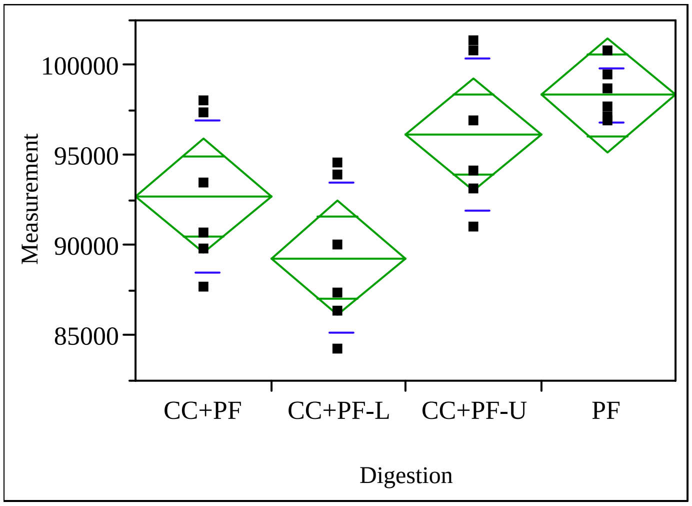
Table 3.5 contains the total amount of solids present in the six original samples to undergo the DWPF CC digestion, the amount of undissolved or precipitated solids recovered by filtration of the digestion solutions, the amount of each element (cations only) present in the undissolved or precipitated solids, the percent of the undissolved or precipitated solids that is Al, and the percent of Al that is from *boehmite* which was spiked into the sample. The percent Al in the undissolved solids from *boehmite* was calculated by determining the amount of *boehmite* present per gram of sludge solids and dividing the weight of Al measured from the PF digestion of undissolved/precipitated solids from the DWPF CC method digestions by the calculated amount of Al from *boehmite* spiked into the parent sample. In each case, more than 80% of the Al recovered was from *boehmite*. X-ray diffraction data were not obtained on any portion of the insoluble/precipitated solids.

Table 3.5 ICP-ES analysis results of the undissolved/precipitated solids from the DWPF CC digestions of *boemite* spiked SB4 simulant sludge.

Sample #	1	2	3	4	5	6
Amount of Solids in Original DWPF CC Digestions						
(g)	0.905	0.911	0.920	0.927	0.940	0.903
Amount of Undissolved or Precipitated Solids in DWPF CC Digestions Recovered by Filtration						
(g)	0.1323	0.1170	0.1268	0.1211	0.1377	0.1145
Amount of Each Element in Undissolved or Precipitated Solids in DWPF CC Digestions (mg) Determined by ICP-ES Analysis						
Al (mg)	27.0	20.2	24.0	21.1	23.7	21.9
Ca (mg)	7.3	5.8	6.4	6.5	6.6	6.6
Cr (mg)	1.1	1.0	1.0	1.0	1.1	1.0
Fe (mg)	3.2	2.2	2.7	2.3	2.5	2.2
K (mg)	0.6	0.4	<MDL	<MDL	0.5	<MDL
Mg (mg)	5.4	3.8	4.7	4.4	4.5	4.4
Mn (mg)	2.8	2.0	2.5	2.1	2.2	2.0
Ni (mg)	0.1	0.04	0.1	0.04	0.1	0.05
Ti (mg)	0.03	0.02	0.02	0.03	0.03	0.03
Sum (mg)	47.4	35.5	41.4	37.4	41.4	38.1
% of Al in Undissolved/Precipitated Solids	56.9	57.1	57.9	56.3	57.2	57.4
% of Al from *Boehmite* in solids (assuming all of it is insoluble)	84.1	112.8	96.3	110.3	99.5	103.6

Statistically significant differences in concentration were noted in Table 3.4 for Al, Ca, K and Mg - all of which are seen in the solids recovered from filtration of the DWPF CC digestion solutions. However, it is difficult to assess the error associated with performing sequential digestions of material; therefore, the data were re-evaluated with the help of another statistical comparison for Al. The Al data from the PF digestion were compared to the average and upper and lower bounds (obtained by adding the standard deviation to the average concentration of six samples or subtracting the standard deviation from the average concentration) of data obtained from the DWPF CC plus the PF digestions. This comparison gives a more complete picture of the variability associated with multiple digestions on the same material (Figure 3.1).

Figure 3.1 Statistical comparison of the ICP-ES measured upper and lower bounds (percent relative standard deviation) of SB4 simulant sludge slurry spiked with *boehmite* digested by the DWPF CC + PF digestion of the undissolved solids in the DWPF CC solutions *versus* PF)



The lower concentration bound and the average concentration range of Al from the CC+PF experiments are statistically different at the 5% significance level from the PF only digestion. However, the upper concentration range of Al from the CC+PF digestions and the PF digestion only are not statistically different. If the DWPF lab were to adopt this digestion strategy, it would be necessary for DWPF engineering to determine the allowable variability in the measurements of the concentration of Al. It should be noted that the Al concentration determined in the DWPF CC digestion before digestion of the filtered solids was approximately 2.5% lower than the combined Al result after digestion of the undissolved solids.

The ICP-ES results of analysis of the ARG standards that were digested in tandem along with the *boehmite* spiked SB4 simulant are shown in Table 3.6. Statistically significant differences in the means compared to the ARG reference values werenoted for B, Mg, and Ni. Several elements were found to be in the undissolved solids in the DWPF CC method digestion solutions which were recovered by filtration and dissolved by the subsequent PF. The elements were Al, Ca, Fe, Mg, Mn and Ni. The elements in which there was a statistical difference between digestion results in the first set of ARG samples digested along with the SB4 simulant containing only *gibbsite* were Al, Ca, Cr and Mn. It was somewhat surprising to identify so many elements from species that either did not dissolve or precipitated from the DWPF CC digestions performed on the ARG standards. In many cases, though, it was such a small amount of material that did not dissolve that it had no impact on the final results and statistical analysis. In the current case, only 2 of the 6 elements that had species which may not have dissolved fully during the CC digestion actually ended up having statistically different means when compared to the PF data. Calcium is a contaminant in the fusion reagents and so the higher average concentration measured by ICP-ES in the PF samples was expected. The PF digestion is performed in a Zr crucible, a source of Zr contamination. The higher potassium concentration in the PF was unexpected and the reason for it is unclear.

Table 3.6 ICP-ES analysis results from digested (DWPF CC + PF digestion of the undissolved solids in the DWPF CC Method solutions *versus* PF) ARG samples and statistical comparison of the data to the ARG reference value. Values are presented on a wt% dried solids basis.

Element	Wt % from DWPF CC+PF of Undissolved Solids *	% RSD#	Wt % from PF Digestion*	% RSD#	Statistically Different Means (Combined Digestion Result versus Reference Value, at a 5% Significance Level)	DWPF CC + PF of Undissolved Solids Result Ratio to True Value	PF Digestion Result Ratio to True Value
Al	2.45E+00	3.2E+00	2.38E+00	4.8E+00	No	0.98	0.95
B	2.80E+00	9.8E-01	2.66E+00	6.0E+00	Yes	1.04	0.99
Ca	9.98E-01	4.9E+00	1.16E+00	6.2E+00	No	0.98	1.14
Cr	6.79E-02	4.9E+00	6.67E-02	4.0E+00	No	1.06	1.04
Cu	<5.00E-02	NA	<1.78E-02	NA	NA	NA	NA
Fe	9.88E+00	7.0E-01	9.67E+00	4.7E+00	No	1.01	0.99
Li	2.13E+00	8.6E+00	1.47E+00	4.7E+00	No	0.94	0.99
K	1.50E+00	8.1E-01	2.52E+00	9.0E+00	No	1.01	1.12
Mg	4.93E-01	7.7E-01	4.79E-01	5.1E+00	Yes	0.95	0.92
Mn	1.45E+00	3.8E-01	1.43E+00	5.3E+00	No	1.00	0.98
Na	8.21E+00	5.9E-01	NA	NA	NA	0.96	NA
Ni	8.60E-01	8.4E-01	7.98E-01	4.8E+00	Yes	1.04	0.96
Si	2.11E+01	1.8E+00	2.19E+01	5.0E+00	No	0.94	0.98
Ti	7.01E-01	1.1E+00	6.89E-01	4.6E+00	No	1.02	1.00
U^	NA	NA	NA	NA	NA	NA	NA
Zr	1.05E-01	3.0E+00	NA	NA	NA	1.09	NA

*Three samples were digested by each method and analyzed by ICP-ES. #% RSD = percent relative standard deviation.
^Uranium is not present in the simulant sludge.

The ICP-ES results from analysis of the multi-element standards submitted with the *boehmite* spiked samples are summarized in Table 3.7. One purified *boehmite* sample was dissolved using the PF digestion along with this sample batch. The recovery of Al on an elemental basis based on ICP-ES data was 97.4%.

Table 3.7 Multi-element ICP-ES standard results. Standards were analyzed as part of the sample batch that included he samples in Table 3.4 and Table 3.6.

		DWPF CC + PF Sample Batch Analysis				PF Sample Batch Analysis			
Element	Reference Value	Result (mg/L) Run 1*	Delta	Result (mg/L) Run 2^	Delta	Result (mg/L) Run 1*	Delta	Result (mg/L) Run 1^	Delta
Al	4	3.9	-2.3	3.7	-7.3	3.7	-6.8	4.0	0.5
B	20	19.9	-0.5	19.5	-2.5	19.9	-0.5	19.5	-2.5
Fe	4	4.0	0.2	4.0	-1.0	4.03	0.8	4.0	0.2
Li	10	10.2	2.0	10.4	4.0	10.4	4.0	10.3	3.0
K	10	9.4	-6.2	9.4	-5.6	10.8	8.0	12.6	26.0
Si	50	49.9	-0.2	48.4	-3.2	49.4	-1.2	49.3	-1.4
Na	81	81.2	0.2	80.8	-0.2	NA	NA	NA	NA

*Multi-ement standard analyzed within the first few samples of the batch. ^Muli-element standard analyzed at the end of the sample batch.

The ICP-ES results from analysis of duplicate digestions of feldspar and triplicate digestions of purified *boehmite* are shown in Table 3.8 and Table 3.9. The iron oxide Fe_2O_3 result in Table 3.8 is high. The reason is unclear but Fe is present at such a low level in the feldspar that a minor amount of contaminant would result in a large difference versus the Fe_2O_3 reference value. A low sodium oxide (Na_2O) value was obtained and is noted in Table 3.8 for the 2nd sample. Again, the reason the measured sodium value is low is unclear, but most likely it is an ICP problem rather than a problem with the digestion because the other elements are close to their given reference values. In both cases, the measured Al values agree reasonably well compared to the standard reference value for this element.

Table 3.8 ICP-ES results from analysis of digested feldspar. Feldspar was digested in duplicate and in tandem with the samples listed in Table 3.6, Table 3.7 and Table 3.9.

DWPF CC + PF of Feldspar					
Element	Wt% Oxide Reference Value	Sample 1 Wt%	Delta	Sample 2 Wt%	Delta
Al_2O_3	20.5	19.8	-3.3	19.9	-2.8
BaO	0.26	0.26	-1.7	0.26	1.8
CaO	2.14	2.15	0.5	2.24	4.8
Fe_2O_3	0.06	0.09	44.1	0.26	326.2
Na_2O	6.2	5.73	-7.6	3.12	-49.6
SiO_2	65.2	69.4	6.4	68.3	4.7

Purified *boehmite* was digested in triplicate as quality control samples. The results are listed in Table 3.9. In all cases the measured *boehmite* concentration is low. This can be attributed to the difficulty encountered trying to transfer the *boehmite* out of the bottle into a filter to collect the undissolved solids. The *boehmite* powder kept sticking to the sides of the plastic bottle and could not be easily rinsed out.

Table 3.9 ICP-ES results from analysis of digested purified *boehmit*e. Purified *boehmite* was digested in triplicate and in tandem with the samples listed in Table 3.6, Table 3.7 and Table 3.8.

Element	Wt% Elemental Reference Value	Sample 1 Wt%	Delta	Sample 2 Wt%	Delta	Sample 3 Wt%	Delta
Al	44.98	31.5	-30.0	37.6	-16.3	34.8	-22.7

4.0 CONCLUSIONS

The CC + PF digestion of SB4 simulant sludge has shown this method can be used as a possible alternative to the DWPF CC method now being used for process control samples. The experimental results from digestion of SB4 simulant spiked with *boehmite* indicate the following:

- The measured and combined Al concentration from the CC + PF digestion of SB4 simulant was close to the PF only method indicating that this method will digest all aluminum hydroxide species present. The additional steps necessary to perform the combined digestion method is not expected to significantly increase the burden of the cell technician as PF digestions are already currently performed at DWPF.
- The CC + PF method will result in decreased turnaround times for the DWPF Lab versus the PF only digestion method because only the insoluble solids from the CC digestion will undergo fusion. This will result in a time savings as only milligrams of material will be dried and digested instead of multi-gram quantities of sludge slurry.
- Compounds that tend to precipitate from the DWPF CC digestion solutions (mixed metal fluoride salts) will be digested and measured resulting in a better determination of the Al concentration in process samples over the current digestion method.

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5.0 RECOMMENDATIONS

The following recommendations are based upon ICP-ES results and statistical analysis of those results from experimental digestion tests conducted with SB4 simulant sludge slurry. It is recommended that the DWPF:

- Pursue a side-by-side development and comparison of the combined digestion method (DWPF CC plus PF of undissolved solids) in the SRNL shielded cells and at the DWPF Lab using radioactive sludge.

- Perform periodic analysis (X-ray diffraction) of solids that may be in the DWPF CC analytical process digestion samples out at DWPF to help further refine the digestion method.

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6.0 REFERENCES

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6. JMP Statistical Discovery Software *v* 6.02; SAS Institute Inc., Cary, NC, 2005.

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7.0 ACKNOWLEDGEMENTS

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Distribution
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R.E. Edwards, 773-A
T.B. Edwards, 999-W
C.C. Herman, 999-W
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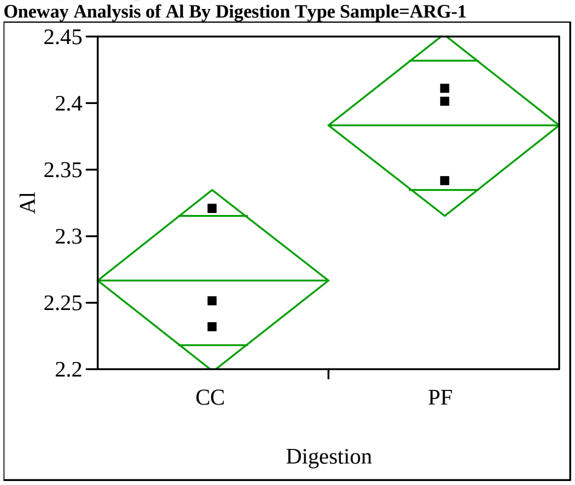
ADS Files

APPENDIX A

JMP One-Way Analysis Plots of ARG Digested by PF and DWPF CC

Fit Y by X Group

Oneway Analysis of Al By Digestion Type Sample=ARG-1



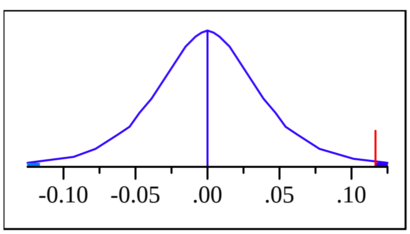
Oneway Anova
Summary of Fit

Rsquare	0.735736
Adj Rsquare	0.66967
Root Mean Square Error	0.042817
Mean of Response	2.325
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.116667	t Ratio	3.337119
Std Err Dif	0.034960	DF	4
Upper CL Dif	0.213732	Prob > |t|	0.0289
Lower CL Dif	0.019601	Prob > t	0.0145
Confidence	0.95	Prob < t	0.9855



Analysis of Variance

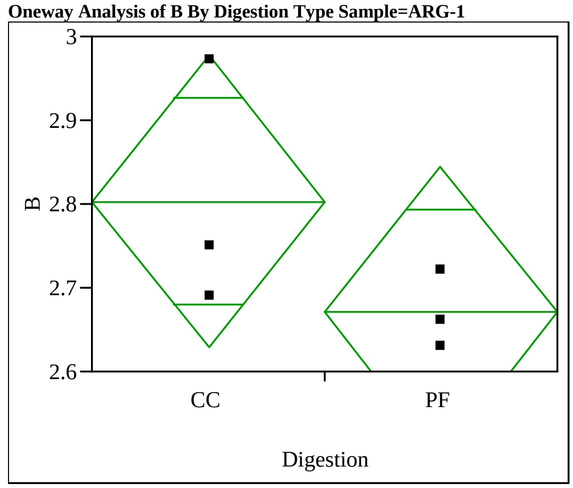
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02041667	0.020417	11.1364	0.0289
Error	4	0.00733333	0.001833		
C. Total	5	0.02775000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	2.26667	0.02472	2.1980	2.3353
PF	3	2.38333	0.02472	2.3147	2.4520

Std Error uses a pooled estimate of error variance

Oneway Analysis of B By Digestion Type Sample=ARG-1



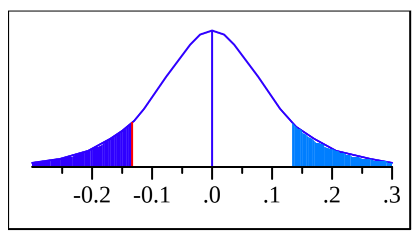
Oneway Anova
Summary of Fit

Rsquare	0.358744
Adj Rsquare	0.19843
Root Mean Square Error	0.109163
Mean of Response	2.736667
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.13333	t Ratio	-1.49592
Std Err Dif	0.08913	DF	4
Upper CL Dif	0.11414	Prob > |t|	0.2090
Lower CL Dif	-0.38080	Prob > t	0.8955
Confidence	0.95	Prob < t	0.1045



Analysis of Variance

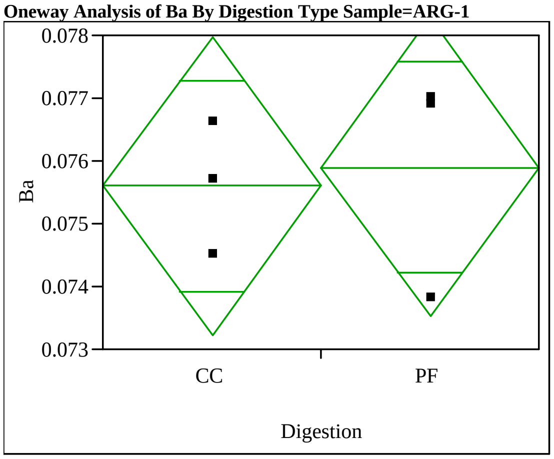
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02666667	0.026667	2.2378	0.2090
Error	4	0.04766667	0.011917		
C. Total	5	0.07433333			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	2.80333	0.06303	2.6283	2.9783
PF	3	2.67000	0.06303	2.4950	2.8450

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ba By Digestion Type Sample=ARG-1



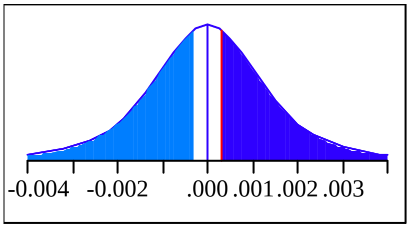
Oneway Anova
Summary of Fit

Rsquare	0.015042
Adj Rsquare	-0.2312
Root Mean Square Error	0.001487
Mean of Response	0.07575
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.00030	t Ratio	0.247156
Std Err Dif	0.00121	DF	4
Upper CL Dif	0.00367	Prob > |t|	0.8170
Lower CL Dif	-0.00307	Prob > t	0.4085
Confidence	0.95	Prob < t	0.5915



Analysis of Variance

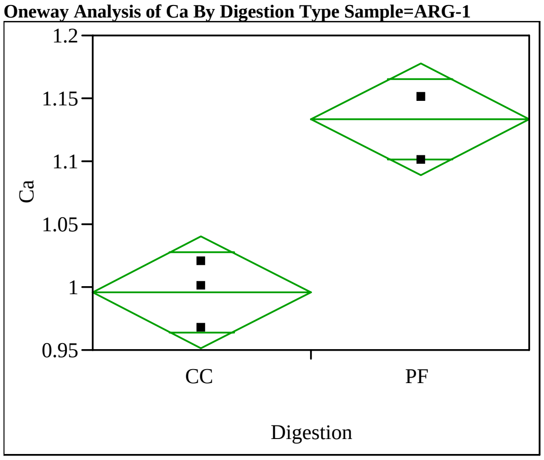
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	1.35e-7	1.35e-7	0.0611	0.8170
Error	4	0.00000884	2.21e-6		
C. Total	5	8.975e-6			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.075600	0.00086	0.07322	0.07798
PF	3	0.075900	0.00086	0.07352	0.07828

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ca By Digestion Type Sample=ARG-1



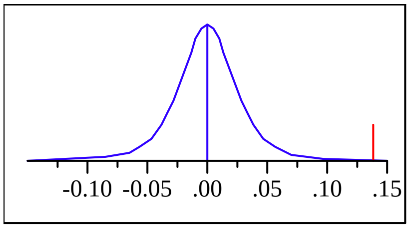
Oneway Anova
Summary of Fit

Rsquare	0.901694
Adj Rsquare	0.877118
Root Mean Square Error	0.027836
Mean of Response	1.0645
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.137667	t Ratio	6.057177
Std Err Dif	0.022728	DF	4
Upper CL Dif	0.200769	Prob > |t|	0.0037
Lower CL Dif	0.074564	Prob > t	0.0019
Confidence	0.95	Prob < t	0.9981



Analysis of Variance

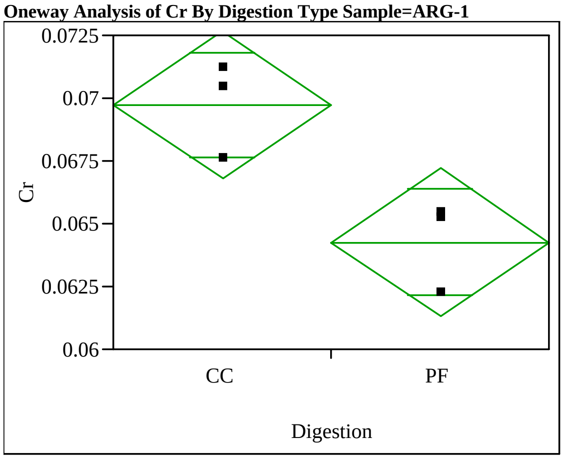
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02842817	0.028428	36.6894	0.0037
Error	4	0.00309933	0.000775		
C. Total	5	0.03152750			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.99567	0.01607	0.9510	1.0403
PF	3	1.13333	0.01607	1.0887	1.1780

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cr By Digestion Type Sample=ARG-1



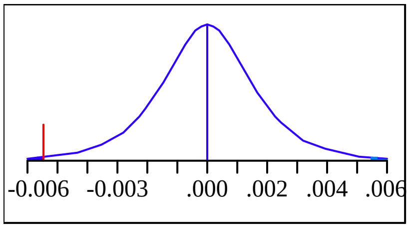
Oneway Anova
Summary of Fit

Rsquare	0.76758
Adj Rsquare	0.709475
Root Mean Square Error	0.001842
Mean of Response	0.067
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.00547	t Ratio	-3.63459
Std Err Dif	0.00150	DF	4
Upper CL Dif	-0.00129	Prob > |t|	0.0221
Lower CL Dif	-0.00964	Prob > t	0.9890
Confidence	0.95	Prob < t	0.0110



Analysis of Variance

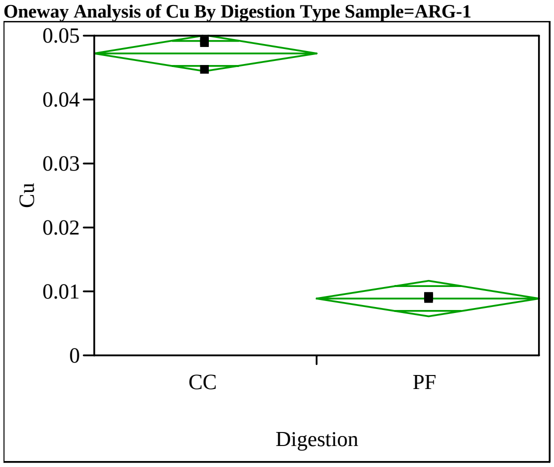
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00004483	0.000045	13.2102	0.0221
Error	4	0.00001357	3.393e-6		
C. Total	5	0.00005840			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.069733	0.00106	0.06678	0.07269
PF	3	0.064267	0.00106	0.06131	0.06722

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cu By Digestion Type Sample=ARG-1



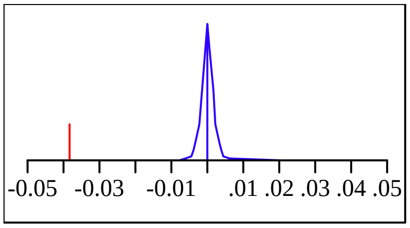
Oneway Anova
Summary of Fit

Rsquare	0.994667
Adj Rsquare	0.993334
Root Mean Square Error	0.001726
Mean of Response	0.028055
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.03849	t Ratio	-27.3139
Std Err Dif	0.00141	DF	4
Upper CL Dif	-0.03458	Prob > |t|	<.0001
Lower CL Dif	-0.04240	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

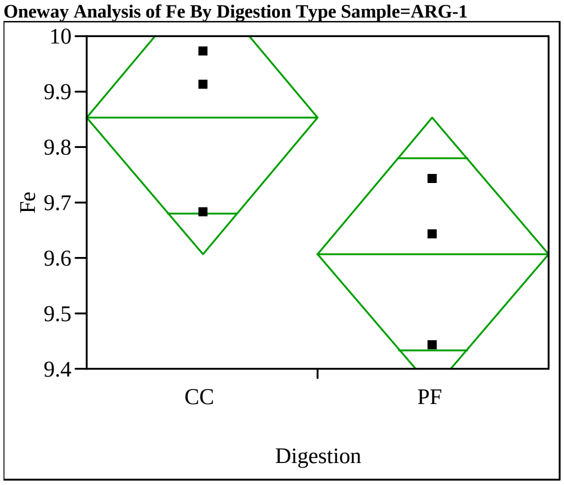
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00222222	0.002222	746.0494	<.0001
Error	4	0.00001191	2.979e-6		
C. Total	5	0.00223413			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.047300	0.00100	0.04453	0.05007
PF	3	0.008810	0.00100	0.00604	0.01158

Std Error uses a pooled estimate of error variance

Oneway Analysis of Fe By Digestion Type Sample=ARG-1



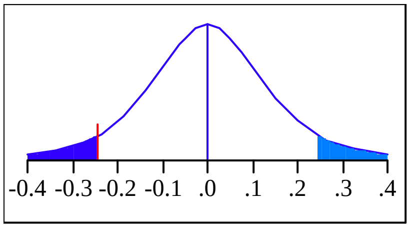
Oneway Anova
Summary of Fit

Rsquare	0.493867
Adj Rsquare	0.367334
Root Mean Square Error	0.152916
Mean of Response	9.73
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.24667	t Ratio	-1.97562
Std Err Dif	0.12486	DF	4
Upper CL Dif	0.09999	Prob > |t|	0.1194
Lower CL Dif	-0.59332	Prob > t	0.9403
Confidence	0.95	Prob < t	0.0597



Analysis of Variance

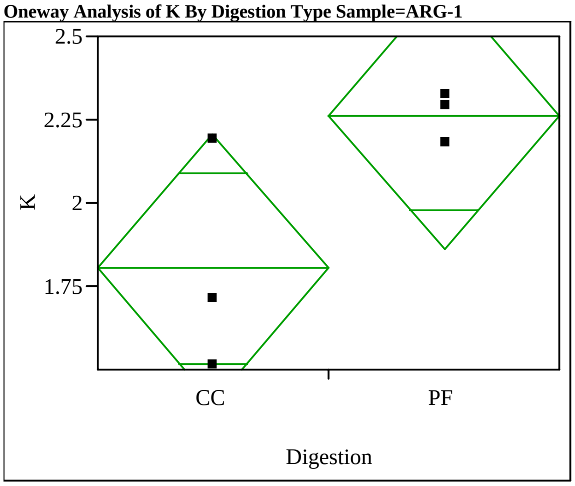
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.09126667	0.091267	3.9031	0.1194
Error	4	0.09353333	0.023383		
C. Total	5	0.18480000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	9.85333	0.08829	9.6082	10.098
PF	3	9.60667	0.08829	9.3615	9.852

Std Error uses a pooled estimate of error variance

Oneway Analysis of K By Digestion Type Sample=ARG-1



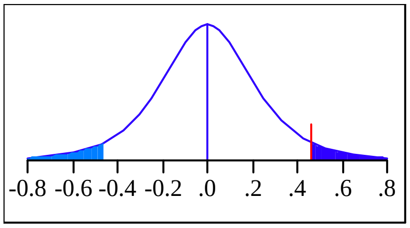
Oneway Anova
Summary of Fit

Rsquare	0.554378
Adj Rsquare	0.442973
Root Mean Square Error	0.252554
Mean of Response	2.033333
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.4600	t Ratio	2.230745
Std Err Dif	0.2062	DF	4
Upper CL Dif	1.0325	Prob > |t|	0.0895
Lower CL Dif	-0.1125	Prob > t	0.0448
Confidence	0.95	Prob < t	0.9552



Analysis of Variance

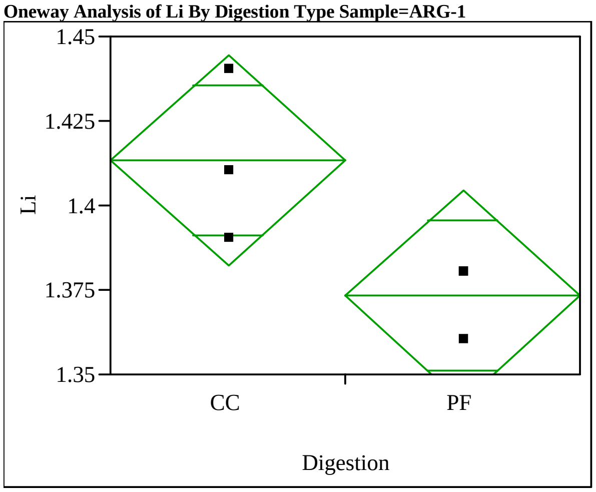
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.31740000	0.317400	4.9762	0.0895
Error	4	0.25513333	0.063783		
C. Total	5	0.57253333			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	1.80333	0.14581	1.3985	2.2082
PF	3	2.26333	0.14581	1.8585	2.6682

Std Error uses a pooled estimate of error variance

Oneway Analysis of Li By Digestion Type Sample=ARG-1



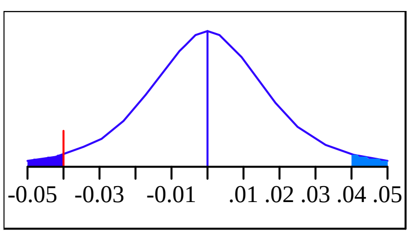
Oneway Anova
Summary of Fit

Rsquare	0.610169
Adj Rsquare	0.512712
Root Mean Square Error	0.019579
Mean of Response	1.393333
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.04000	t Ratio	-2.50217
Std Err Dif	0.01599	DF	4
Upper CL Dif	0.00438	Prob > |t|	0.0666
Lower CL Dif	-0.08438	Prob > t	0.9667
Confidence	0.95	Prob < t	0.0333



Analysis of Variance

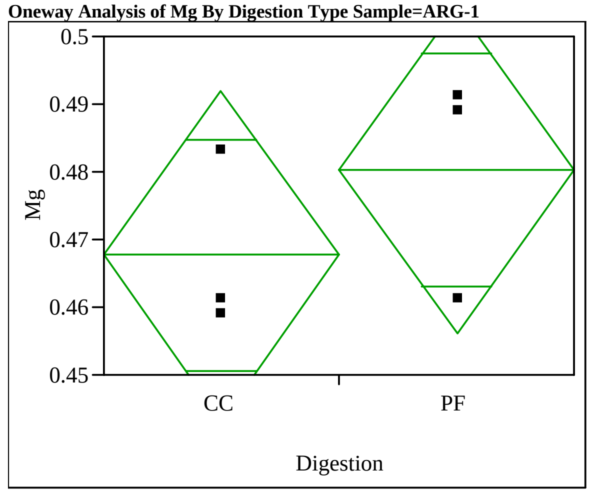
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00240000	0.002400	6.2609	0.0666
Error	4	0.00153333	0.000383		
C. Total	5	0.00393333			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	1.41333	0.01130	1.3819	1.4447
PF	3	1.37333	0.01130	1.3419	1.4047

Std Error uses a pooled estimate of error variance

Oneway Analysis of Mg By Digestion Type Sample=ARG-1



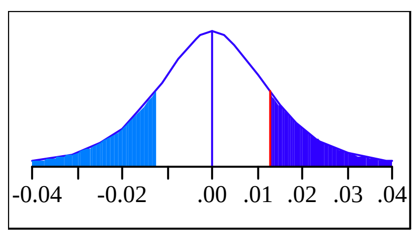
Oneway Anova
Summary of Fit

Rsquare	0.20783
Adj Rsquare	0.009787
Root Mean Square Error	0.015144
Mean of Response	0.474
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.01267	t Ratio	1.024411
Std Err Dif	0.01236	DF	4
Upper CL Dif	0.04700	Prob > |t|	0.3635
Lower CL Dif	-0.02166	Prob > t	0.1818
Confidence	0.95	Prob < t	0.8182



Analysis of Variance

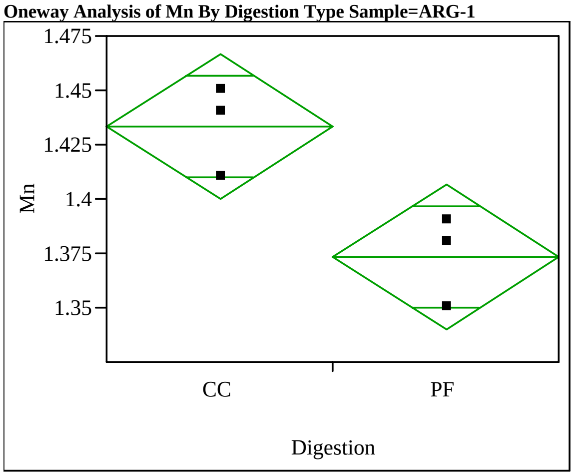
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00024067	0.000241	1.0494	0.3635
Error	4	0.00091733	0.000229		
C. Total	5	0.00115800			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.467667	0.00874	0.44339	0.49194
PF	3	0.480333	0.00874	0.45606	0.50461

Std Error uses a pooled estimate of error variance

Oneway Analysis of Mn By Digestion Type Sample=ARG-1



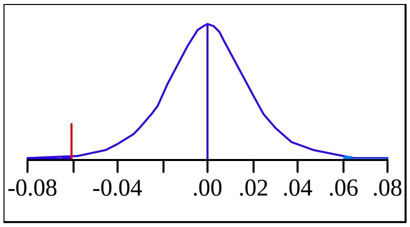
Oneway Anova
Summary of Fit

Rsquare	0.757009
Adj Rsquare	0.696262
Root Mean Square Error	0.020817
Mean of Response	1.403333
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.06000	t Ratio	-3.53009
Std Err Dif	0.01700	DF	4
Upper CL Dif	-0.01281	Prob > |t|	0.0242
Lower CL Dif	-0.10719	Prob > t	0.9879
Confidence	0.95	Prob < t	0.0121



Analysis of Variance

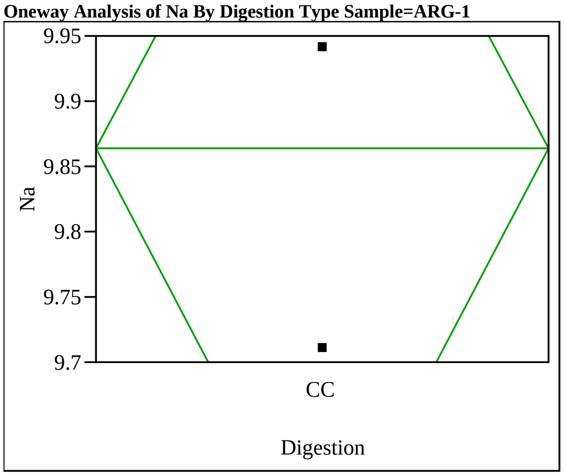
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00540000	0.005400	12.4615	0.0242
Error	4	0.00173333	0.000433		
C. Total	5	0.00713333			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	1.43333	0.01202	1.4000	1.4667
PF	3	1.37333	0.01202	1.3400	1.4067

Std Error uses a pooled estimate of error variance

Oneway Analysis of Na By Digestion Type Sample=ARG-1



Missing Rows
3

Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.132791
Mean of Response	9.863333
Observations (or Sum Wgts)	3

Analysis of Variance

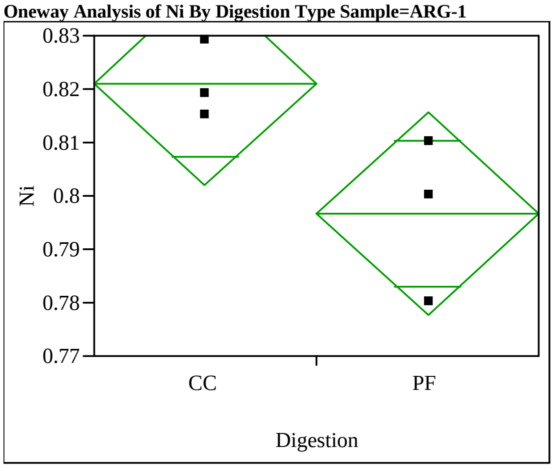
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	2	0.03526667	0.017633		
C. Total	2	0.03526667			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	9.86333	0.07667	9.5335	10.193

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ni By Digestion Type Sample=ARG-1



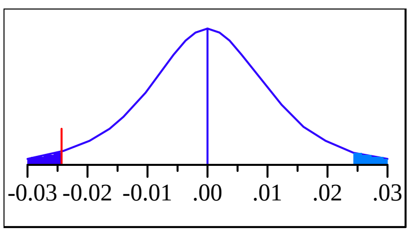
Oneway Anova
Summary of Fit

Rsquare	0.60882
Adj Rsquare	0.511025
Root Mean Square Error	0.011944
Mean of Response	0.808833
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.02433	t Ratio	-2.49509
Std Err Dif	0.00975	DF	4
Upper CL Dif	0.00274	Prob > |t|	0.0671
Lower CL Dif	-0.05141	Prob > t	0.9664
Confidence	0.95	Prob < t	0.0336



Analysis of Variance

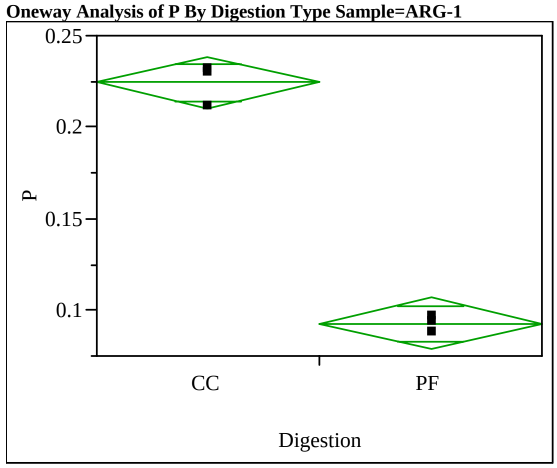
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00088817	0.000888	6.2255	0.0671
Error	4	0.00057067	0.000143		
C. Total	5	0.00145883			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.821000	0.00690	0.80185	0.84015
PF	3	0.796667	0.00690	0.77752	0.81581

Std Error uses a pooled estimate of error variance

Oneway Analysis of P By Digestion Type Sample=ARG-1



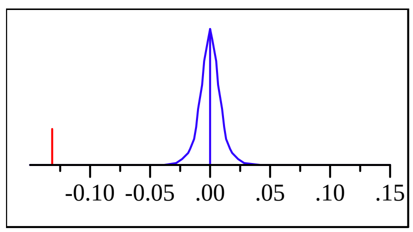
Oneway Anova
Summary of Fit

Rsquare	0.988192
Adj Rsquare	0.98524
Root Mean Square Error	0.008816
Mean of Response	0.158483
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.13170	t Ratio	-18.2966
Std Err Dif	0.00720	DF	4
Upper CL Dif	-0.11171	Prob > |t|	<.0001
Lower CL Dif	-0.15169	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

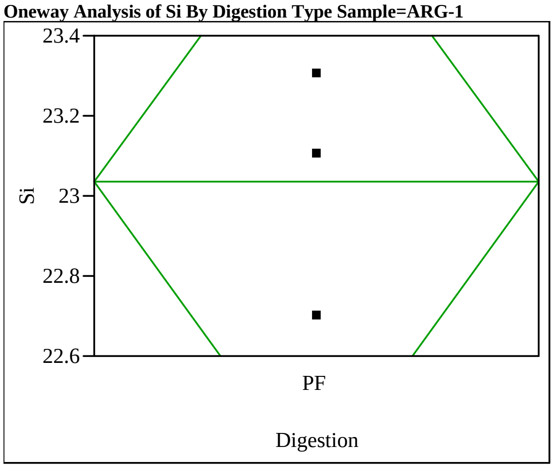
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02601734	0.026017	334.7644	<.0001
Error	4	0.00031087	0.000078		
C. Total	5	0.02632821			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.224333	0.00509	0.21020	0.23846
PF	3	0.092633	0.00509	0.07850	0.10676

Std Error uses a pooled estimate of error variance

Oneway Analysis of Si By Digestion Type Sample=ARG-1



Missing Rows
3

Oneway Anova

Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.305505
Mean of Response	23.03333
Observations (or Sum Wgts)	3

Analysis of Variance

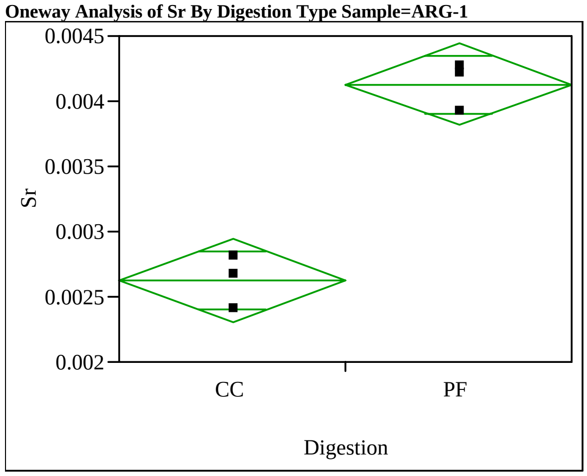
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	2	0.18666667	0.093333		
C. Total	2	0.18666667			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
PF	3	23.0333	0.17638	22.274	23.792

Std Error uses a pooled estimate of error variance

Oneway Analysis of Sr By Digestion Type Sample=ARG-1



Oneway Anova

Summary of Fit

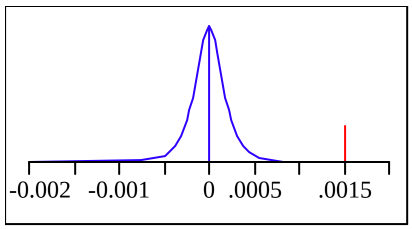
Rsquare	0.956475
Adj Rsquare	0.945593
Root Mean Square Error	0.000196
Mean of Response	0.003378
Observations (or Sum Wgts)	6

t Test

PF-CC

Assuming equal variances

Difference	0.001503	t Ratio	9.375509
Std Err Dif	0.000160	DF	4
Upper CL Dif	0.001949	Prob > |t|	0.0007
Lower CL Dif	0.001058	Prob > t	0.0004
Confidence	0.95	Prob < t	0.9996



Analysis of Variance

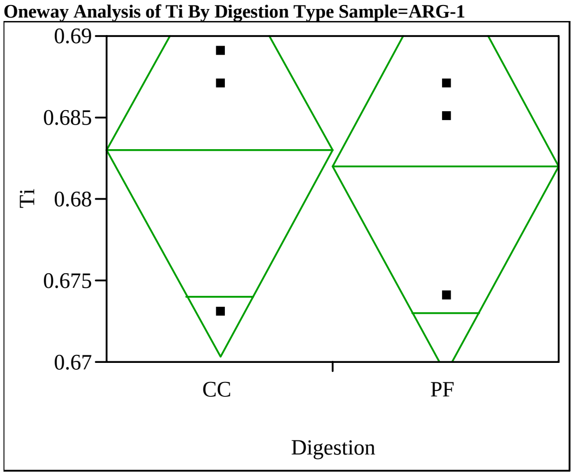
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00000339	3.39e-6	87.9002	0.0007
Error	4	1.54267e-7	3.8567e-8		
C. Total	5	3.54428e-6			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.002627	0.00011	0.00231	0.00294
PF	3	0.004130	0.00011	0.00382	0.00444

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ti By Digestion Type Sample=ARG-1



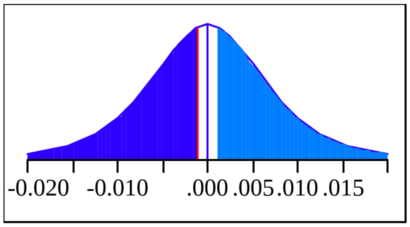
Oneway Anova
Summary of Fit

Rsquare	0.005964
Adj Rsquare	-0.24254
Root Mean Square Error	0.007906
Mean of Response	0.6825
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	-0.00100	t Ratio	-0.15492
Std Err Dif	0.00645	DF	4
Upper CL Dif	0.01692	Prob > |t|	0.8844
Lower CL Dif	-0.01892	Prob > t	0.5578
Confidence	0.95	Prob < t	0.4422



Analysis of Variance

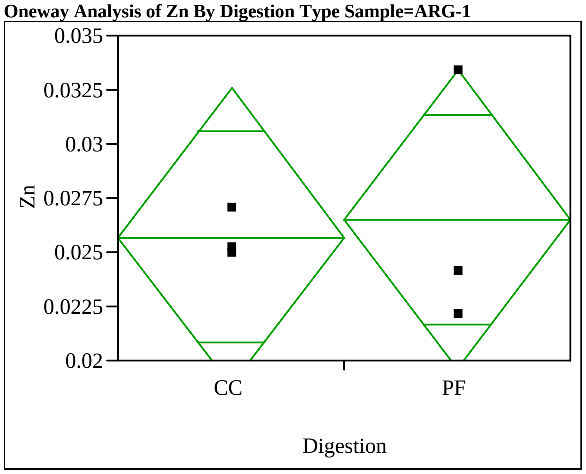
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00000150	1.5e-6	0.0240	0.8844
Error	4	0.00025000	0.000062		
C. Total	5	0.00025150			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.683000	0.00456	0.67033	0.69567
PF	3	0.682000	0.00456	0.66933	0.69467

Std Error uses a pooled estimate of error variance

Oneway Analysis of Zn By Digestion Type Sample=ARG-1



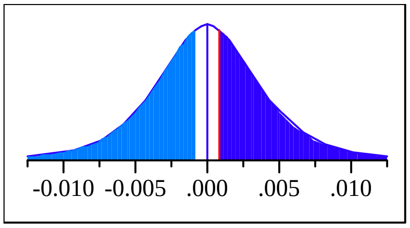
Oneway Anova
Summary of Fit

Rsquare	0.012817
Adj Rsquare	-0.23398
Root Mean Square Error	0.004299
Mean of Response	0.0261
Observations (or Sum Wgts)	6

t Test
PF-CC

Assuming equal variances

Difference	0.00080	t Ratio	0.22789
Std Err Dif	0.00351	DF	4
Upper CL Dif	0.01055	Prob > |t|	0.8309
Lower CL Dif	-0.00895	Prob > t	0.4155
Confidence	0.95	Prob < t	0.5845



Analysis of Variance

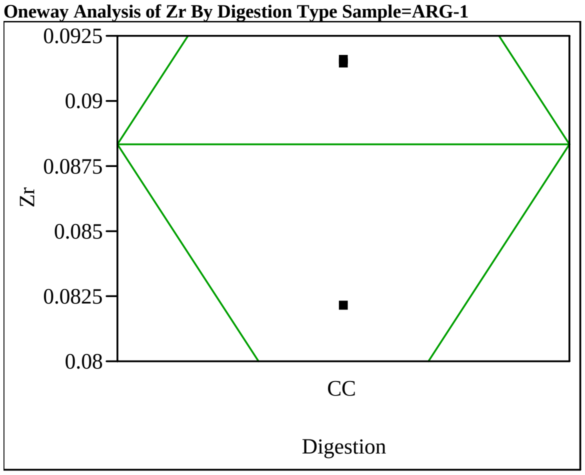
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00000096	9.6e-7	0.0519	0.8309
Error	4	0.00007394	0.000018		
C. Total	5	0.00007490			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.025700	0.00248	0.01881	0.03259
PF	3	0.026500	0.00248	0.01961	0.03339

Std Error uses a pooled estimate of error variance

Oneway Analysis of Zr By Digestion Type Sample=ARG-1



Missing Rows
3

Oneway Anova

Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.005398
Mean of Response	0.088333
Observations (or Sum Wgts)	3

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	2	0.00005829	0.000029		
C. Total	2	0.00005829			

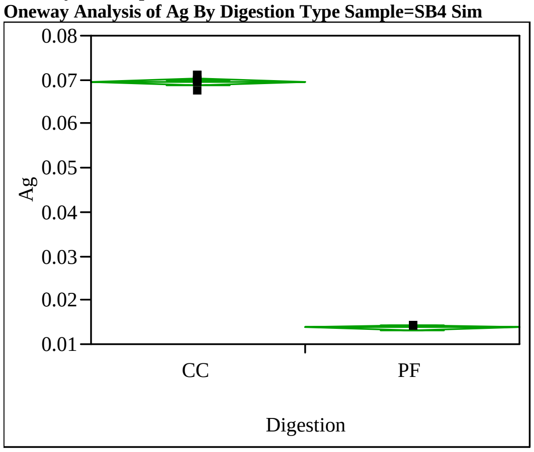
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	3	0.088333	0.00312	0.07492	0.10174

Std Error uses a pooled estimate of error variance

Fit Y by X Group

Oneway Analysis of Ag By Digestion Type Sample=SB4 Sim



Oneway Anova

Summary of Fit

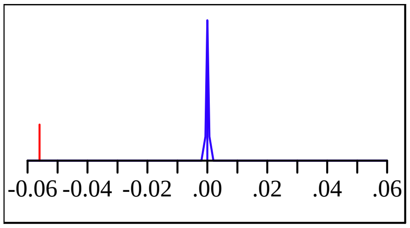
Rsquare	0.999242
Adj Rsquare	0.999166
Root Mean Square Error	0.000841
Mean of Response	0.041575
Observations (or Sum Wgts)	12

t Test

PF-CC

Assuming equal variances

Difference	-0.05575	t Ratio	-114.8
Std Err Dif	0.00049	DF	10
Upper CL Dif	-0.05467	Prob > |t|	0.0000
Lower CL Dif	-0.05683	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00932419	0.009324	13179.06	<.0001
Error	10	0.00000708	7.075e-7		
C. Total	11	0.00933126			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.069450	0.00034	0.06868	0.07022
PF	6	0.013700	0.00034	0.01293	0.01447

Std Error uses a pooled estimate of error variance

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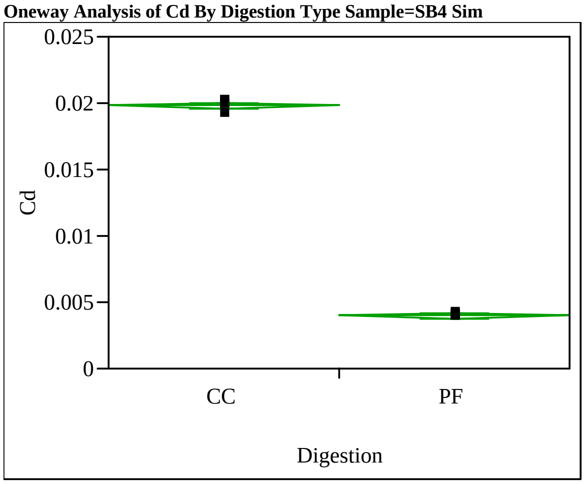
APPENDIX B

JMP One-Way Analysis Plots of SB4 Simulant Digested by PF and DWPF CC

Oneway Analysis of Cd By Digestion Type Sample=SB4 Sim



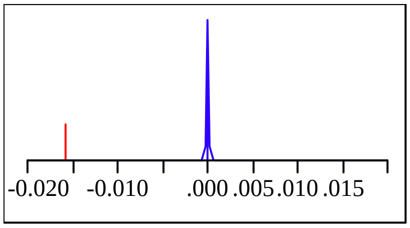
Oneway Anova
Summary of Fit

Rsquare	0.999238
Adj Rsquare	0.999161
Root Mean Square Error	0.000239
Mean of Response	0.011885
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.01583	t Ratio	-114.482
Std Err Dif	0.00014	DF	10
Upper CL Dif	-0.01552	Prob > |t|	0.0000
Lower CL Dif	-0.01614	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

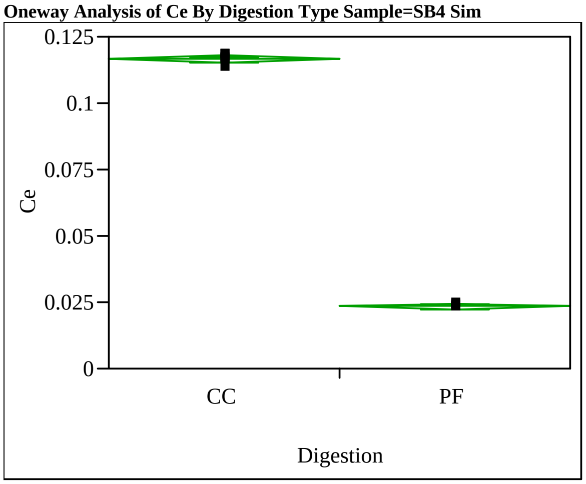
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00075177	0.000752	13106.11	<.0001
Error	10	0.00000057	5.736e-8		
C. Total	11	0.00075234			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.019800	0.0001	0.01958	0.02002
PF	6	0.003970	0.0001	0.00375	0.00419

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ce By Digestion Type Sample=SB4 Sim



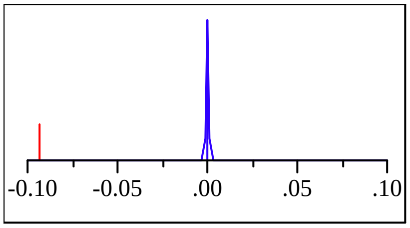
Oneway Anova
Summary of Fit

Rsquare	0.999283
Adj Rsquare	0.999212
Root Mean Square Error	0.001367
Mean of Response	0.069917
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.09317	t Ratio	-118.089
Std Err Dif	0.00079	DF	10
Upper CL Dif	-0.09141	Prob > |t|	0.0000
Lower CL Dif	-0.09492	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

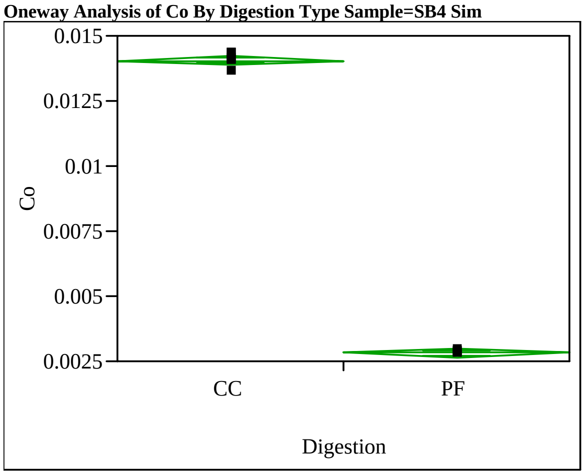
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02604008	0.026040	13945.06	<.0001
Error	10	0.00001867	1.867e-6		
C. Total	11	0.02605876			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.116500	0.00056	0.11526	0.11774
PF	6	0.023333	0.00056	0.02209	0.02458

Std Error uses a pooled estimate of error variance

Oneway Analysis of Co By Digestion Type Sample=SB4 Sim



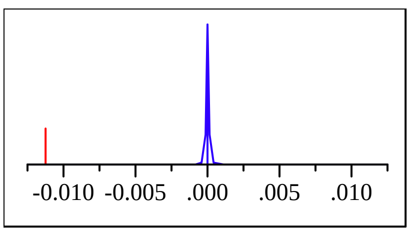
Oneway Anova
Summary of Fit

Rsquare	0.999074
Adj Rsquare	0.998982
Root Mean Square Error	0.000187
Mean of Response	0.008432
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.01124	t Ratio	-103.893
Std Err Dif	0.00011	DF	10
Upper CL Dif	-0.01100	Prob > |t|	<.0001
Lower CL Dif	-0.01148	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

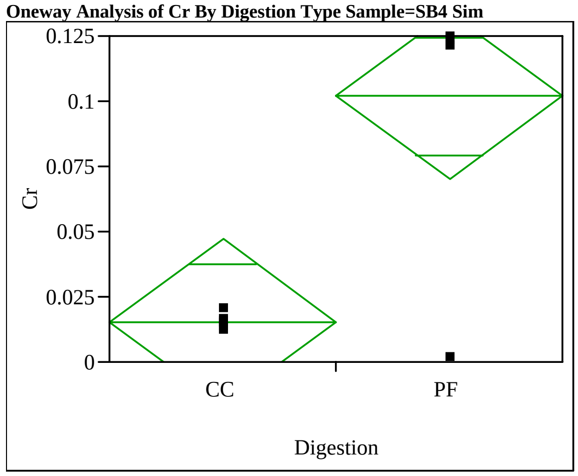
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00037879	0.000379	10793.73	<.0001
Error	10	0.00000035	3.509e-8		
C. Total	11	0.00037914			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.014050	7.65e-5	0.01388	0.01422
PF	6	0.002813	7.65e-5	0.00264	0.00298

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cr By Digestion Type Sample=SB4 Sim



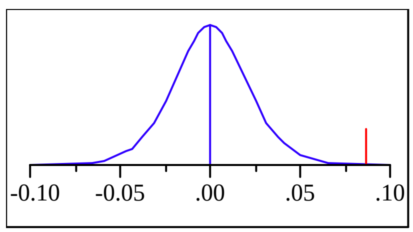
Oneway Anova
Summary of Fit

Rsquare	0.648994
Adj Rsquare	0.613893
Root Mean Square Error	0.034932
Mean of Response	0.058511
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	0.086722	t Ratio	4.299942
Std Err Dif	0.020168	DF	10
Upper CL Dif	0.131659	Prob > |t|	0.0016
Lower CL Dif	0.041784	Prob > t	0.0008
Confidence	0.95	Prob < t	0.9992



Analysis of Variance

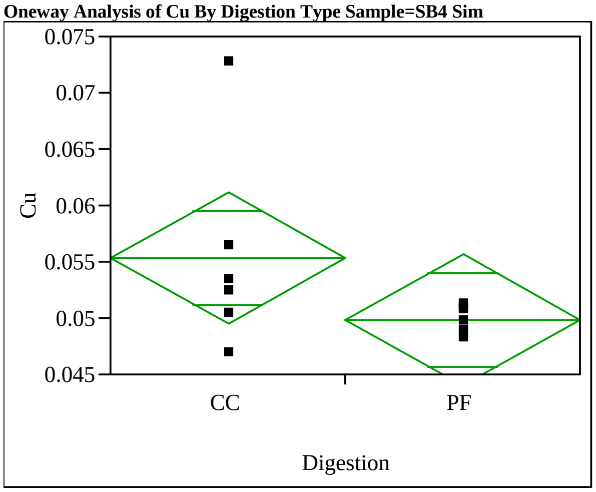
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02256194	0.022562	18.4895	0.0016
Error	10	0.01220257	0.001220		
C. Total	11	0.03476451			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.015150	0.01426	-0.0166	0.04693
PF	6	0.101872	0.01426	0.0701	0.13365

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cu By Digestion Type Sample=SB4 Sim



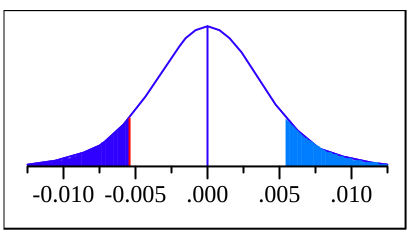
Oneway Anova
Summary of Fit

Rsquare	0.177293
Adj Rsquare	0.095022
Root Mean Square Error	0.00645
Mean of Response	0.0526
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.00547	t Ratio	-1.46799
Std Err Dif	0.00372	DF	10
Upper CL Dif	0.00283	Prob > |t|	0.1728
Lower CL Dif	-0.01376	Prob > t	0.9136
Confidence	0.95	Prob < t	0.0864



Analysis of Variance

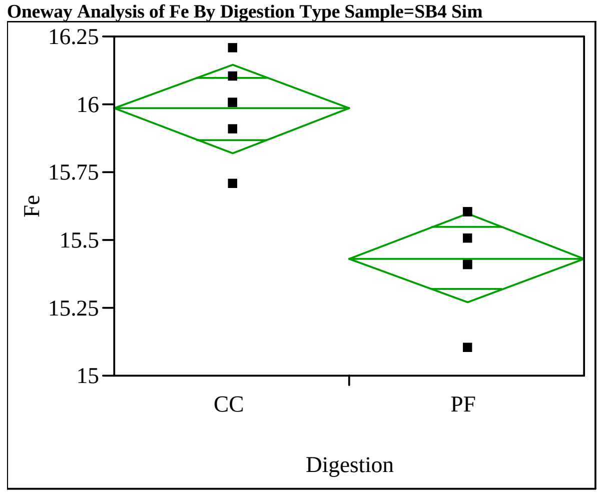
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00008965	0.000090	2.1550	0.1728
Error	10	0.00041603	0.000042		
C. Total	11	0.00050568			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.055333	0.00263	0.04947	0.06120
PF	6	0.049867	0.00263	0.04400	0.05573

Std Error uses a pooled estimate of error variance

Oneway Analysis of Fe By Digestion Type Sample=SB4 Sim



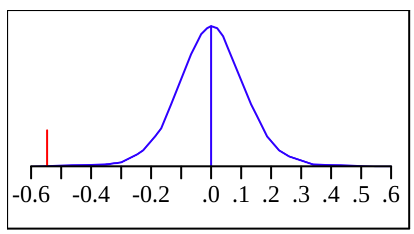
Oneway Anova
Summary of Fit

Rsquare	0.738305
Adj Rsquare	0.712136
Root Mean Square Error	0.179351
Mean of Response	15.70833
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.55000	t Ratio	-5.31154
Std Err Dif	0.10355	DF	10
Upper CL Dif	-0.31928	Prob > |t|	0.0003
Lower CL Dif	-0.78072	Prob > t	0.9998
Confidence	0.95	Prob < t	0.0002



Analysis of Variance

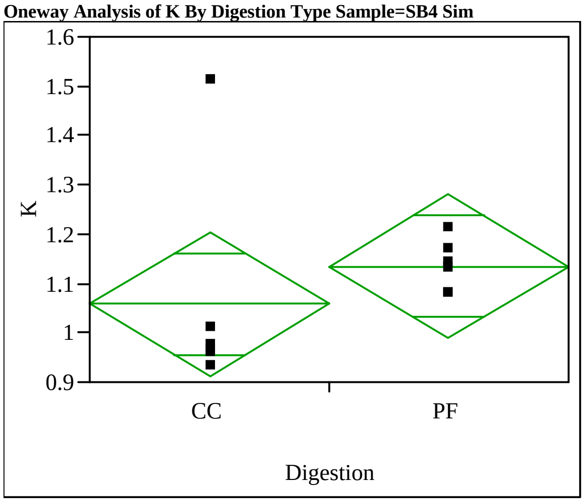
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.9075000	0.907500	28.2124	0.0003
Error	10	0.3216667	0.032167		
C. Total	11	1.2291667			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	15.9833	0.07322	15.820	16.146
PF	6	15.4333	0.07322	15.270	15.596

Std Error uses a pooled estimate of error variance

Oneway Analysis of K By Digestion Type Sample=SB4 Sim



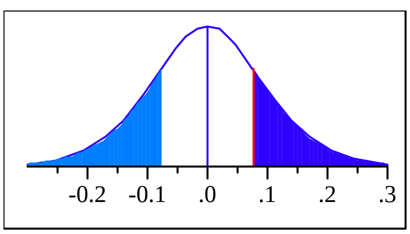
Oneway Anova
Summary of Fit

Rsquare	0.063724
Adj Rsquare	-0.0299
Root Mean Square Error	0.16166
Mean of Response	1.0965
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	0.07700	t Ratio	0.824989
Std Err Dif	0.09333	DF	10
Upper CL Dif	0.28496	Prob > |t|	0.4286
Lower CL Dif	-0.13096	Prob > t	0.2143
Confidence	0.95	Prob < t	0.7857



Analysis of Variance

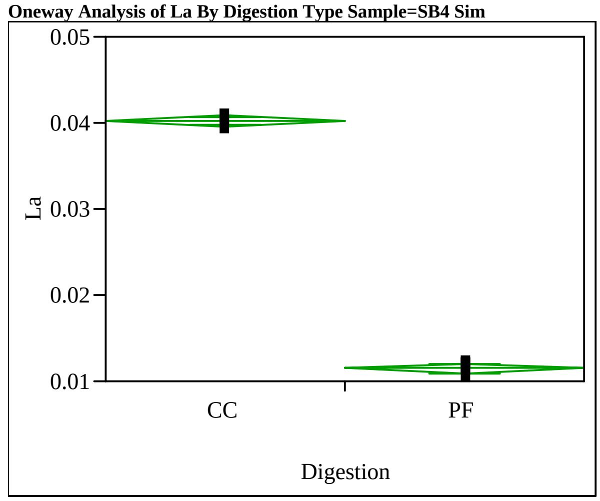
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.01778700	0.017787	0.6806	0.4286
Error	10	0.26134000	0.026134		
C. Total	11	0.27912700			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	1.05800	0.06600	0.91095	1.2051
PF	6	1.13500	0.06600	0.98795	1.2821

Std Error uses a pooled estimate of error variance

Oneway Analysis of La By Digestion Type Sample=SB4 Sim



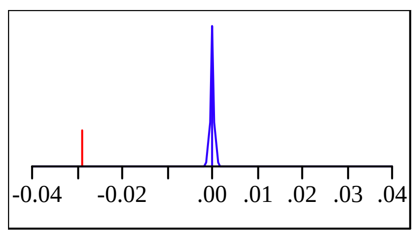
Oneway Anova
Summary of Fit

Rsquare	0.997966
Adj Rsquare	0.997762
Root Mean Square Error	0.000711
Mean of Response	0.025825
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.02875	t Ratio	-70.0387
Std Err Dif	0.00041	DF	10
Upper CL Dif	-0.02784	Prob > |t|	<.0001
Lower CL Dif	-0.02966	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

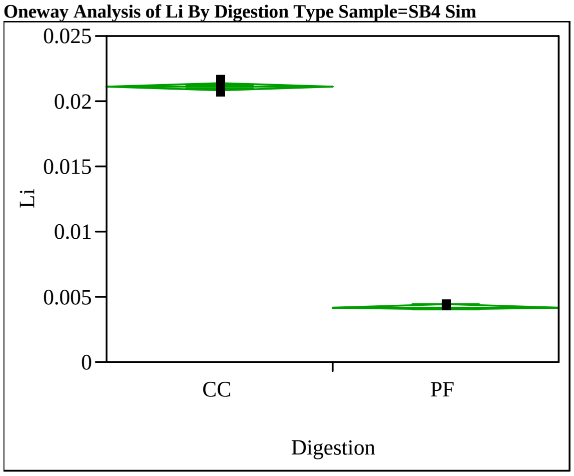
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00247969	0.002480	4905.415	<.0001
Error	10	0.00000506	5.055e-7		
C. Total	11	0.00248474			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.040200	0.00029	0.03955	0.04085
PF	6	0.011450	0.00029	0.01080	0.01210

Std Error uses a pooled estimate of error variance

Oneway Analysis of Li By Digestion Type Sample=SB4 Sim



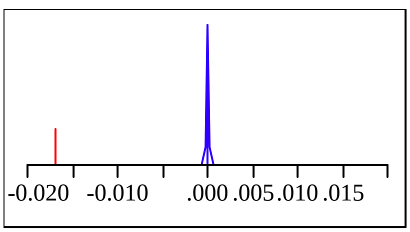
Oneway Anova
Summary of Fit

Rsquare	0.999245
Adj Rsquare	0.999169
Root Mean Square Error	0.000254
Mean of Response	0.012676
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.01688	t Ratio	-115.035
Std Err Dif	0.00015	DF	10
Upper CL Dif	-0.01655	Prob > |t|	0.0000
Lower CL Dif	-0.01721	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

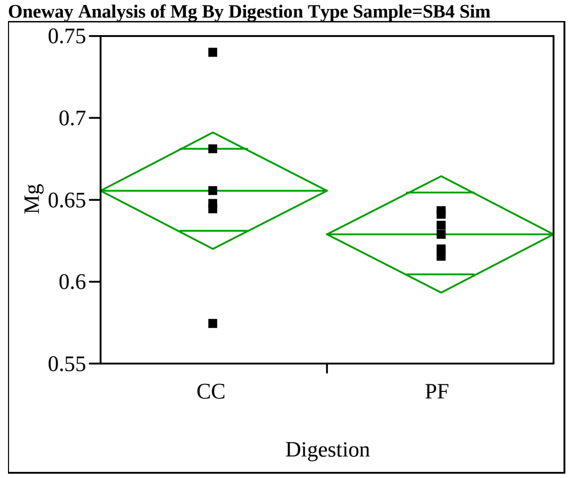
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00085497	0.000855	13233.15	<.0001
Error	10	0.00000065	6.461e-8		
C. Total	11	0.00085562			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.021117	0.00010	0.02089	0.02135
PF	6	0.004235	0.00010	0.00400	0.00447

Std Error uses a pooled estimate of error variance

Oneway Analysis of Mg By Digestion Type Sample=SB4 Sim



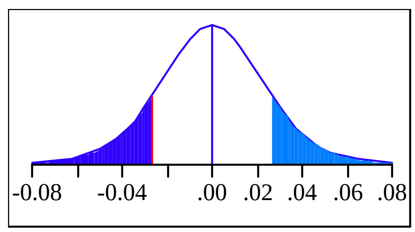
Oneway Anova
Summary of Fit

Rsquare	0.122798
Adj Rsquare	0.035078
Root Mean Square Error	0.039038
Mean of Response	0.642667
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.02667	t Ratio	-1.18317
Std Err Dif	0.02254	DF	10
Upper CL Dif	0.02355	Prob > |t|	0.2641
Lower CL Dif	-0.07689	Prob > t	0.8679
Confidence	0.95	Prob < t	0.1321



Analysis of Variance

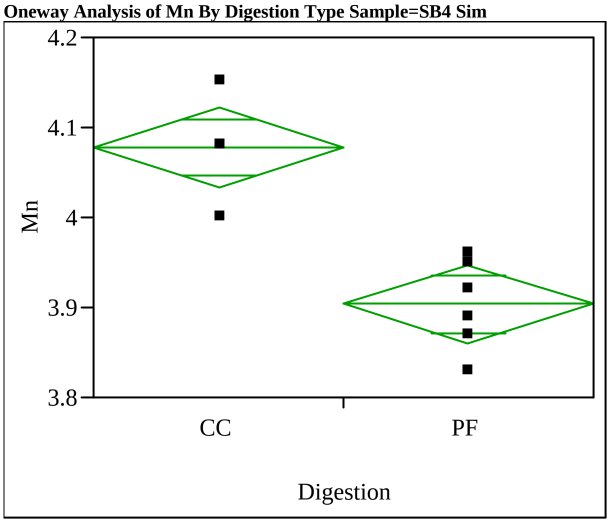
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00213333	0.002133	1.3999	0.2641
Error	10	0.01523933	0.001524		
C. Total	11	0.01737267			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.656000	0.01594	0.62049	0.69151
PF	6	0.629333	0.01594	0.59382	0.66484

Std Error uses a pooled estimate of error variance

Oneway Analysis of Mn By Digestion Type Sample=SB4 Sim



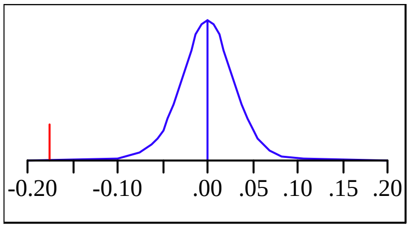
Oneway Anova
Summary of Fit

Rsquare	0.795512
Adj Rsquare	0.775063
Root Mean Square Error	0.048597
Mean of Response	3.990833
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.17500	t Ratio	-6.2372
Std Err Dif	0.02806	DF	10
Upper CL Dif	-0.11248	Prob > |t|	<.0001
Lower CL Dif	-0.23752	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

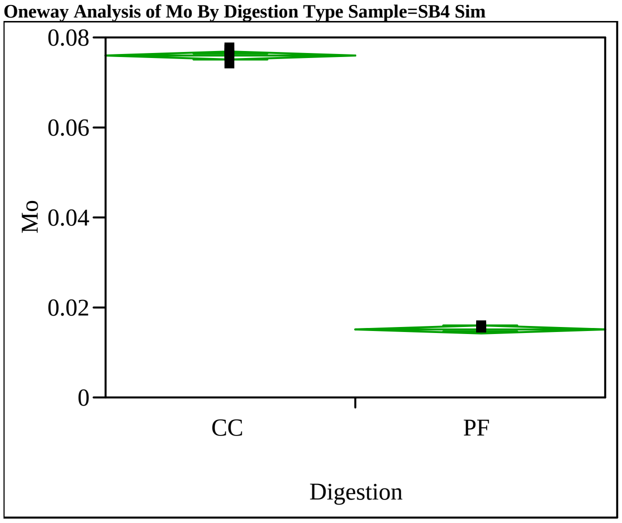
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.09187500	0.091875	38.9026	<.0001
Error	10	0.02361667	0.002362		
C. Total	11	0.11549167			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	4.07833	0.01984	4.0341	4.1225
PF	6	3.90333	0.01984	3.8591	3.9475

Std Error uses a pooled estimate of error variance

Oneway Analysis of Mo By Digestion Type Sample=SB4 Sim



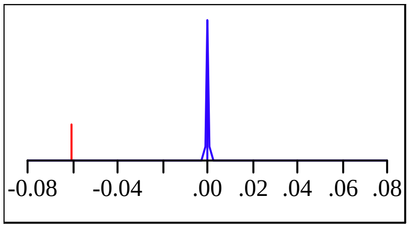
Oneway Anova
Summary of Fit

Rsquare	0.999188
Adj Rsquare	0.999107
Root Mean Square Error	0.000948
Mean of Response	0.045567
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.06073	t Ratio	-110.924
Std Err Dif	0.00055	DF	10
Upper CL Dif	-0.05951	Prob > |t|	0.0000
Lower CL Dif	-0.06195	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

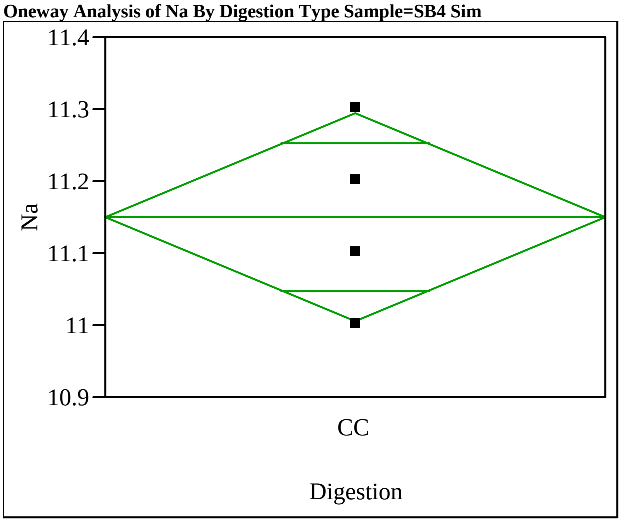
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.01106561	0.011066	12304.24	<.0001
Error	10	0.00000899	8.993e-7		
C. Total	11	0.01107461			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.075933	0.00039	0.07507	0.07680
PF	6	0.015200	0.00039	0.01434	0.01606

Std Error uses a pooled estimate of error variance

Oneway Analysis of Na By Digestion Type Sample=SB4 Sim



Missing Rows
6

Oneway Anova

Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.13784
Mean of Response	11.15
Observations (or Sum Wgts)	6

Analysis of Variance

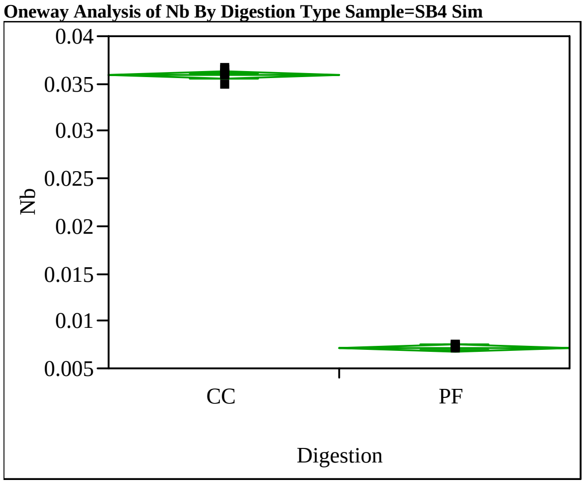
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	5	0.09500000	0.019000		
C. Total	5	0.09500000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	11.1500	0.05627	11.005	11.295

Std Error uses a pooled estimate of error variance

Oneway Analysis of Nb By Digestion Type Sample=SB4 Sim



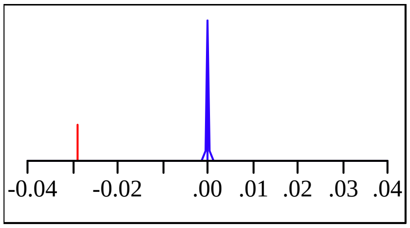
Oneway Anova
Summary of Fit

Rsquare	0.999216
Adj Rsquare	0.999137
Root Mean Square Error	0.00044
Mean of Response	0.021534
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.02870	t Ratio	-112.869
Std Err Dif	0.00025	DF	10
Upper CL Dif	-0.02813	Prob > |t|	0.0000
Lower CL Dif	-0.02926	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

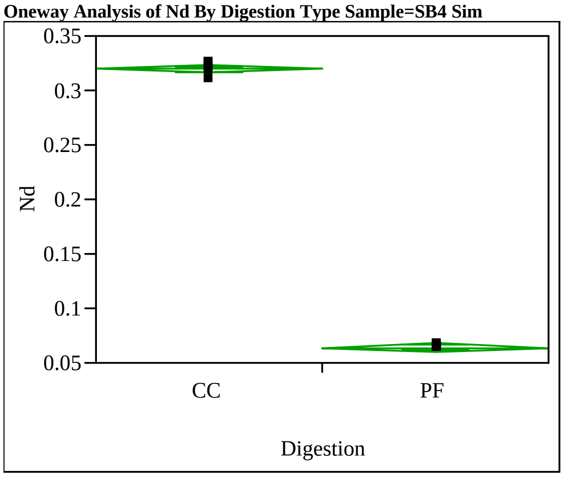
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00247078	0.002471	12739.39	<.0001
Error	10	0.00000194	1.939e-7		
C. Total	11	0.00247272			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.035883	0.00018	0.03548	0.03628
PF	6	0.007185	0.00018	0.00678	0.00759

Std Error uses a pooled estimate of error variance

Oneway Analysis of Nd By Digestion Type Sample=SB4 Sim



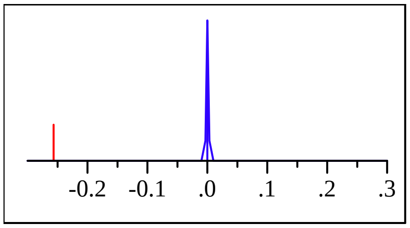
Oneway Anova
Summary of Fit

Rsquare	0.999188
Adj Rsquare	0.999106
Root Mean Square Error	0.003994
Mean of Response	0.191967
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.25573	t Ratio	-110.905
Std Err Dif	0.00231	DF	10
Upper CL Dif	-0.25060	Prob > |t|	0.0000
Lower CL Dif	-0.26087	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

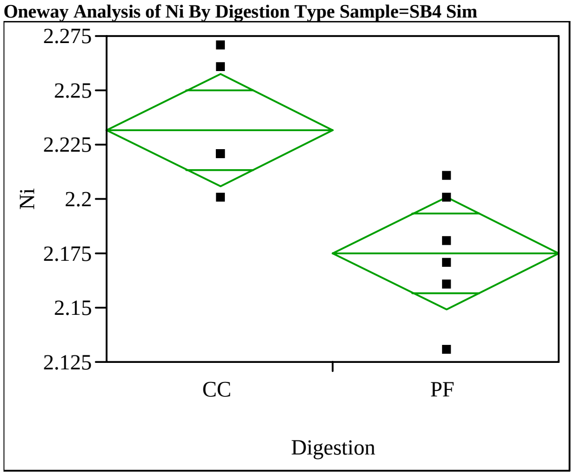
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.19619861	0.196199	12299.83	<.0001
Error	10	0.00015951	0.000016		
C. Total	11	0.19635813			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.319833	0.00163	0.31620	0.32347
PF	6	0.064100	0.00163	0.06047	0.06773

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ni By Digestion Type Sample=SB4 Sim



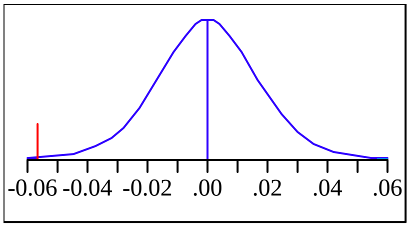
Oneway Anova
Summary of Fit

Rsquare	0.551527
Adj Rsquare	0.506679
Root Mean Square Error	0.027988
Mean of Response	2.203333
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.05667	t Ratio	-3.50683
Std Err Dif	0.01616	DF	10
Upper CL Dif	-0.02066	Prob > |t|	0.0057
Lower CL Dif	-0.09267	Prob > t	0.9972
Confidence	0.95	Prob < t	0.0028



Analysis of Variance

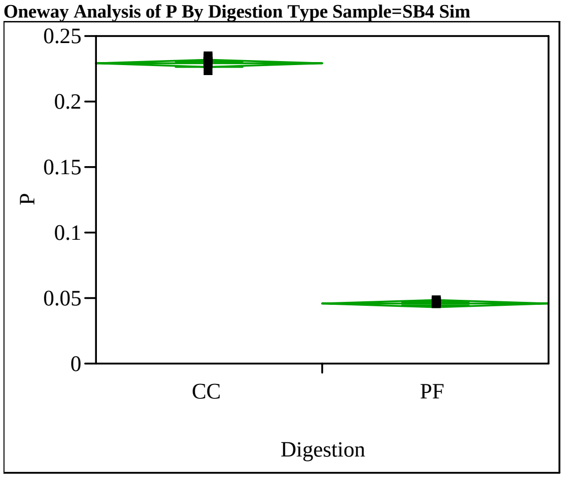
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00963333	0.009633	12.2979	0.0057
Error	10	0.00783333	0.000783		
C. Total	11	0.01746667			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	2.23167	0.01143	2.2062	2.2571
PF	6	2.17500	0.01143	2.1495	2.2005

Std Error uses a pooled estimate of error variance

Oneway Analysis of P By Digestion Type Sample=SB4 Sim



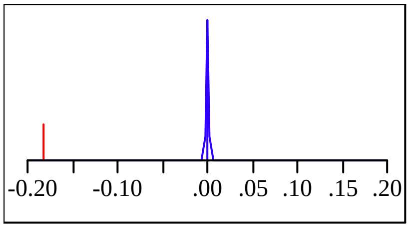
Oneway Anova
Summary of Fit

Rsquare	0.999211
Adj Rsquare	0.999132
Root Mean Square Error	0.002817
Mean of Response	0.137325
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.18302	t Ratio	-112.538
Std Err Dif	0.00163	DF	10
Upper CL Dif	-0.17939	Prob > |t|	0.0000
Lower CL Dif	-0.18664	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

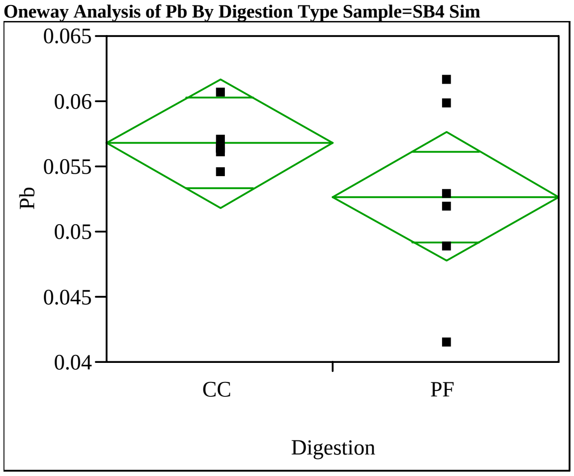
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.10048530	0.100485	12664.88	<.0001
Error	10	0.00007934	7.934e-6		
C. Total	11	0.10056464			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.228833	0.00115	0.22627	0.23140
PF	6	0.045817	0.00115	0.04325	0.04838

Std Error uses a pooled estimate of error variance

Oneway Analysis of Pb By Digestion Type Sample=SB4 Sim



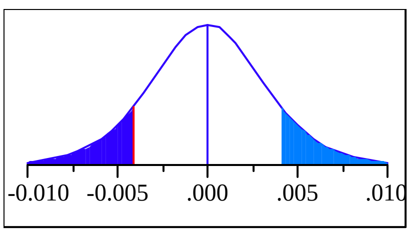
Oneway Anova
Summary of Fit

Rsquare	0.146564
Adj Rsquare	0.061221
Root Mean Square Error	0.005397
Mean of Response	0.054708
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.00408	t Ratio	-1.31048
Std Err Dif	0.00312	DF	10
Upper CL Dif	0.00286	Prob > |t|	0.2193
Lower CL Dif	-0.01103	Prob > t	0.8903
Confidence	0.95	Prob < t	0.1097



Analysis of Variance

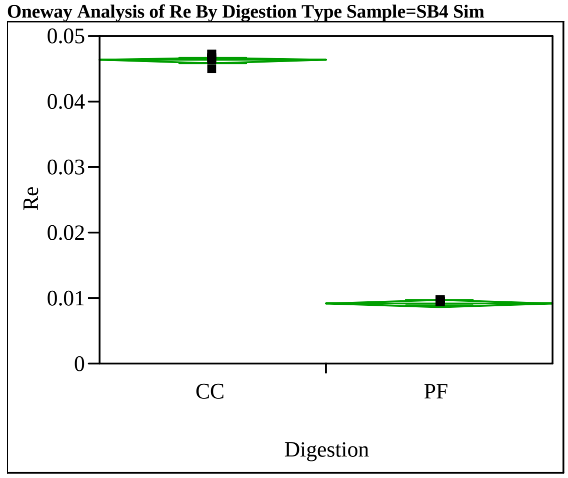
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00005002	0.000050	1.7173	0.2193
Error	10	0.00029127	0.000029		
C. Total	11	0.00034129			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.056750	0.00220	0.05184	0.06166
PF	6	0.052667	0.00220	0.04776	0.05758

Std Error uses a pooled estimate of error variance

Oneway Analysis of Re By Digestion Type Sample=SB4 Sim



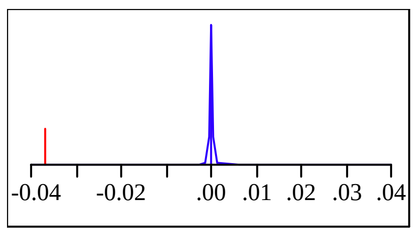
Oneway Anova
Summary of Fit

Rsquare	0.999163
Adj Rsquare	0.999079
Root Mean Square Error	0.000586
Mean of Response	0.02776
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.03698	t Ratio	-109.245
Std Err Dif	0.00034	DF	10
Upper CL Dif	-0.03623	Prob > |t|	0.0000
Lower CL Dif	-0.03773	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

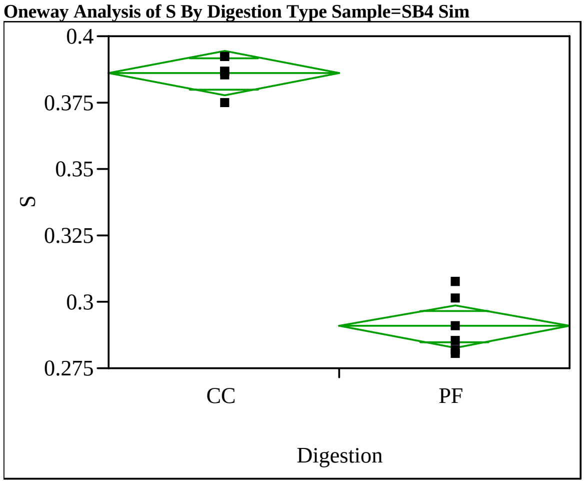
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00410256	0.004103	11934.38	<.0001
Error	10	0.00000344	3.438e-7		
C. Total	11	0.00410600			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.046250	0.00024	0.04572	0.04678
PF	6	0.009270	0.00024	0.00874	0.00980

Std Error uses a pooled estimate of error variance

Oneway Analysis of S By Digestion Type Sample=SB4 Sim



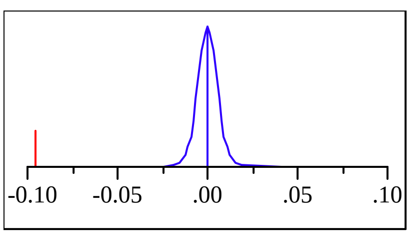
Oneway Anova
Summary of Fit

Rsquare	0.970351
Adj Rsquare	0.967387
Root Mean Square Error	0.009111
Mean of Response	0.33825
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.09517	t Ratio	-18.091
Std Err Dif	0.00526	DF	10
Upper CL Dif	-0.08345	Prob > |t|	<.0001
Lower CL Dif	-0.10689	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

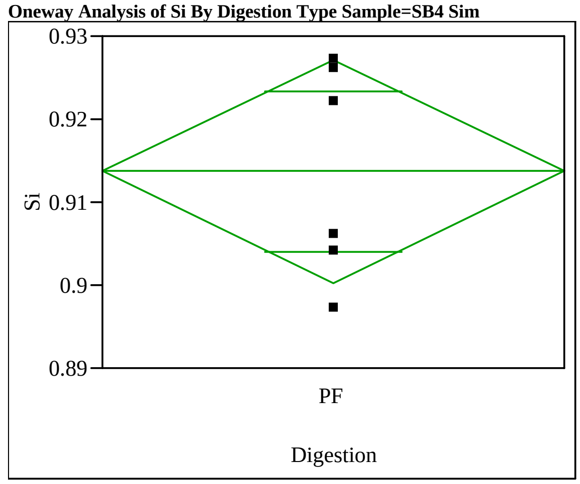
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.02717008	0.027170	327.2847	<.0001
Error	10	0.00083017	0.000083		
C. Total	11	0.02800025			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.385833	0.00372	0.37755	0.39412
PF	6	0.290667	0.00372	0.28238	0.29895

Std Error uses a pooled estimate of error variance

Oneway Analysis of Si By Digestion Type Sample=SB4 Sim



Missing Rows
6

Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	0
Root Mean Square Error	0.012879
Mean of Response	0.913667
Observations (or Sum Wgts)	6

Analysis of Variance

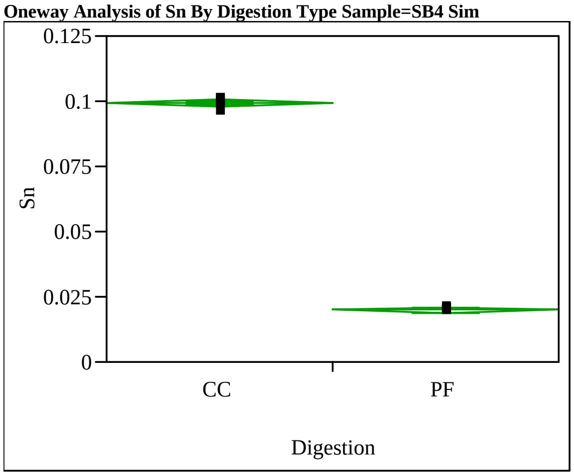
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	5	0.00082933	0.000166		
C. Total	5	0.00082933			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
PF	6	0.913667	0.00526	0.90015	0.92718

Std Error uses a pooled estimate of error variance

Oneway Analysis of Sn By Digestion Type Sample=SB4 Sim



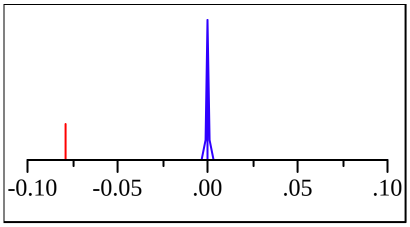
Oneway Anova
Summary of Fit

Rsquare	0.999062
Adj Rsquare	0.998969
Root Mean Square Error	0.001331
Mean of Response	0.059517
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.07930	t Ratio	-103.22
Std Err Dif	0.00077	DF	10
Upper CL Dif	-0.07759	Prob > |t|	<.0001
Lower CL Dif	-0.08101	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001



Analysis of Variance

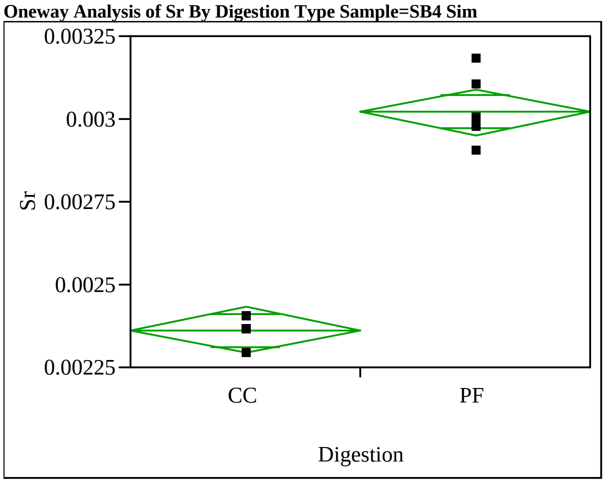
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.01886547	0.018865	10654.44	<.0001
Error	10	0.00001771	1.771e-6		
C. Total	11	0.01888318			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.099167	0.00054	0.09796	0.10038
PF	6	0.019867	0.00054	0.01866	0.02108

Std Error uses a pooled estimate of error variance

Oneway Analysis of Sr By Digestion Type Sample=SB4 Sim



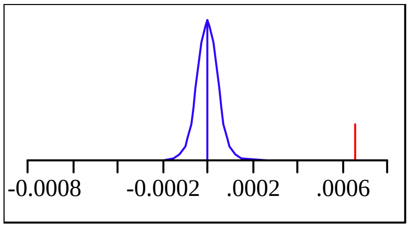
Oneway Anova
Summary of Fit

Rsquare	0.956825
Adj Rsquare	0.952508
Root Mean Square Error	7.679e-5
Mean of Response	0.002692
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	0.000660	t Ratio	14.8868
Std Err Dif	0.000044	DF	10
Upper CL Dif	0.000759	Prob > |t|	<.0001
Lower CL Dif	0.000561	Prob > t	<.0001
Confidence	0.95	Prob < t	1.0000



Analysis of Variance

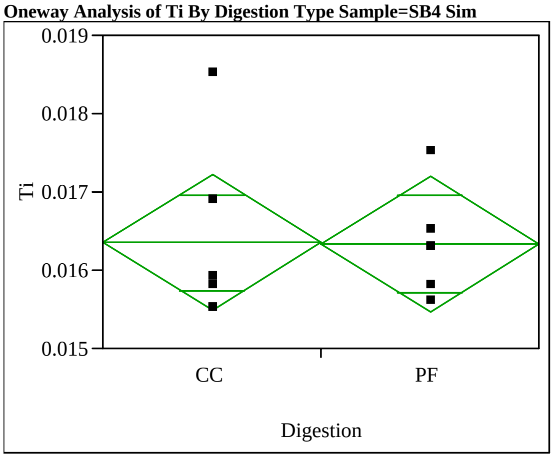
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	1.3068e-6	1.3068e-6	221.6167	<.0001
Error	10	5.89667e-8	5.8967e-9		
C. Total	11	1.36577e-6			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.002362	3.13e-5	0.00229	0.00243
PF	6	0.003022	3.13e-5	0.00295	0.00309

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ti By Digestion Type Sample=SB4 Sim



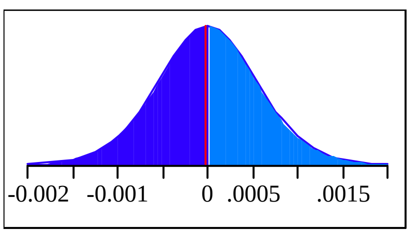
Oneway Anova
Summary of Fit

Rsquare	9.168e-5
Adj Rsquare	-0.0999
Root Mean Square Error	0.000953
Mean of Response	0.016342
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-1.67e-5	t Ratio	-0.03028
Std Err Dif	0.00055	DF	10
Upper CL Dif	0.00121	Prob > |t|	0.9764
Lower CL Dif	-0.00124	Prob > t	0.5118
Confidence	0.95	Prob < t	0.4882



Analysis of Variance

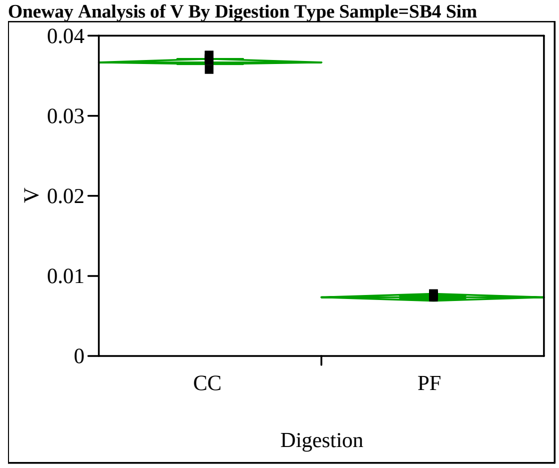
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	8.3333e-10	8.333e-10	0.0009	0.9764
Error	10	9.08833e-6	9.0883e-7		
C. Total	11	9.08917e-6			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.016350	0.00039	0.01548	0.01722
PF	6	0.016333	0.00039	0.01547	0.01720

Std Error uses a pooled estimate of error variance

Oneway Analysis of V By Digestion Type Sample=SB4 Sim



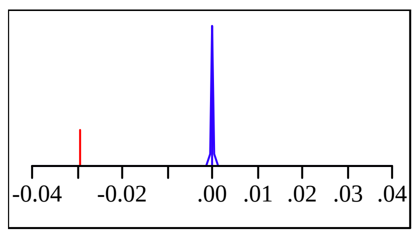
Oneway Anova
Summary of Fit

Rsquare	0.999166
Adj Rsquare	0.999082
Root Mean Square Error	0.000465
Mean of Response	0.022067
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.02940	t Ratio	-109.434
Std Err Dif	0.00027	DF	10
Upper CL Dif	-0.02880	Prob > |t|	0.0000
Lower CL Dif	-0.03000	Prob > t	1.0000
Confidence	0.95	Prob < t	0.0000



Analysis of Variance

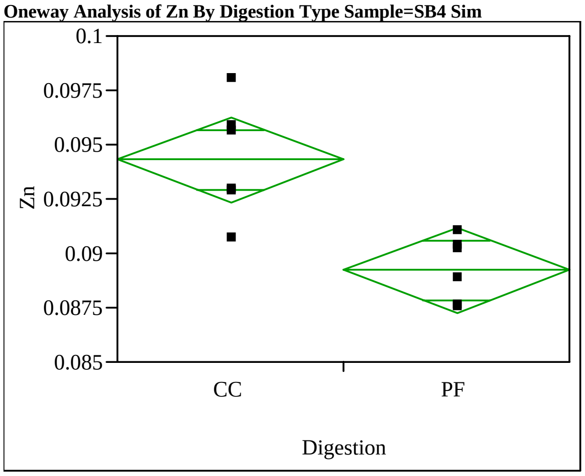
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00259308	0.002593	11975.80	<.0001
Error	10	0.00000217	2.165e-7		
C. Total	11	0.00259525			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.036767	0.00019	0.03634	0.03719
PF	6	0.007367	0.00019	0.00694	0.00779

Std Error uses a pooled estimate of error variance

Oneway Analysis of Zn By Digestion Type Sample=SB4 Sim



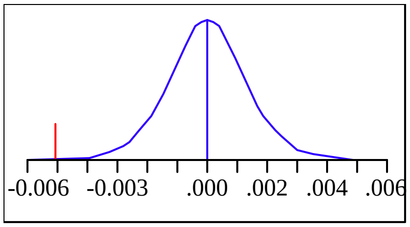
Oneway Anova
Summary of Fit

Rsquare	0.626804
Adj Rsquare	0.589485
Root Mean Square Error	0.002141
Mean of Response	0.091767
Observations (or Sum Wgts)	12

t Test
PF-CC

Assuming equal variances

Difference	-0.00507	t Ratio	-4.09824
Std Err Dif	0.00124	DF	10
Upper CL Dif	-0.00231	Prob > |t|	0.0022
Lower CL Dif	-0.00782	Prob > t	0.9989
Confidence	0.95	Prob < t	0.0011



Analysis of Variance

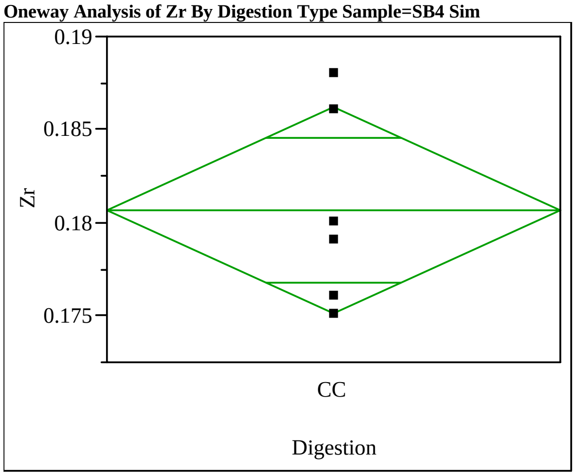
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	0.00007701	0.000077	16.7956	0.0022
Error	10	0.00004585	4.585e-6		
C. Total	11	0.00012287			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.094300	0.00087	0.09235	0.09625
PF	6	0.089233	0.00087	0.08729	0.09118

Std Error uses a pooled estimate of error variance

Oneway Analysis of Zr By Digestion Type Sample=SB4 Sim



Missing Rows
6
Oneway Anova
Summary of Fit

Rsquare	1.11e-16
Adj Rsquare	1.11e-16
Root Mean Square Error	0.005279
Mean of Response	0.180667
Observations (or Sum Wgts)	6

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	0	0.00000000	.	.	.
Error	5	0.00013933	0.000028		
C. Total	5	0.00013933			

Means for Oneway Anova

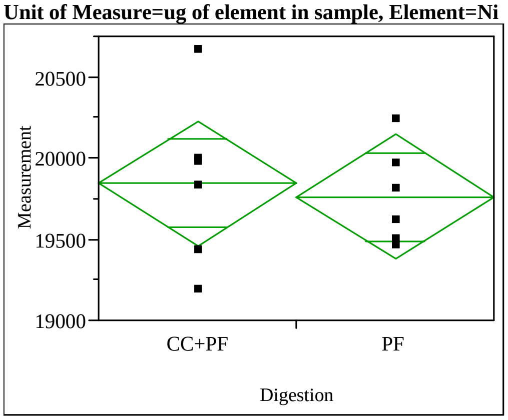
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC	6	0.180667	0.00216	0.17513	0.18621

Std Error uses a pooled estimate of error variance

APPENDIX C

JMP One-Way Analysis Plots of SB4 spiked with *Boehmite* Digested by DWPF CC + PF and PF

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Ni



Oneway Anova
Summary of Fit

Rsquare	0.011927
Adj Rsquare	-0.08688
Root Mean Square Error	421.3985
Mean of Response	19802.8
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	-84.53	t Ratio	-0.34743
Std Err Dif	243.29	DF	10
Upper CL Dif	457.57	Prob > |t|	0.7355
Lower CL Dif	-626.62	Prob > t	0.6323
Confidence	0.95	Prob < t	0.3677

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	21435.1	21435	0.1207	0.7355
Error	10	1775766.7	177577		
C. Total	11	1797201.8			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	19845.1	172.04	19462	20228
PF	6	19760.5	172.04	19377	20144

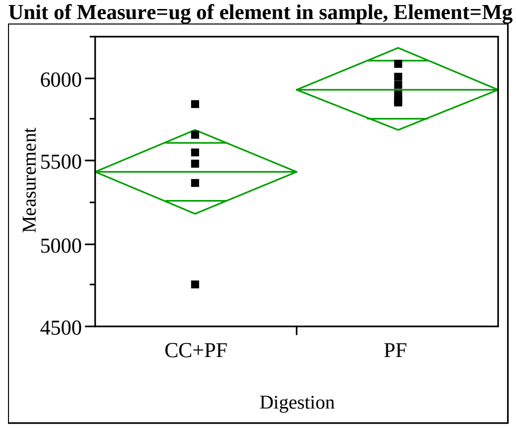
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	-84.53	t Ratio	-0.34743
Std Err Dif	243.29	DF	8.094249
Upper CL Dif	475.37	Prob > |t|	0.7371
Lower CL Dif	-644.43	Prob > t	0.6314
Confidence	0.95	Prob < t	0.3686

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Mg



Oneway Anova
Summary of Fit

Rsquare	0.500855
Adj Rsquare	0.450941
Root Mean Square Error	273.1399
Mean of Response	5682.998
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	499.537	t Ratio	3.167691
Std Err Dif	157.697	DF	10
Upper CL Dif	850.908	Prob > |t|	0.0100
Lower CL Dif	148.165	Prob > t	0.0050
Confidence	0.95	Prob < t	0.9950

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	748610.6	748611	10.0343	0.0100
Error	10	746054.0	74605		
C. Total	11	1494664.7			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	5433.23	111.51	5184.8	5681.7
PF	6	5932.77	111.51	5684.3	6181.2

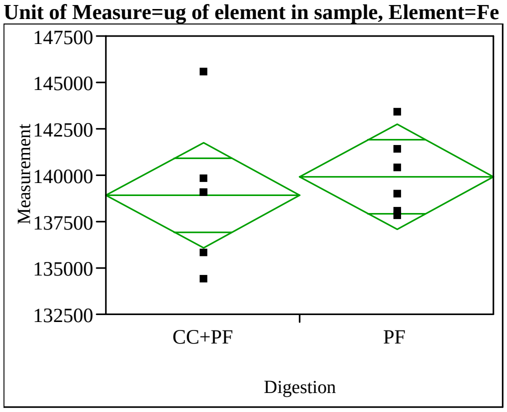
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	499.537	t Ratio	3.167691
Std Err Dif	157.697	DF	5.582569
Upper CL Dif	892.511	Prob > |t|	0.0214
Lower CL Dif	106.562	Prob > t	0.0107
Confidence	0.95	Prob < t	0.9893

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Fe



Oneway Anova
Summary of Fit

Rsquare	0.033043
Adj Rsquare	-0.06365
Root Mean Square Error	3125.612
Mean of Response	139417.6
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	1054.9	t Ratio	0.584572
Std Err Dif	1804.6	DF	10
Upper CL Dif	5075.7	Prob > |t|	0.5718
Lower CL Dif	-2965.9	Prob > t	0.2859
Confidence	0.95	Prob < t	0.7141

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	3338463	3338463	0.3417	0.5718
Error	10	97694502	9769450		
C. Total	11	101032965			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	138890	1276.0	136047	141733
PF	6	139945	1276.0	137102	142788

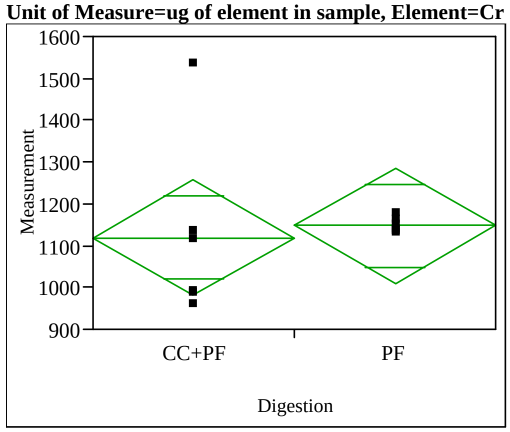
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	1054.9	t Ratio	0.584572
Std Err Dif	1804.6	DF	7.802025
Upper CL Dif	5234.7	Prob > |t|	0.5753
Lower CL Dif	-3124.9	Prob > t	0.2877
Confidence	0.95	Prob < t	0.7123

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Cr



Oneway Anova
Summary of Fit

Rsquare	0.009875
Adj Rsquare	-0.08914
Root Mean Square Error	152.9819
Mean of Response	1133.153
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	27.89	t Ratio	0.315808
Std Err Dif	88.32	DF	10
Upper CL Dif	224.69	Prob > |t|	0.7586
Lower CL Dif	-168.90	Prob > t	0.3793
Confidence	0.95	Prob < t	0.6207

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	2334.14	2334.1	0.0997	0.7586
Error	10	234034.71	23403.5		
C. Total	11	236368.85			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower
CC+PF	6	1119.21	62.455	9
PF	6	1147.10	62.455	10

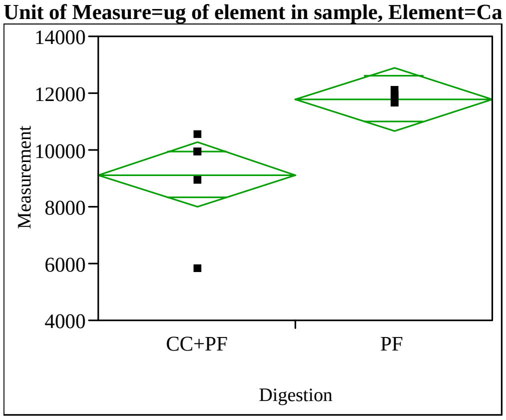
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	27.89	t Ratio	0.315808
Std Err Dif	88.32	DF	5.066365
Upper CL Dif	254.05	Prob > |t|	0.7647
Lower CL Dif	-198.26	Prob > t	0.3824
Confidence	0.95	Prob < t	0.6176

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Ca



Oneway Anova
Summary of Fit

Rsquare	0.581804
Adj Rsquare	0.539984
Root Mean Square Error	1232.175
Mean of Response	10465.36
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	2653.45	t Ratio	3.729909
Std Err Dif	711.40	DF	10
Upper CL Dif	4238.54	Prob > |t|	0.0039
Lower CL Dif	1068.35	Prob > t	0.0020
Confidence	0.95	Prob < t	0.9980

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	21122311	21122311	13.9122	0.0039
Error	10	15182558	1518255.8		
C. Total	11	36304869			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	9138.6	503.03	8018	10259
PF	6	11792.1	503.03	10671	12913

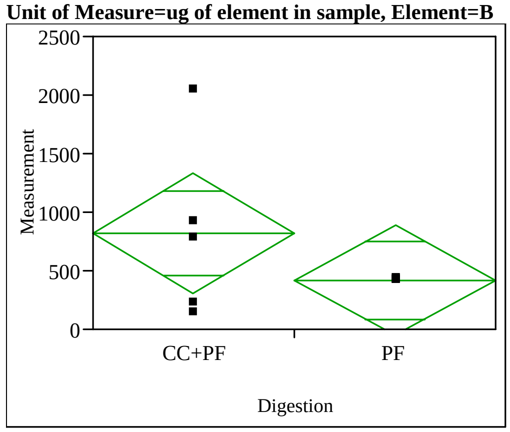
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	2653.45	t Ratio	3.729909
Std Err Dif	711.40	DF	5.108314
Upper CL Dif	4470.55	Prob > |t|	0.0131
Lower CL Dif	836.34	Prob > t	0.0065
Confidence	0.95	Prob < t	0.9935

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=B



Oneway Anova
Summary of Fit

Rsquare	0.157705
Adj Rsquare	0.064116
Root Mean Square Error	509.0479
Mean of Response	602.4793
Observations (or Sum Wgts)	11

t Test
PF-CC+PF

Assuming equal variances

Difference	-400.1	t Ratio	-1.29811
Std Err Dif	308.2	DF	9
Upper CL Dif	297.2	Prob > |t|	0.2265
Lower CL Dif	-1097.4	Prob > t	0.8867
Confidence	0.95	Prob < t	0.1133

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	436656.9	436657	1.6851	0.2265
Error	9	2332168.1	259130		
C. Total	10	2768825.0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	5	820.734	227.65	305.7	1335.7
PF	6	420.600	207.82	-49.5	890.7

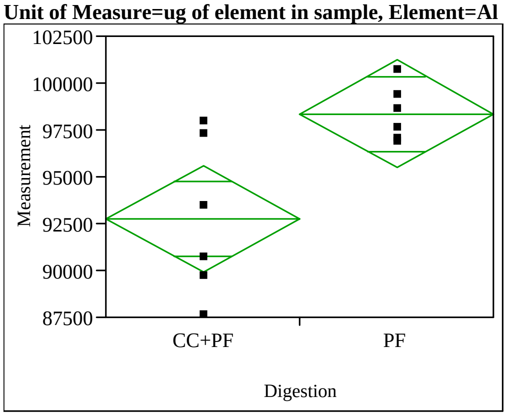
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	-400.1	t Ratio	-1.17178
Std Err Dif	341.5	DF	4.000476
Upper CL Dif	547.9	Prob > |t|	0.3063
Lower CL Dif	-1348.2	Prob > t	0.8468
Confidence	0.95	Prob < t	0.1532

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Al



Oneway Anova
Summary of Fit

Rsquare	0.486512
Adj Rsquare	0.435164
Root Mean Square Error	3151.754
Mean of Response	95543.33
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	5601.11	t Ratio	3.078095
Std Err Dif	1819.67	DF	10
Upper CL Dif	9655.57	Prob > |t|	0.0117
Lower CL Dif	1546.64	Prob > t	0.0058
Confidence	0.95	Prob < t	0.9942

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	94117132	94117132	9.4747	0.0117
Error	10	99335538	9933553.8		
C. Total	11	193452670			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	92742.8	1286.7	89876	95610
PF	6	98343.9	1286.7	95477	101211

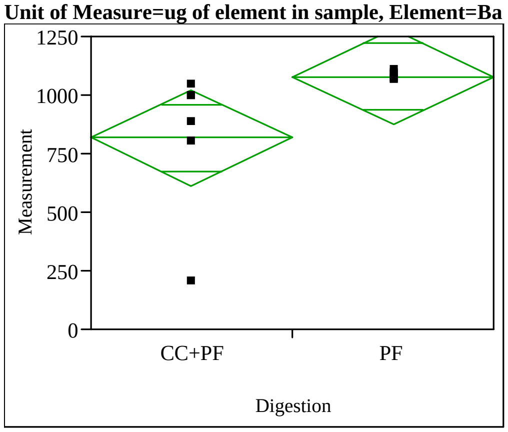
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	5601.1	t Ratio	3.078095
Std Err Dif	1819.7	DF	6.265084
Upper CL Dif	10008.4	Prob > |t|	0.0205
Lower CL Dif	1193.8	Prob > t	0.0103
Confidence	0.95	Prob < t	0.9897

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Ba



Oneway Anova
Summary of Fit

Rsquare	0.291622
Adj Rsquare	0.220785
Root Mean Square Error	222.8193
Mean of Response	947.7578
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	261.02	t Ratio	2.028981
Std Err Dif	128.64	DF	10
Upper CL Dif	547.66	Prob > |t|	0.0699
Lower CL Dif	-25.62	Prob > t	0.0350
Confidence	0.95	Prob < t	0.9650

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	204390.90	204391	4.1168	0.0699
Error	10	496484.53	49648		
C. Total	11	700875.43			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	817.25	90.966	614.56	1019.9
PF	6	1078.27	90.966	875.58	1281.0

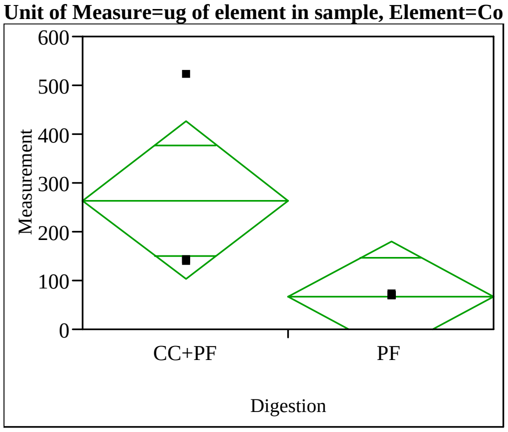
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	261.02	t Ratio	2.028981
Std Err Dif	128.64	DF	5.027493
Upper CL Dif	591.17	Prob > |t|	0.0979
Lower CL Dif	-69.13	Prob > t	0.0490
Confidence	0.95	Prob < t	0.9510

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Co



Oneway Anova
Summary of Fit

Rsquare	0.447646
Adj Rsquare	0.368738
Root Mean Square Error	117.5397
Mean of Response	132.7867
Observations (or Sum Wgts)	9

t Test
PF-CC+PF

Assuming equal variances

Difference	-197.96	t Ratio	-2.38181
Std Err Dif	83.11	DF	7
Upper CL Dif	-1.43	Prob > |t|	0.0488
Lower CL Dif	-394.49	Prob > t	0.9756
Confidence	0.95	Prob < t	0.0244

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	78376.32	78376.3	5.6730	0.0488
Error	7	96709.13	13815.6		
C. Total	8	175085.45			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	3	264.760	67.862	104.3	425.23
PF	6	66.800	47.985	-46.7	180.27

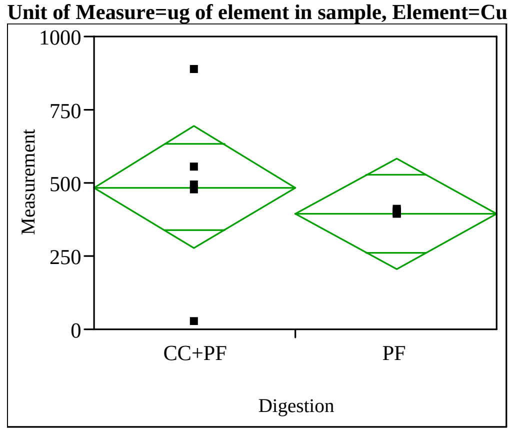
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	-197.96	t Ratio	-1.5593
Std Err Dif	126.95	DF	2.000044
Upper CL Dif	348.27	Prob > |t|	0.2593
Lower CL Dif	-744.19	Prob > t	0.8704
Confidence	0.95	Prob < t	0.1296

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Cu



Oneway Anova
Summary of Fit

Rsquare	0.052882
Adj Rsquare	-0.05235
Root Mean Square Error	205.8122
Mean of Response	435.34
Observations (or Sum Wgts)	11

t Test
PF-CC+PF

Assuming equal variances

Difference	-88.34	t Ratio	-0.70888
Std Err Dif	124.63	DF	9
Upper CL Dif	193.58	Prob > |t|	0.4963
Lower CL Dif	-370.27	Prob > t	0.7518
Confidence	0.95	Prob < t	0.2482

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	21285.76	21285.8	0.5025	0.4963
Error	9	381227.78	42358.6		
C. Total	10	402513.55			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	5	483.528	92.042	275.31	691.74
PF	6	395.183	84.022	205.11	585.26

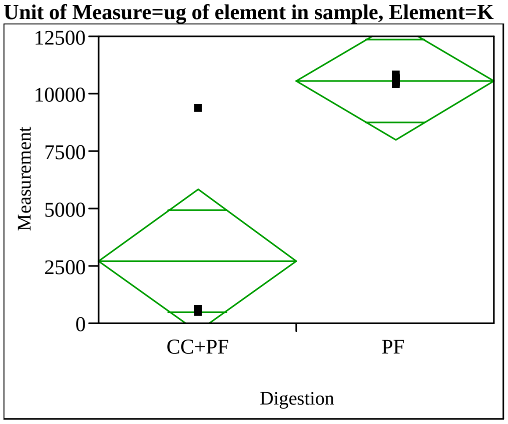
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	-88.34	t Ratio	-0.63994
Std Err Dif	138.05	DF	4.002573
Upper CL Dif	294.85	Prob > |t|	0.5570
Lower CL Dif	-471.54	Prob > t	0.7215
Confidence	0.95	Prob < t	0.2785

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=K



Oneway Anova
Summary of Fit

Rsquare	0.716224
Adj Rsquare	0.680752
Root Mean Square Error	2696.596
Mean of Response	7404.734
Observations (or Sum Wgts)	10

t Test
PF-CC+PF

Assuming equal variances

Difference	7821.5	t Ratio	4.493472
Std Err Dif	1740.6	DF	8
Upper CL Dif	11835.5	Prob > |t|	0.0020
Lower CL Dif	3807.6	Prob > t	0.0010
Confidence	0.95	Prob < t	0.9990

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	146823571	146823571	20.1913	0.0020
Error	8	58173026	7271628.3		
C. Total	9	204996597			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	4	2711.8	1348.3	-397	5821
PF	6	10533.4	1100.9	7995	13072

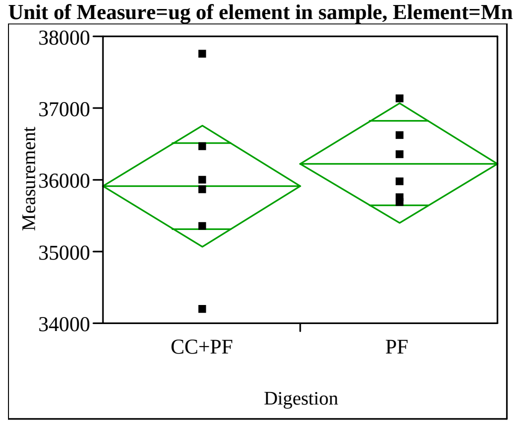
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	7821.5	t Ratio	3.554783
Std Err Dif	2200.3	DF	3.00537
Upper CL Dif	14816.8	Prob > |t|	0.0379
Lower CL Dif	826.3	Prob > t	0.0189
Confidence	0.95	Prob < t	0.9811

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Mn



Oneway Anova
Summary of Fit

Rsquare	0.033856
Adj Rsquare	-0.06276
Root Mean Square Error	925.4271
Mean of Response	36074.61
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	316.3	t Ratio	0.591966
Std Err Dif	534.3	DF	10
Upper CL Dif	1506.8	Prob > |t|	0.5670
Lower CL Dif	-874.2	Prob > t	0.2835
Confidence	0.95	Prob < t	0.7165

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	300108.6	300109	0.3504	0.5670
Error	10	8564153.3	856415		
C. Total	11	8864261.9			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	35916.5	377.80	35075	36758
PF	6	36232.8	377.80	35391	37075

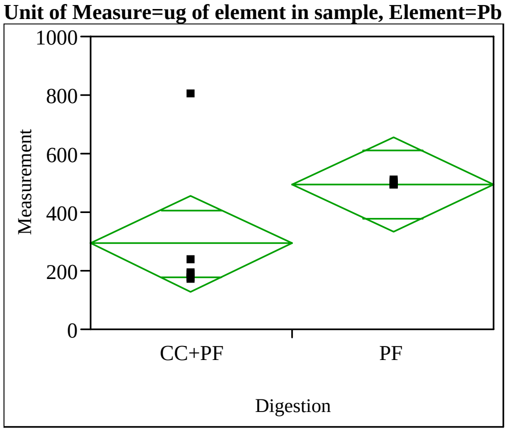
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	316.3	t Ratio	0.591966
Std Err Dif	534.3	DF	7.086401
Upper CL Dif	1576.6	Prob > |t|	0.5723
Lower CL Dif	-944.0	Prob > t	0.2861
Confidence	0.95	Prob < t	0.7139

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Pb



Oneway Anova
Summary of Fit

Rsquare	0.281053
Adj Rsquare	0.209158
Root Mean Square Error	177.6512
Mean of Response	393.0702
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	202.79	t Ratio	1.977177
Std Err Dif	102.57	DF	10
Upper CL Dif	431.33	Prob > |t|	0.0762
Lower CL Dif	-25.74	Prob > t	0.0381
Confidence	0.95	Prob < t	0.9619

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	123375.00	123375	3.9092	0.0762
Error	10	315599.31	31560		
C. Total	11	438974.32			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	291.674	72.526	130.08	453.27
PF	6	494.467	72.526	332.87	656.06

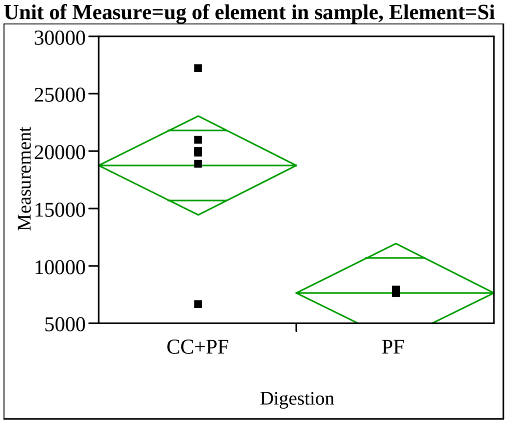
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	202.79	t Ratio	1.977177
Std Err Dif	102.57	DF	5.009035
Upper CL Dif	466.31	Prob > |t|	0.1049
Lower CL Dif	-60.72	Prob > t	0.0524
Confidence	0.95	Prob < t	0.9476

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Si



Oneway Anova
Summary of Fit

Rsquare	0.622461
Adj Rsquare	0.584707
Root Mean Square Error	4740.289
Mean of Response	13218.9
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	-11113	t Ratio	-4.06046
Std Err Dif	2737	DF	10
Upper CL Dif	-5015	Prob > |t|	0.0023
Lower CL Dif	-17211	Prob > t	0.9989
Confidence	0.95	Prob < t	0.0011

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	370476415	370476415	16.4874	0.0023
Error	10	224703395	22470340		
C. Total	11	595179810			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	18775.3	1935.2	14463	23087
PF	6	7662.6	1935.2	3351	11974

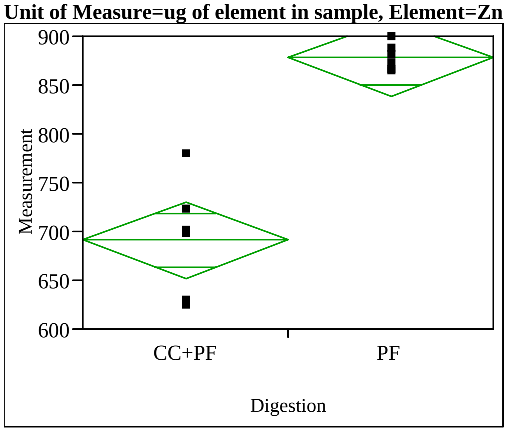
Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	-11113	t Ratio	-4.06046
Std Err Dif	2737	DF	5.003058
Upper CL Dif	-4079	Prob > |t|	0.0097
Lower CL Dif	-18147	Prob > t	0.9951
Confidence	0.95	Prob < t	0.0049

Oneway Analysis of Measurement By Digestion
Unit of Measure=ug of element in sample, Element=Zn



Oneway Anova
Summary of Fit

Rsquare	0.851777
Adj Rsquare	0.836954
Root Mean Square Error	42.67093
Mean of Response	784.3883
Observations (or Sum Wgts)	12

t Test
PF-CC+PF

Assuming equal variances

Difference	186.757	t Ratio	7.580619
Std Err Dif	24.636	DF	10
Upper CL Dif	241.649	Prob > |t|	<.0001
Lower CL Dif	131.864	Prob > t	<.0001
Confidence	0.95	Prob < t	1.0000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Digestion	1	104634.16	104634	57.4658	<.0001
Error	10	18208.08	1821		
C. Total	11	122842.24			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
CC+PF	6	691.010	17.420	652.20	729.82
PF	6	877.767	17.420	838.95	916.58

Std Error uses a pooled estimate of error variance

t Test
PF-CC+PF

Assuming unequal variances

Difference	186.757	t Ratio	7.580619
Std Err Dif	24.636	DF	5.518763
Upper CL Dif	248.336	Prob > |t|	0.0004
Lower CL Dif	125.177	Prob > t	0.0002
Confidence	0.95	Prob < t	0.9998

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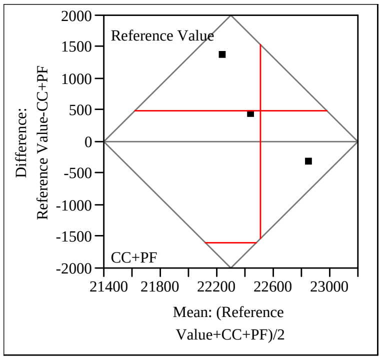
APPENDIX D

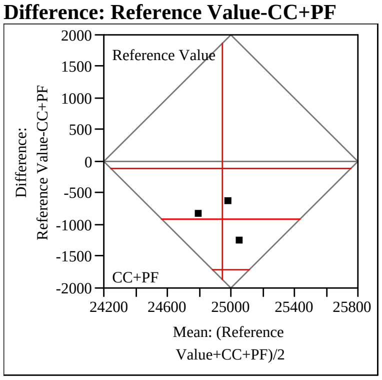
JMP One-Way Analysis Plots of ARG digested with *Boehmite* spiked SB4 simulant by DWPF CC + PF and PF

Matched Pairs Element=Al
Difference: Reference Value-CC+PF



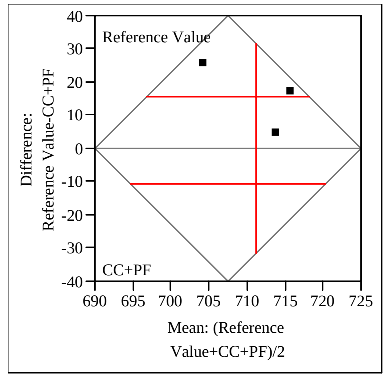
Reference Value	22755	t-Ratio	1.005361
CC+PF	22268.3	DF	2
Mean Difference	486.707	Prob > |t|	0.4206
Std Error	484.111	Prob > t	0.2103
Upper95%	2569.67	Prob < t	0.7897
Lower95%	-1596.3		
N	3		
Correlation	-0.7783		

Matched Pairs Element=B
Difference: Reference Value-CC+PF



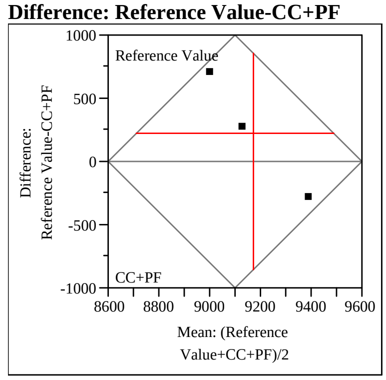
Reference Value	24484.4	t-Ratio	-4.95177
CC+PF	25404.6	DF	2
Mean Difference	-920.25	Prob > |t|	0.0384
Std Error	185.843	Prob > t	0.9808
Upper95%	-120.63	Prob < t	0.0192
Lower95%	-1719.9		
N	3		
Correlation	-0.2379		

Matched Pairs Element=Ba
Difference: Reference Value-CC+PF



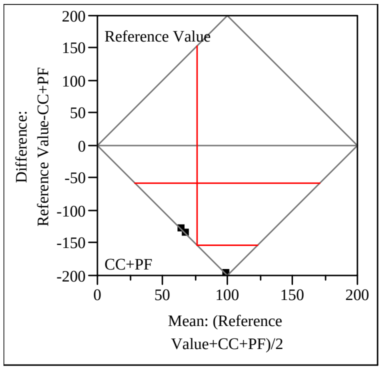
Reference Value	719.058	t-Ratio	2.547199
CC+PF	703.467	DF	2
Mean Difference	15.5913	Prob > |t|	0.1257
Std Error	6.12097	Prob > t	0.0629
Upper95%	41.9277	Prob < t	0.9371
Lower95%	-10.745		
N	3		
Correlation	0.20519		

Matched Pairs Element=Ca
Difference: Reference Value-CC+PF



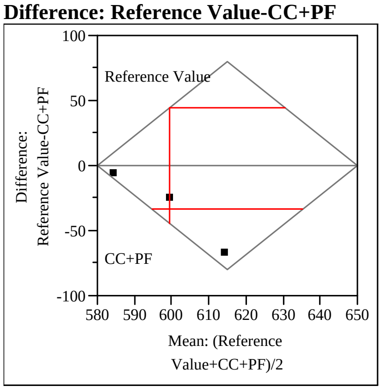
Reference Value	9284.04	t-Ratio	0.785388
CC+PF	9058.4	DF	2
Mean Difference	225.64	Prob > |t|	0.5145
Std Error	287.298	Prob > t	0.2572
Upper95%	1461.78	Prob < t	0.7428
Lower95%	-1010.5		
N	3		
Correlation	-0.8552		

Matched Pairs Element=Co
Difference: Reference Value-CC+PF



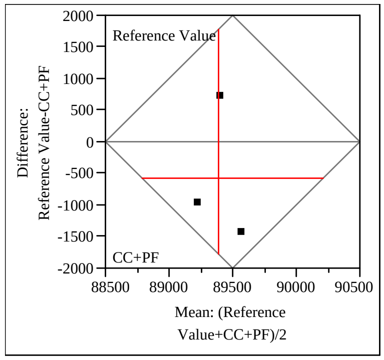
Reference Value	0	t-Ratio	-6.95197
CC+PF	153.997	DF	2
Mean Difference	-154	Prob > |t|	0.0201
Std Error	22.1515	Prob > t	0.9900
Upper95%	-58.686	Prob < t	0.0100
Lower95%	-249.31		
N	3		
Correlation	0		

Matched Pairs Element=Cr
Difference: Reference Value-CC+PF



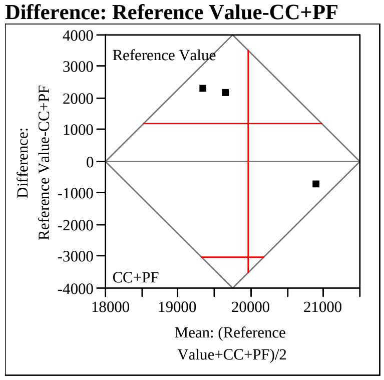
Reference Value	582.528	t-Ratio	-1.86183
CC+PF	616.21	DF	2
Mean Difference	-33.682	Prob > |t|	0.2037
Std Error	18.0908	Prob > t	0.8982
Upper95%	44.1566	Prob < t	0.1018
Lower95%	-111.52		
N	3		
Correlation	-0.2116		

Matched Pairs Element=Fe
Difference: Reference Value-CC+PF



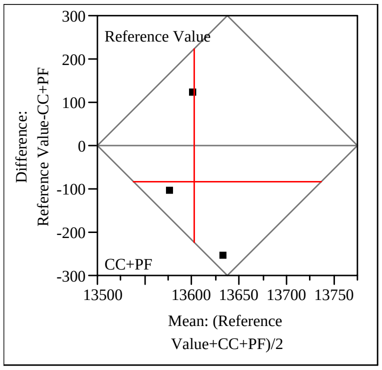
Reference Value	89108.6	t-Ratio	-0.86912
CC+PF	89679.5	DF	2
Mean Difference	-570.94	Prob > |t|	0.4764
Std Error	656.917	Prob > t	0.7618
Upper95%	2255.55	Prob < t	0.2382
Lower95%	-3397.4		
N	3		
Correlation	-0.8307		

Matched Pairs Element=K
Difference: Reference Value-CC+PF



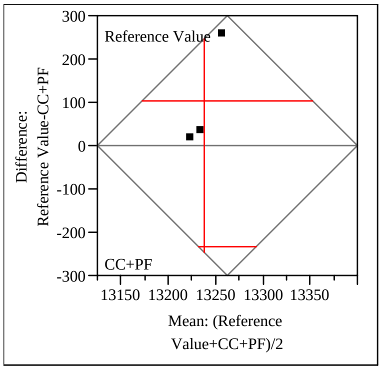
Reference Value	20570.5	t-Ratio	1.230693
CC+PF	19351.3	DF	2
Mean Difference	1219.21	Prob > |t|	0.3435
Std Error	990.67	Prob > t	0.1718
Upper95%	5481.72	Prob < t	0.8282
Lower95%	-3043.3		
N	3		
Correlation	-0.2973		

Matched Pairs Element=Li
Difference: Reference Value-CC+PF



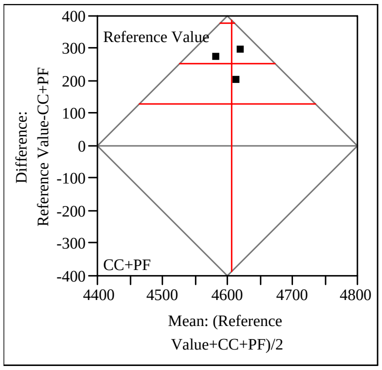
Reference Value	13562	t-Ratio	-0.74989
CC+PF	13643.9	DF	2
Mean Difference	-81.96	Prob > |t|	0.5315
Std Error	109.297	Prob > t	0.7342
Upper95%	388.305	Prob < t	0.2658
Lower95%	-552.23		
N	3		
Correlation	-0.8618		

Matched Pairs Element=Mn
Difference: Reference Value-CC+PF



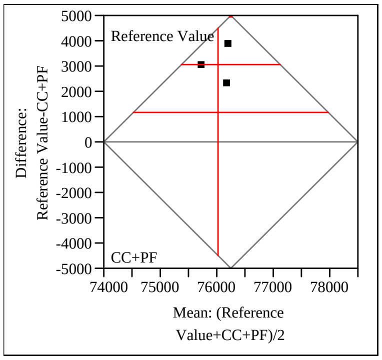
Reference Value	13288.9	t-Ratio	1.312592
CC+PF	13187.1	DF	2
Mean Difference	101.837	Prob > |t|	0.3197
Std Error	77.5844	Prob > t	0.1599
Upper95%	435.655	Prob < t	0.8401
Lower95%	-231.98		
N	3		
Correlation	-0.9914		

Matched Pairs Element=Mg
Difference: Reference Value-CC+PF



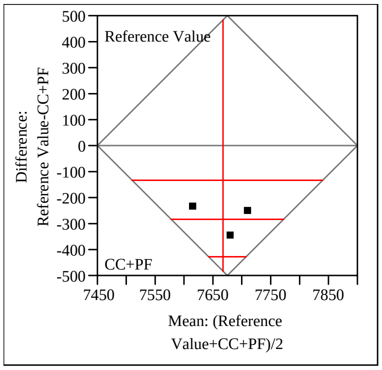
Reference Value	4733.04	t-Ratio	8.756483
CC+PF	4478.39	DF	2
Mean Difference	254.653	Prob > |t|	0.0128
Std Error	29.0817	Prob > t	0.0064
Upper95%	379.782	Prob < t	0.9936
Lower95%	129.525		
N	3		
Correlation	-0.2202		

Matched Pairs Element=Na
Difference: Reference Value-CC+PF



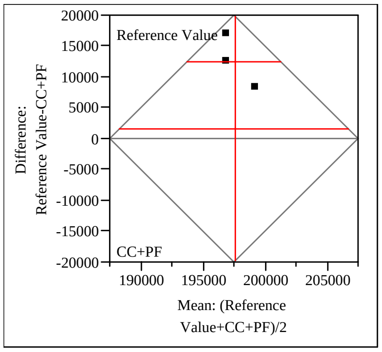
Reference Value	77549	t-Ratio	6.899629
CC+PF	74520	DF	2
Mean Difference	3029.08	Prob > |t|	0.0204
Std Error	439.021	Prob > t	0.0102
Upper95%	4918.03	Prob < t	0.9898
Lower95%	1140.13		
N	3		
Correlation	-0.3324		

Matched Pairs Element=Ni
Difference: Reference Value-CC+PF



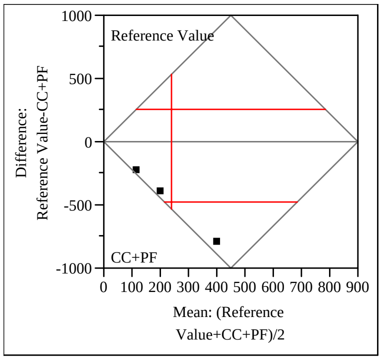
Reference Value	7527.35	t-Ratio	-8.24734
CC+PF	7808.08	DF	2
Mean Difference	-280.73	Prob > |t|	0.0144
Std Error	34.0388	Prob > t	0.9928
Upper95%	-134.27	Prob < t	0.0072
Lower95%	-427.19		
N	3		
Correlation	0.49671		

Matched Pairs Element=Si
Difference: Reference Value-CC+PF



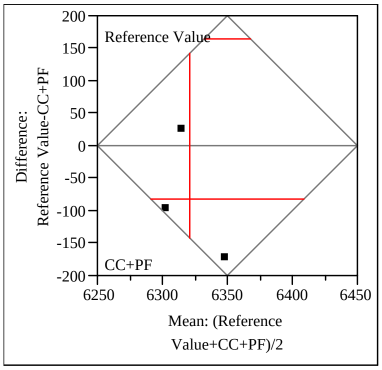
Reference Value	203885	t-Ratio	4.930434
CC+PF	191372	DF	2
Mean Difference	12513.1	Prob > |t|	0.0388
Std Error	2537.92	Prob > t	0.0194
Upper95%	23432.9	Prob < t	0.9806
Lower95%	1593.26		
N	3		
Correlation	-0.6918		

Matched Pairs Element=Sn
Difference: Reference Value-CC+PF



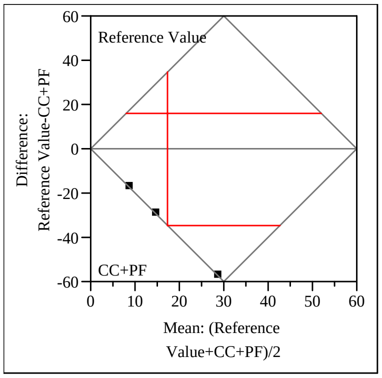
Reference Value	0	t-Ratio	-2.81037
CC+PF	477.563	DF	2
Mean Difference	-477.56	Prob > |t|	0.1067
Std Error	169.929	Prob > t	0.9466
Upper95%	253.581	Prob < t	0.0534
Lower95%	-1208.7		
N	3		
Correlation	0		

Matched Pairs Element=Ti
Difference: Reference Value-CC+PF



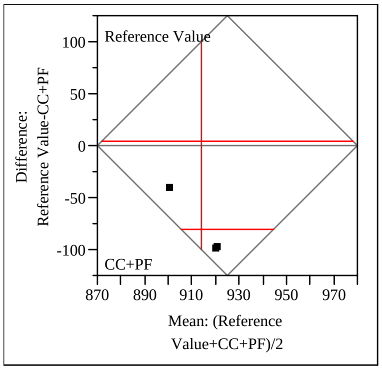
Reference Value	6280.38	t-Ratio	-1.43705
CC+PF	6362.55	DF	2
Mean Difference	-82.173	Prob > |t|	0.2873
Std Error	57.1819	Prob > t	0.8564
Upper95%	163.861	Prob < t	0.1436
Lower95%	-328.21		
N	3		
Correlation	-0.712		

Matched Pairs Element=V
Difference: Reference Value-CC+PF



Reference Value	0	t-Ratio	-2.95848
CC+PF	34.7433	DF	2
Mean Difference	-34.743	Prob > |t|	0.0978
Std Error	11.7436	Prob > t	0.9511
Upper95%	15.7855	Prob < t	0.0489
Lower95%	-85.272		
N	3		
Correlation	0		

Matched Pairs Element=Zr
Difference: Reference Value-CC+PF



Reference Value	873.792	t-Ratio	-4.11975
CC+PF	953.883	DF	2
Mean Difference	-80.091	Prob > |t|	0.0542
Std Error	19.4408	Prob > t	0.9729
Upper95%	3.55588	Prob < t	0.0271
Lower95%	-163.74		
N	3		
Correlation	-0.9937		