

# **STATISTICAL REVIEW OF CESIUM CARBONATE FUSION MEASUREMENTS OF ARCHIVED GLASS SAMPLES FROM THE DWPF PROCESS**

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July 2006

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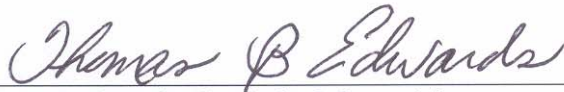
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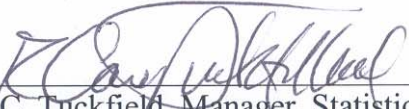


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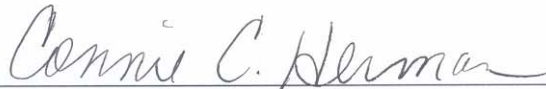
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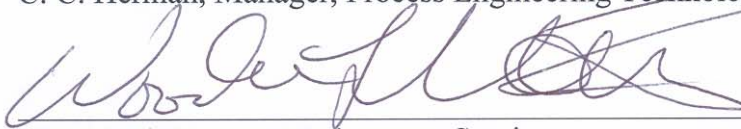
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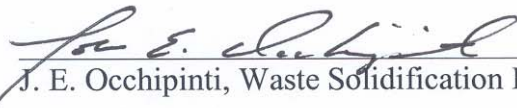
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## EXECUTIVE SUMMARY

This technical report provides a statistical review of measurements that the Defense Waste Processing Facility (DWPF) Laboratory (Lab) generated by Inductively-Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) for samples of archived Slurry Mix Evaporator (SME) glass that were prepared using a cesium carbonate ( $\text{Cs}_2\text{CO}_3$ ) fusion dissolution method. Measurements were generated by both of the Lab's ICPs, which are designated as M-13 and M-14. Comparisons between the two ICPs suggest that for the ARG-1 measurements the M-14 provided lower (on average) concentration measurements for about 13 of the 16 elements reported by DWPF with Cr, Mn, Ni, and Zr being statistically lower (at a 5% significance level) for the ARG-1 samples. One of the three exceptions is Li, for which the M-14 yielded (on average) higher concentration measurements than the M-13. For the SME samples, the M-14 and M-13 measurements had statistically different (at the 5% significance level) means for B, Ca, Cr, Cu, Fe, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr with the M-14 being lower than the M-13 for all of these elements except for Li.

For the screened SME samples, statistically significant (at the 5% level) differences between the means of the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations are seen for Ca ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), Cr ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), Cu ( $\text{Cs}_2\text{CO}_3$  higher for M-13), K ( $\text{Cs}_2\text{CO}_3$  higher for M-13), Mn ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Ni ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Si ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Ti ( $\text{Cs}_2\text{CO}_3$  higher for M-13), U ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), and Zr ( $\text{Cs}_2\text{CO}_3$  lower for M-14).

The  $\text{Cs}_2\text{CO}_3$  fusion method provided elemental of glass samples made from SME batches that were statistically comparable to the DWPF prototypic dissolution methods when the  $\text{Cs}_2\text{CO}_3$  solutions were analyzed by the M-13. However, when the same solutions were analyzed with the M-15, the elemental analyses from the  $\text{Cs}_2\text{CO}_3$  method were slightly lower and more prone to fail the 95% sum of oxides criterion imposed by DWPF's Product Composition Control System (PCCS). The  $\text{Cs}_2\text{CO}_3$  fusion method dissolutions also provided slightly lower waste loading values than the prototypic dissolutions because of slightly higher lithium values when the M-14 was used. In essence, subtle differences in the analyses from the M-13 and M-14 ICPs make it difficult to assess the viability of the  $\text{Cs}_2\text{CO}_3$  fusion method to replace the prototypic dissolution methods. The DWPF Laboratory should investigate the causes of these subtle differences between the M-13 and M-14 ICPs as the effort to employ the  $\text{Cs}_2\text{CO}_3$  fusion method for SME elemental analyses continues.

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## LIST OF ACRONYMS

AD	Analytical Development
Cs <sub>2</sub> CO <sub>3</sub>	Cesium Carbonate
DWPF	Defense Waste Processing Facility
ICP-AES	Inductively-Coupled Plasma – Atomic Emission Spectroscopy
JMP	Pronounced “jump.” It’s a statistical software package, a registered trademark of SAS Institute, Inc.
MAR	Measurement Acceptability Region
MFT	Melter Feed Tank
PCCS	Product Composition Control System
PET	Process Engineering Technology
SCS	Statistical Consulting Section
SME	Slurry Mix Evaporator
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TT&QA	Task Technical and Quality Assurance (plan)
TTR	Technical Task Request
WL	Waste Loading



## 1.0 INTRODUCTION AND BACKGROUND

The Statistical Consulting Section (SCS) of the Savannah River National Laboratory (SRNL) is working with Analytical Development (AD) and Process Engineering Technology (PET) at SRNL to pursue opportunities for improvements in analytical turnaround times at the Defense Waste Processing Facility (DWPF) Laboratory (Lab) that were identified as part of a Six Sigma study of that facility's operation. The Six Sigma study identified several steps that needed to be taken to assess the viability of sampling and analytical methods that were proposed for use in characterizing the contents of DWPF's Slurry Mix Evaporator (SME) tank [1]. Two phases of work have already been completed as reported in [2], [3], and [4]. The success of these phases has led to the recommendation for a Phase III effort as defined in technical task requests (TTRs) [5] and [6]. SRNL issued a task technical and quality assurance (TT&QA) plan [7] to address the scope of work defined in the first of the two TTRs [5].

The TT&QA plan [7] covers the statistical review of two sets of data. The first is a set of elemental analyses of archived SME (i.e., powdered glass) samples that DWPF Lab prepared using the proposed cesium carbonate ( $\text{Cs}_2\text{CO}_3$ ) fusion method and subsequently measured by inductively-coupled plasma – atomic emission spectroscopy (ICP-AES). The statistical review of these measurements and of their comparison to the original (prototypic) measurements of these production samples is the focus of this report. The second set of measurements is to be from a true side-by-side testing of the two analytical protocols: the DWPF Lab's current protocol that uses peanut vials and the dual dissolution methods of peroxide fusion and mixed acid versus the new protocol that relies on a large bottle sampling approach and the  $\text{Cs}_2\text{CO}_3$  fusion dissolution method. The statistical review of this second set of measurements is to be provided in a separate report.

## 2.0 RESULTS

This section provides the statistical review of the measurements. In addition to the general review of the measurements, there are some specific issues that are considered. These include

- (1) Is there any indication of a significant bias between the  $\text{Cs}_2\text{CO}_3$  fusion method and the DWPF Lab's current method for any element?
- (2) Are there any issues related to the particular ICP used?
- (3) What would the best 4 of 6 samples mean for the elemental analyses generated by the  $\text{Cs}_2\text{CO}_3$  fusion method? (Should all 6 samples be used?)
- (4) What is the impact of the  $\text{Cs}_2\text{CO}_3$  results on the process and product quality predictions (i.e., on the SME acceptability decisions) for the batches under study?
- (5) What is the impact of the  $\text{Cs}_2\text{CO}_3$  results on the estimated waste loadings (WLs) for the SME batches under study?

The statistical analyses presented in this paper were conducted using JMP Version 5.1.2 from SAS Institute, Inc. [8].

### 2.1 Overview of Measurements and Initial Plots

Table A1 in Appendix A lists the measurement data that are the subject of the statistical review provided in this report. Measurements of glass samples from 7 different SME batches are shown in this table. The batch numbers are indicated and the 6 process samples for each batch are

labeled as rep1 through rep 6. The “rep” samples and the ARG-1 (standard glass) samples, which appear at the beginning and end of each block of measurements, were prepared using the  $\text{Cs}_2\text{CO}_3$  fusion dissolution method, and the elemental composition of the prepared samples were determined by ICP-AES by both Leeman ICPs (the instruments are designated as the M-13 and M-14 ICPs and these labels are indicated in the headings for the measurements in Table A1).

Other information is also provided as part of the block of results. For the ARG-1 samples, the known or reference values for the elemental composition of the ARG-1 standard are provided. For the “rep” samples, the prototypic measurements (derived from samples prepared by peroxide fusion and mixed acid dissolution methods) are provided. The latter data were the “production” measurements generated by the DWPF Lab as the SME batches were originally processed. Situations where the prototypic measurements were rejected by the DWPF Lab are also indicated as part of Table A1. The rejected measurements were excluded from the analyses that follow, and this is indicated by the use of the descriptor “screened prototypic” to represent these measurements.

Exhibit A1 in Appendix A provides initial plots of these measurements. For the ARG-1 measurements, even though a “place-holder” is provided for the screened prototypic grouping, no data were generated for ARG-1 by the prototypic preparation methods for this study. The following observations are noted from this exhibit: the measurements for both ARG-1 samples for batch 362 are low for several elements (e.g., Al, B, Fe, Mg, Mn, and Si), the measurements for rep 5 of batch 357 are high for several elements, one of the K measurements by the M-13 is higher for batch 359. The sums of oxides for the ARG-1 and SME rep samples are plotted in this exhibit by grouping by batch. The ARG-1 results for batch 362 and the  $\text{Cs}_2\text{CO}_3$  results for the SME rep samples for batch 357 (both ICPs) and for batch 359 (the M-14 ICP) fall below the 95% working limit for these sums.

## **2.2 Initial Comparisons among the M-13, M-14, and Screened Prototypic Measurements**

Exhibit A2 in Appendix A provides initial comparisons using all of the  $\text{Cs}_2\text{CO}_3$  measurement data along with the screened prototypic measurements. This exhibit provides the results of pair-wise tests for differing means for M-13 versus M-14 for the ARG-1 data and pair-wise tests for differing means for M-13 versus M-14, for M-13 versus screened prototypic, and for M-14 versus screened prototypic for the SME rep samples. For each of these tests, a value for  $\text{Prob} > |t|$  of 0.05 or less indicates that the two means being compared are statistically different at a significance level of 5%.

For the ARG-1 comparisons, the means being compared are those for the ICPs (i.e., the M-13 and M-14). In this case, the samples were prepared using the  $\text{Cs}_2\text{CO}_3$  method. Statistically significant (at the 5% level) differences between the means of the ICPs are seen for Cr, Mn, Ni, U, and Zr. Note that there is no U in the ARG-1 glass, so the U difference is probably due to a difference in the calibrations of the two instruments. Another observation of note for the ARG-1 results is that the M-14 ICP yields average measurements that are often (13 of the 16 elements) less than those provided by the M-13 ICP with the exceptions being the measurements for Li, Ti, and Zr.

For the SME rep samples, comparisons are made between the two ICPs for the  $\text{Cs}_2\text{CO}_3$  prepared process samples and between the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations. In the latter case, the  $\text{Cs}_2\text{CO}_3$  results are represented in turn by the M-13 measurements and by the M-14 measurements. From the exhibit, statistically significant (at the 5% level) differences between

the means of the two ICPs are seen for B, Ca, Cr, Cu, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr. From the exhibit, statistically significant (at the 5% level) differences between the means of the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations are seen for Ca (the  $\text{Cs}_2\text{CO}_3$  M-13 is higher), Cr (the  $\text{Cs}_2\text{CO}_3$  results for both ICPs are higher), Cu (the  $\text{Cs}_2\text{CO}_3$  M-13 is higher), K (the  $\text{Cs}_2\text{CO}_3$  M-13 is higher), Mg (the  $\text{Cs}_2\text{CO}_3$  M-13 is higher), and U (the  $\text{Cs}_2\text{CO}_3$  results for both ICPs are higher).

To make sure that the potential outliers noted in the previous section for the  $\text{Cs}_2\text{CO}_3$  measurements are not adversely affecting the results of these pair-wise comparisons, Exhibit A3 in Appendix A was prepared. In this exhibit, the pair-wise comparisons of Exhibit A2 were repeated with the ARG-1 data for batch 362 and with the rep 5 measurements for batch 357 excluded from the analysis. For the ARG-1 comparisons, recall that the means being compared are those for the ICPs (i.e., the M-13 and M-14). For the screened  $\text{Cs}_2\text{CO}_3$  data, statistically significant (at the 5% level) differences between the means of the ICPs are seen for Cr, Mn, Ni, U, and Zr. As before, note that there is no U in the ARG-1 glass, so the U difference is probably due to a difference in the calibrations of the two instruments. For the screened SME rep samples, comparisons are made between the two ICPs for the  $\text{Cs}_2\text{CO}_3$  prepared process samples and between the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations. In the latter case, the  $\text{Cs}_2\text{CO}_3$  results are represented in turn by the M-13 measurements and by the M-14 measurements. From Exhibit A3, statistically significant (at the 5% level) differences between the means of the two ICPs are seen for B, Ca, Cr, Cu, Fe, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr. From the exhibit, statistically significant (at the 5% level) differences between the means of the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations are seen for Ca (both ICPs), Cr (both ICPs), Cu (M-13), K (M-13), Mn (M-14), Ni (M-14), Si (M-14), Ti (M-13), U (both ICPs), and Zr (M-14).

### 2.3 Uncertainties of M-13 and M-14 Measurements Based Upon the ARG-1 Results

The ARG-1 measurements of Table A1 provide an opportunity for an investigation into the uncertainties, both bias and precision errors, in the  $\text{Cs}_2\text{CO}_3$  measurements for the two ICPs. Using the screened ARG-1 values (i.e., excluding the ARG1 results for batch 362), a components-of-variance analysis was conducted using JMP, and the results are summarized in Table 2-1.

The upper portion of the table covers the M-13 results while the lower covers the M-14. The first column indicates the element, the second column provides the ARG-1 reference concentration in wt% for the element, the next column provides the average measurement in wt%, the next column provides the percent bias (% bias), and the next column provides the number of observations (n). An estimate of the between batch variance (Estimate of Between Var), an estimate of the within batch variance (Estimate of Within Var), and an estimate of the total variance (Estimate of Total Var) are the next columns. The three columns that follow provide the percent relative standard deviation (%RSD) for each of the sources of variation (Between, Within, and Total). The Mean Squares Between Batch and its degrees of freedom (DoF) follow next. From all of this information, a 95% confidence interval for the mean is computed and shown next. Finally, the 95% uncertainty interval for a single measurement when made using the indicated ICP for the element is shown. The interpretation of these intervals may be illustrated using the M-13 Al results. The uncertainty interval is (7.16%, 8.81%), which implies that, for a single Al measurement using the M-13 ICP, the true value may be 7.16% lower or 8.81% higher than the measurement itself.

Table 2-1. Details of ARG-1 Analyses by ICP and the Resulting ICP Uncertainties

Analyte	wt%	M-13	% Bias	n	Estimate of	Estimate of	Estimate of	Estimate of	Estimate of	Estimate of	Mean Squares	DoF	95% Confidence Interval for Mean		95% Measurement Uncertainty	
	Ref. Value	Avg. wt%e			(B)etween Var	(W)ithin Var	(T)otal Var.	%RSD (B)	%RSD(W)	%RSD(T)	Between Batch		Lower	Upper	% Lower	% Upper
Al	2.5	2.418	-3.27%	12	0	0.004533	0.004533	0.00%	2.78%	2.78%	0.00327	5	2.376	2.461	7.16%	8.81%
B	2.69	2.606	-3.13%	12	0	0.007842	0.007842	0.00%	3.40%	3.40%	0.00681	5	2.545	2.667	8.74%	10.37%
Ca	1.02	1.056	3.51%	12	0.000413	0.007342	0.007755	1.92%	8.12%	8.34%	0.00817	5	0.989	1.123	23.55%	21.44%
Cr	0.06	0.160	166.67%	12	0.00069	0.0005	0.00119	16.42%	13.98%	21.56%	0.00188	5	0.128	0.192	99.48%	55.42%
Cu	0.0001	0.009	9067.00%	12	0.000028	0.000092	0.00012	57.72%	104.63%	119.50%	0.00015	5	0.000	0.018	365.49%	307.18%
Fe	9.79	9.866	0.77%	12	0.002335	0.070158	0.072493	0.49%	2.68%	2.73%	0.07483	5	9.663	10.069	7.56%	7.02%
K	2.25	2.414	7.30%	12	0	0.474458	0.474458	0.00%	28.53%	28.53%	0.29103	5	2.014	2.814	76.98%	73.34%
Li	1.49	1.468	-1.51%	12	0.003572	0.000592	0.004164	4.07%	1.66%	4.40%	0.00774	5	1.402	1.533	11.67%	11.30%
Mg	0.52	0.522	0.32%	12	0	0.000317	0.000317	0.00%	3.41%	3.41%	0.00029	5	0.509	0.534	9.19%	8.77%
Mn	1.46	1.430	-2.05%	12	0.000318	0.002383	0.002701	1.25%	3.41%	3.63%	0.00302	5	1.389	1.471	9.37%	9.34%
Na	8.53	8.608	0.92%	12	0	0.1004	0.1004	0.00%	3.68%	3.68%	0.05679	5	8.431	8.785	9.92%	9.46%
Ni	0.83	0.832	0.20%	12	0.000208	0.000717	0.000925	1.73%	3.22%	3.66%	0.00113	5	0.807	0.857	9.93%	9.40%
Si	22.39	22.998	2.71%	12	0	0.534125	0.534125	0.00%	3.18%	3.18%	0.24085	5	22.633	23.362	9.20%	8.17%
Ti	0.69	0.683	-1.09%	12	0	0.000225	0.000225	0.00%	2.20%	2.20%	0.00013	5	0.674	0.691	5.65%	5.65%
U	0	0.126		12	0.037603	0.033242	0.070845	154.10%	144.89%	211.52%	0.10845	5	-0.119	0.370	-	-
Zr	0.1	0.110	10.00%	12	0.000002	0.000017	0.000019	1.29%	3.75%	3.96%	0.00002	5	0.107	0.113	15.82%	10.19%
Sum Ox	99.3	100.863	1.57%	12	0	9.239066	9.239066	0.00%	3.01%	3.01%	2.51214	5	99.687	102.039	8.21%	7.75%

Analyte	wt%	M-14	% Bias	n	Estimate of	Estimate of	Estimate of	Estimate of	Estimate of	Estimate of	Mean Squares	DoF	95% Confidence Interval for Mean		95% Measurement Uncertainty	
	Ref. Value	Avg. wt%e			Between Var	Within Var	Total Variance	%RSD (B)	%RSD(W)	%RSD(T)	Between Batch		Lower	Upper	% Lower	% Upper
Al	2.5	2.408	-3.67%	12	0.000147	0.0053	0.005447	0.50%	3.02%	3.06%	0.00559	5	2.353	2.464	7.88%	9.97%
B	2.69	2.575	-4.28%	12	0.001358	0.002683	0.004041	1.43%	2.01%	2.47%	0.0054	5	2.520	2.630	6.35%	9.14%
Ca	1.02	1.039	1.88%	12	0	0.006708	0.006708	0.00%	7.88%	7.88%	0.00557	5	0.984	1.095	21.49%	20.26%
Cr	0.06	0.139	131.95%	12	0.000375	0.000358	0.000733	13.91%	13.60%	19.45%	0.00111	5	0.114	0.164	89.85%	50.01%
Cu	0.0001	-0.002	-1770.00%	12	0.000098	0.000017	0.000115	592.78%	246.89%	642.14%	0.00021	5	-0.012	0.009	1736.12%	1650.68%
Fe	9.79	9.750	-0.41%	12	0	0.0466	0.0466	0.00%	2.21%	2.21%	0.03232	5	9.617	9.883	5.77%	5.69%
K	2.25	2.190	-2.67%	12	0.004423	0.059033	0.063456	3.04%	11.09%	11.50%	0.06788	5	1.997	2.383	30.19%	29.57%
Li	1.49	1.508	1.23%	12	0	0.001217	0.001217	0.00%	2.31%	2.31%	0.00113	5	1.483	1.533	6.60%	5.95%
Mg	0.52	0.515	-0.96%	12	0	0.000283	0.000283	0.00%	3.27%	3.27%	0.0002	5	0.505	0.525	8.46%	8.40%
Mn	1.46	1.409	-3.48%	12	0.000655	0.002958	0.003613	1.82%	3.86%	4.27%	0.00427	5	1.361	1.458	10.96%	13.03%
Na	8.53	8.419	-1.30%	12	0.075873	0.147542	0.223415	3.27%	4.56%	5.61%	0.29929	5	8.013	8.825	14.85%	14.43%
Ni	0.83	0.802	-3.41%	12	0	0.00085	0.00085	0.00%	3.64%	3.64%	0.00041	5	0.787	0.817	9.35%	10.80%
Si	22.39	22.607	0.97%	12	0	0.447317	0.447317	0.00%	2.96%	2.96%	0.25783	5	22.230	22.983	8.05%	7.61%
Ti	0.69	0.690	0.00%	12	0.000075	0.00035	0.000425	1.26%	2.71%	2.99%	0.0005	5	0.673	0.707	8.05%	7.68%
U	0	-0.154		12	0.04815	0.014108	0.062258	-142.33%	-77.04%	-161.84%	0.11041	5	-0.401	0.092	-	-
Zr	0.1	0.101	0.83%	12	0.000023	0.000042	0.000065	4.76%	6.43%	8.00%	0.00009	5	0.094	0.108	21.99%	20.55%
Sum Ox	99.3	98.833	-0.47%	12	0	6.843413	6.843413	0.00%	2.65%	2.65%	3.15096	5	97.515	100.150	6.86%	6.80%

## 2.4 PCCS MAR Results for the M-13, M-14, and Prototypic Measurements

During DWPF operation, the measured chemical compositions of the SME samples (i.e., the rep measurements in this study) are entered into DWPF's Product Composition Control System (PCCS) to facilitate the SME acceptability decision. PCCS is used to make the acceptability decision for each process batch of HLW prior to its being transferred to the melter. The chemical composition measurements of the SME samples are judged against various acceptance criteria, and the glass compositions that satisfy the most stringent of these criteria are designated as being in the Measurement Acceptance (or Acceptability) Region (MAR) of PCCS. The criteria rely directly on chemical composition measurements of the glass or on predictions from models that relate important process (e.g., viscosity or liquidus temperature) or product quality (specifically, the durability of the final waste form) properties of the glass to the chemical composition measurements. SME batches that satisfy the MAR criteria are deemed acceptable and are transferred to the melter for processing via the Melter Feed Tank (MFT). The technical basis for SME acceptability is provided by reference [9], and this document has been in place to support the DWPF since it began radioactive operation in 1996.

For this study, there is an opportunity to compare PCCS outcomes from the  $\text{Cs}_2\text{CO}_3$  results for both the M-13 and the M-14 with the outcomes from the screened prototypic results. Before providing the comparisons, it should be noted that the PCCS results being discussed do not necessarily reflect the production results for the batches presented here. As part of its production process, DWPF utilizes the best 4 of 6 SME sample results. No attempt was made to duplicate this process as part of this study. For the comparisons presented here, all of the available prototypic measurements were used except for the values excluded as a result of the screening process discussed above.

Table 2-2 provides a subset of the PCCS predictions and measurement assessments for each of the three categories of measurements. The SME batch is indicated in this table along with the category of measurements being evaluated. Columns are provided for predictions of the normalized leachate for boron in grams/liter (g/L), of liquidus temperature ( $T_L$ ) in °C, viscosity in Poise (P), sum of oxides in weight percent (wt%),  $\text{Al}_2\text{O}_3$  concentration in wt%,  $\text{Cr}_2\text{O}_3$  concentration in wt%, and the sum of alkali content (denoted by  $\text{R}_2\text{O}$ ) in wt%.

**Table 2-2. PCCS Results by Measurement Category by SME Batch**

Batch	Category of Measurements	NL[B (g/L)]	T <sub>L</sub> Prediction (°C)	Viscosity Prediction (P)	Sum of Oxide (wt%)s	Al <sub>2</sub> O <sub>3</sub> wt%	Cr <sub>2</sub> O <sub>3</sub> wt%	R <sub>2</sub> O wt%
354	M-13	0.69	978.4	61.00	100.38	6.15	0.17	17.15
354	M-14	1.00	951.7	47.17	99.34	6.32	0.14	18.14
354	Screened Prototypic	0.53	959.1	68.42	97.60	6.24	0.09	16.41
357	M-13	0.43	963.3	75.72	93.41	5.51	0.20	15.51
357	M-14	0.46	954.5	73.17	93.86	5.44	0.18	15.71
357	Screened Prototypic	0.48	928.2	73.65	96.22	5.70	0.09	16.03
359	M-13	0.56	979.6	73.89	96.25	5.62	0.26	16.37
359	M-14	0.39	970.3	79.21	94.25	5.56	0.20	15.36
359	Screened Prototypic	0.55	930.0	78.85	99.46	5.69	0.09	16.48
361	M-13	0.75	901.6	65.02	96.89	5.48	0.11	17.09
361	M-14	0.65	906.3	65.73	96.26	5.59	0.11	16.74
361	Screened Prototypic	0.57	896.1	77.29	96.97	5.27	0.08	16.44
362	M-13	0.52	954.5	85.68	100.35	5.55	0.22	16.35
362	M-14	0.48	942.9	82.19	97.48	5.56	0.20	16.05
362	Screened Prototypic	0.44	903.2	90.40	96.60	5.36	0.08	15.76
364	M-13	0.55	993.4	77.66	99.48	6.03	0.23	16.53
364	M-14	0.50	969.4	72.49	97.13	5.69	0.19	16.14
364	Screened Prototypic	0.63	919.7	63.04	95.67	5.66	0.09	16.68
365	M-13	0.54	1000.4	67.55	97.74	5.97	0.18	16.40
365	M-14	0.55	972.8	57.96	94.05	5.95	0.15	16.30
365	Screened Prototypic	0.53	959.4	66.10	97.91	6.15	0.08	16.32

The acceptability of these predictions and the other MAR criteria for each of the measurement categories is summarized in Table 2-3. This table indicates using a short-hand notation those criteria that are not met. The lsum designation indicates that the low of sum of oxides constraint is not met; the Cr<sub>2</sub>O<sub>3</sub> designation indicates that the chromium oxide solubility limit criterion is exceeded. As seen in Table 2-3, low sum of oxides is a problem for the Cs<sub>2</sub>CO<sub>3</sub> measurements from both ICPs for batch 357 and for the M-14 ICP for batch 359 and for batch 365. The M-14 Cs<sub>2</sub>CO<sub>3</sub> measurements leading to more low of sum of oxides problems may be a reflection of the tendency of the M-14 to provide lower concentration measurements for most of the oxides as compared to the M-13 as was noted above. The Cr<sub>2</sub>O<sub>3</sub> solubility constraint is not met by the M-13 measurements for batch 359 and for batch 364; these may have been calibration issues for the M-13. Thus, in general, the Cs<sub>2</sub>CO<sub>3</sub> measurements met the PCCS MAR criteria.

**Table 2-3. PCCS MAR Results by Measurement Category by SME Batch**

Type of Sample	Batch	Method	MAR Status
SME	354	M-13	
SME	354	M-14	
SME	354	Screened Prototypic	
SME	357	M-13	lsum
SME	357	M-14	lsum
SME	357	Screened Prototypic	
SME	359	M-13	Cr <sub>2</sub> O <sub>3</sub>
SME	359	M-14	lsum
SME	359	Screened Prototypic	
SME	361	M-13	
SME	361	M-14	
SME	361	Screened Prototypic	
SME	362	M-13	
SME	362	M-14	
SME	362	Screened Prototypic	
SME	364	M-13	Cr <sub>2</sub> O <sub>3</sub>
SME	364	M-14	
SME	364	Screened Prototypic	
SME	365	M-13	
SME	365	M-14	lsum
SME	365	Screened Prototypic	

## 2.5 Comparisons of WLs

Another important use of the SME measurements at DWPF is in the determination of the waste loading (WL) attained for the SME batch. The targeting of the WL takes place during the blending of the SME; this involves the heel of the previous SME batch, a transfer from the current batch in the Sludge Adjustment and Receipt Tank (SRAT), and the addition of frit. But it is in the SME that the attained WL is determined.

The measurements of the rep samples in this study provide an opportunity for additional estimations of attained WL and for comparisons among these values and the values recorded at blending and for the original SME batches. Figure 2-1 provides a plot of these various WLs by batch, and Table 2-4 provides a listing of these values.

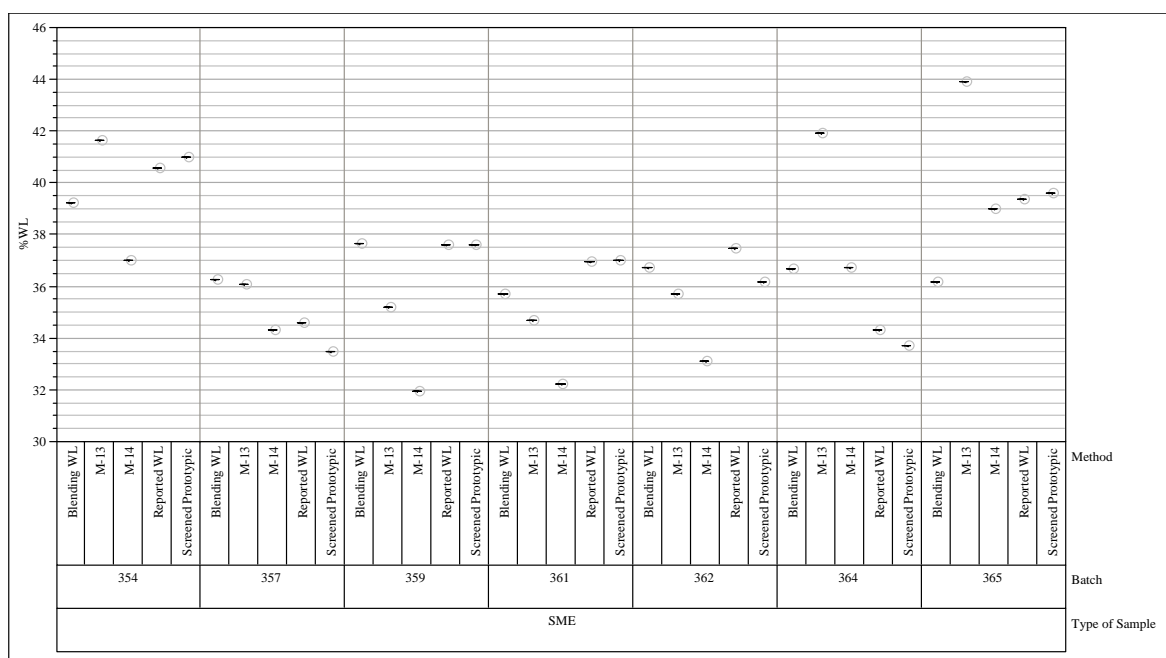


Figure 2-1. Waste Loading Comparisons

Table 2-4. Waste Loading Comparisons

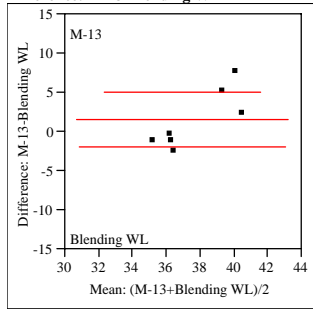
Type of Sample	Batch	Blending WL	M-13	M-14	Reported WL	Screened Prototype
SME	354	39.25	41.67	37.05	40.62	41.03
SME	357	36.29	36.10	34.33	34.63	33.49
SME	359	37.66	35.23	31.96	37.63	37.62
SME	361	35.71	34.71	32.26	36.96	37.01
SME	362	36.76	35.74	33.13	37.51	36.19
SME	364	36.70	41.93	36.74	34.32	33.74
SME	365	36.18	43.96	39.04	39.41	39.63

From Table 2-4, it appears that for the  $\text{Cs}_2\text{CO}_3$  data the WLs determined using the M-14 measurements are consistently lower than those determined using the M-13 measurements. This is probably a consequence of the M-14 providing a larger Li concentration (on average) as compared to the M-13 for these measurements. This is also reflected in the Li measurements for ARG-1 (see Table 2-1). A higher Li concentration leads to a lower estimation of WL (all other things being equal).

Figure 2-2 provides pair-wise statistical comparisons among these WLs values. As suggested by the discussion in the preceding paragraph, the M-14 yielded a statistically lower (~3.5 points lower) estimated WL value for the batches covered in this study as compared to the M-13. While the WLs values are somewhat noisy and other differences in the averages range a high of 2.37 points to a low of 0.02 points, there are no other statistically significant differences at the 5% significance level.

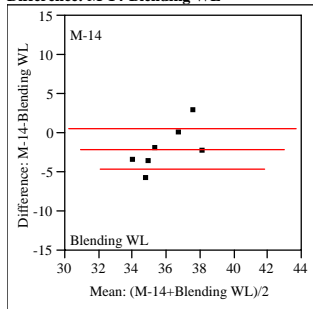


**Difference: M-13-Blending WL**



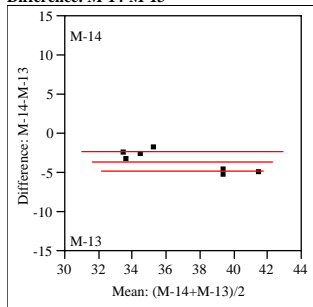
M-13	38.4771	t-Ratio	1.082969
Blending WL	36.9357	DF	6
Mean Difference	1.54143	Prob >  t	0.3204
Std Error	1.42334	Prob > t	0.1602
Upper95%	5.0242	Prob < t	0.8398
Lower95%	-1.9413		
N	7		
Correlation	0.24326		

**Difference: M-14-Blending WL**



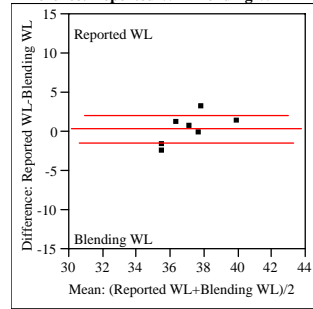
M-14	34.93	t-Ratio	-1.91089
Blending WL	36.9357	DF	6
Mean Difference	-2.0057	Prob >  t	0.1046
Std Error	1.04963	Prob > t	0.9477
Upper95%	0.56263	Prob < t	0.0523
Lower95%	-4.5741		
N	7		
Correlation	0.16543		

**Difference: M-14-M-13**



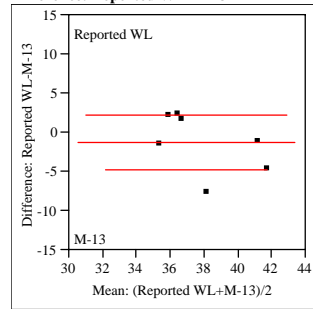
M-14	34.93	t-Ratio	-6.91493
M-13	38.4771	DF	6
Mean Difference	-3.5471	Prob >  t	0.0005
Std Error	0.51297	Prob > t	0.9998
Upper95%	-2.292	Prob < t	0.0002
Lower95%	-4.8023		
N	7		
Correlation	0.97636		

**Difference: Reported WL-Blending WL**



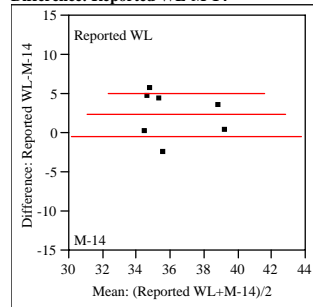
Reported WL	37.2971	t-Ratio	0.500053
Blending WL	36.9357	DF	6
Mean Difference	0.36143	Prob >  t	0.6348
Std Error	0.72278	Prob > t	0.3174
Upper95%	2.13001	Prob < t	0.6826
Lower95%	-1.4072		
N	7		
Correlation	0.5574		

**Difference: Reported WL-M-13**



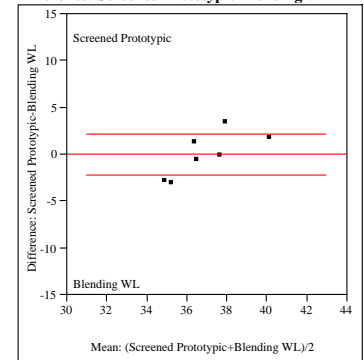
Reported WL	37.2971	t-Ratio	-0.82421
M-13	38.4771	DF	6
Mean Difference	-1.18	Prob >  t	0.4413
Std Error	1.43167	Prob > t	0.7793
Upper95%	2.32317	Prob < t	0.2207
Lower95%	-4.6832		
N	7		
Correlation	0.33401		

**Difference: Reported WL-M-14**



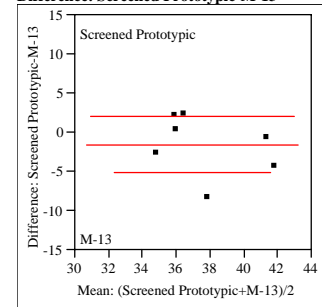
Reported WL	37.2971	t-Ratio	2.106671
M-14	34.93	DF	6
Mean Difference	2.36714	Prob >  t	0.0797
Std Error	1.12364	Prob > t	0.0399
Upper95%	5.11659	Prob < t	0.9601
Lower95%	-0.3823		
N	7		
Correlation	0.30609		

**Difference: Screened Prototypic-Blending WL**



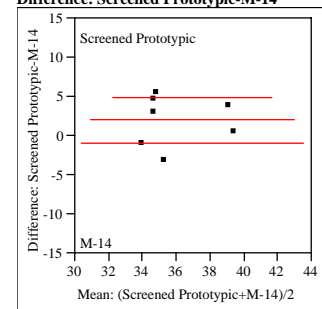
Screened Prototypic	36.9586	t-Ratio	0.025523
Blending WL	36.9357	DF	6
Mean Difference	0.02286	Prob >  t	0.9805
Std Error	0.89556	Prob > t	0.4902
Upper95%	2.21422	Prob < t	0.5098
Lower95%	-2.1685		
N	7		
Correlation	0.54859		

**Difference: Screened Prototypic-M-13**



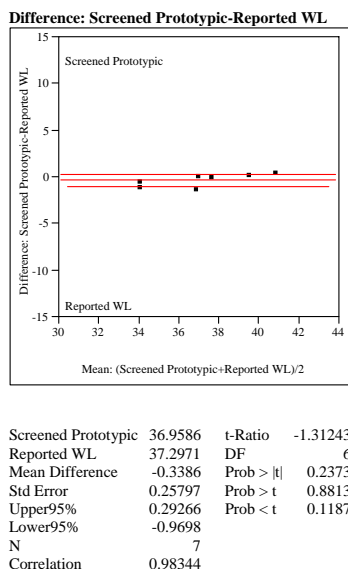
Screened Prototypic	36.9586	t-Ratio	-1.04846
M-13	38.4771	DF	6
Mean Difference	-1.5186	Prob >  t	0.3348
Std Error	1.44839	Prob > t	0.8326
Upper95%	2.02551	Prob < t	0.1674
Lower95%	-5.0627		
N	7		
Correlation	0.37667		

**Difference: Screened Prototypic-M-14**



Screened Prototypic	36.9586	t-Ratio	1.682565
M-14	34.93	DF	6
Mean Difference	2.02857	Prob >  t	0.1434
Std Error	1.20564	Prob > t	0.0717
Upper95%	4.97867	Prob < t	0.9283
Lower95%	-0.9215		
N	7		
Correlation	0.33183		

**Figure 2-2. Pair-Wise Statistical Comparisons of WLs**



**Figure 2-2. Pair-Wise Statistical Comparisons of WLs**  
(continued)

### 3.0 CONCLUSIONS

This report provides a statistical review of measurements that the DWPF Lab generated by ICP-AES for samples of archived SME glass that were prepared using a  $\text{Cs}_2\text{CO}_3$  fusion dissolution method. Measurements were generated by both of the Lab's ICPs. Comparisons between the two ICPs for the  $\text{Cs}_2\text{CO}_3$ -prepared samples suggest that, for the ARG-1 measurements, the M-14 provided lower concentration measurements for about 13 of the 16 elements reported by DWPF with Cr, Mn, Ni, and Zr being statistically lower (at a 5% significance level) for the ARG-1 samples. One of the three exceptions is Li, for which the M-14 yielded (on average) higher concentration measurements than the M-13. For the  $\text{Cs}_2\text{CO}_3$ -prepared SME samples, the M-14 and M-13 measurements had statistically different (at the 5% significance level) means for B, Ca, Cr, Cu, Fe, Li, Mg, Mn, Na, Ni, Si, Ti, U, and Zr with the M-14 being lower than the M-13 for all of these elements except for Li.

For the screened SME samples, statistically significant (at the 5% level) differences between the means of the  $\text{Cs}_2\text{CO}_3$  and prototypic preparations are seen for Ca ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), Cr ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), Cu ( $\text{Cs}_2\text{CO}_3$  higher for M-13), K ( $\text{Cs}_2\text{CO}_3$  higher for M-13), Mn ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Ni ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Si ( $\text{Cs}_2\text{CO}_3$  lower for M-14), Ti ( $\text{Cs}_2\text{CO}_3$  higher for M-13), U ( $\text{Cs}_2\text{CO}_3$  higher for both ICPs), and Zr ( $\text{Cs}_2\text{CO}_3$  lower for M-14).

The  $\text{Cs}_2\text{CO}_3$  fusion method provided elemental of glass samples made from SME batches that were statistically comparable to the DWPF prototypic dissolution methods when the  $\text{Cs}_2\text{CO}_3$  solutions were analyzed by the M-13. However, when the same solutions were analyzed with the M-15, the elemental analyses from the  $\text{Cs}_2\text{CO}_3$  method were slightly lower and more prone to fail the 95% sum of oxides criterion imposed by DWPF's Product Composition Control System (PCCS). The  $\text{Cs}_2\text{CO}_3$  fusion method dissolutions also provided slightly lower waste loading values than the prototypic dissolutions because of slightly higher lithium values when the M-14 was used. In essence, subtle differences in the analyses from the M-13 and M-14 ICPs make it difficult to assess the viability of the  $\text{Cs}_2\text{CO}_3$  fusion method to

replace the prototypic dissolution methods. The DWPF Laboratory should investigate the causes of these subtle differences between the M-13 and M-14 ICPs as the effort to employ the  $\text{Cs}_2\text{CO}_3$  fusion method for SME elemental analyses continues.

In these analyses all of the  $\text{Cs}_2\text{CO}_3$  measurements available for each of the SME batches were used in the comparisons, and there is no reason that use of all 6 samples might not be adopted as the standard operating procedures for the measurements from a  $\text{Cs}_2\text{CO}_3$  -prepared SME samples. A step can be incorporated into such procedures to screen for potential outliers in the measurements.

## 4.0 REFERENCES

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- [4] Edwards, TB and RN Mahannah, "Phase II of a Six Sigma Initiative to Study DWPF Slurry Mix Evaporator Analytical Turnaround Times: Comparisons of Weight Percent Total Solids Measurements at DWPF of Large Bottle Samples versus Peanut Vial Samples," WSRC-TR-2005-00492, Revision 0, October 2005.
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## **Appendix A. Supplemental Data Tables and Exhibits**

Table A1. ICP-AES Elemental Measurements for Samples Prepared by Cs<sub>2</sub>CO<sub>3</sub> Fusion, Peroxide Fusion, and Mixed Acid

Run 1LIMS ID 103454Batch 354																							
Weight Percents																							
	ARG 1			rep 1			rep 2			rep 3			rep 4			rep 5			rep 6			ARG-2	
	M-13	M-14	Known	M-13	M-14	103454	M-13	M-14	103454	M-13	M-14	103454	M-13	M-14	103454	M-13	M-14	103454	M-13	M-14	103454	M-13	M-14
Al	2.32	2.42	2.5	3.15	3.29	3.33	3.22	3.29	3.45	3.29	3.37	3.30	3.13	3.24	3.20	3.24	3.28	3.21	3.50	3.59	3.32	2.36	2.44
B	2.62	2.60	2.69	1.37	1.31	1.38	1.44	1.36	1.36	1.45	1.40	1.40	1.44	1.39	1.40	1.42	1.41	1.42	1.60	1.57	1.42	2.68	2.64
Ca	0.99	1.02	1.02	0.79	0.81	0.76	0.79	0.82	0.84	0.78	0.80	0.76	0.75	0.76	0.73	0.77	0.80	0.74	0.85	0.88	0.73	0.98	1.04
Cr	0.12	0.10	0.06	0.13	0.12	0.06	0.10	0.08	0.06	0.13	0.11	0.06	0.09	0.07	0.06	0.13	0.11	0.06	0.12	0.10	0.06	0.11	0.09
Cu	0.01	0.01	-----	0.01	0.02	0.01	0.01	0.03	0.01	0.02	0.03	0.01	0.01	0.03	0.01	0.01	0.02	0.01	0.02	0.04	0.01	0.00	0.02
Fe	9.93	9.53	9.79	8.64	8.15	8.61	8.79	8.42	9.04	8.94	8.76	8.49	8.51	8.40	8.13	8.60	8.62	8.15	9.57	9.45	8.33	10.32	10.15
K	1.94	2.13	2.25	0.03	0.41	0.10	-0.09	0.27	0.14	-0.19	0.33	0.02	-0.17	0.38	0.00	-0.19	0.71	0.00	0.65	0.44	0.00	2.85	2.72
Li	1.44	1.47	1.49	2.01	2.19	2.13	2.13	2.24	2.11	2.11	2.28	2.24	2.11	2.25	2.05	2.13	2.25	2.07	2.40	2.55	2.07	1.38	1.54
Mg	0.53	0.50	0.52	0.74	0.69	0.73	0.73	0.69	0.89	0.73	0.70	0.70	0.71	0.69	0.67	0.73	0.72	0.68	0.79	0.78	0.69	0.54	0.52
Mn	1.47	1.42	1.46	1.67	1.59	1.71	1.71	1.62	1.95	1.71	1.70	1.65	1.64	1.62	1.58	1.68	1.68	1.61	1.88	1.86	1.62	1.52	1.51
Na	8.42	8.36	8.53	8.55	8.73	8.77	9.23	9.37	8.84	9.65	9.81	8.70	9.65	9.84	8.73	8.22	8.34	8.86	10.40	10.40	8.68	9.08	9.17
Ni	0.85	0.79	0.83	0.45	0.40	0.50	0.47	0.42	0.53	0.50	0.45	0.48	0.48	0.45	0.45	0.46	0.42	0.45	0.53	0.52	0.46	0.86	0.85
Si	23.03	21.39	22.39	22.00	20.57	23.06	22.86	21.53	22.83	23.67	23.35	23.12	23.00	22.35	23.32	23.41	22.57	23.32	26.75	25.99	24.10	23.55	22.92
Ti	0.67	0.67	0.69	0.05	0.05	0.04	0.05	0.05	0.04	0.05	0.06	0.04	0.05	0.06	0.04	0.05	0.06	0.04	0.05	0.06	0.04	0.67	0.72
U	-0.06	0.04	-----	3.13	3.19	2.79	3.11	3.23	2.92	3.14	3.33	2.73	2.99	3.34	2.58	3.14	3.42	2.66	3.37	3.66	2.68	-0.10	0.44
Zr	0.11	0.10	0.1	0.07	0.07	0.07	0.08	0.08	0.07	0.08	0.09	0.07	0.08	0.09	0.07	0.08	0.08	0.07	0.09	0.09	0.07	0.11	0.12

Run 2LIMS ID 104161Batch 361																							
Weight Percents																							
	ARG 1			rep 1			rep 2			rep 3			rep 4			rep 5			rep 6			ARG-2	
	M-13	M-14	Known	M-13	M-14	104161	M-13	M-14	104161	M-13	M-14	104161	M-13	M-14	104161	M-13	M-14	104161	M-13	M-14	104161	M-13	M-14
Al	2.40	2.47	2.50	2.73	2.80	2.59	2.63	2.63	2.73	3.01	3.10	2.60	2.81	2.88	2.90	3.05	3.11	2.84	3.16	3.23	3.09	2.47	2.50
B	2.60	2.61	2.69	1.56	1.56	1.53	1.44	1.41	1.49	1.42	1.39	1.44	1.40	1.36	1.43	1.70	1.67	1.41	1.39	1.34	1.35	2.66	2.68
Ca	1.06	1.03	1.02	0.66	0.64	0.58	0.66	0.65	0.63	0.76	0.76	0.61	0.71	0.69	0.68	0.76	0.77	0.67	0.79	0.79	0.72	1.07	1.07
Cr	0.15	0.15	0.06	0.07	0.07	0.06	0.08	0.08	0.05	0.07	0.07	0.04	0.08	0.08	0.05	0.06	0.06	0.05	0.11	0.11	0.06	0.13	0.13
Cu	0.01	0.01	-----	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.00	0.00
Fe	9.81	9.86	9.79	6.89	6.89	6.77	6.71	6.66	7.05	7.81	7.68	6.76	7.21	7.10	7.69	7.61	7.55	7.70	7.65	7.87	8.33	9.64	9.75
K	2.10	2.07	2.25	-0.12	0.00	0.23	-0.05	0.07	0.06	0.47	0.00	0.21	0.45	0.16	0.24	0.40	0.10	0.30	0.65	0.22	0.34	2.64	2.48
Li	1.46	1.53	1.49	2.45	2.48	2.34	2.27	2.32	2.32	2.28	2.27	2.19	2.08	2.23	2.19	2.62	2.74	2.21	2.11	2.20	2.08	1.43	1.56
Mg	0.51	0.52	0.52	0.57	0.56	0.54	0.60	0.59	0.62	0.69	0.67	0.58	0.62	0.61	0.64	0.69	0.68	0.67	0.68	0.68	0.68	0.52	0.52
Mn	1.43	1.42	1.46	1.31	1.31	1.27	1.36	1.34	1.42	1.56	1.54	1.36	1.39	1.38	1.48	1.57	1.58	1.53	1.61	1.55	1.63	1.47	1.46
Na	8.48	8.47	8.53	8.35	8.25	7.97	8.12	8.13	8.51	8.95	8.56	8.28	8.50	8.29	8.62	9.63	9.26	8.60	8.87	8.79	8.67	8.95	8.84
Ni	0.81	0.79	0.83	0.38	0.37	0.38	0.38	0.37	0.40	0.44	0.43	0.38	0.39	0.41	0.43	0.44	0.43	0.44	0.46	0.44	0.48	0.83	0.83
Si	22.51	22.61	22.39	26.18	26.27	25.81	22.54	22.56	25.20	22.09	21.81	24.63	22.69	22.59	24.17	27.27	26.85	23.55	22.39	22.26	23.26	23.12	22.51
Ti	0.69	0.70	0.69	0.05	0.05	0.05	0.05	0.04	0.05	0.05	0.05	0.05	0.04	0.04	0.05	0.05	0.05	0.05	0.05	0.04	0.05	0.69	0.72
U	0.01	-0.09	-----	2.51	2.54	2.49	2.46	2.47	2.49	2.83	2.97	2.41	2.71	2.84	2.84	2.80	2.91	2.77	3.05	3.14	3.08	-0.15	0.00
Zr	0.11	0.11	0.10	0.09	0.08	0.08	0.08	0.07	0.08	0.09	0.09	0.08	0.08	0.08	0.08	0.09	0.08	0.08	0.08	0.08	0.08	0.10	0.10

Table A1. ICP-AES Elemental Measurements for Samples Prepared by Cs<sub>2</sub>CO<sub>3</sub> Fusion, Peroxide Fusion, and Mixed Acid

Run 3										LIMS ID 104213										Batch 362									
Weight Percents																													
	ARG 1			rep 1			rep 2			rep 3			rep 4			rep 5			rep 6			ARG-2							
	M-13	M-14	Known	M-13	M-14	6604	M-13	M-14	6603	M-13	M-14	6601	M-13	M-14	6605	M-13	M-14	6600	M-13	M-14	104213	M-13	M-14						
Al	0.28	0.31	2.50	3.08	3.10	3.03	3.21	3.23	3.16	2.82	2.82	2.56	3.01	3.02	2.97	2.82	2.81	2.95	2.69	2.66	2.46	2.14	2.06						
B	0.37	0.35	2.69	1.42	1.36	1.41	1.94	1.91	1.35	1.56	1.52	1.49	1.42	1.42	1.47	1.50	1.45	1.51	1.58	1.53	1.50	2.23	2.20						
Ca	0.14	0.13	1.02	0.74	0.73	0.74	0.80	0.77	0.75	0.65	0.63	0.56	0.73	0.69	0.70	0.64	0.60	0.65	0.62	0.60	0.53	0.91	0.89						
Cr	0.08	0.08	0.06	0.17	0.16	0.07	0.14	0.12	0.06	0.11	0.09	0.05	0.13	0.12	0.05	0.16	0.14	0.04	0.21	0.19	0.04	0.15	0.14						
Cu	-0.02	-0.01	-----	0.01	0.01	0.02	0.01	0.01	0.02	0.01	0.01	0.02	0.01	0.01	0.02	0.01	0.01	0.02	0.01	0.01	0.02	0.00	0.00						
Fe	1.15	1.20	9.79	8.14	7.91	7.89	8.49	8.10	8.05	7.05	6.79	6.60	7.56	7.43	7.35	7.05	6.79	2.86	6.62	6.36	6.11	8.58	8.41						
K	0.01	0.27	2.25	0.09	-0.05	0.10	0.04	0.03	0.14	-0.02	-0.05	0.06	0.37	0.04	0.04	-0.02	-0.12	0.16	0.11	0.00	0.08	1.82	1.94						
Li	0.20	0.22	1.49	2.22	2.27	2.15	2.17	2.17	2.14	2.43	2.49	2.17	2.29	2.31	2.24	2.33	2.31	2.38	2.40	2.44	2.25	1.29	1.31						
Mg	0.07	0.07	0.52	0.70	0.68	0.68	0.73	0.71	0.71	0.61	0.58	0.56	0.65	0.64	0.64	0.58	0.55	0.57	0.58	0.54	0.52	0.47	0.44						
Mn	0.17	0.18	1.46	1.65	1.57	1.60	1.69	1.63	1.67	1.39	1.32	1.28	1.48	1.45	1.47	1.37	1.28	0.97	1.30	1.24	1.18	1.27	1.23						
Na	1.05	1.12	8.53	8.74	8.71	8.59	8.75	8.68	7.01	8.30	8.15	7.89	8.50	8.36	8.39	7.75	7.41	7.90	8.14	7.93	7.81	7.32	7.20						
Ni	0.03	0.04	0.83	0.45	0.44	0.44	0.46	0.44	0.46	0.42	0.39	0.37	0.43	0.41	0.41	0.41	0.40	0.30	0.40	0.36	0.35	0.73	0.70						
Si	0.50	0.60	22.39	24.26	23.93	24.25	23.53	22.92	23.79	27.38	26.30	25.53	24.66	24.08	25.20	26.09	25.05	25.80	27.06	26.27	25.26	20.23	19.58						
Ti	0.08	0.09	0.69	0.04	0.05	0.04	0.04	0.05	0.04	0.04	0.05	0.04	0.04	0.05	0.04	0.04	0.05	0.02	0.04	0.05	0.04	0.60	0.59						
U	-0.23	-0.25	-----	2.93	2.74	2.66	3.21	2.87	2.83	2.46	2.22	2.15	2.85	2.61	2.51	2.52	2.28	2.10	2.39	2.12	1.94	0.00	-0.21						
Zr	0.00	0.01	0.10	0.09	0.09	0.10	0.09	0.09	0.08	0.10	0.10	0.09	0.09	0.09	0.09	0.10	0.09	0.09	0.09	0.09	0.09	0.09	0.09						
* ARG 1and ARG 2 - Muffle Furnace malfunctioned										rejected MA			rejected FS																
Run 4										LIMS ID 103793										Batch 357									
Weight Percents																													
	ARG 1			rep 1			rep 2			rep 3			rep 4			rep 5			rep 6			ARG-2							
	M-13	M-14	Known	M-13	M-14	5713	M-13	M-14	5745	M-13	M-14	5715	M-13	M-14	5742	M-13	M-14	5714	M-13	M-14	5739	M-13	M-14						
Al	2.49	2.53	2.50	2.90	2.91	3.04	2.82	2.83	2.92	2.91	2.87	3.09	2.98	2.95	2.97	4.86	4.72	2.98	2.96	2.84	3.11	2.36	2.31						
B	2.66	2.65	2.69	1.33	1.36	1.42	1.36	1.39	1.46	1.34	1.35	1.42	1.37	1.41	1.41	3.22	3.45	1.45	1.32	1.36	1.39	2.38	2.53						
Ca	1.32	1.28	1.02	0.73	0.70	0.72	0.72	0.68	0.69	0.74	0.69	0.74	0.72	0.67	0.69	1.52	1.50	0.70	0.75	0.72	0.77	1.03	1.00						
Cr	0.17	0.16	0.06	0.11	0.09	0.06	0.12	0.10	0.06	0.14	0.12	0.06	0.14	0.12	0.05	0.16	0.16	0.06	0.17	0.17	0.06	0.15	0.14						
Cu	0.00	0.00	-----	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.00	0.00						
Fe	10.10	10.10	9.79	7.51	7.46	7.96	7.19	7.24	7.48	7.33	7.43	8.08	7.46	7.56	7.51	13.91	15.67	7.62	7.26	7.61	7.62	9.60	9.71						
K	2.33	2.31	2.25	-0.04	-0.10	0.01	-0.12	-0.13	0.00	-0.22	-0.03	0.05	-0.21	0.10	0.05	1.92	1.76	0.01	-0.12	0.30	0.08	1.94	2.33						
Li	1.56	1.56	1.49	2.19	2.21	2.26	2.28	2.26	2.34	2.02	2.26	2.35	2.04	2.29	2.30	3.63	3.47	2.33	2.34	2.20	2.31	1.60	1.50						
Mg	0.53	0.53	0.52	0.65	0.63	0.66	0.62	0.61	0.64	0.63	0.63	0.64	0.59	0.60	0.58	1.01	1.04	0.65	0.65	0.67	0.67	0.48	0.50						
Mn	1.46	1.43	1.46	1.47	1.46	1.54	1.43	1.41	1.50	1.46	1.46	1.51	1.41	1.44	1.41	2.52	2.62	1.50	1.49	1.50	1.55	1.33	1.36						
Na	8.92	8.82	8.53	8.34	8.15	6.75	8.16	7.83	7.86	7.98	8.01	8.02	7.78	7.74	7.96	14.95	14.88	8.34	8.55	8.51	8.65	8.47	8.37						
Ni	0.82	0.83	0.83	0.43	0.42	0.46	0.41	0.43	0.44	0.42	0.42	0.46	0.42	0.41	0.43	1.15	1.18	0.43	0.42	0.43	0.43	0.76	0.79						
Si	23.54	23.30	22.39	22.90	23.45	24.08	23.32	23.06	23.92	23.22	23.32	24.18	23.29	23.13	23.17	44.75	46.44	24.16	22.80	23.50	23.68	21.43	22.54						
Ti	0.71	0.71	0.69	0.06	0.05	0.06	0.06	0.05	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.62	0.61	0.06	0.06	0.06	0.06	0.67	0.69						
U	-0.02	-0.28	-----	2.86	2.54	2.53	2.64	2.47	2.40	2.78	2.56	2.52	2.72	2.57	2.47	2.69	2.43	2.46	2.66	2.46	2.53	-0.02	-0.30						
Zr	0.11	0.10	0.10	0.09	0.08	0.07	0.08	0.08	0.09	0.09	0.08	0.09	0.09	0.08	0.09	0.18	0.18	0.09	0.09	0.08	0.09	0.11	0.10						
										rejected MA																			

Table A1. ICP-AES Elemental Measurements for Samples Prepared by Cs<sub>2</sub>CO<sub>3</sub> Fusion, Peroxide Fusion, and Mixed Acid

Run 5			LIMS ID 103992			Batch 359																																			
Weight Percents																																									
	ARG 1			rep 1			6057			rep 2			6015			rep 3			6059			rep 4			6066			rep 5			6058			rep 6			6056			ARG-2	
	M-13	M-14	Known	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14	103992	M-13	M-14						
Al	2.41	2.43	2.50	2.92	2.94	3.02	3.03	2.95	2.92	3.00	3.01	3.10	3.12	3.12	2.74	2.73	2.61	2.86	3.06	3.03	3.00	2.48	2.34																		
B	2.61	2.59	2.69	1.35	1.38	1.44	1.41	1.38	1.48	1.34	1.30	1.43	1.39	1.39	1.35	1.45	1.52	1.71	1.34	1.37	1.44	2.54	2.49																		
Ca	1.01	1.00	1.02	0.74	0.75	0.72	0.77	0.73	0.71	0.76	0.74	0.76	0.79	0.74	0.66	0.64	0.60	0.64	0.80	0.74	0.74	1.06	1.00																		
Cr	0.18	0.15	0.06	0.12	0.09	0.06	0.13	0.09	0.06	0.10	0.08	0.06	0.33	0.26	0.06	0.16	0.13	0.06	0.22	0.18	0.07	0.23	0.18																		
Cu	0.01	-0.01	-----	0.03	0.01	0.02	0.02	0.00	0.02	0.02	0.01	0.02	0.04	0.00	0.02	0.03	0.00	0.01	0.04	0.00	0.01	0.03	-0.01																		
Fe	9.86	9.62	9.79	7.73	7.71	7.93	7.61	7.43	7.86	7.72	7.55	8.32	7.97	8.16	7.55	6.02	6.41	6.85	7.49	7.54	7.93	9.56	9.65																		
K	2.14	1.84	2.25	0.19	-0.39	-0.04	0.13	0.48	-0.05	0.37	-0.09	-0.03	0.14	-0.21	-0.06	0.69	-0.39	-0.03	0.39	-0.11	-0.01	4.19	2.17																		
Li	1.48	1.49	1.49	2.13	2.23	2.24	2.20	2.30	2.19	2.13	2.16	2.16	2.17	2.26	1.94	2.39	2.42	2.61	2.20	2.23	2.19	1.51	1.49																		
Mg	0.53	0.51	0.52	0.71	0.69	0.72	0.71	0.67	0.70	0.71	0.69	0.74	0.70	0.69	0.68	0.55	0.55	0.60	0.72	0.70	0.73	0.52	0.49																		
Mn	1.46	1.43	1.46	1.57	1.60	1.64	1.55	1.54	1.59	1.58	1.58	1.67	1.59	1.57	1.52	1.19	1.23	1.36	1.19	1.11	1.61	1.40	1.41																		
Na	8.44	8.05	8.53	8.23	8.15	8.79	8.28	7.96	8.66	8.35	8.08	8.94	8.54	8.16	8.49	7.87	6.94	7.87	8.76	7.98	8.86	8.95	7.32																		
Ni	0.82	0.82	0.83	0.45	0.42	0.46	0.46	0.40	0.45	0.45	0.42	0.47	0.45	0.42	0.43	0.36	0.32	0.39	0.42	0.39	0.45	0.85	0.77																		
Si	23.37	22.93	22.39	24.14	23.57	24.84	23.96	22.89	25.01	21.97	21.68	24.33	23.20	22.96	22.96	25.22	25.88	29.02	23.56	24.51	24.95	22.71	23.13																		
Ti	0.69	0.70	0.69	0.06	0.05	0.06	0.07	0.05	0.06	0.06	0.05	0.06	0.07	0.05	0.06	0.07	0.05	0.07	0.07	0.05	0.06	0.68	0.68																		
U	0.17	-0.42	-----	2.82	2.50	2.49	2.80	2.58	2.38	3.07	2.79	2.59	3.13	2.55	2.28	2.45	1.74	2.06	3.14	2.44	2.40	0.53	-0.42																		
Zr	0.11	0.10	0.10	0.10	0.09	0.10	0.09	0.08	0.10	0.09	0.08	0.10	0.10	0.09	0.10	0.10	0.09	0.10	0.08	0.06	0.10	0.12	0.10																		
													rejected FS											rejected FS & MA																	
Run 6			LIMS ID 104369			Batch 364																																			
Weight Percents																																									
	ARG 1			rep 1			6914			rep 2			6920			rep 3			6926			rep 4			6917			rep 5			6911			rep 6			6923			ARG-2	
	M-13	M-14	Known	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14	104369	M-13	M-14						
Al	2.42	2.36	2.50	3.05	2.94	2.99	3.21	3.01	3.01	3.21	3.12	3.00	3.25	3.03	3.01	3.20	2.99	3.00	3.24	2.98	2.96	2.48	2.29																		
B	2.58	2.52	2.69	1.38	1.38	1.34	1.41	1.36	1.36	1.41	1.37	1.39	1.38	1.32	1.33	1.43	1.40	1.39	1.37	1.35	1.33	2.58	2.50																		
Ca	1.05	1.00	1.02	0.75	0.72	0.70	0.76	0.72	0.69	0.80	0.76	0.76	0.82	0.76	0.73	0.77	0.73	0.73	0.80	0.76	0.73	1.03	1.01																		
Cr	0.15	0.12	0.06	0.15	0.12	0.05	0.15	0.13	0.06	0.18	0.15	0.06	0.17	0.14	0.06	0.16	0.12	0.06	0.15	0.11	0.06	0.20	0.17																		
Cu	0.01	-0.01	-----	0.03	0.01	0.02	0.03	0.01	0.02	0.03	0.01	0.02	0.05	0.02	0.02	0.03	0.01	0.02	0.04	0.01	0.02	0.03	-0.01																		
Fe	9.80	9.55	9.79	7.78	7.90	7.69	8.21	8.24	7.87	8.16	7.91	7.83	8.21	8.43	8.00	7.97	7.91	7.88	8.04	7.95	7.62	9.60	9.59																		
K	2.00	2.09	2.25	0.05	-0.11	0.09	0.15	-0.18	0.09	0.41	-0.12	0.09	0.36	-0.27	0.09	0.20	-0.29	0.09	0.31	-0.28	0.13	2.44	1.82																		
Li	1.43	1.52	1.49	2.07	2.19	2.27	2.08	2.23	2.29	2.14	2.18	2.29	2.05	2.16	2.20	2.12	2.30	2.29	1.95	2.14	2.28	1.42	1.46																		
Mg	0.52	0.51	0.52	0.66	0.68	0.65	0.68	0.67	0.64	0.72	0.72	0.69	0.70	0.71	0.67	0.71	0.70	0.67	0.73	0.73	0.67	0.51	0.55																		
Mn	1.43	1.42	1.46	1.55	1.51	1.48	1.53	1.45	1.51	1.65	1.59	1.59	1.54	1.54	1.55	1.59	1.57	1.57	1.60	1.59	1.55	1.41	1.39																		
Na	8.36	8.64	8.53	8.29	8.35	8.34	8.49	8.48	8.52	8.93	8.94	8.84	8.79	8.68	8.86	8.81	8.57	8.59	9.11	8.84	8.83	8.32	8.25																		
Ni	0.84	0.78	0.83	0.45	0.42	0.41	0.49	0.46	0.44	0.47	0.43	0.44	0.51	0.46	0.44	0.47	0.43	0.44	0.49	0.46	0.43	0.84	0.78																		
Si	22.84	21.67	22.39	23.27	23.28	23.08	24.38	23.91	23.78	24.01	23.86	23.07	23.82	23.98	22.96	24.83	23.83	23.98	23.99	23.92	22.63	22.96	22.79																		
Ti	0.69	0.67	0.69	0.05	0.04	0.05	0.05	0.04	0.05	0.05	0.04	0.05	0.05	0.04	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.66	0.66																		
U	0.32	-0.13	-----	3.24	2.80	2.54	3.43	2.87	2.61	3.34	2.87	2.64	3.65	2.89	2.65	3.46	2.83	2.63	3.51	2.75	2.58	0.62	-0.11																		
Zr	0.11	0.10	0.10	0.09	0.08	0.09	0.10	0.08	0.09	0.09	0.08	0.09	0.10	0.08	0.10	0.10	0.08	0.09	0.10	0.08	0.09	0.11	0.10																		



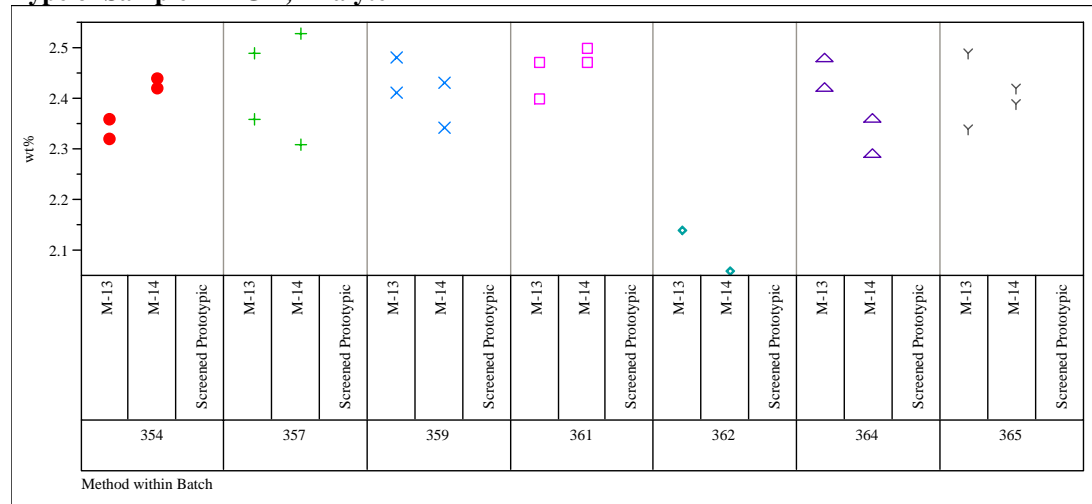
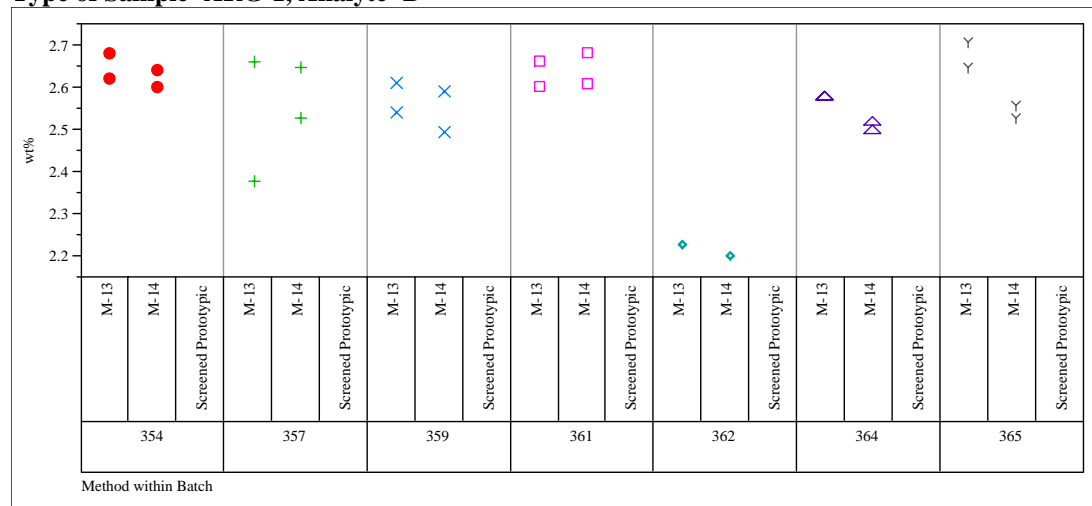
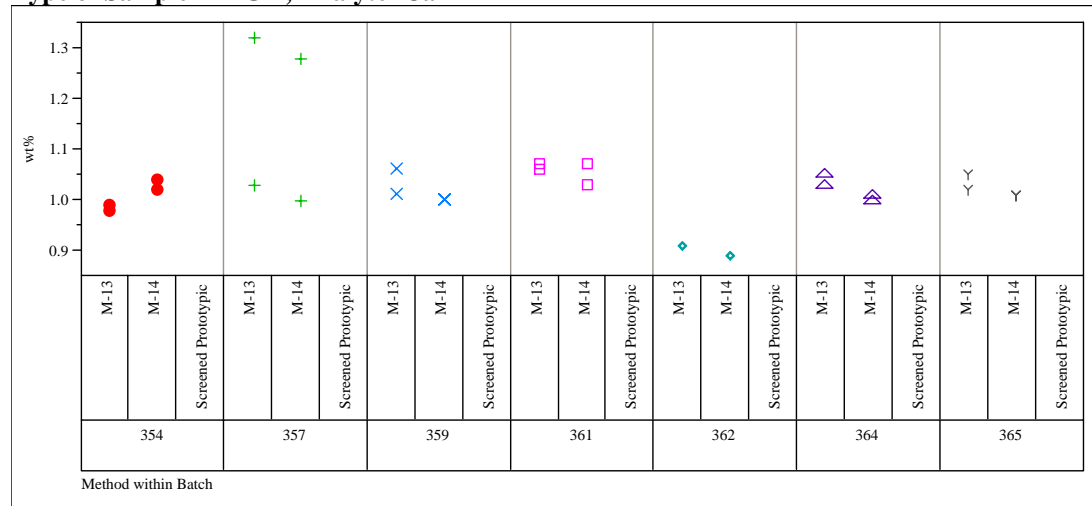
Table A1. ICP-AES Elemental Measurements for Samples Prepared by Cs<sub>2</sub>CO<sub>3</sub> Fusion, Peroxide Fusion, and Mixed Acid

Run 7 LIMS ID 104455      Batch 365

Weight Percents

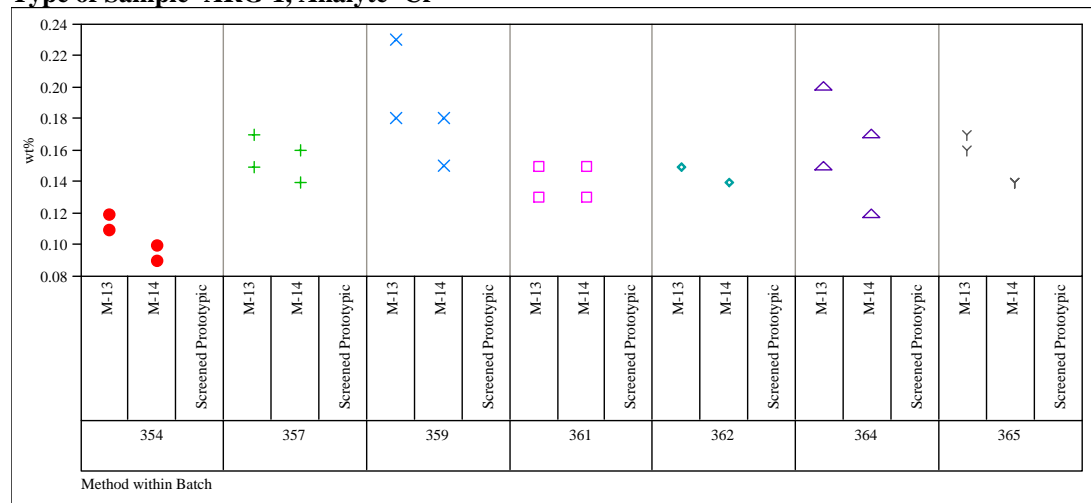
	ARG 1			rep 1			rep 2			rep 3			rep 4			rep 5			rep 6			ARG-2	
	M-13	M-14	Known	M-13	M-14	104455	M-13	M-14	104455	M-13	M-14	104455	M-13	M-14	104455	M-13	M-14	104455	M-13	M-14	104455	M-13	M-14
Al	2.34	2.42	2.50	2.59	2.63	3.12	3.24	3.32	3.52	3.28	3.26	3.22	3.24	3.22	3.16	3.40	3.32	3.26	3.20	3.14	3.15	2.49	2.39
B	2.65	2.53	2.69	1.16	1.10	1.27	1.37	1.37	1.44	1.33	1.29	1.38	1.35	1.32	1.30	1.39	1.33	1.39	1.40	1.33	1.35	2.71	2.56
Ca	1.02	1.01	1.02	0.68	0.65	0.70	0.83	0.82	0.90	0.84	0.82	0.80	0.86	0.82	0.82	0.88	0.87	0.77	0.78	0.75	0.78	1.05	1.01
Cr	0.16	0.14	0.06	0.11	0.09	0.05	0.12	0.10	0.06	0.12	0.10	0.06	0.11	0.09	0.08	0.13	0.10	0.05	0.15	0.12	0.06	0.17	0.14
Cu	0.00	-0.01	-----	0.01	0.00	0.01	0.02	0.01	0.02	0.02	0.01	0.02	0.02	0.01	0.01	0.02	0.00	0.02	0.03	0.01	0.01	0.01	-0.01
Fe	9.82	9.70	9.79	7.20	6.94	8.14	8.78	8.44	9.05	9.03	8.70	8.47	8.81	8.43	8.61	9.39	8.84	8.70	8.95	8.47	8.43	10.35	9.79
K	2.12	2.11	2.25	0.06	-0.04	-0.19	0.21	0.03	-0.06	0.19	-0.20	-0.29	0.39	-0.16	-0.05	0.24	0.05	-0.23	0.19	-0.16	-0.02	2.28	2.21
Li	1.45	1.47	1.49	1.80	1.83	2.01	2.05	2.14	2.45	2.03	2.14	2.18	2.08	2.16	2.10	2.10	2.17	2.14	1.99	2.19	2.09	1.45	1.51
Mg	0.52	0.52	0.52	0.65	0.64	0.73	0.82	0.78	0.83	0.81	0.79	0.76	0.83	0.78	0.76	0.88	0.83	0.78	0.78	0.75	0.73	0.55	0.51
Mn	1.42	1.40	1.46	1.40	1.37	1.68	1.42	1.34	1.80	1.39	1.26	1.77	1.59	1.44	1.73	1.55	1.38	1.77	1.60	1.52	1.72	1.36	1.26
Na	8.31	8.22	8.53	7.43	7.39	8.82	8.86	9.14	8.68	8.99	8.99	8.83	9.12	9.16	8.75	9.55	9.53	9.07	8.67	8.61	8.48	8.60	8.52
Ni	0.82	0.78	0.83	0.40	0.36	0.45	0.48	0.45	0.50	0.48	0.46	0.47	0.48	0.45	0.50	0.52	0.43	0.49	0.52	0.45	0.48	0.88	0.81
Si	22.99	22.21	22.39	19.49	19.04	21.71	23.59	22.11	25.02	23.11	22.57	23.58	23.78	22.11	21.70	24.53	23.32	22.96	24.06	23.20	22.95	23.92	23.28
Ti	0.69	0.67	0.69	0.05	0.04	0.04	0.06	0.05	0.05	0.06	0.04	0.05	0.06	0.05	0.05	0.06	0.04	0.05	0.06	0.04	0.05	0.68	0.69
U	-0.09	-0.28	-----	2.59	2.32	2.89	3.38	3.25	3.31	3.32	3.24	2.85	3.39	3.12	3.03	3.56	3.21	3.02	3.33	3.01	2.93	0.30	-0.30
Zr	0.11	0.09	0.10	0.07	0.06	0.09	0.07	0.06	0.10	0.07	0.05	0.09	0.08	0.06	0.09	0.08	0.06	0.09	0.09	0.07	0.09	0.11	0.09

rejected  
FS & MA

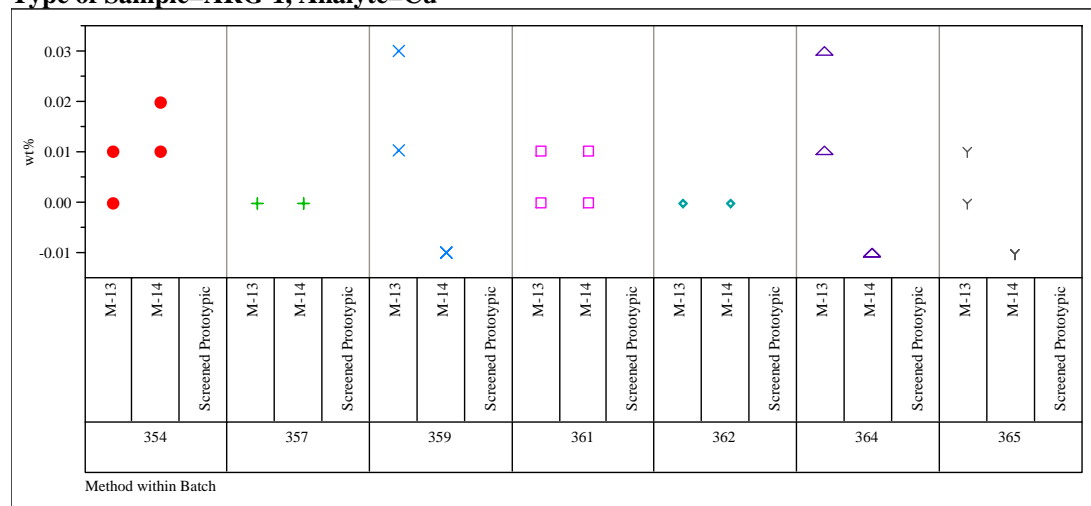
**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=ARG-1, Analyte=Al****Type of Sample=ARG-1, Analyte=B****Type of Sample=ARG-1, Analyte=Ca**

**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element**

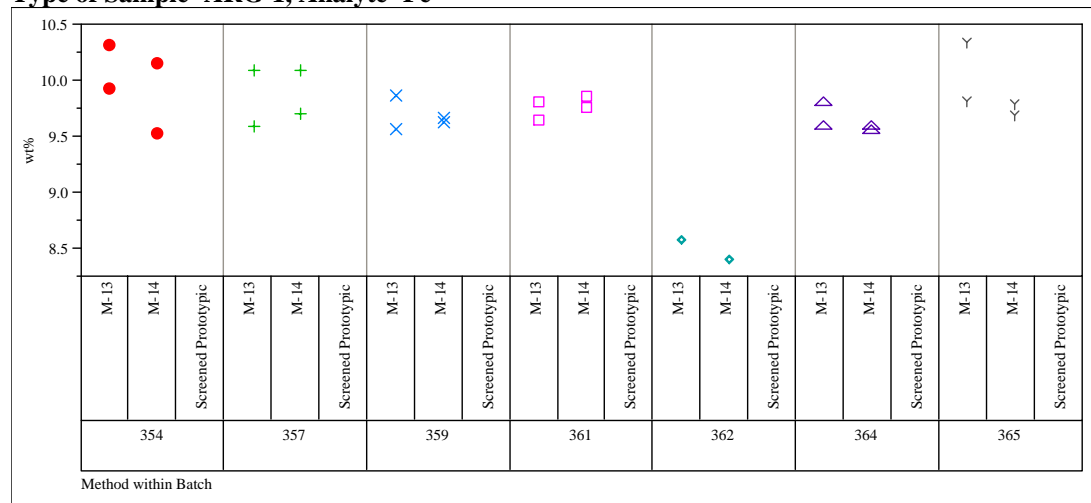
**Type of Sample=ARG-1, Analyte=Cr**

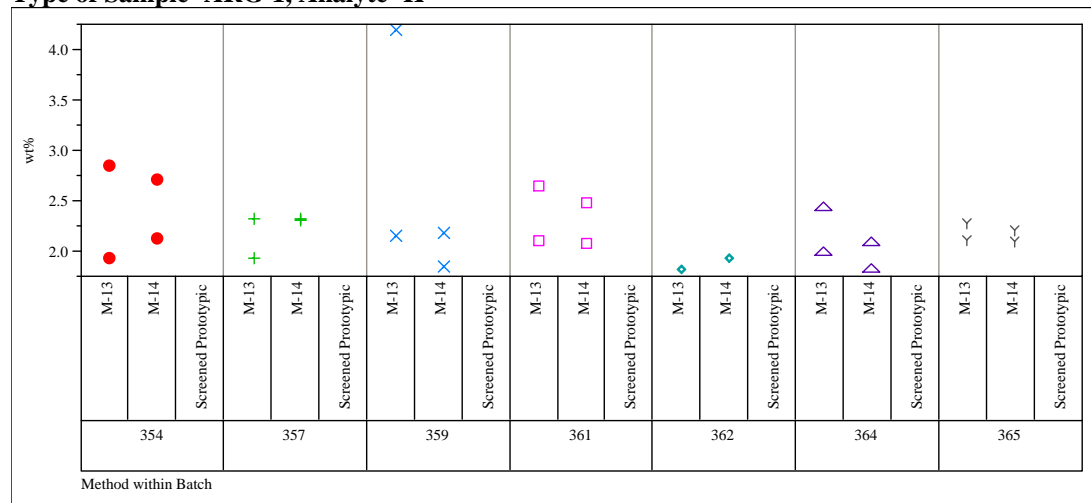
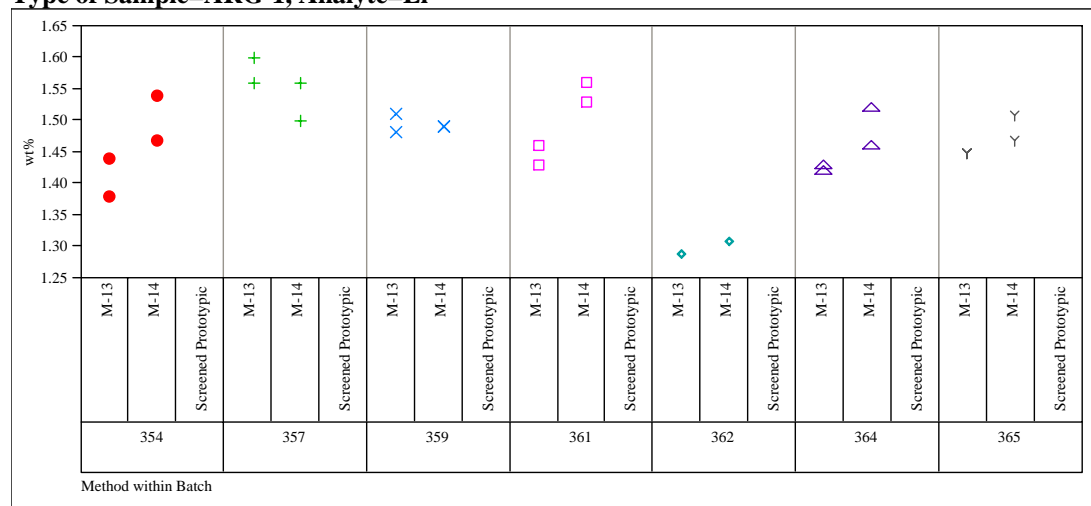
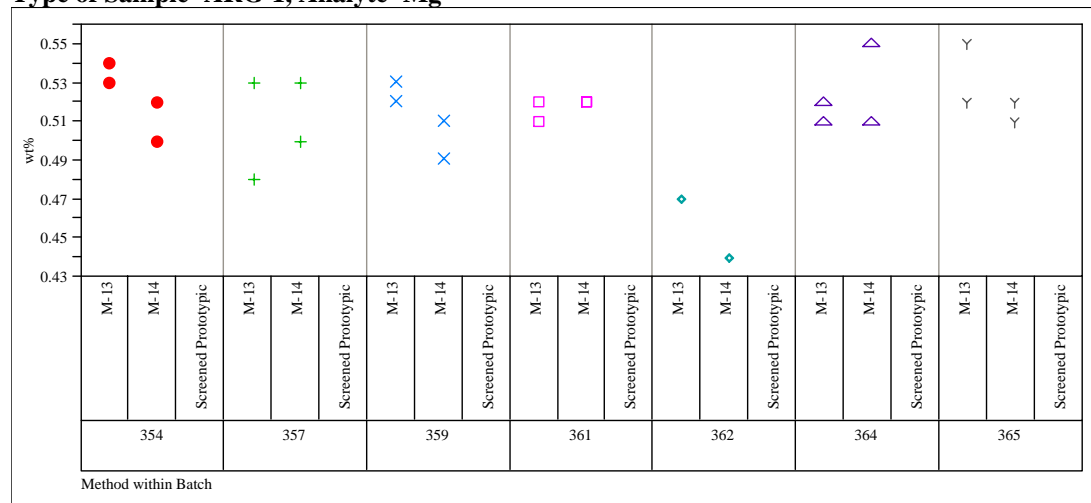


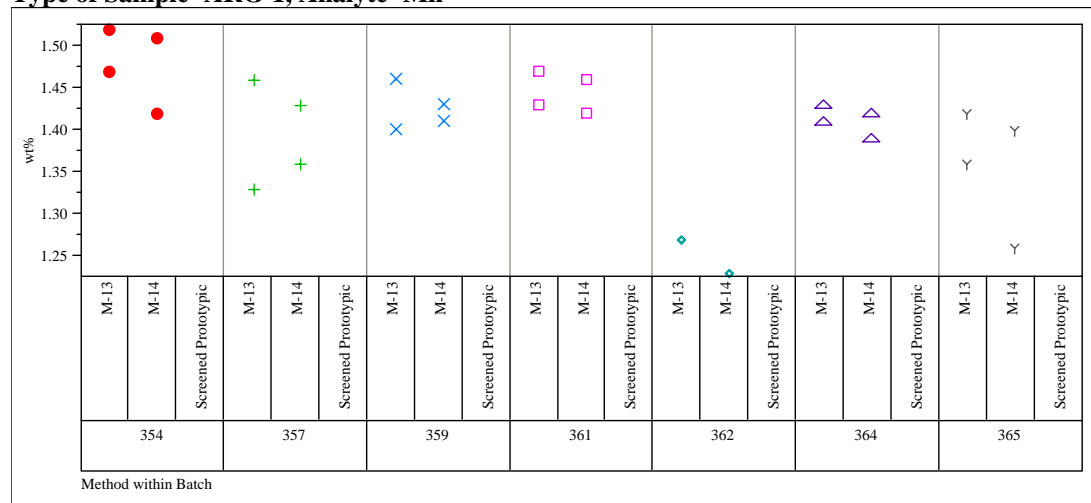
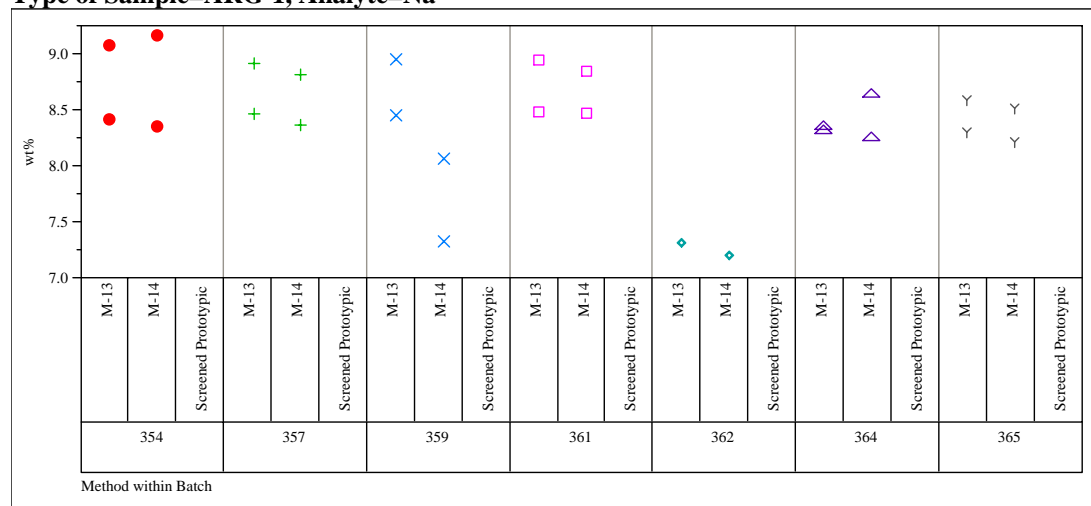
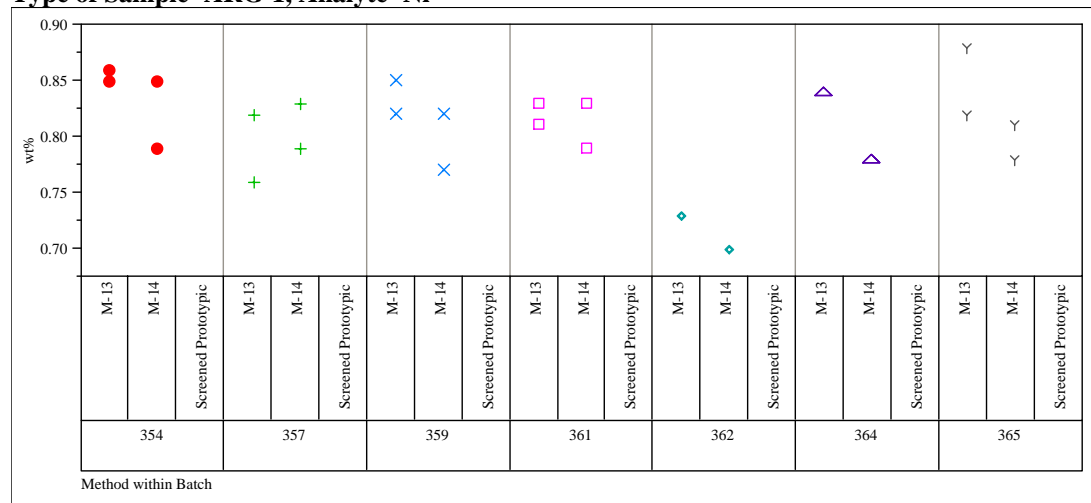
**Type of Sample=ARG-1, Analyte=Cu**



**Type of Sample=ARG-1, Analyte=Fe**

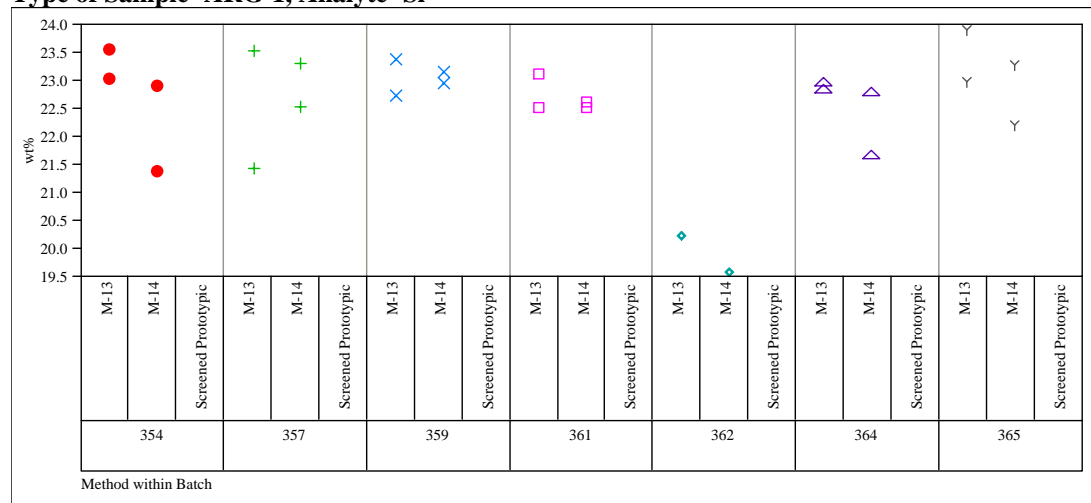


**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=ARG-1, Analyte=K****Type of Sample=ARG-1, Analyte=Li****Type of Sample=ARG-1, Analyte=Mg**

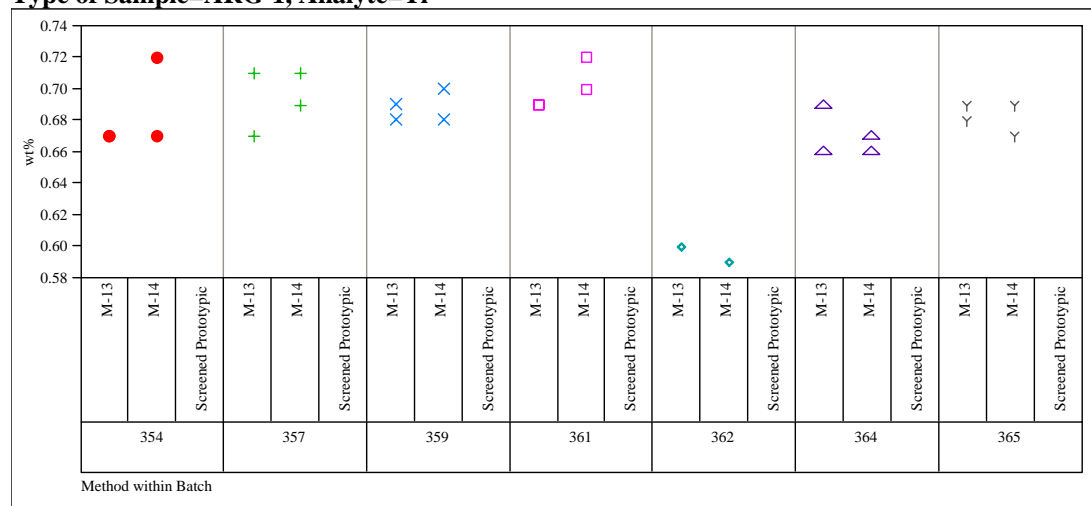
**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=ARG-1, Analyte=Mn****Type of Sample=ARG-1, Analyte=Na****Type of Sample=ARG-1, Analyte=Ni**

**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element**

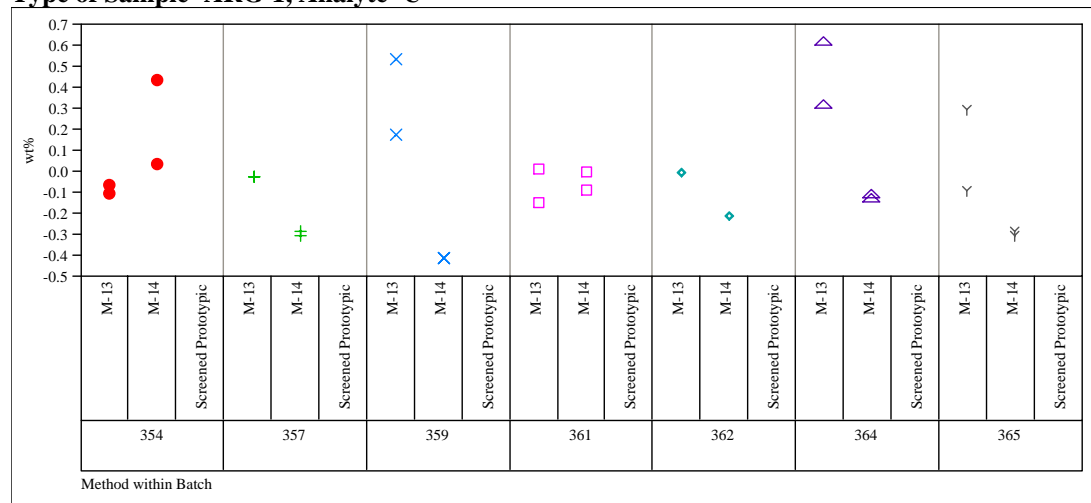
**Type of Sample=ARG-1, Analyte=Si**



**Type of Sample=ARG-1, Analyte=Ti**

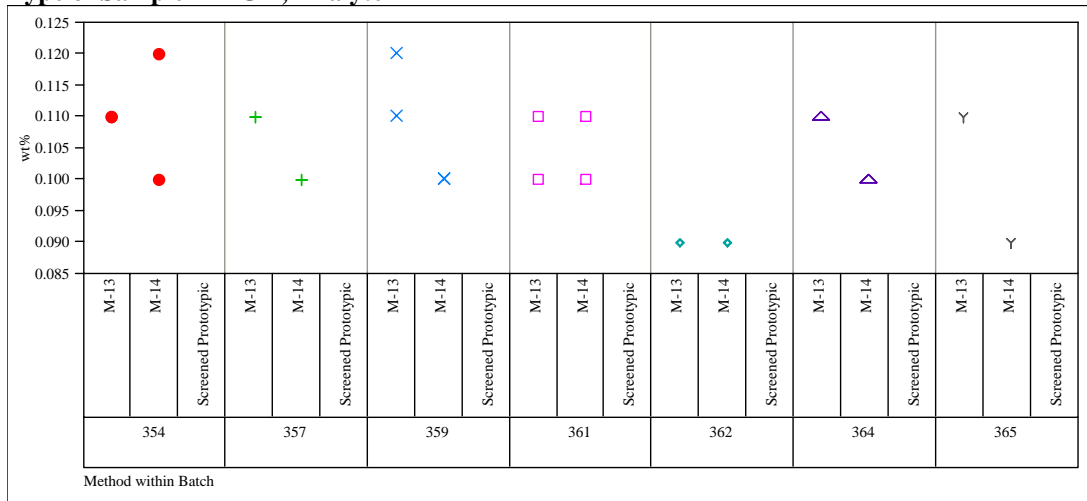


**Type of Sample=ARG-1, Analyte=U**

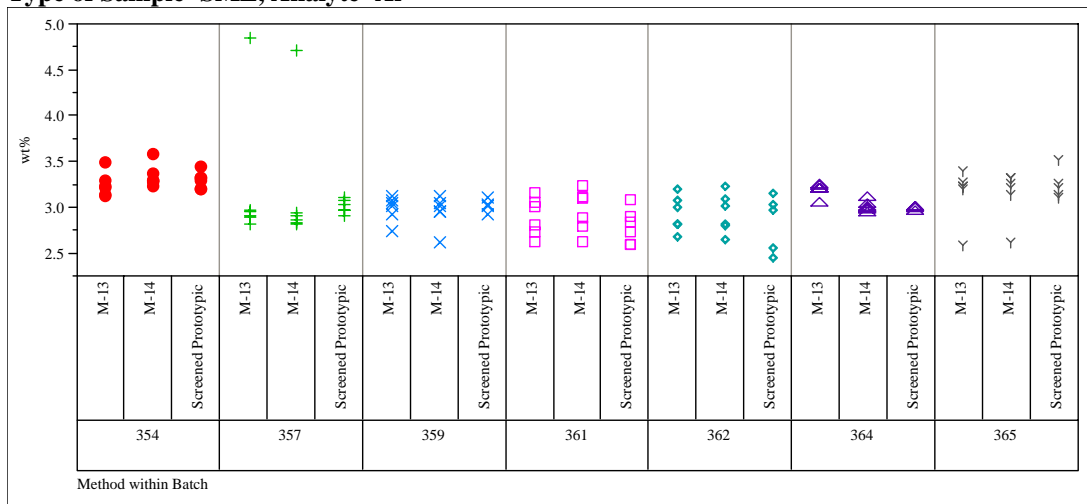


**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element**

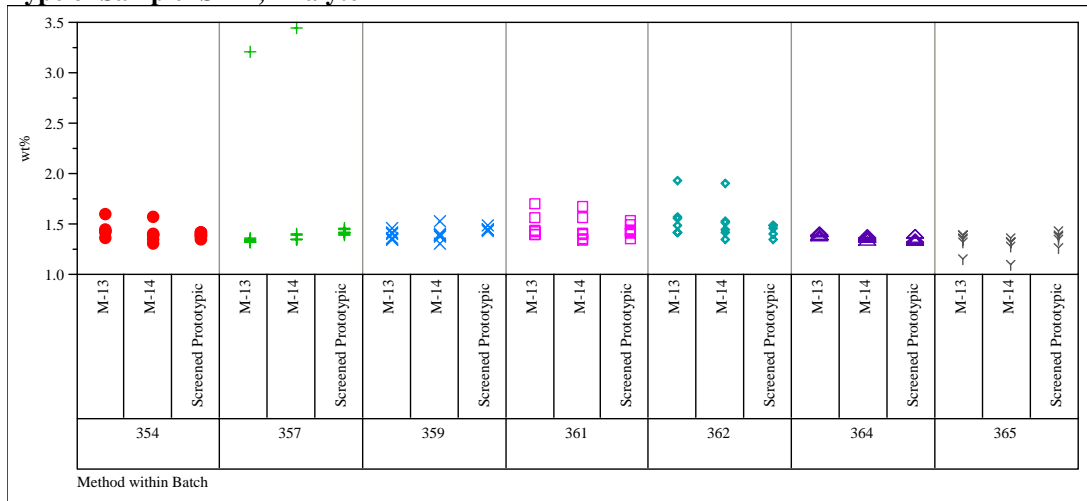
**Type of Sample=ARG-1, Analyte=Zr**

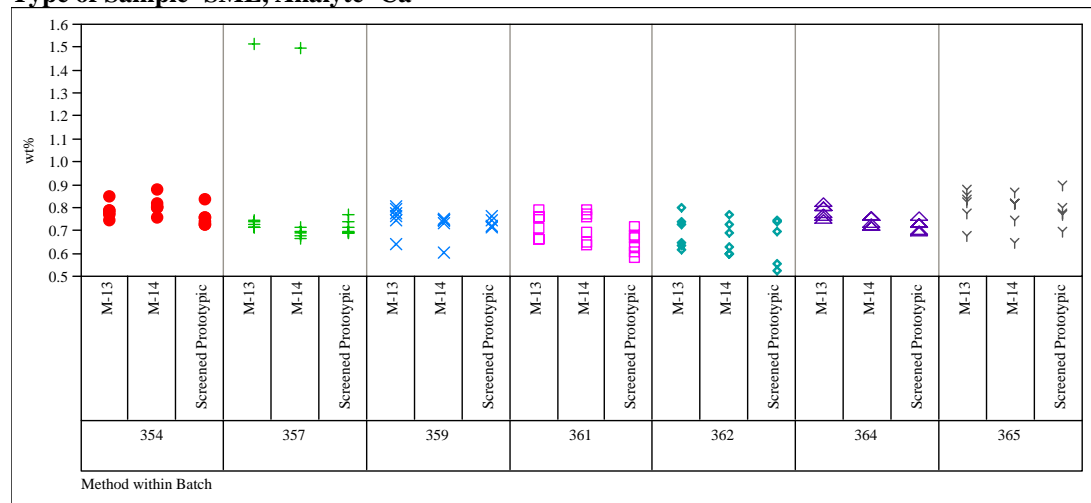
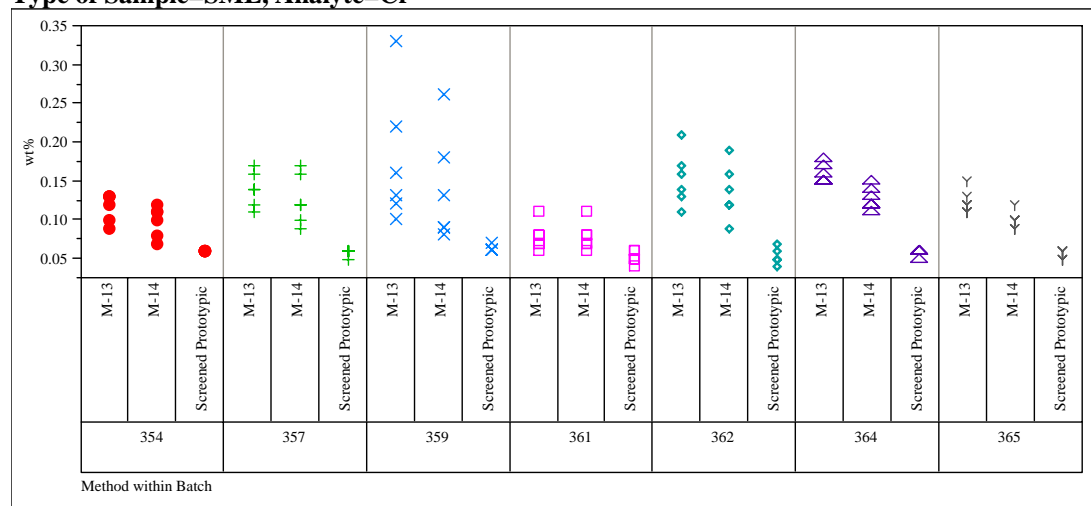
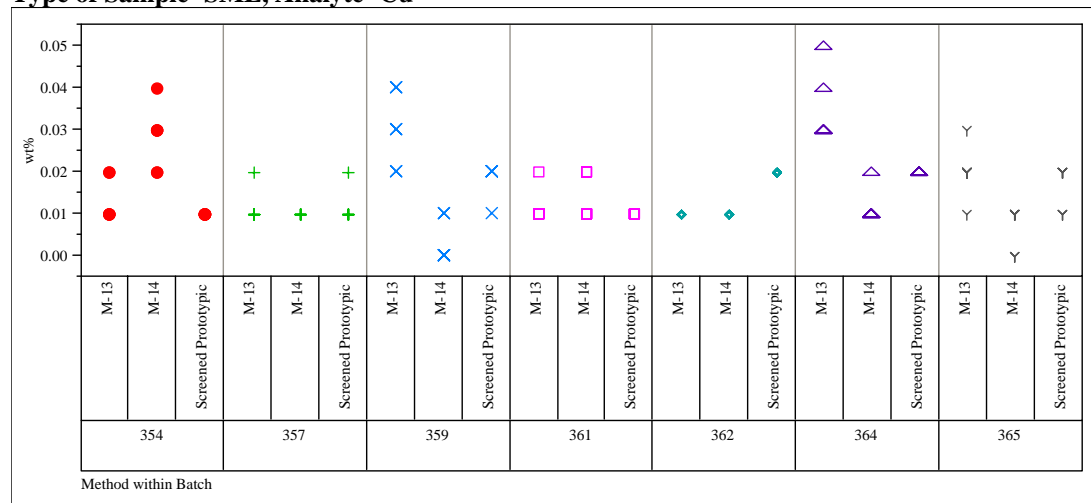


**Type of Sample=SME, Analyte=Al**

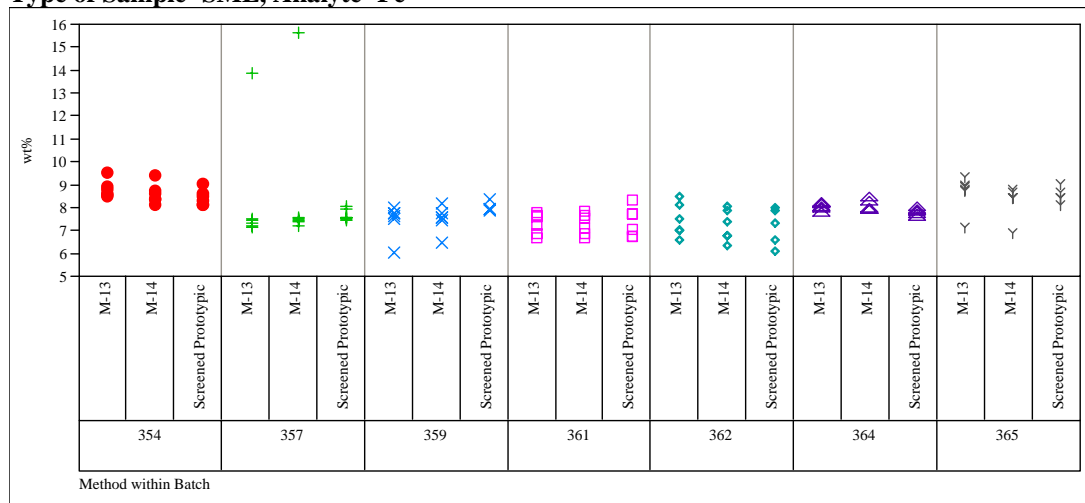
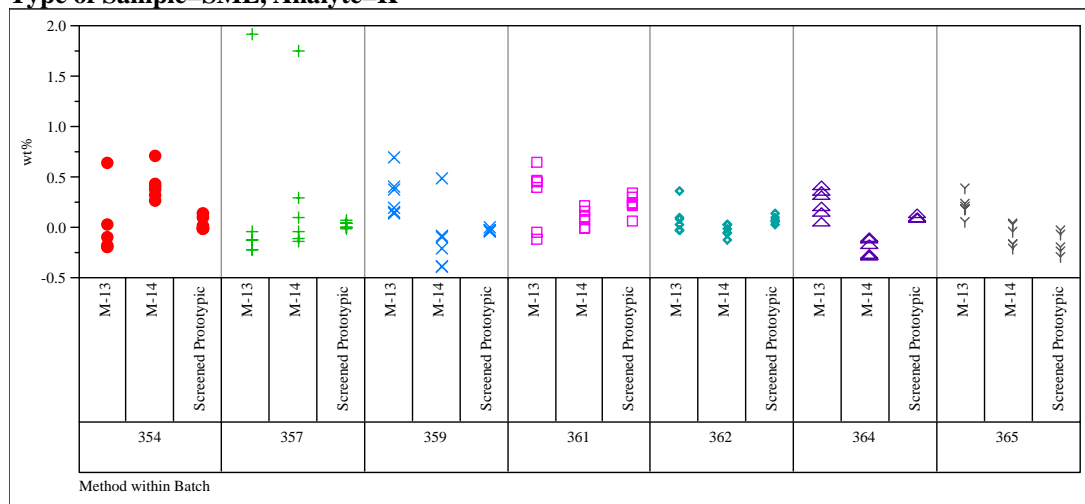
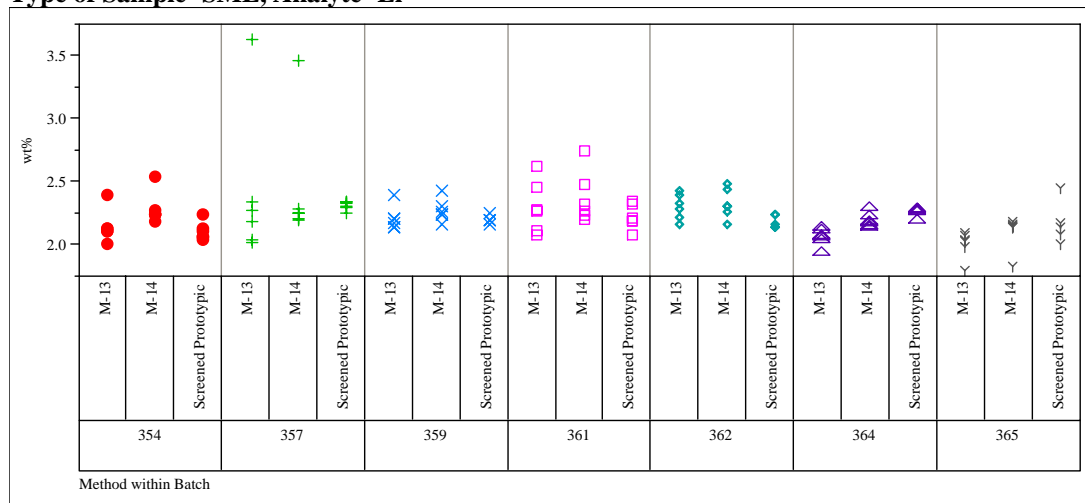


**Type of Sample=SME, Analyte=B**



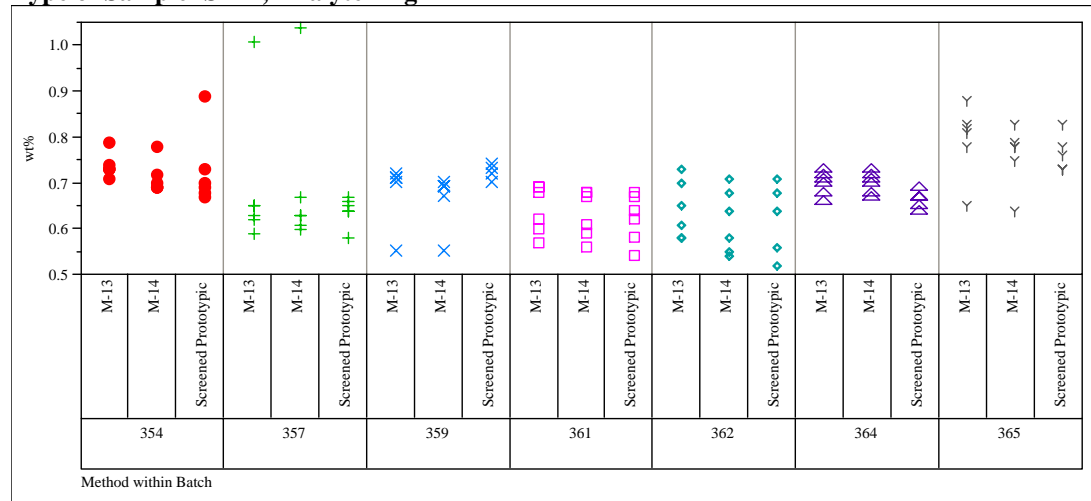
**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=SME, Analyte=Ca****Type of Sample=SME, Analyte=Cr****Type of Sample=SME, Analyte=Cu**



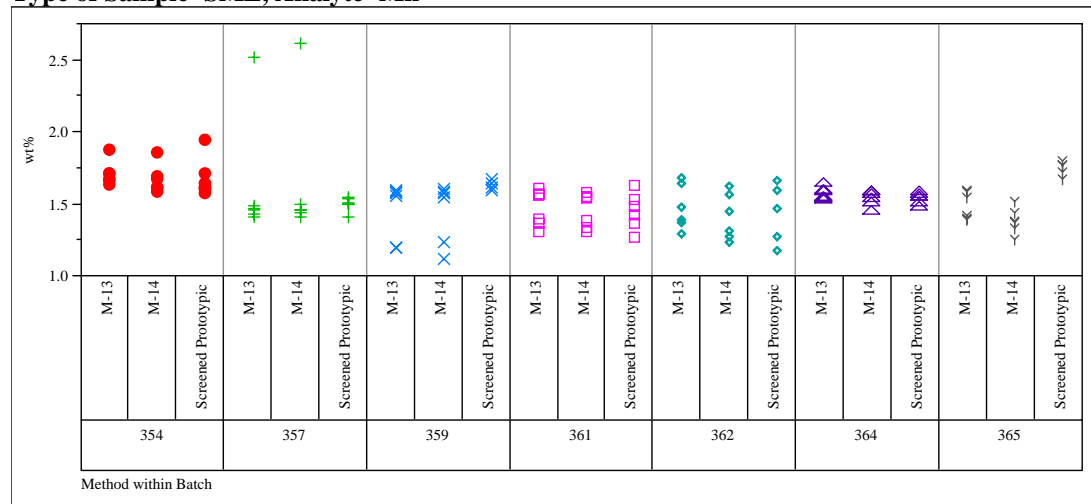
**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=SME, Analyte=Fe****Type of Sample=SME, Analyte=K****Type of Sample=SME, Analyte=Li**

**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element**

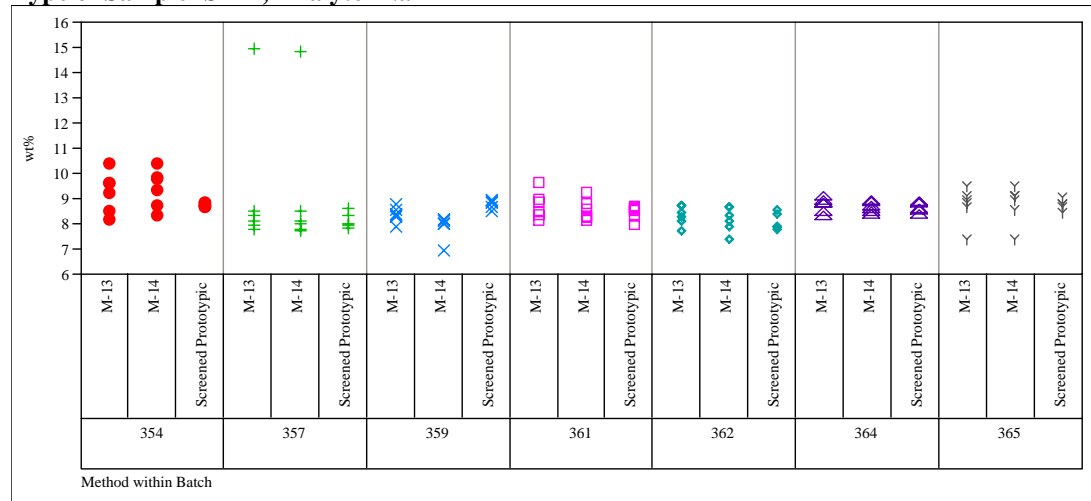
**Type of Sample=SME, Analyte=Mg**

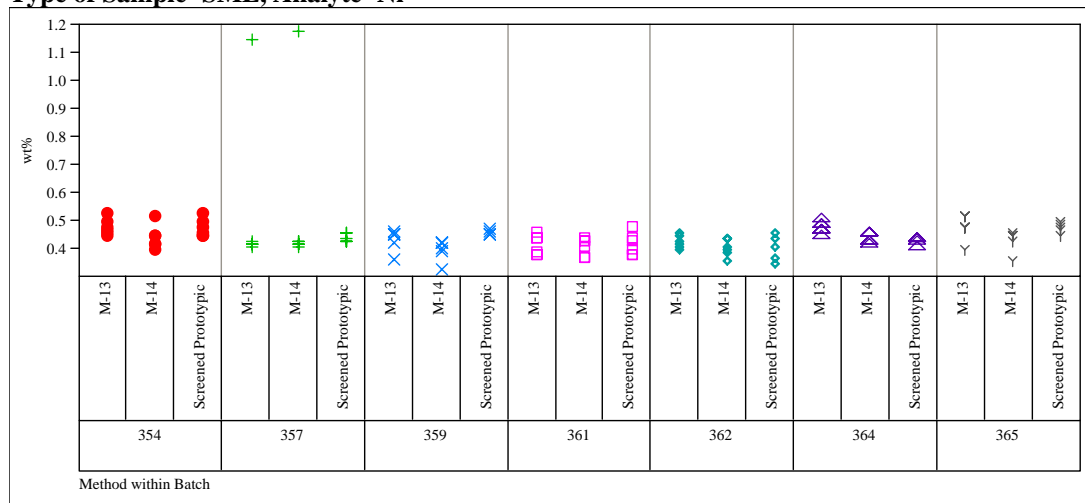
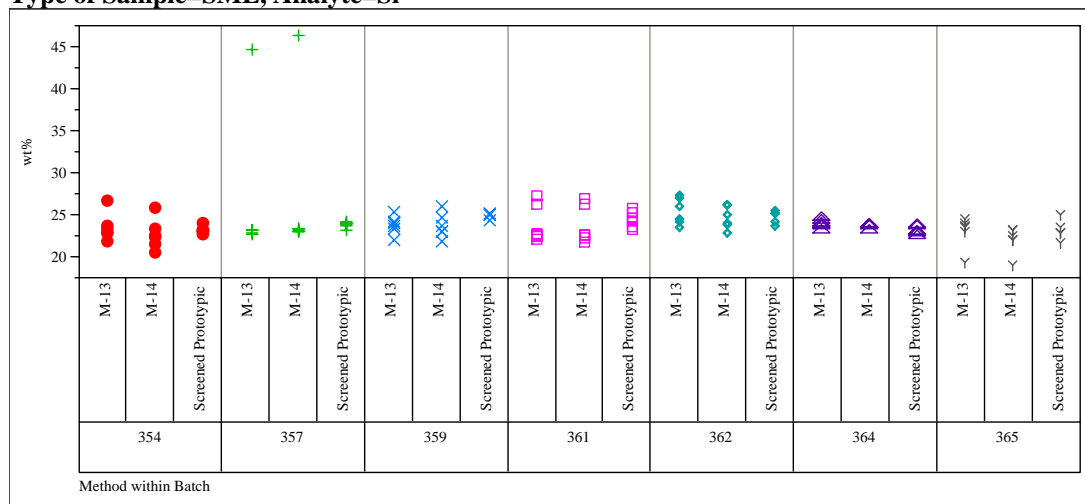
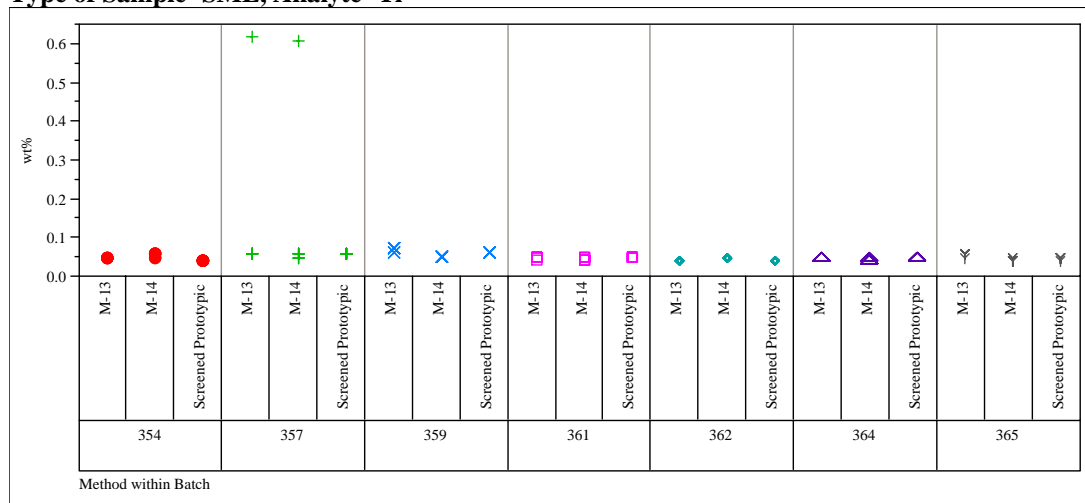


**Type of Sample=SME, Analyte=Mn**



**Type of Sample=SME, Analyte=Na**



**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=SME, Analyte=Ni****Type of Sample=SME, Analyte=Si****Type of Sample=SME, Analyte=Ti**

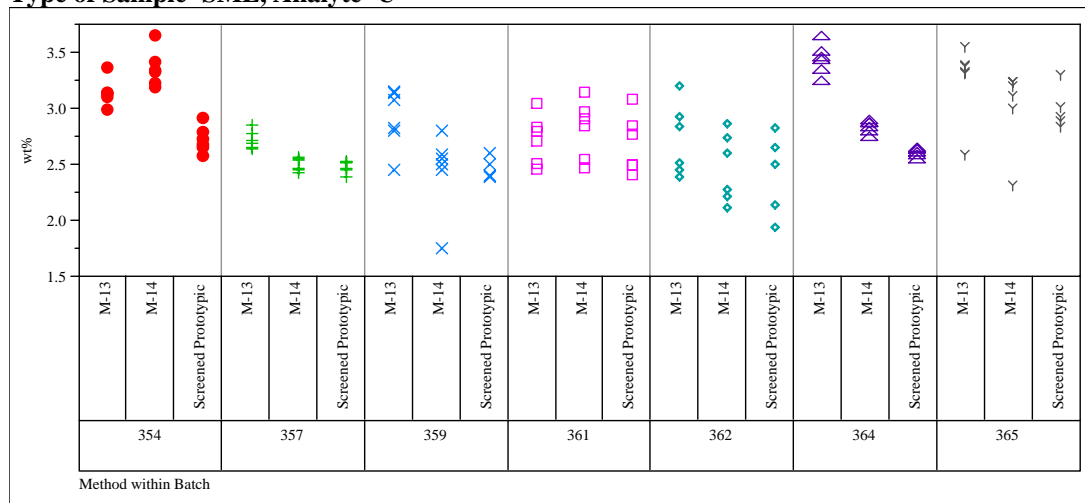
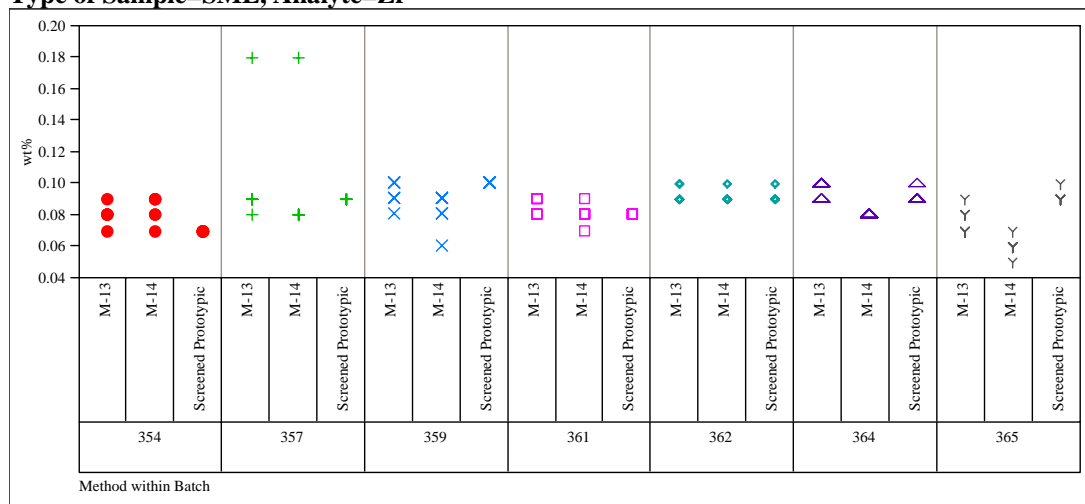
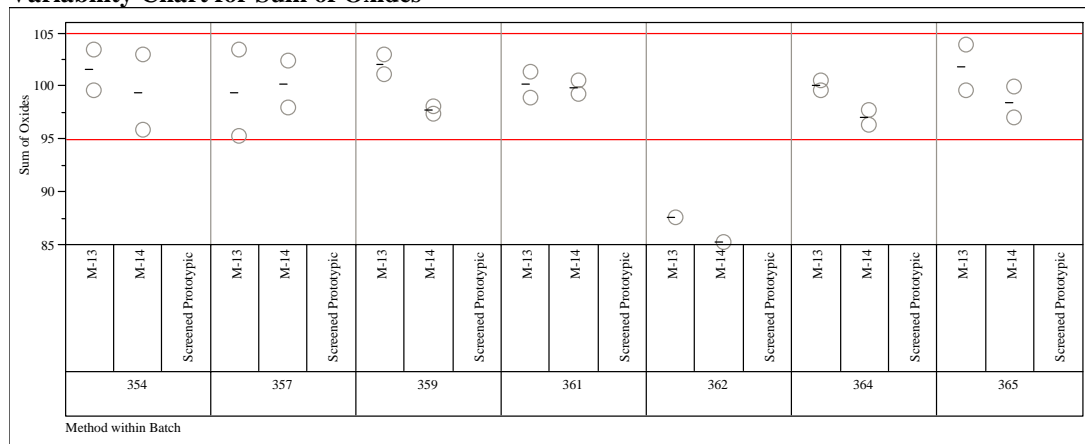
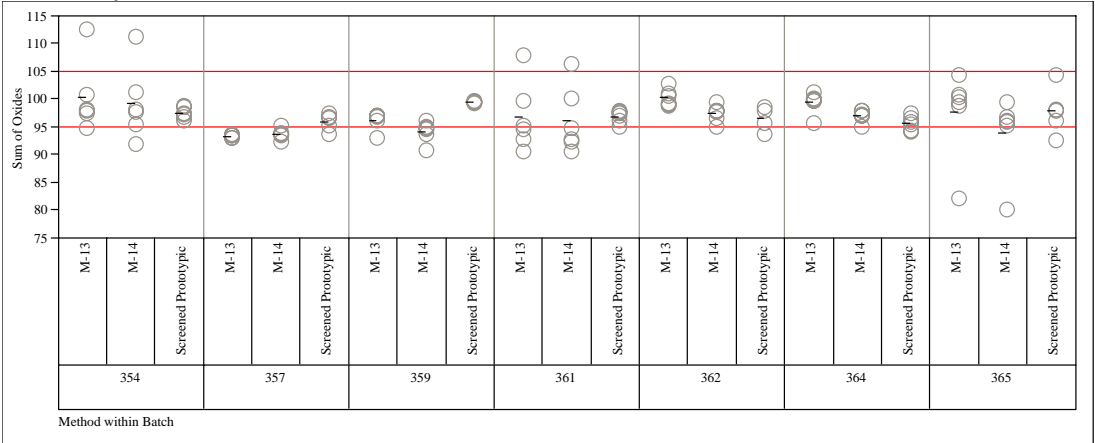
**Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element****Type of Sample=SME, Analyte=U****Type of Sample=SME, Analyte=Zr****Type of Sample=ARG-1****Variability Chart for Sum of Oxides**

Exhibit A1. Initial Plots of Measurements by Type of Sample and by Element

Type of Sample=SME  
Variability Chart for Sum of Oxides

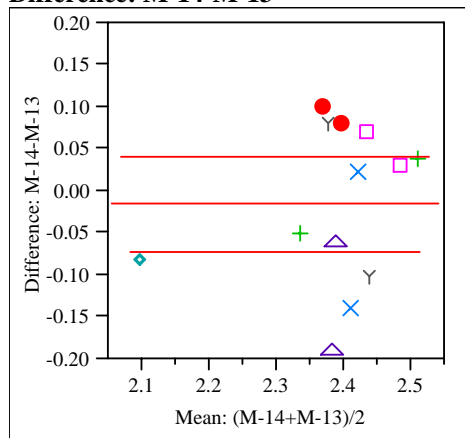


**Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample**

**Type/ Analyte/UoM=ARG-1/Al/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

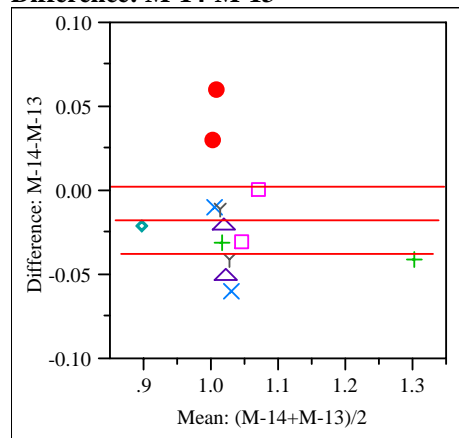


M-14	2.38154	t-Ratio	-0.59097
M-13	2.39692	DF	12
Mean Difference	-0.0154	Prob >  t	0.5655
Std Error	0.02603	Prob > t	0.7172
Upper95%	0.04134	Prob < t	0.2828
Lower95%	-0.0721		
N	13		
Correlation	0.64414		

**Type/ Analyte/UoM=ARG-1/Ca/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

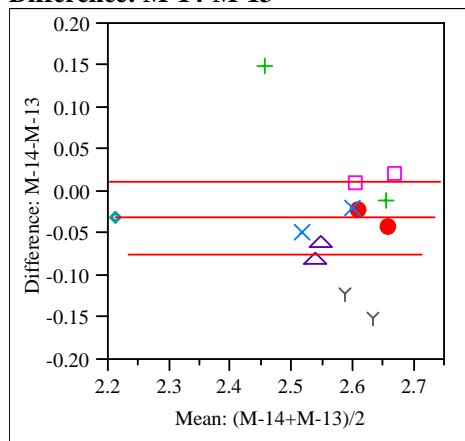


M-14	1.02769	t-Ratio	-1.86267
M-13	1.04462	DF	12
Mean Difference	-0.0169	Prob >  t	0.0872
Std Error	0.00909	Prob > t	0.9564
Upper95%	0.00287	Prob < t	0.0436
Lower95%	-0.0367		
N	13		
Correlation	0.93651		

**Type/ Analyte/UoM=ARG-1/B/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

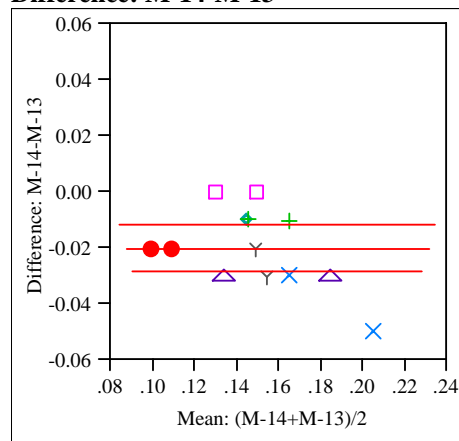


M-14	2.54615	t-Ratio	-1.52517
M-13	2.57692	DF	12
Mean Difference	-0.0308	Prob >  t	0.1531
Std Error	0.02017	Prob > t	0.9234
Upper95%	0.01319	Prob < t	0.0766
Lower95%	-0.0747		
N	13		
Correlation	0.83906		

**Type/ Analyte/UoM=ARG-1/Cr/wt%**

**Matched Pairs**

**Difference: M-14-M-13**



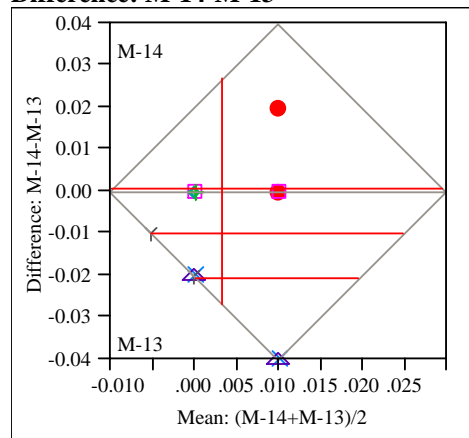
M-14	0.13923	t-Ratio	-5.09902
M-13	0.15923	DF	12
Mean Difference	-0.02	Prob >  t	0.0003
Std Error	0.00392	Prob > t	0.9999
Upper95%	-0.0115	Prob < t	0.0001
Lower95%	-0.0285		
N	13		
Correlation	0.90712		

# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=ARG-1/Cu/wt%

Matched Pairs

Difference: M-14-M-13

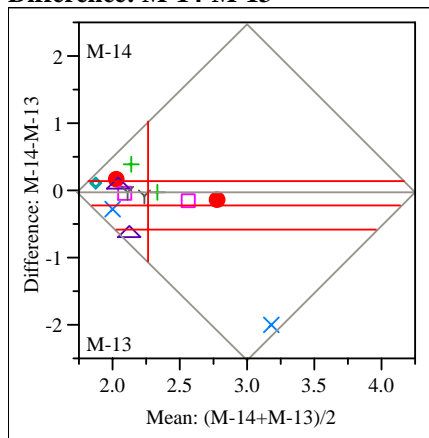


M-14	-0.0015	t-Ratio	-2.08167
M-13	0.00846	DF	12
Mean Difference	-0.01	Prob >  t	0.0594
Std Error	0.0048	Prob > t	0.9703
Upper95%	0.00047	Prob < t	0.0297
Lower95%	-0.0205		
N	13		
Correlation	-0.4195		

Type/ Analyte/UoM=ARG-1/K/wt%

Matched Pairs

Difference: M-14-M-13

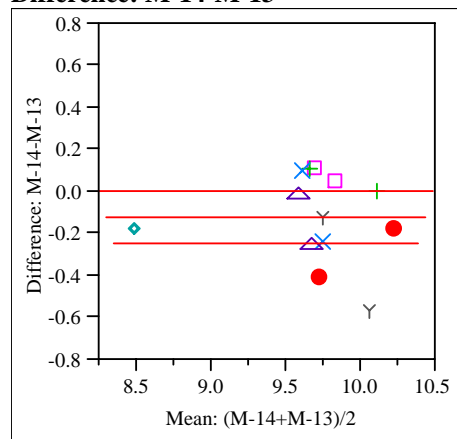


M-14	2.17077	t-Ratio	-1.18988
M-13	2.36846	DF	12
Mean Difference	-0.1977	Prob >  t	0.2571
Std Error	0.16614	Prob > t	0.8714
Upper95%	0.16431	Prob < t	0.1286
Lower95%	-0.5597		
N	13		
Correlation	0.28768		

Type/ Analyte/UoM=ARG-1/Fe/wt%

Matched Pairs

Difference: M-14-M-13

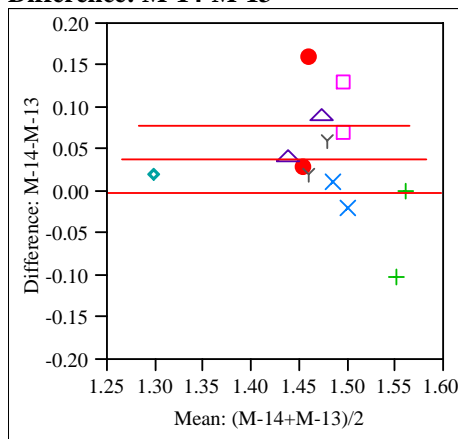


M-14	9.64692	t-Ratio	-2.09137
M-13	9.76692	DF	12
Mean Difference	-0.12	Prob >  t	0.0584
Std Error	0.05738	Prob > t	0.9708
Upper95%	0.00502	Prob < t	0.0292
Lower95%	-0.245		
N	13		
Correlation	0.88492		

Type/ Analyte/UoM=ARG-1/Li/wt%

Matched Pairs

Difference: M-14-M-13



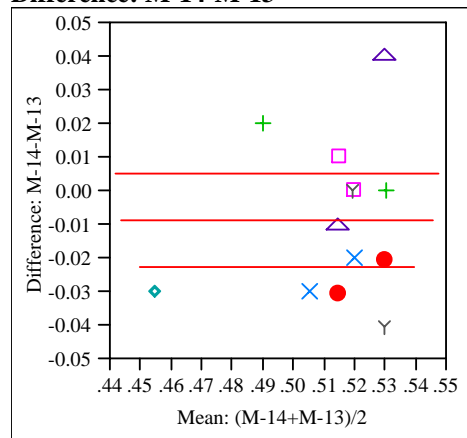
M-14	1.49308	t-Ratio	2.130556
M-13	1.45385	DF	12
Mean Difference	0.03923	Prob >  t	0.0545
Std Error	0.01841	Prob > t	0.0273
Upper95%	0.07935	Prob < t	0.9727
Lower95%	-0.0009		
N	13		
Correlation	0.57098		

# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=ARG-1/Mg/wt%

Matched Pairs

Difference: M-14-M-13

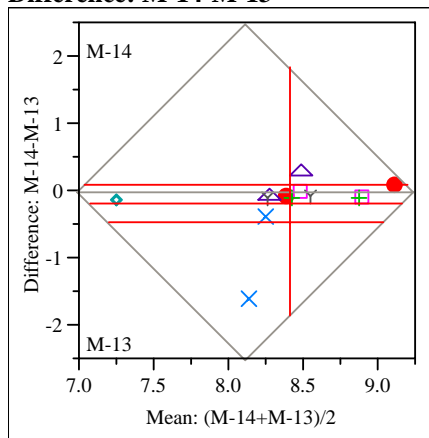


M-14	0.50923	t-Ratio	-1.32424
M-13	0.51769	DF	12
Mean Difference	-0.0085	Prob >  t	0.2101
Std Error	0.00639	Prob > t	0.8950
Upper95%	0.00546	Prob < t	0.1050
Lower95%	-0.0224		
N	13		
Correlation	0.54203		

Type/ Analyte/UoM=ARG-1/Na/wt%

Matched Pairs

Difference: M-14-M-13

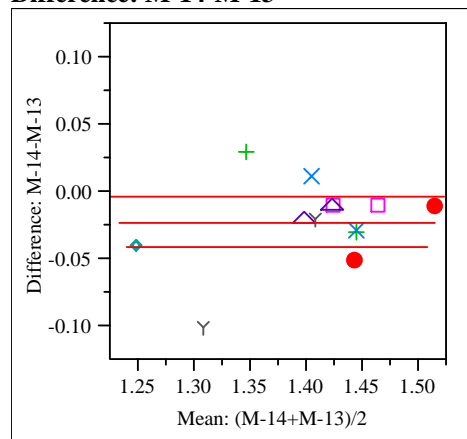


M-14	8.32538	t-Ratio	-1.44486
M-13	8.50923	DF	12
Mean Difference	-0.1838	Prob >  t	0.1741
Std Error	0.12724	Prob > t	0.9130
Upper95%	0.09339	Prob < t	0.0870
Lower95%	-0.4611		
N	13		
Correlation	0.60512		

Type/ Analyte/UoM=ARG-1/Mn/wt%

Matched Pairs

Difference: M-14-M-13

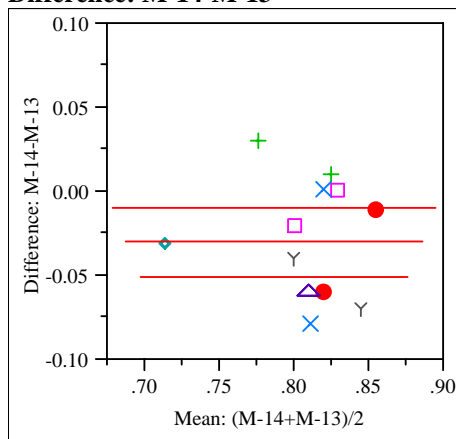


M-14	1.39538	t-Ratio	-2.58353
M-13	1.41769	DF	12
Mean Difference	-0.0223	Prob >  t	0.0239
Std Error	0.00863	Prob > t	0.9880
Upper95%	-0.0035	Prob < t	0.0120
Lower95%	-0.0411		
N	13		
Correlation	0.91208		

Type/ Analyte/UoM=ARG-1/Ni/wt%

Matched Pairs

Difference: M-14-M-13



M-14	0.79385	t-Ratio	-3.1225
M-13	0.82385	DF	12
Mean Difference	-0.03	Prob >  t	0.0088
Std Error	0.00961	Prob > t	0.9956
Upper95%	-0.0091	Prob < t	0.0044
Lower95%	-0.0509		
N	13		
Correlation	0.60419		

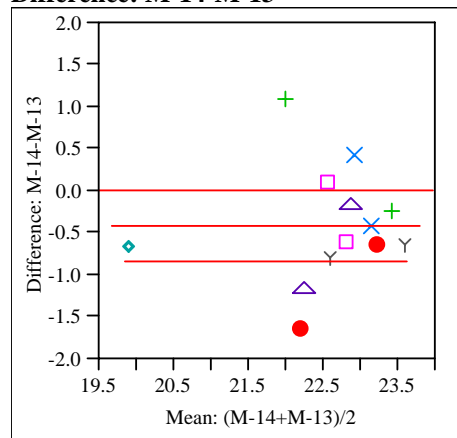


### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=ARG-1/Si/wt%

Matched Pairs

Difference: M-14-M-13

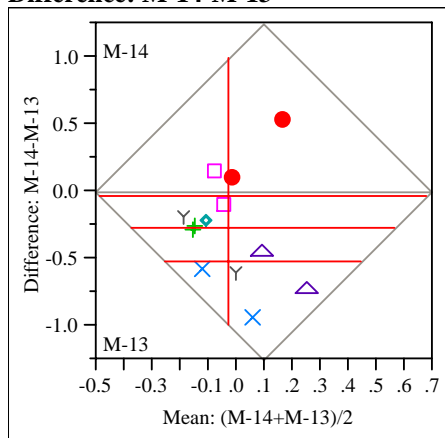


M-14	22.3738	t-Ratio	-2.13529
M-13	22.7846	DF	12
Mean Difference	-0.4108	Prob >  t	0.0540
Std Error	0.19237	Prob > t	0.9730
Upper95%	0.00837	Prob < t	0.0270
Lower95%	-0.8299		
N	13		
Correlation	0.75913		

Type/ Analyte/UoM=ARG-1/U/wt%

Matched Pairs

Difference: M-14-M-13

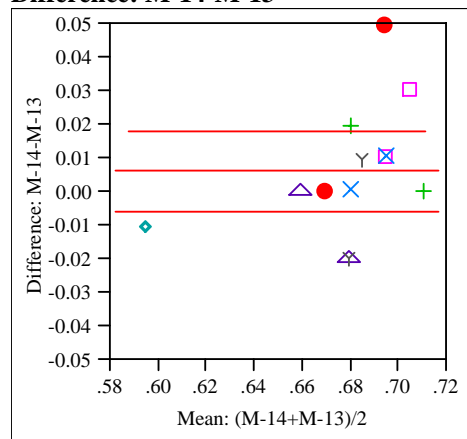


M-14	-0.1585	t-Ratio	-2.47154
M-13	0.11615	DF	12
Mean Difference	-0.2746	Prob >  t	0.0294
Std Error	0.11111	Prob > t	0.9853
Upper95%	-0.0325	Prob < t	0.0147
Lower95%	-0.5167		
N	13		
Correlation	-0.3811		

Type/ Analyte/UoM=ARG-1/Ti/wt%

Matched Pairs

Difference: M-14-M-13

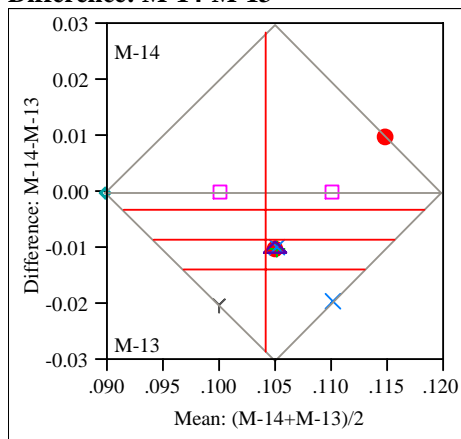


M-14	0.68231	t-Ratio	1.144806
M-13	0.67615	DF	12
Mean Difference	0.00615	Prob >  t	0.2746
Std Error	0.00538	Prob > t	0.1373
Upper95%	0.01787	Prob < t	0.8627
Lower95%	-0.0056		
N	13		
Correlation	0.82239		

Type/ Analyte/UoM=ARG-1/Zr/wt%

Matched Pairs

Difference: M-14-M-13



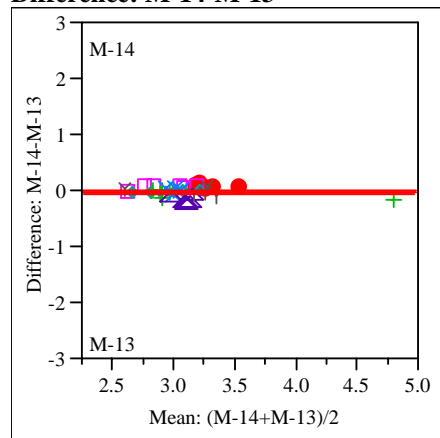
M-14	0.1	t-Ratio	-3.39467
M-13	0.10846	DF	12
Mean Difference	-0.0085	Prob >  t	0.0053
Std Error	0.00249	Prob > t	0.9973
Upper95%	-0.003	Prob < t	0.0027
Lower95%	-0.0139		
N	13		
Correlation	0.29637		

# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=SME/Al/wt%

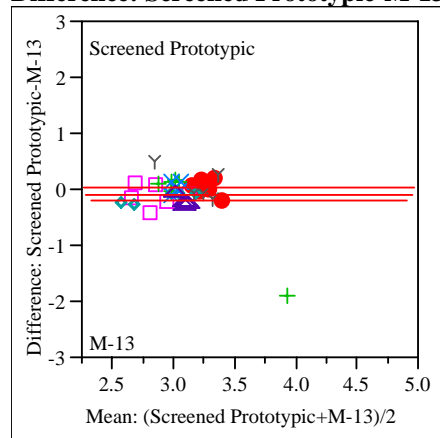
Matched Pairs

Difference: M-14-M-13



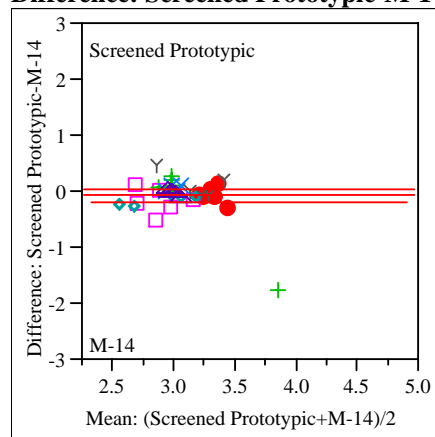
M-14	3.09026	t-Ratio	-1.01123
M-13	3.10632	DF	37
Mean Difference	-0.0161	Prob >  t	0.3185
Std Error	0.01587	Prob > t	0.8408
Upper95%	0.01611	Prob < t	0.1592
Lower95%	-0.0482		
N	38		
Correlation	0.96224		

Difference: Screened Prototypic-M-13



Screened Prototypic	3.02974	t-Ratio	-1.34086
M-13	3.10632	DF	37
Mean Difference	-0.0766	Prob >  t	0.1881
Std Error	0.05711	Prob > t	0.9059
Upper95%	0.03914	Prob < t	0.0941
Lower95%	-0.1923		
N	38		
Correlation	0.35369		

Difference: Screened Prototypic-M-14

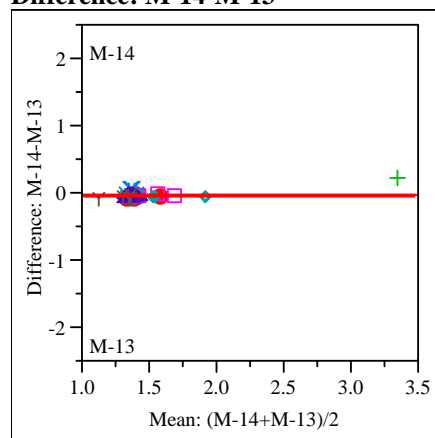


Screened Prototypic	3.02974	t-Ratio	-1.13719
M-14	3.09026	DF	37
Mean Difference	-0.0605	Prob >  t	0.2628
Std Error	0.05322	Prob > t	0.8686
Upper95%	0.04732	Prob < t	0.1314
Lower95%	-0.1684		
N	38		
Correlation	0.41276		

Type/ Analyte/UoM=SME/B/wt%

Matched Pairs

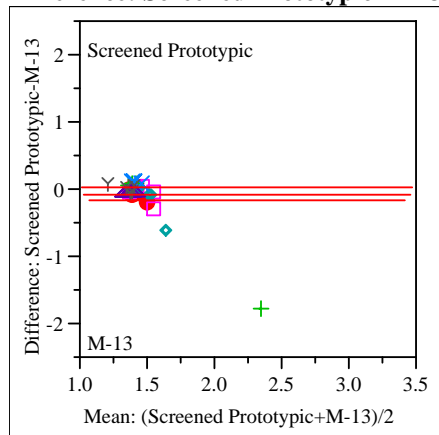
Difference: M-14-M-13



M-14	1.45421	t-Ratio	-2.14697
M-13	1.47263	DF	37
Mean Difference	-0.0184	Prob >  t	0.0384
Std Error	0.00858	Prob > t	0.9808
Upper95%	-0.001	Prob < t	0.0192
Lower95%	-0.0358		
N	38		
Correlation	0.99404		

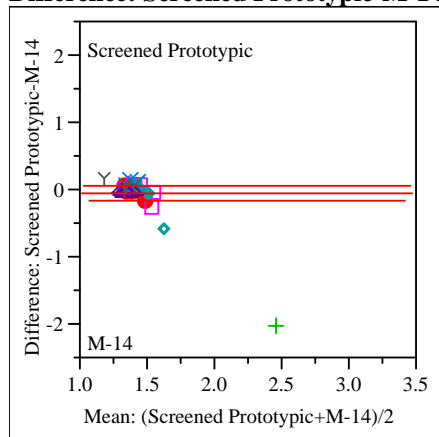
# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

## Difference: Screened Prototypic-M-13



Screened Prototypic	1.40947	t-Ratio	-1.25321
M-13	1.47263	DF	37
Mean Difference	-0.0632	Prob >  t	0.2180
Std Error	0.0504	Prob > t	0.8910
Upper95%	0.03896	Prob < t	0.1090
Lower95%	-0.1653		
N	38		
Correlation	0.20292		

## Difference: Screened Prototypic-M-14

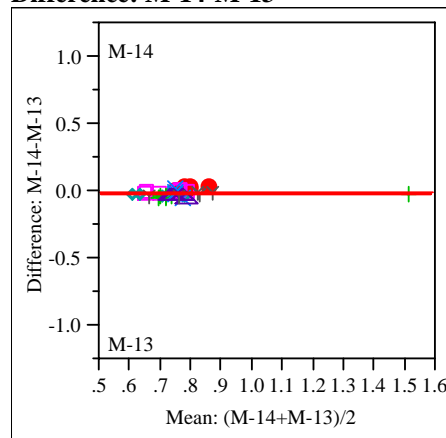


Screened Prototypic	1.40947	t-Ratio	-0.79498
M-14	1.45421	DF	37
Mean Difference	-0.0447	Prob >  t	0.4317
Std Error	0.05627	Prob > t	0.7842
Upper95%	0.06929	Prob < t	0.2158
Lower95%	-0.1588		
N	38		
Correlation	0.22806		

## Type/ Analyte/UoM=SME/Ca/wt%

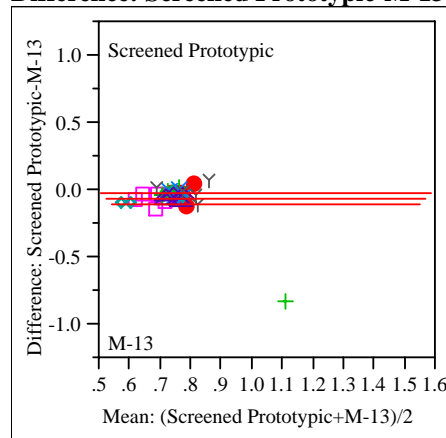
### Matched Pairs

## Difference: M-14-M-13



M-14	0.75974	t-Ratio	-4.56394
M-13	0.77816	DF	37
Mean Difference	-0.0184	Prob >  t	<.0001
Std Error	0.00404	Prob > t	1.0000
Upper95%	-0.0102	Prob < t	<.0001
Lower95%	-0.0266		
N	38		
Correlation	0.98387		

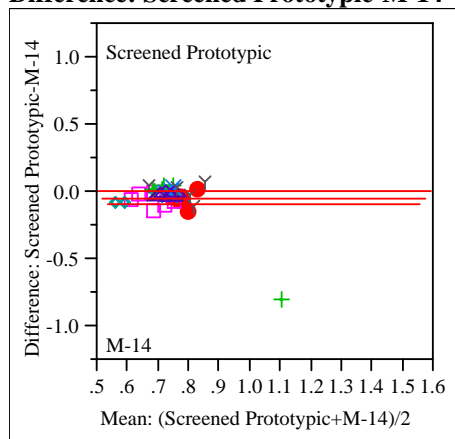
## Difference: Screened Prototypic-M-13



Screened Prototypic	0.71737	t-Ratio	-2.79041
M-13	0.77816	DF	37
Mean Difference	-0.0608	Prob >  t	0.0083
Std Error	0.02179	Prob > t	0.9959
Upper95%	-0.0166	Prob < t	0.0041
Lower95%	-0.1049		
N	38		
Correlation	0.27577		

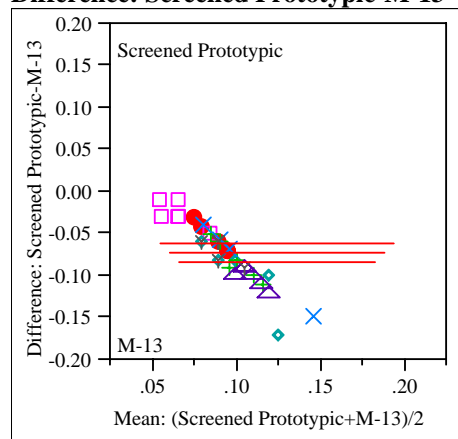
### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

**Difference: Screened Prototypic-M-14**



Screened Prototypic	0.71737	t-Ratio	-1.91799
M-14	0.75974	DF	37
Mean Difference	-0.0424	Prob >  t	0.0629
Std Error	0.02209	Prob > t	0.9686
Upper95%	0.00239	Prob < t	0.0314
Lower95%	-0.0871		
N	38		
Correlation	0.29065		

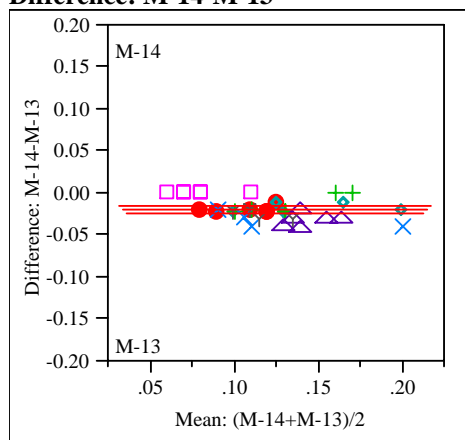
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.05711	t-Ratio	-12.9341
M-13	0.12974	DF	37
Mean Difference	-0.0726	Prob >  t	<.0001
Std Error	0.00562	Prob > t	1.0000
Upper95%	-0.0613	Prob < t	<.0001
Lower95%	-0.084		
N	38		
Correlation	0.30625		

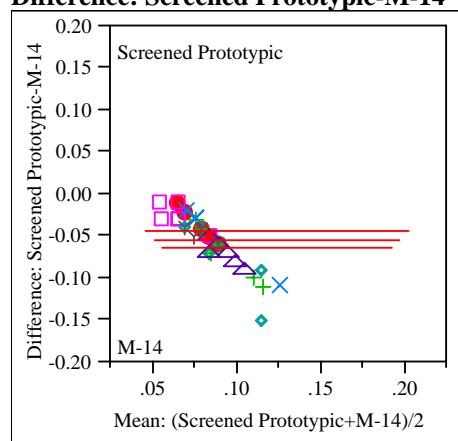
#### Type/ Analyte/UoM=SME/Cr/wt% Matched Pairs

**Difference: M-14-M-13**



M-14	0.11105	t-Ratio	-9.33364
M-13	0.12974	DF	37
Mean Difference	-0.0187	Prob >  t	<.0001
Std Error	0.002	Prob > t	1.0000
Upper95%	-0.0146	Prob < t	<.0001
Lower95%	-0.0227		
N	38		
Correlation	0.94187		

**Difference: Screened Prototypic-M-14**



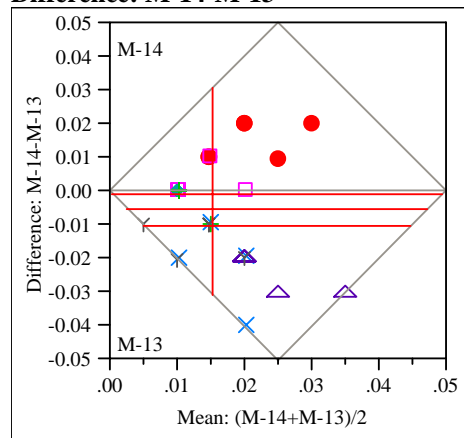
Screened Prototypic	0.05711	t-Ratio	-10.8268
M-14	0.11105	DF	37
Mean Difference	-0.0539	Prob >  t	<.0001
Std Error	0.00498	Prob > t	1.0000
Upper95%	-0.0439	Prob < t	<.0001
Lower95%	-0.064		
N	38		
Correlation	0.2375		

### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=SME/Cu/wt%

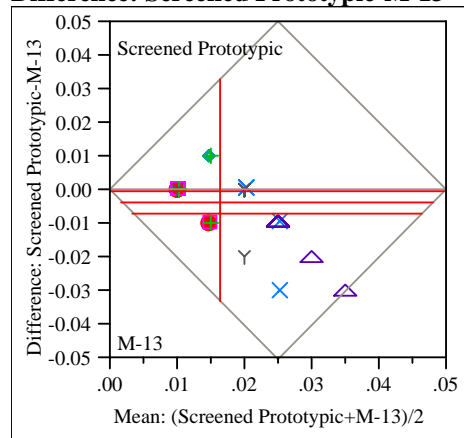
Matched Pairs

Difference: M-14-M-13



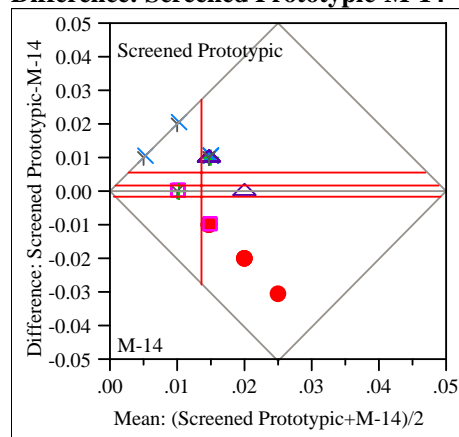
M-14	0.01263	t-Ratio	-2.38648
M-13	0.01816	DF	37
Mean Difference	-0.0055	Prob >  t	0.0222
Std Error	0.00232	Prob > t	0.9889
Upper95%	-0.0008	Prob < t	0.0111
Lower95%	-0.0102		
N	38		
Correlation	-0.0935		

Difference: Screened Prototypic-M-13



Screened Prototypic	0.01474	t-Ratio	-2.12174
M-13	0.01816	DF	37
Mean Difference	-0.0034	Prob >  t	0.0406
Std Error	0.00161	Prob > t	0.9797
Upper95%	-0.0002	Prob < t	0.0203
Lower95%	-0.0067		
N	38		
Correlation	0.36803		

Difference: Screened Prototypic-M-14

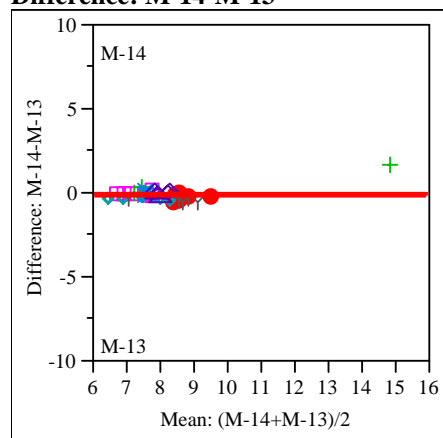


Screened Prototypic	0.01474	t-Ratio	1.135676
M-14	0.01263	DF	37
Mean Difference	0.00211	Prob >  t	0.2634
Std Error	0.00185	Prob > t	0.1317
Upper95%	0.00586	Prob < t	0.8683
Lower95%	-0.0017		
N	38		
Correlation	-0.3563		

Type/ Analyte/UoM=SME/Fe/wt%

Matched Pairs

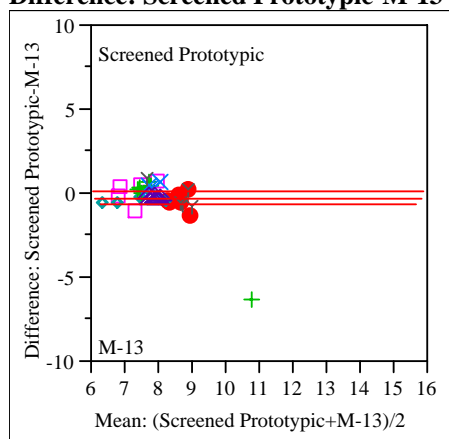
Difference: M-14-M-13



M-14	8.02816	t-Ratio	-1.17362
M-13	8.09789	DF	37
Mean Difference	-0.0697	Prob >  t	0.2480
Std Error	0.05942	Prob > t	0.8760
Upper95%	0.05066	Prob < t	0.1240
Lower95%	-0.1901		
N	38		
Correlation	0.97537		

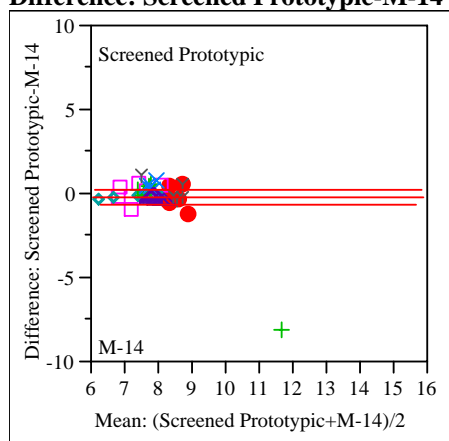
# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

## Difference: Screened Prototypic-M-13



Screened Prototypic	7.86947	t-Ratio	-1.25545
M-13	8.09789	DF	37
Mean Difference	-0.2284	Prob >  t	0.2172
Std Error	0.18194	Prob > t	0.8914
Upper95%	0.14023	Prob < t	0.1086
Lower95%	-0.5971		
N	38		
Correlation	0.4031		

## Difference: Screened Prototypic-M-14

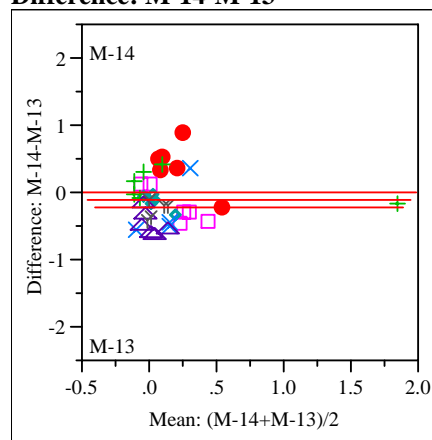


Screened Prototypic	7.86947	t-Ratio	-0.70199
M-14	8.02816	DF	37
Mean Difference	-0.1587	Prob >  t	0.4871
Std Error	0.22605	Prob > t	0.7565
Upper95%	0.29934	Prob < t	0.2435
Lower95%	-0.6167		
N	38		
Correlation	0.28511		

## Type/ Analyte/UoM=SME/K/wt%

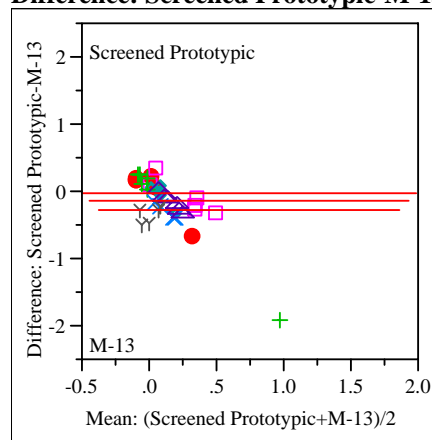
### Matched Pairs

## Difference: M-14-M-13



M-14	0.08632	t-Ratio	-1.66781
M-13	0.18658	DF	37
Mean Difference	-0.1003	Prob >  t	0.1038
Std Error	0.06012	Prob > t	0.9481
Upper95%	0.02154	Prob < t	0.0519
Lower95%	-0.2221		
N	38		
Correlation	0.50081		

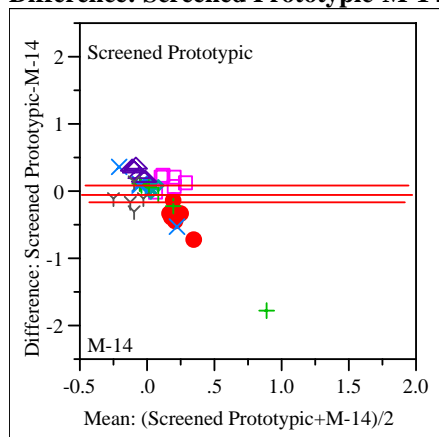
## Difference: Screened Prototypic-M-13



Screened Prototypic	0.05053	t-Ratio	-2.18099
M-13	0.18658	DF	37
Mean Difference	-0.1361	Prob >  t	0.0356
Std Error	0.06238	Prob > t	0.9822
Upper95%	-0.0097	Prob < t	0.0178
Lower95%	-0.2624		
N	38		
Correlation	0.08505		

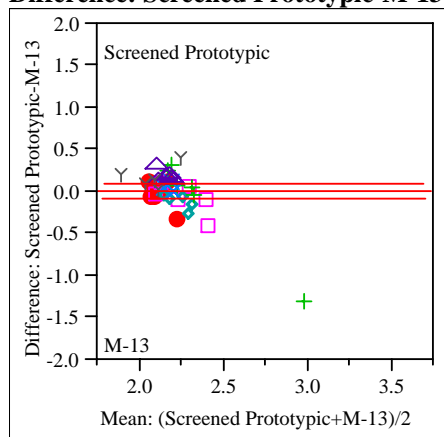
### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

**Difference: Screened Prototypic-M-14**



Screened Prototypic	0.05053	t-Ratio	-0.57144
M-14	0.08632	DF	37
Mean Difference	-0.0358	Prob >  t	0.5712
Std Error	0.06263	Prob > t	0.7144
Upper95%	0.09111	Prob < t	0.2856
Lower95%	-0.1627		
N	38		
Correlation	0.02108		

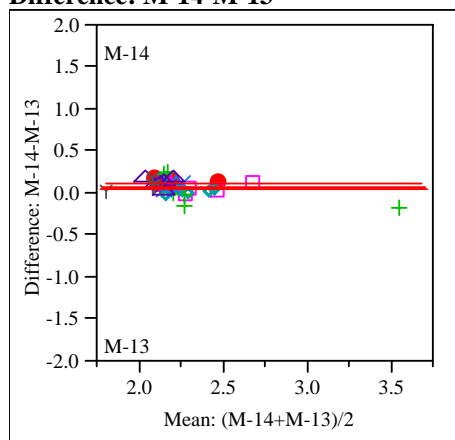
**Difference: Screened Prototypic-M-13**



Screened Prototypic	2.21342	t-Ratio	0.210762
M-13	2.20395	DF	37
Mean Difference	0.00947	Prob >  t	0.8342
Std Error	0.04495	Prob > t	0.4171
Upper95%	0.10055	Prob < t	0.5829
Lower95%	-0.0816		
N	38		
Correlation	0.25322		

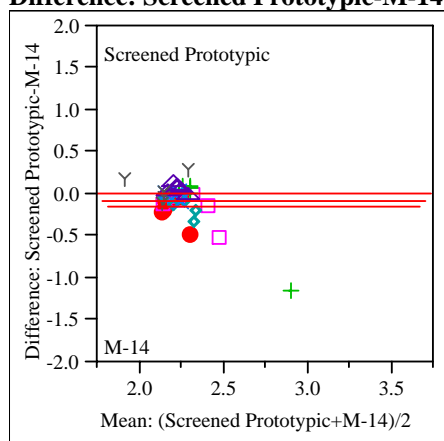
#### Type/ Analyte/UoM=SME/Li/wt% Matched Pairs

**Difference: M-14-M-13**



M-14	2.28842	t-Ratio	5.898778
M-13	2.20395	DF	37
Mean Difference	0.08447	Prob >  t	<.0001
Std Error	0.01432	Prob > t	<.0001
Upper95%	0.11349	Prob < t	1.0000
Lower95%	0.05546		
N	38		
Correlation	0.95637		

**Difference: Screened Prototypic-M-14**



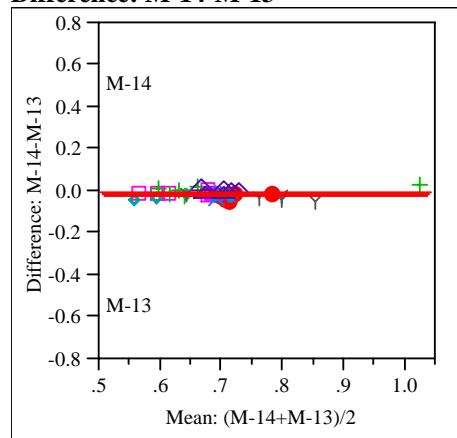
Screened Prototypic	2.21342	t-Ratio	-1.93158
M-14	2.28842	DF	37
Mean Difference	-0.075	Prob >  t	0.0611
Std Error	0.03883	Prob > t	0.9695
Upper95%	0.00367	Prob < t	0.0305
Lower95%	-0.1537		
N	38		
Correlation	0.24365		

# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=SME/Mg/wt%

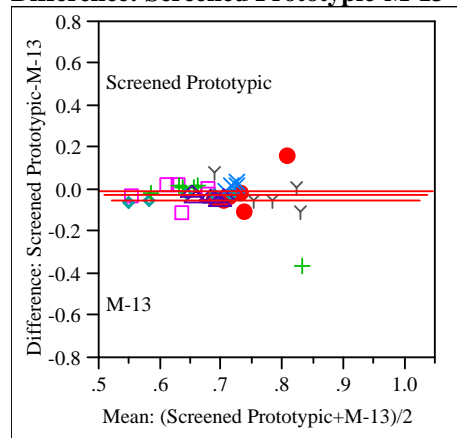
Matched Pairs

Difference: M-14-M-13



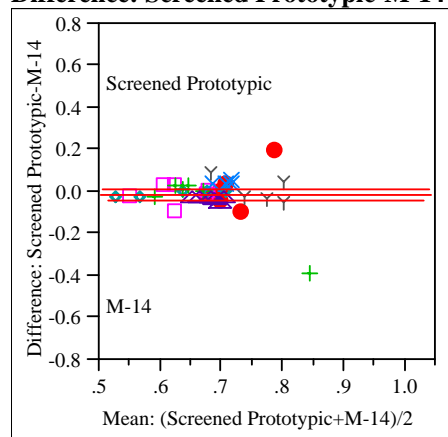
M-14	0.68789	t-Ratio	-4.80835
M-13	0.70237	DF	37
Mean Difference	-0.0145	Prob >  t	<.0001
Std Error	0.00301	Prob > t	1.0000
Upper95%	-0.0084	Prob < t	<.0001
Lower95%	-0.0206		
N	38		
Correlation	0.9765		

Difference: Screened Prototypic-M-13



Screened Prototypic	0.67763	t-Ratio	-2.0702
M-13	0.70237	DF	37
Mean Difference	-0.0247	Prob >  t	0.0455
Std Error	0.01195	Prob > t	0.9773
Upper95%	-0.0005	Prob < t	0.0227
Lower95%	-0.0489		
N	38		
Correlation	0.57334		

Difference: Screened Prototypic-M-14

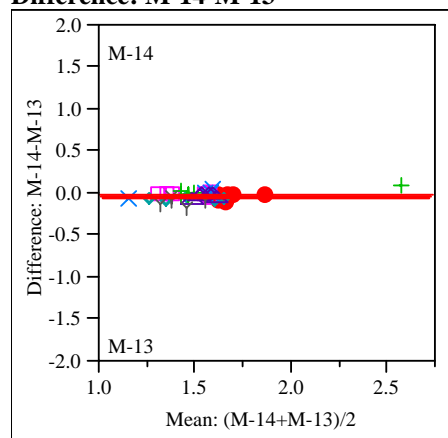


Screened Prototypic	0.67763	t-Ratio	-0.78278
M-14	0.68789	DF	37
Mean Difference	-0.0103	Prob >  t	0.4387
Std Error	0.01311	Prob > t	0.7806
Upper95%	0.0163	Prob < t	0.2194
Lower95%	-0.0368		
N	38		
Correlation	0.48918		

Type/ Analyte/UoM=SME/Mn/wt%

Matched Pairs

Difference: M-14-M-13

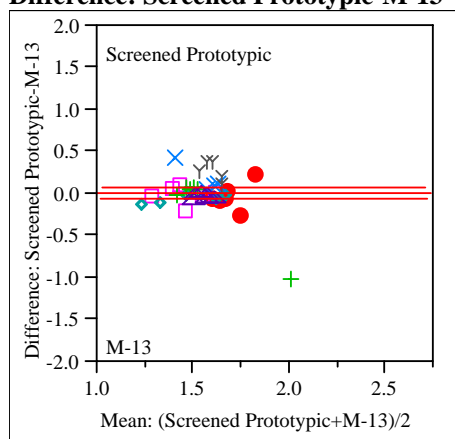


M-14	1.52158	t-Ratio	-4.2376
M-13	1.555	DF	37
Mean Difference	-0.0334	Prob >  t	0.0001
Std Error	0.00789	Prob > t	0.9999
Upper95%	-0.0174	Prob < t	<.0001
Lower95%	-0.0494		
N	38		
Correlation	0.9809		



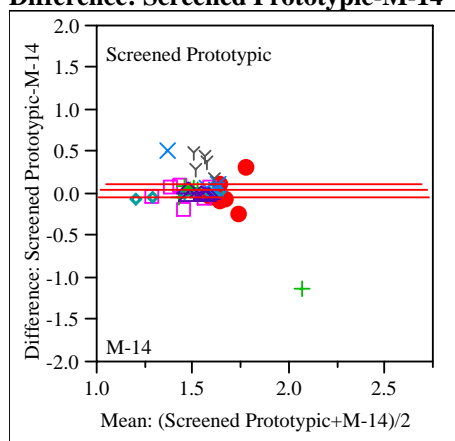
# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

## Difference: Screened Prototypic-M-13



Screened Prototypic	1.56632	t-Ratio	0.307174
M-13	1.555	DF	37
Mean Difference	0.01132	Prob >  t	0.7604
Std Error	0.03684	Prob > t	0.3802
Upper95%	0.08596	Prob < t	0.6198
Lower95%	-0.0633		
N	38		
Correlation	0.24699		

## Difference: Screened Prototypic-M-14

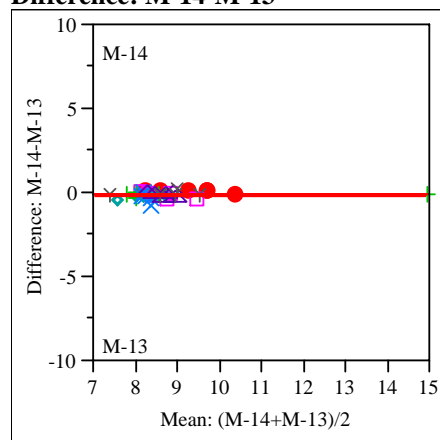


Screened Prototypic	1.56632	t-Ratio	1.06427
M-14	1.52158	DF	37
Mean Difference	0.04474	Prob >  t	0.2941
Std Error	0.04204	Prob > t	0.1471
Upper95%	0.12991	Prob < t	0.8529
Lower95%	-0.0404		
N	38		
Correlation	0.1409		

## Type/ Analyte/UoM=SME/Na/wt%

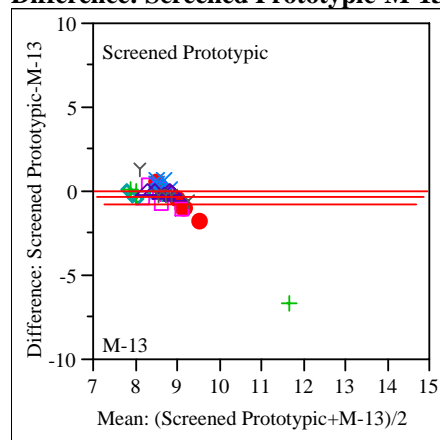
### Matched Pairs

## Difference: M-14-M-13



M-14	8.71447	t-Ratio	-3.07824
M-13	8.81711	DF	37
Mean Difference	-0.1026	Prob >  t	0.0039
Std Error	0.03334	Prob > t	0.9980
Upper95%	-0.0351	Prob < t	0.0020
Lower95%	-0.1702		
N	38		
Correlation	0.98585		

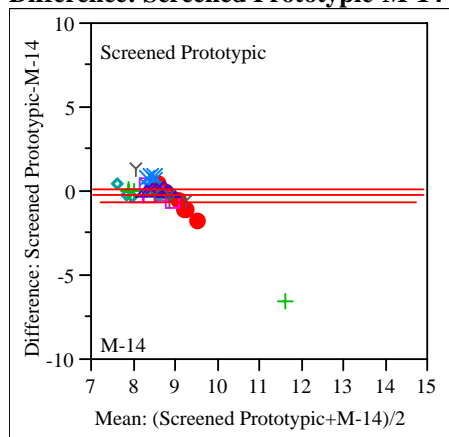
## Difference: Screened Prototypic-M-13



Screened Prototypic	8.53263	t-Ratio	-1.48665
M-13	8.81711	DF	37
Mean Difference	-0.2845	Prob >  t	0.1456
Std Error	0.19135	Prob > t	0.9272
Upper95%	0.10324	Prob < t	0.0728
Lower95%	-0.6722		
N	38		
Correlation	0.15509		

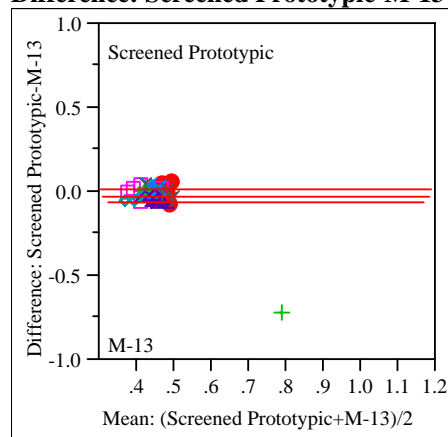
### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

**Difference: Screened Prototypic-M-14**



Screened Prototypic	8.53263	t-Ratio	-0.9293
M-14	8.71447	DF	37
Mean Difference	-0.1818	Prob >  t	0.3588
Std Error	0.19568	Prob > t	0.8206
Upper95%	0.21464	Prob < t	0.1794
Lower95%	-0.5783		
N	38		
Correlation	0.1814		

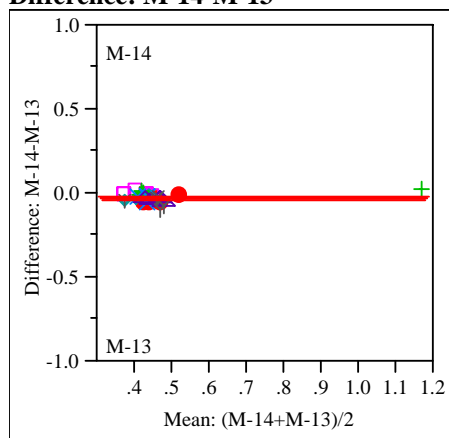
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.44421	t-Ratio	-1.29947
M-13	0.46974	DF	37
Mean Difference	-0.0255	Prob >  t	0.2018
Std Error	0.01964	Prob > t	0.8991
Upper95%	0.01428	Prob < t	0.1009
Lower95%	-0.0653		
N	38		
Correlation	0.11715		

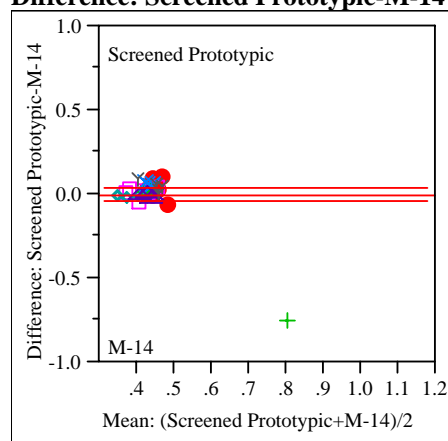
#### Type/ Analyte/UoM=SME/Ni/wt% Matched Pairs

**Difference: M-14-M-13**



M-14	0.44421	t-Ratio	-6.43276
M-13	0.46974	DF	37
Mean Difference	-0.0255	Prob >  t	<.0001
Std Error	0.00397	Prob > t	1.0000
Upper95%	-0.0175	Prob < t	<.0001
Lower95%	-0.0336		
N	38		
Correlation	0.98177		

**Difference: Screened Prototypic-M-14**



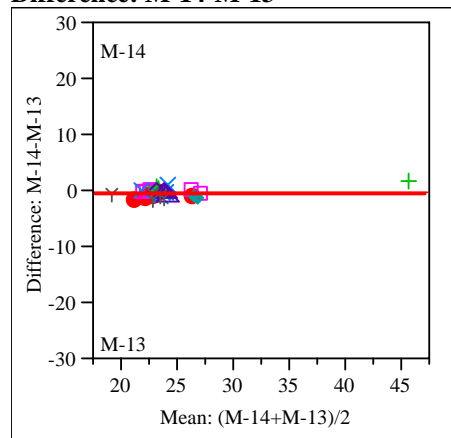
Screened Prototypic	0.44421	t-Ratio	6.91e-17
M-14	0.44421	DF	37
Mean Difference	1.5e-18	Prob >  t	1.0000
Std Error	0.02113	Prob > t	0.5000
Upper95%	0.04281	Prob < t	0.5000
Lower95%	-0.0428		
N	38		
Correlation	0.04359		

# Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

Type/ Analyte/UoM=SME/Si/wt%

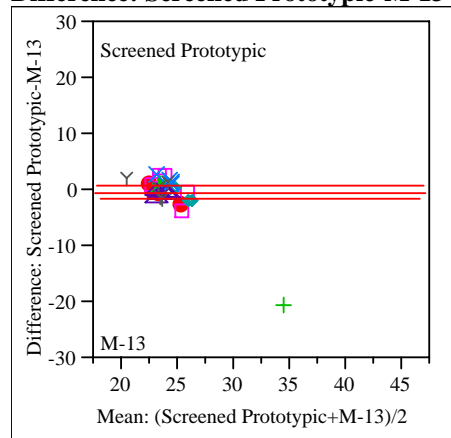
Matched Pairs

Difference: M-14-M-13



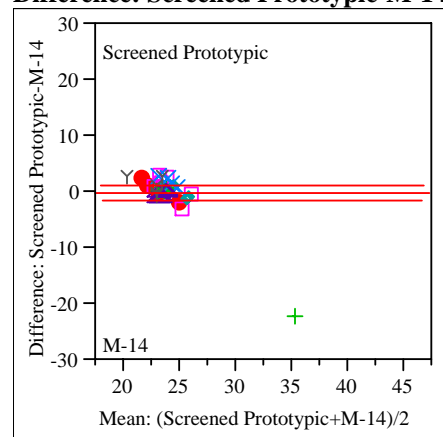
M-14	23.9676	t-Ratio	-3.46968
M-13	24.335	DF	37
Mean Difference	-0.3674	Prob >  t	0.0013
Std Error	0.10588	Prob > t	0.9993
Upper95%	-0.1528	Prob < t	0.0007
Lower95%	-0.5819		
N	38		
Correlation	0.98916		

Difference: Screened Prototypic-M-13



Screened Prototypic	23.9063	t-Ratio	-0.72539
M-13	24.335	DF	37
Mean Difference	-0.4287	Prob >  t	0.4728
Std Error	0.59097	Prob > t	0.7636
Upper95%	0.76873	Prob < t	0.2364
Lower95%	-1.6261		
N	38		
Correlation	0.23339		

Difference: Screened Prototypic-M-14

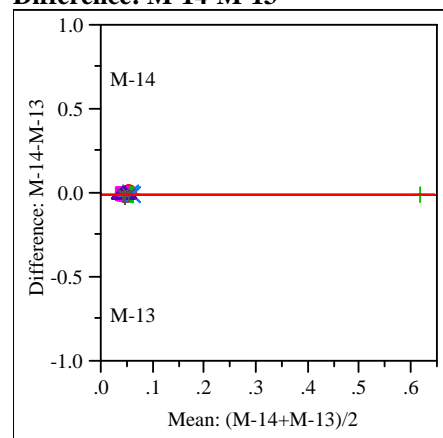


Screened Prototypic	23.9063	t-Ratio	-0.09563
M-14	23.9676	DF	37
Mean Difference	-0.0613	Prob >  t	0.9243
Std Error	0.64115	Prob > t	0.5378
Upper95%	1.23778	Prob < t	0.4622
Lower95%	-1.3604		
N	38		
Correlation	0.2255		

Type/ Analyte/UoM=SME/Ti/wt%

Matched Pairs

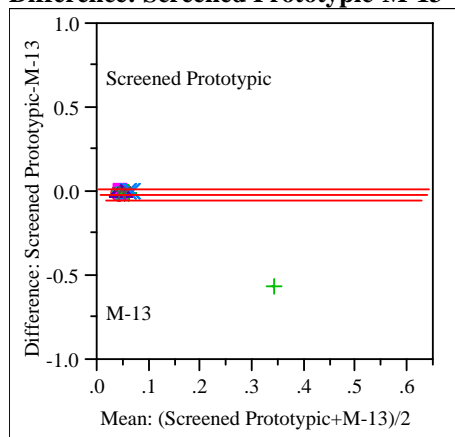
Difference: M-14-M-13



M-14	0.06368	t-Ratio	-2.27596
M-13	0.06737	DF	37
Mean Difference	-0.0037	Prob >  t	0.0287
Std Error	0.00162	Prob > t	0.9856
Upper95%	-0.0004	Prob < t	0.0144
Lower95%	-0.007		
N	38		
Correlation	0.99417		

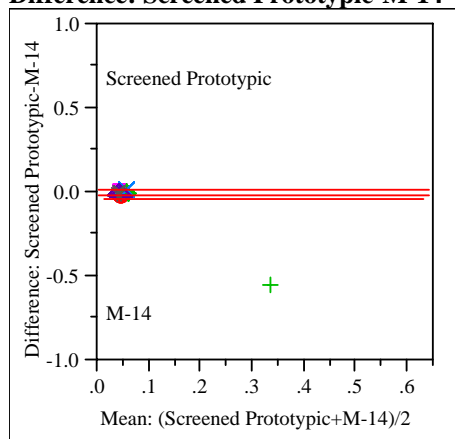
### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.04947	t-Ratio	-1.21932
M-13	0.06737	DF	37
Mean Difference	-0.0179	Prob >  t	0.2304
Std Error	0.01468	Prob > t	0.8848
Upper95%	0.01184	Prob < t	0.1152
Lower95%	-0.0476		
N	38		
Correlation	0.29076		

**Difference: Screened Prototypic-M-14**

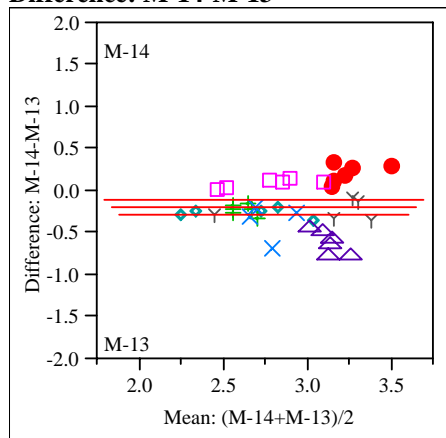


Screened Prototypic	0.04947	t-Ratio	-0.97491
M-14	0.06368	DF	37
Mean Difference	-0.0142	Prob >  t	0.3359
Std Error	0.01458	Prob > t	0.8320
Upper95%	0.01532	Prob < t	0.1680
Lower95%	-0.0437		
N	38		
Correlation	0.22608		

**Type/ Analyte/UoM=SME/U/wt%**

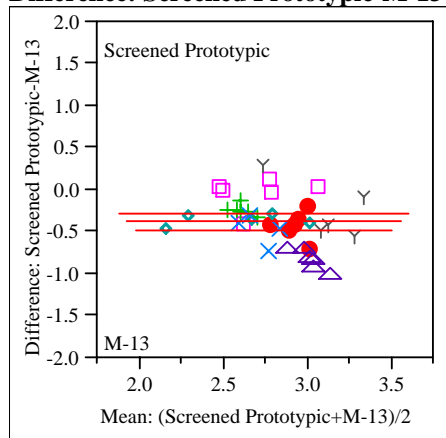
**Matched Pairs**

**Difference: M-14-M-13**



M-14	2.81526	t-Ratio	-4.00986
M-13	3.00184	DF	37
Mean Difference	-0.1866	Prob >  t	0.0003
Std Error	0.04653	Prob > t	0.9999
Upper95%	-0.0923	Prob < t	0.0001
Lower95%	-0.2809		
N	38		
Correlation	0.66941		

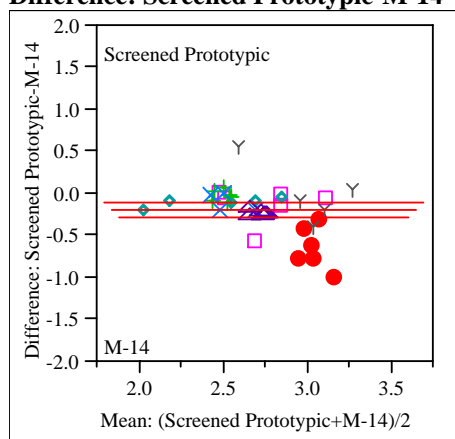
**Difference: Screened Prototypic-M-13**



Screened Prototypic	2.63026	t-Ratio	-7.81457
M-13	3.00184	DF	37
Mean Difference	-0.3716	Prob >  t	<.0001
Std Error	0.04755	Prob > t	1.0000
Upper95%	-0.2752	Prob < t	<.0001
Lower95%	-0.4679		
N	38		
Correlation	0.54611		

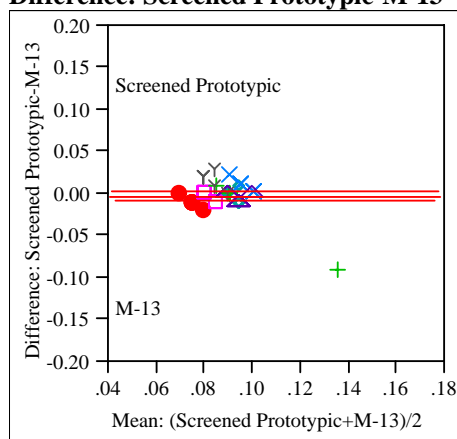
### Exhibit A2. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample

**Difference: Screened Prototypic-M-14**



Screened Prototypic	2.63026	t-Ratio	-4.16951
M-14	2.81526	DF	37
Mean Difference	-0.185	Prob >  t	0.0002
Std Error	0.04437	Prob > t	0.9999
Upper95%	-0.0951	Prob < t	<.0001
Lower95%	-0.2749		
N	38		
Correlation	0.65803		

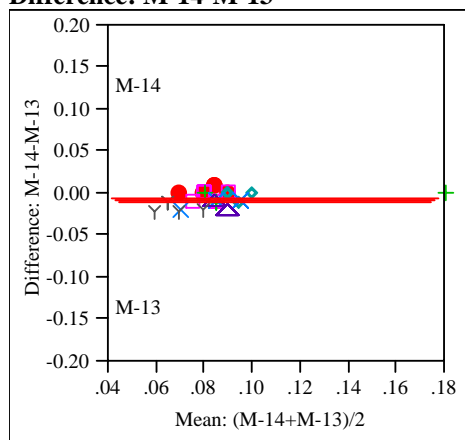
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.08737	t-Ratio	-0.80778
M-13	0.08974	DF	37
Mean Difference	-0.0024	Prob >  t	0.4244
Std Error	0.00293	Prob > t	0.7878
Upper95%	0.00357	Prob < t	0.2122
Lower95%	-0.0083		
N	38		
Correlation	0.2311		

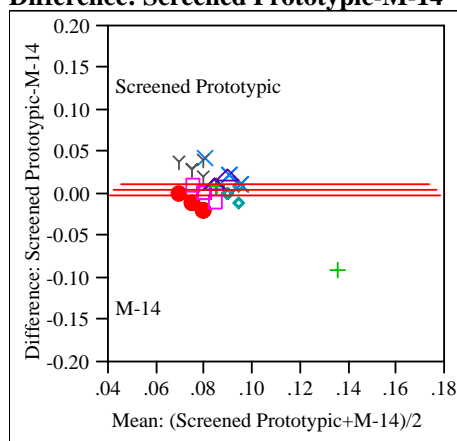
#### Type/ Analyte/UoM=SME/Zr/wt% Matched Pairs

**Difference: M-14-M-13**



M-14	0.08211	t-Ratio	-5.5206
M-13	0.08974	DF	37
Mean Difference	-0.0076	Prob >  t	<.0001
Std Error	0.00138	Prob > t	1.0000
Upper95%	-0.0048	Prob < t	<.0001
Lower95%	-0.0104		
N	38		
Correlation	0.89919		

**Difference: Screened Prototypic-M-14**



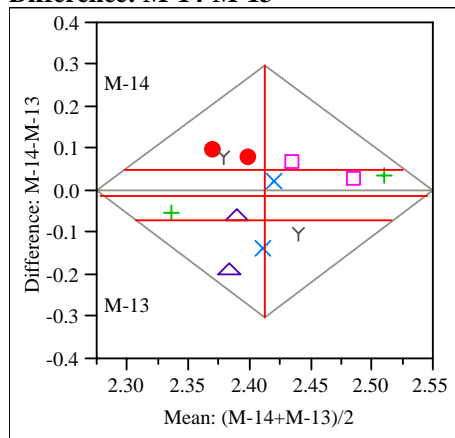
Screened Prototypic	0.08737	t-Ratio	1.464982
M-14	0.08211	DF	37
Mean Difference	0.00526	Prob >  t	0.1514
Std Error	0.00359	Prob > t	0.0757
Upper95%	0.01254	Prob < t	0.9243
Lower95%	-0.002		
N	38		
Correlation	-0.0411		

**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Type/ Analyte/UoM=ARG-1/Al/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

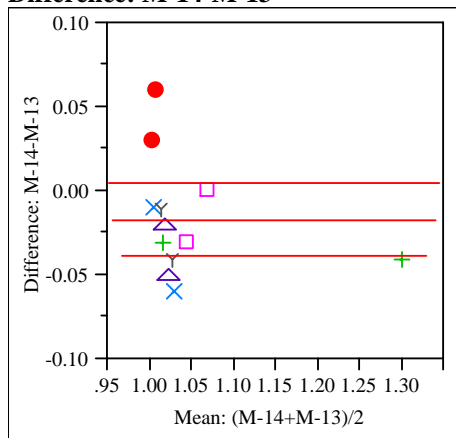


M-14	2.40833	t-Ratio	-0.36116
M-13	2.41833	DF	11
Mean Difference	-0.01	Prob >  t	0.7248
Std Error	0.02769	Prob > t	0.6376
Upper95%	0.05094	Prob < t	0.3624
Lower95%	-0.0709		
N	12		
Correlation	0.0209		

**Type/ Analyte/UoM=ARG-1/Ca/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

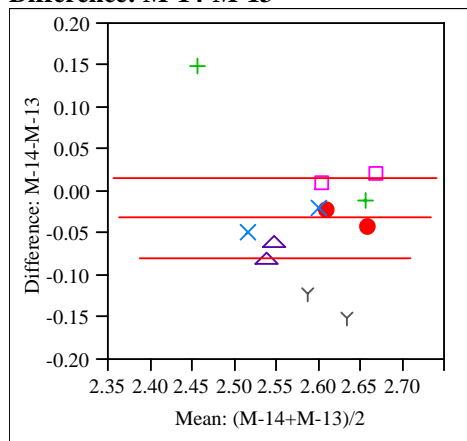


M-14	1.03917	t-Ratio	-1.68812
M-13	1.05583	DF	11
Mean Difference	-0.0167	Prob >  t	0.1195
Std Error	0.00987	Prob > t	0.9402
Upper95%	0.00506	Prob < t	0.0598
Lower95%	-0.0384		
N	12		
Correlation	0.92147		

**Type/ Analyte/UoM=ARG-1/B/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

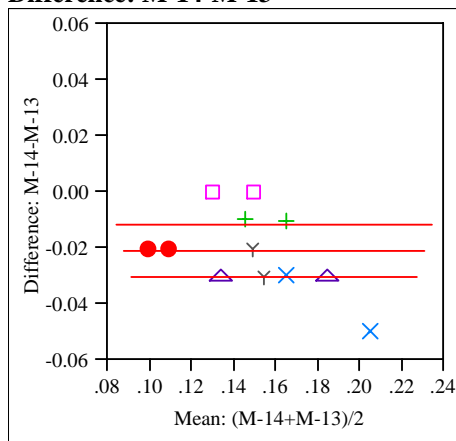


M-14	2.575	t-Ratio	-1.40588
M-13	2.60583	DF	11
Mean Difference	-0.0308	Prob >  t	0.1874
Std Error	0.02193	Prob > t	0.9063
Upper95%	0.01744	Prob < t	0.0937
Lower95%	-0.0791		
N	12		
Correlation	0.51337		

**Type/ Analyte/UoM=ARG-1/Cr/wt%**

**Matched Pairs**

**Difference: M-14-M-13**



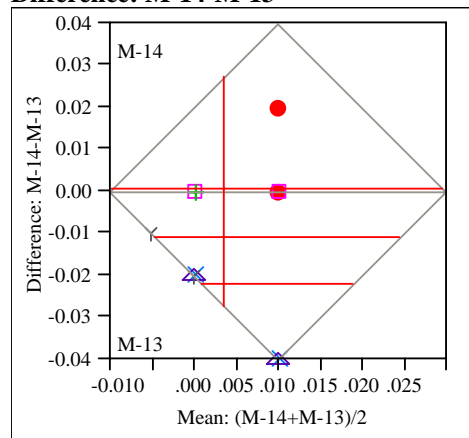
M-14	0.13917	t-Ratio	-5
M-13	0.16	DF	11
Mean Difference	-0.0208	Prob >  t	0.0004
Std Error	0.00417	Prob > t	0.9998
Upper95%	-0.0117	Prob < t	0.0002
Lower95%	-0.03		
N	12		
Correlation	0.91132		

**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Type/ Analyte/UoM=ARG-1/Cu/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

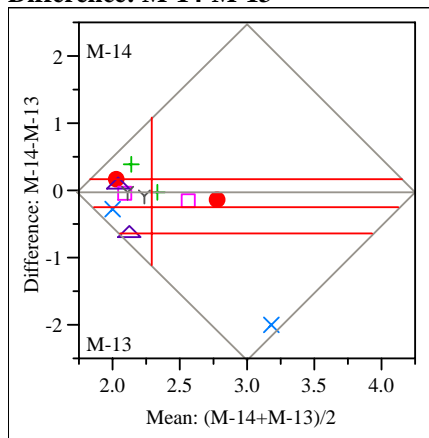


M-14	-0.0017	t-Ratio	-2.10636
M-13	0.00917	DF	11
Mean Difference	-0.0108	Prob >  t	0.0589
Std Error	0.00514	Prob > t	0.9705
Upper95%	0.00049	Prob < t	0.0295
Lower95%	-0.0222		
N	12		
Correlation	-0.4209		

**Type/ Analyte/UoM=ARG-1/K/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

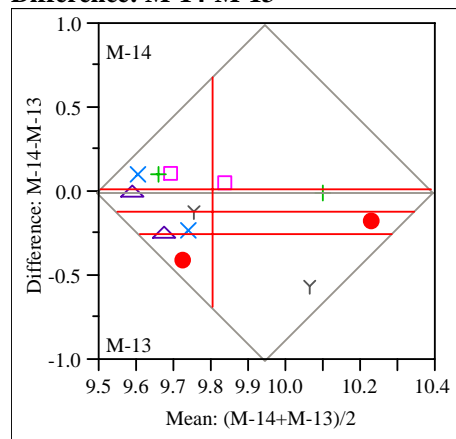


M-14	2.19	t-Ratio	-1.25717
M-13	2.41417	DF	11
Mean Difference	-0.2242	Prob >  t	0.2347
Std Error	0.17831	Prob > t	0.8826
Upper95%	0.16829	Prob < t	0.1174
Lower95%	-0.6166		
N	12		
Correlation	0.23116		

**Type/ Analyte/UoM=ARG-1/Fe/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

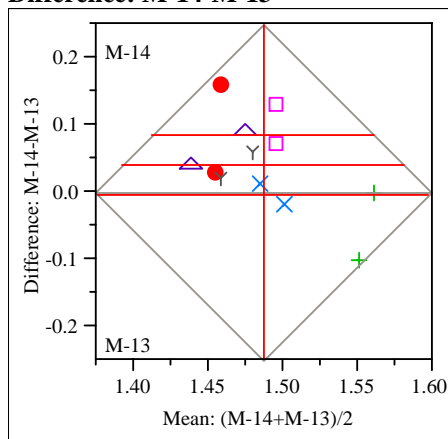


M-14	9.75	t-Ratio	-1.8619
M-13	9.86583	DF	11
Mean Difference	-0.1158	Prob >  t	0.0895
Std Error	0.06221	Prob > t	0.9552
Upper95%	0.0211	Prob < t	0.0448
Lower95%	-0.2528		
N	12		
Correlation	0.61238		

**Type/ Analyte/UoM=ARG-1/Li/wt%**

**Matched Pairs**

**Difference: M-14-M-13**



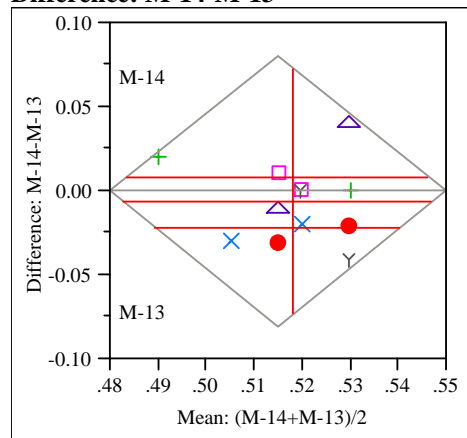
M-14	1.50833	t-Ratio	2.047654
M-13	1.4675	DF	11
Mean Difference	0.04083	Prob >  t	0.0652
Std Error	0.01994	Prob > t	0.0326
Upper95%	0.08472	Prob < t	0.9674
Lower95%	-0.0031		
N	12		
Correlation	0.05769		

**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Type/ Analyte/UoM=ARG-1/Mg/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

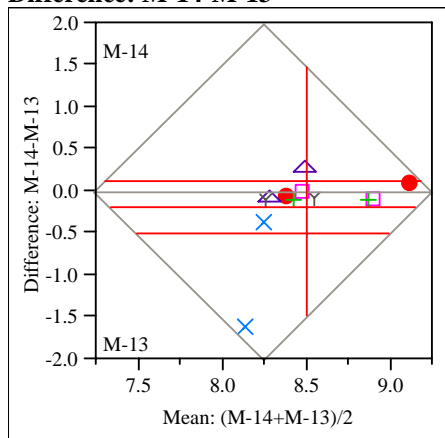


M-14	0.515	t-Ratio	-1
M-13	0.52167	DF	11
Mean Difference	-0.0067	Prob >  t	0.3388
Std Error	0.00667	Prob > t	0.8306
Upper95%	0.00801	Prob < t	0.1694
Lower95%	-0.0213		
N	12		
Correlation	0.03317		

**Type/ Analyte/UoM=ARG-1/Na/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

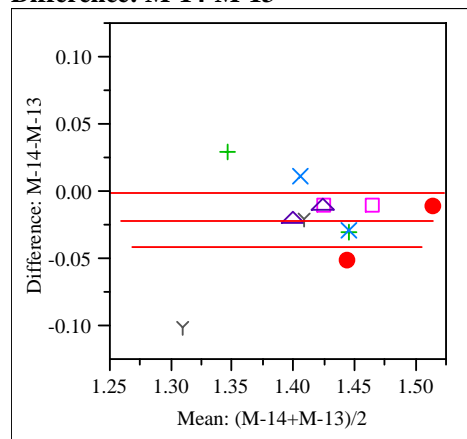


M-14	8.41917	t-Ratio	-1.36874
M-13	8.60833	DF	11
Mean Difference	-0.1892	Prob >  t	0.1984
Std Error	0.13821	Prob > t	0.9008
Upper95%	0.11502	Prob < t	0.0992
Lower95%	-0.4934		
N	12		
Correlation	0.25698		

**Type/ Analyte/UoM=ARG-1/Mn/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

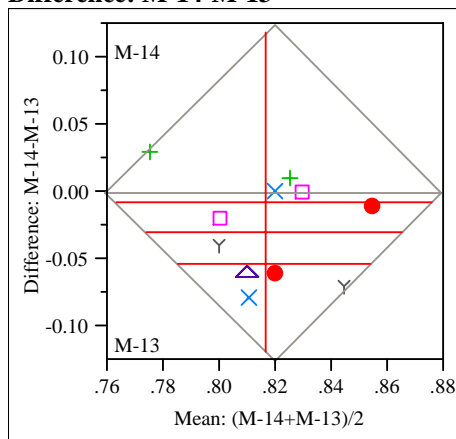


M-14	1.40917	t-Ratio	-2.25251
M-13	1.43	DF	11
Mean Difference	-0.0208	Prob >  t	0.0457
Std Error	0.00925	Prob > t	0.9772
Upper95%	-0.0005	Prob < t	0.0228
Lower95%	-0.0412		
N	12		
Correlation	0.84363		

**Type/ Analyte/UoM=ARG-1/Ni/wt%**

**Matched Pairs**

**Difference: M-14-M-13**



M-14	0.80167	t-Ratio	-2.87228
M-13	0.83167	DF	11
Mean Difference	-0.03	Prob >  t	0.0152
Std Error	0.01044	Prob > t	0.9924
Upper95%	-0.007	Prob < t	0.0076
Lower95%	-0.053		
N	12		
Correlation	0.16171		

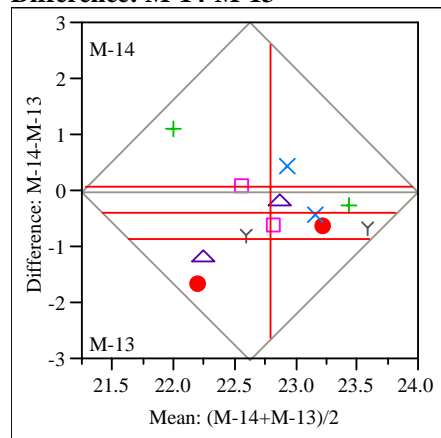


**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Type/ Analyte/UoM=ARG-1/Si/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

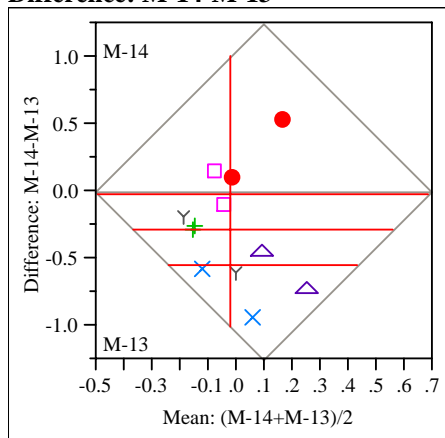


M-14	22.6067	t-Ratio	-1.87897
M-13	22.9975	DF	11
Mean Difference	-0.3908	Prob >  t	0.0870
Std Error	0.208	Prob > t	0.9565
Upper95%	0.06698	Prob < t	0.0435
Lower95%	-0.8486		
N	12		
Correlation	0.31909		

**Type/ Analyte/UoM=ARG-1/U/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

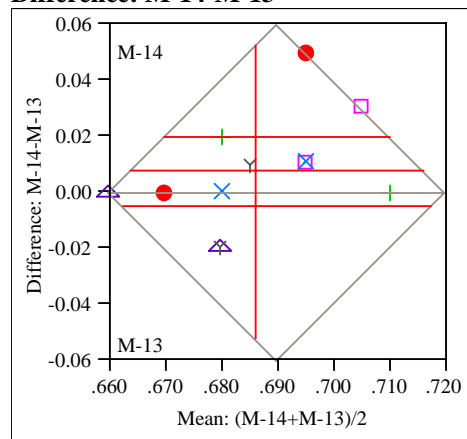


M-14	-0.1542	t-Ratio	-2.32079
M-13	0.12583	DF	11
Mean Difference	-0.28	Prob >  t	0.0405
Std Error	0.12065	Prob > t	0.9797
Upper95%	-0.0145	Prob < t	0.0203
Lower95%	-0.5455		
N	12		
Correlation	-0.3951		

**Type/ Analyte/UoM=ARG-1/Ti/wt%**

**Matched Pairs**

**Difference: M-14-M-13**

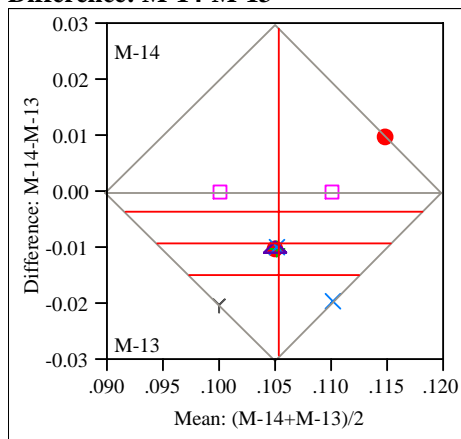


M-14	0.69	t-Ratio	1.325668
M-13	0.6825	DF	11
Mean Difference	0.0075	Prob >  t	0.2118
Std Error	0.00566	Prob > t	0.1059
Upper95%	0.01995	Prob < t	0.8941
Lower95%	-0.005		
N	12		
Correlation	0.39318		

**Type/ Analyte/UoM=ARG-1/Zr/wt%**

**Matched Pairs**

**Difference: M-14-M-13**



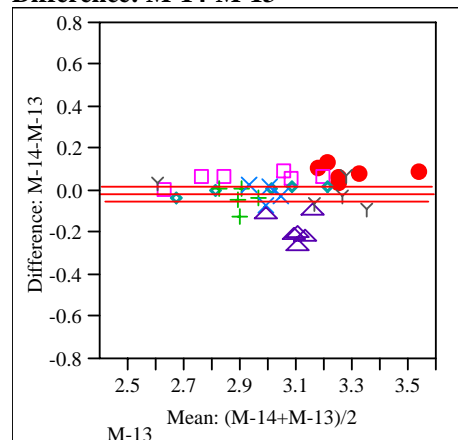
M-14	0.10083	t-Ratio	-3.52693
M-13	0.11	DF	11
Mean Difference	-0.0092	Prob >  t	0.0047
Std Error	0.0026	Prob > t	0.9976
Upper95%	-0.0034	Prob < t	0.0024
Lower95%	-0.0149		
N	12		
Correlation	-2e-13		

### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

Type/ Analyte/UoM=SME/Al/wt%

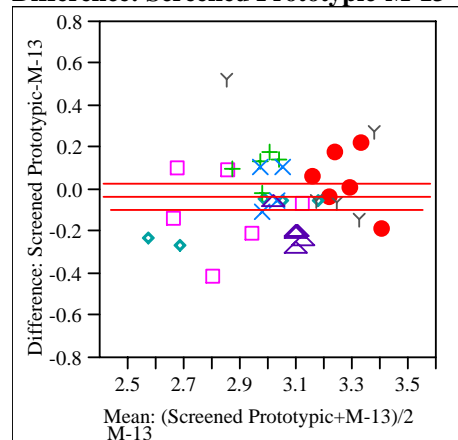
Matched Pairs

Difference: M-14-M-13



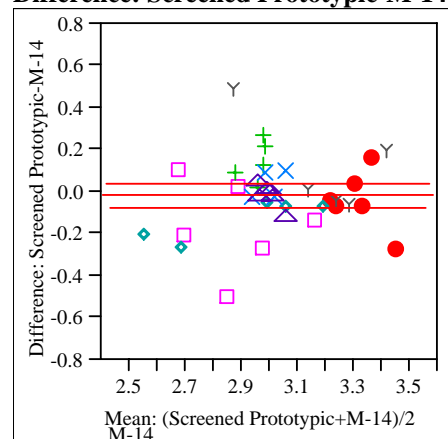
M-14	3.04622	t-Ratio	-0.7968
M-13	3.05892	DF	36
Mean Difference	-0.0127	Prob >  t	0.4308
Std Error	0.01594	Prob > t	0.7846
Upper95%	0.01963	Prob < t	0.2154
Lower95%	-0.045		
N	37		
Correlation	0.8993		

Difference: Screened Prototypic-M-13



Screened Prototypic	3.03108	t-Ratio	-0.91025
M-13	3.05892	DF	36
Mean Difference	-0.0278	Prob >  t	0.3687
Std Error	0.03058	Prob > t	0.8156
Upper95%	0.03419	Prob < t	0.1844
Lower95%	-0.0899		
N	37		
Correlation	0.65749		

Difference: Screened Prototypic-M-14

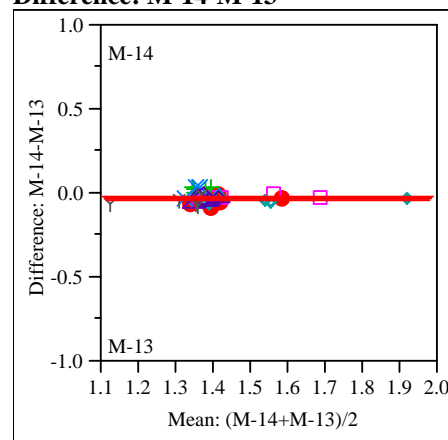


Screened Prototypic	3.03108	t-Ratio	-0.53003
M-14	3.04622	DF	36
Mean Difference	-0.0151	Prob >  t	0.5993
Std Error	0.02856	Prob > t	0.7003
Upper95%	0.04278	Prob < t	0.2997
Lower95%	-0.073		
N	37		
Correlation	0.7086		

Type/ Analyte/UoM=SME/B/wt%

Matched Pairs

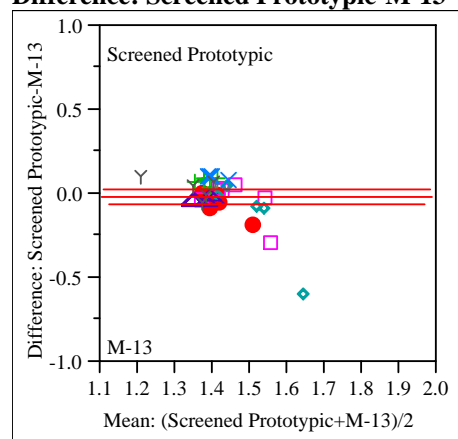
Difference: M-14-M-13



M-14	1.40027	t-Ratio	-4.57962
M-13	1.42541	DF	36
Mean Difference	-0.0251	Prob >  t	<.0001
Std Error	0.00549	Prob > t	1.0000
Upper95%	-0.014	Prob < t	<.0001
Lower95%	-0.0363		
N	37		
Correlation	0.96551		

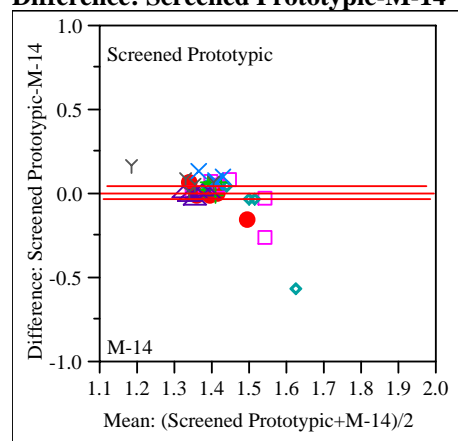
### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

**Difference: Screened Prototypic-M-13**



Screened Prototypic	1.40838	t-Ratio	-0.8167
M-13	1.42541	DF	36
Mean Difference	-0.017	Prob >  t	0.4195
Std Error	0.02085	Prob > t	0.7903
Upper95%	0.02526	Prob < t	0.2097
Lower95%	-0.0593		
N	37		
Correlation	0.22604		

**Difference: Screened Prototypic-M-14**

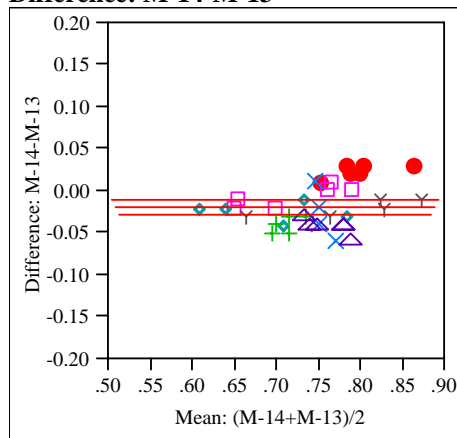


Screened Prototypic	1.40838	t-Ratio	0.407953
M-14	1.40027	DF	36
Mean Difference	0.00811	Prob >  t	0.6857
Std Error	0.01988	Prob > t	0.3429
Upper95%	0.04842	Prob < t	0.6571
Lower95%	-0.0322		
N	37		
Correlation	0.32022		

**Type/ Analyte/UoM=SME/Ca/wt%**

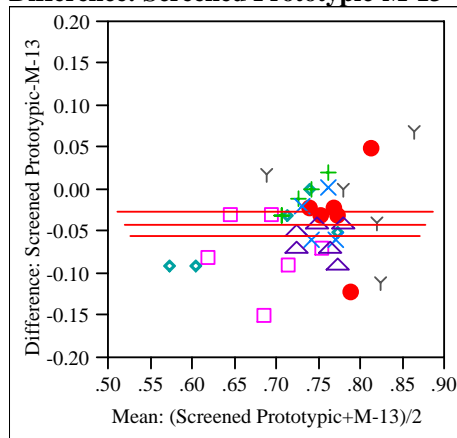
**Matched Pairs**

**Difference: M-14-M-13**



M-14	0.73973	t-Ratio	-4.43217
M-13	0.75811	DF	36
Mean Difference	-0.0184	Prob >  t	<.0001
Std Error	0.00415	Prob > t	1.0000
Upper95%	-0.01	Prob < t	<.0001
Lower95%	-0.0268		
N	37		
Correlation	0.9227		

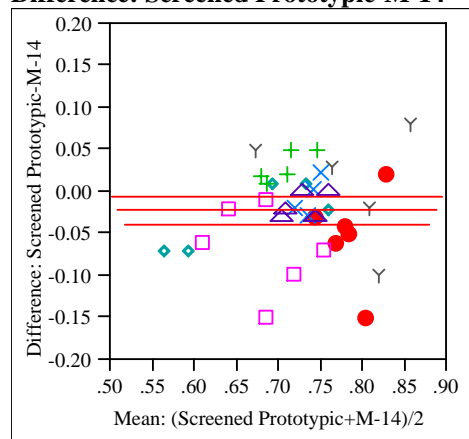
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.71784	t-Ratio	-5.35608
M-13	0.75811	DF	36
Mean Difference	-0.0403	Prob >  t	<.0001
Std Error	0.00752	Prob > t	1.0000
Upper95%	-0.025	Prob < t	<.0001
Lower95%	-0.0555		
N	37		
Correlation	0.76431		

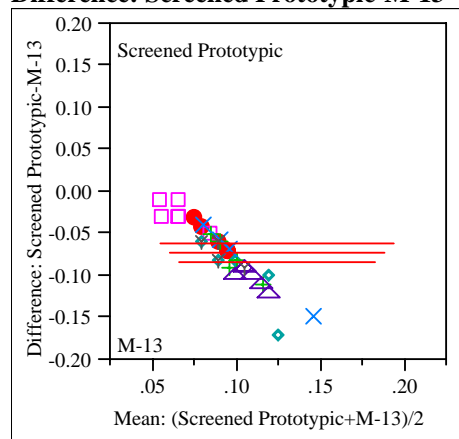
### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

**Difference: Screened Prototypic-M-14**



Screened Prototypic	0.71784	t-Ratio	-2.5711
M-14	0.73973	DF	36
Mean Difference	-0.0219	Prob >  t	0.0144
Std Error	0.00851	Prob > t	0.9928
Upper95%	-0.0046	Prob < t	0.0072
Lower95%	-0.0392		
N	37		
Correlation	0.71185		

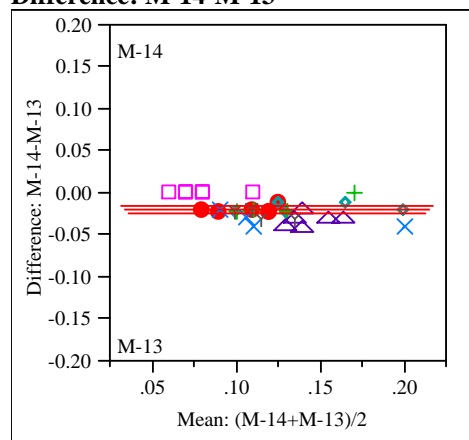
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.05703	t-Ratio	-12.5704
M-13	0.12892	DF	36
Mean Difference	-0.0719	Prob >  t	<.0001
Std Error	0.00572	Prob > t	1.0000
Upper95%	-0.0603	Prob < t	<.0001
Lower95%	-0.0835		
N	37		
Correlation	0.29969		

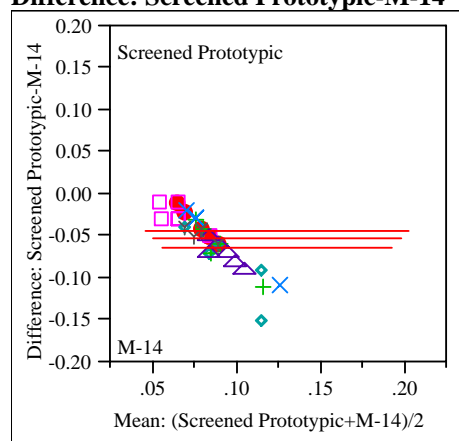
#### Type/ Analyte/UoM=SME/Cr/wt% Matched Pairs

**Difference: M-14-M-13**



M-14	0.10973	t-Ratio	-9.64206
M-13	0.12892	DF	36
Mean Difference	-0.0192	Prob >  t	<.0001
Std Error	0.00199	Prob > t	1.0000
Upper95%	-0.0152	Prob < t	<.0001
Lower95%	-0.0232		
N	37		
Correlation	0.94684		

**Difference: Screened Prototypic-M-14**



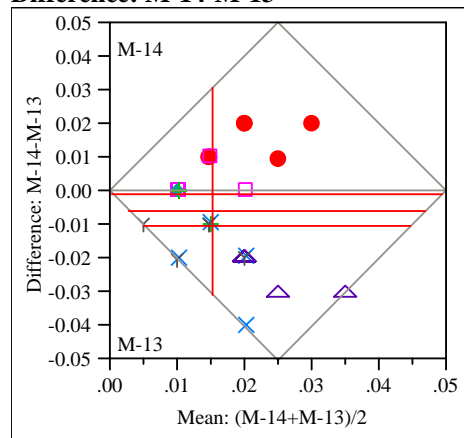
Screened Prototypic	0.05703	t-Ratio	-10.632
M-14	0.10973	DF	36
Mean Difference	-0.0527	Prob >  t	<.0001
Std Error	0.00496	Prob > t	1.0000
Upper95%	-0.0426	Prob < t	<.0001
Lower95%	-0.0628		
N	37		
Correlation	0.22674		

**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Type/ Analyte/UoM=SME/Cu/wt%**

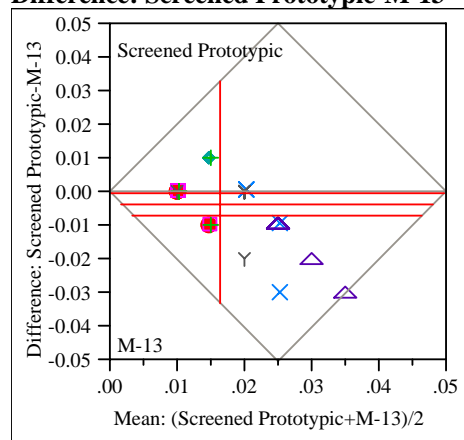
**Matched Pairs**

**Difference: M-14-M-13**



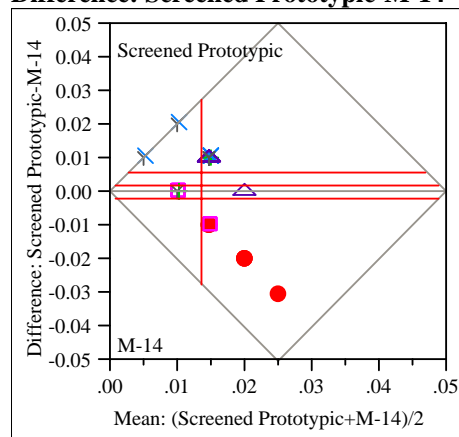
M-14	0.0127	t-Ratio	-2.39059
M-13	0.01838	DF	36
Mean Difference	-0.0057	Prob >  t	0.0222
Std Error	0.00237	Prob > t	0.9889
Upper95%	-0.0009	Prob < t	0.0111
Lower95%	-0.0105		
N	37		
Correlation	-0.101		

**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.01486	t-Ratio	-2.12447
M-13	0.01838	DF	36
Mean Difference	-0.0035	Prob >  t	0.0406
Std Error	0.00165	Prob > t	0.9797
Upper95%	-0.0002	Prob < t	0.0203
Lower95%	-0.0069		
N	37		
Correlation	0.3553		

**Difference: Screened Prototypic-M-14**

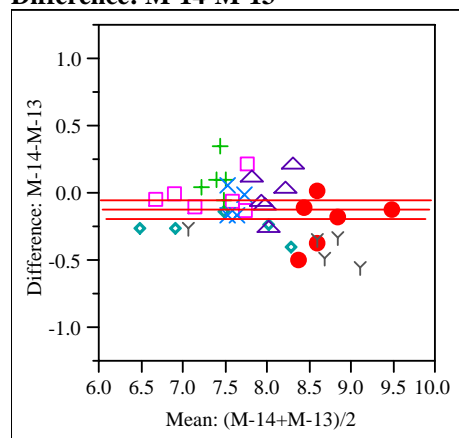


Screened Prototypic	0.01486	t-Ratio	1.135796
M-14	0.0127	DF	36
Mean Difference	0.00216	Prob >  t	0.2635
Std Error	0.0019	Prob > t	0.1318
Upper95%	0.00602	Prob < t	0.8682
Lower95%	-0.0017		
N	37		
Correlation	-0.3692		

**Type/ Analyte/UoM=SME/Fe/wt%**

**Matched Pairs**

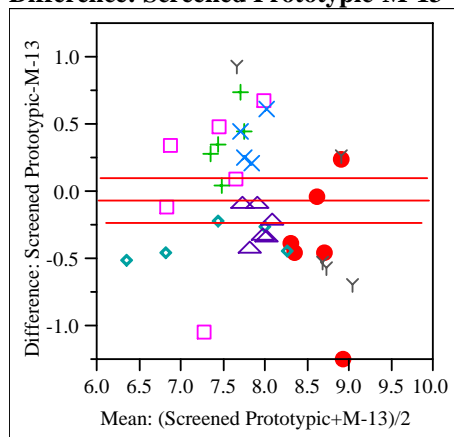
**Difference: M-14-M-13**



M-14	7.82162	t-Ratio	-3.52151
M-13	7.94081	DF	36
Mean Difference	-0.1192	Prob >  t	0.0012
Std Error	0.03385	Prob > t	0.9994
Upper95%	-0.0505	Prob < t	0.0006
Lower95%	-0.1878		
N	37		
Correlation	0.96311		

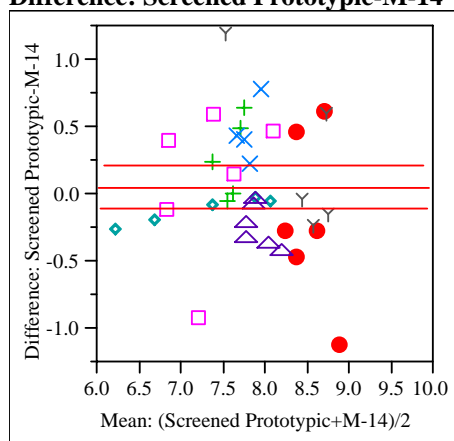
### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

#### Difference: Screened Prototypic-M-13



Screened Prototypic	7.87622	t-Ratio	-0.79438
M-13	7.94081	DF	36
Mean Difference	-0.0646	Prob >  t	0.4322
Std Error	0.08131	Prob > t	0.7839
Upper95%	0.10032	Prob < t	0.2161
Lower95%	-0.2295		
N	37		
Correlation	0.75493		

#### Difference: Screened Prototypic-M-14

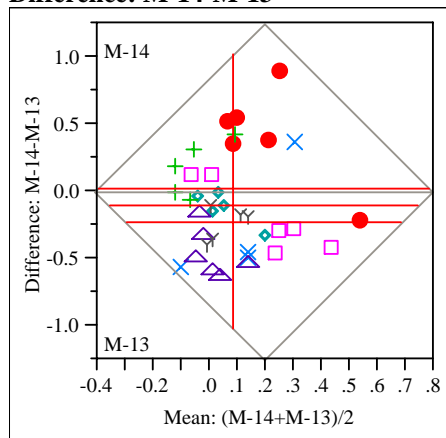


Screened Prototypic	7.87622	t-Ratio	0.709394
M-14	7.82162	DF	36
Mean Difference	0.05459	Prob >  t	0.4826
Std Error	0.07696	Prob > t	0.2413
Upper95%	0.21068	Prob < t	0.7587
Lower95%	-0.1015		
N	37		
Correlation	0.74557		

#### Type/ Analyte/UoM=SME/K/wt%

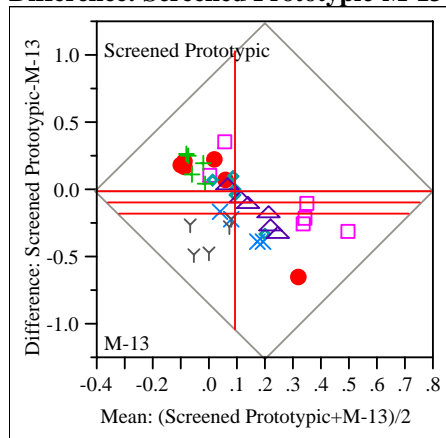
#### Matched Pairs

#### Difference: M-14-M-13



M-14	0.04108	t-Ratio	-1.59776
M-13	0.13973	DF	36
Mean Difference	-0.0986	Prob >  t	0.1188
Std Error	0.06174	Prob > t	0.9406
Upper95%	0.02657	Prob < t	0.0594
Lower95%	-0.2239		
N	37		
Correlation	-0.2048		

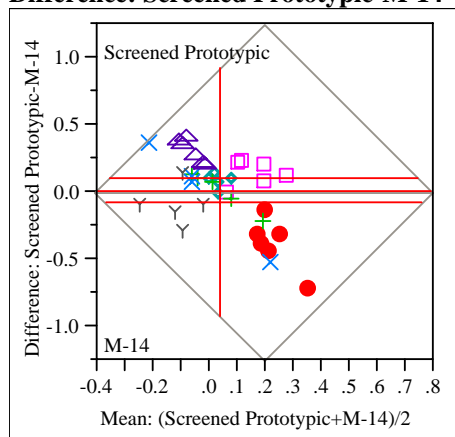
#### Difference: Screened Prototypic-M-13



Screened Prototypic	0.05162	t-Ratio	-2.14885
M-13	0.13973	DF	36
Mean Difference	-0.0881	Prob >  t	0.0384
Std Error	0.041	Prob > t	0.9808
Upper95%	-0.005	Prob < t	0.0192
Lower95%	-0.1713		
N	37		
Correlation	0.19903		

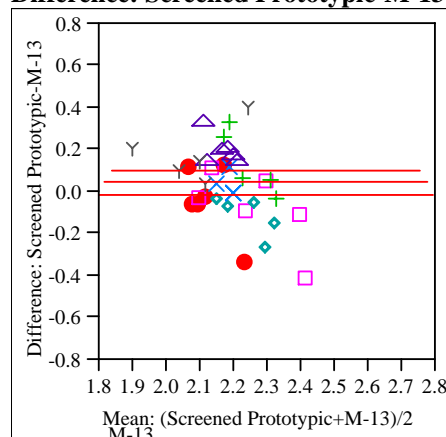
**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements  
for Each Element by Type of Sample after Screening**

**Difference: Screened Prototypic-M-14**



Screened Prototypic	0.05162	t-Ratio	0.243439
M-14	0.04108	DF	36
Mean Difference	0.01054	Prob >  t	0.8090
Std Error	0.0433	Prob > t	0.4045
Upper95%	0.09835	Prob < t	0.5955
Lower95%	-0.0773		
N	37		
Correlation	0.0948		

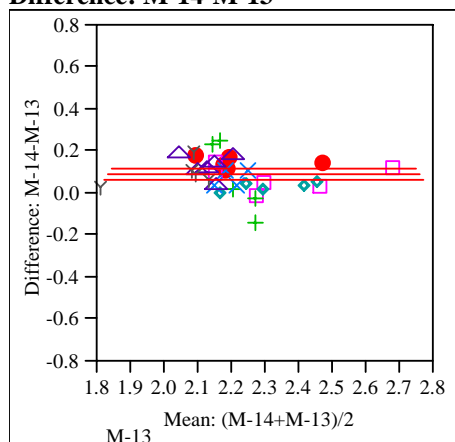
**Difference: Screened Prototypic-M-13**



Screened Prototypic	2.21027	t-Ratio	1.575803
M-13	2.16541	DF	36
Mean Difference	0.04486	Prob >  t	0.1238
Std Error	0.02847	Prob > t	0.0619
Upper95%	0.10261	Prob < t	0.9381
Lower95%	-0.0129		
N	37		
Correlation	0.16539		

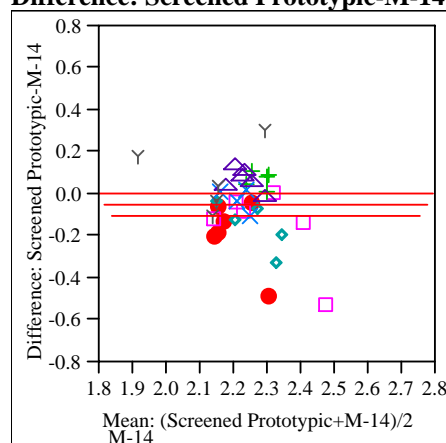
**Type/ Analyte/UoM=SME/Li/wt%  
Matched Pairs**

**Difference: M-14-M-13**



M-14	2.25649	t-Ratio	6.977643
M-13	2.16541	DF	36
Mean Difference	0.09108	Prob >  t	<.0001
Std Error	0.01305	Prob > t	<.0001
Upper95%	0.11755	Prob < t	1.0000
Lower95%	0.06461		
N	37		
Correlation	0.86861		

**Difference: Screened Prototypic-M-14**



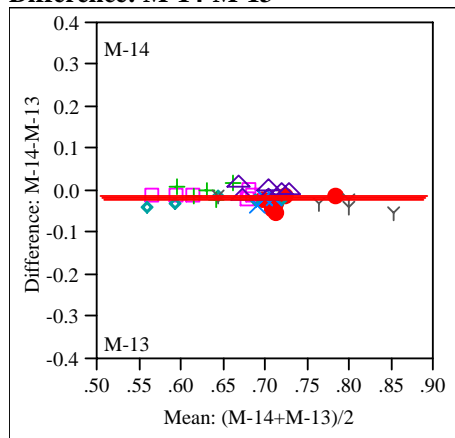
Screened Prototypic	2.21027	t-Ratio	-1.72613
M-14	2.25649	DF	36
Mean Difference	-0.0462	Prob >  t	0.0929
Std Error	0.02677	Prob > t	0.9536
Upper95%	0.00808	Prob < t	0.0464
Lower95%	-0.1005		
N	37		
Correlation	0.14722		

### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

Type/ Analyte/UoM=SME/Mg/wt%

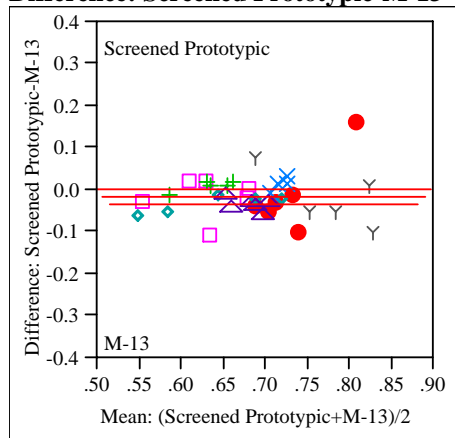
Matched Pairs

Difference: M-14-M-13



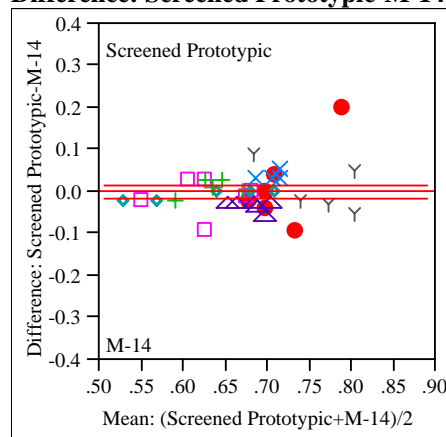
M-14	0.67838	t-Ratio	-5.52869
M-13	0.69405	DF	36
Mean Difference	-0.0157	Prob >  t	<.0001
Std Error	0.00284	Prob > t	1.0000
Upper95%	-0.0099	Prob < t	<.0001
Lower95%	-0.0214		
N	37		
Correlation	0.96942		

Difference: Screened Prototypic-M-13



Screened Prototypic	0.67838	t-Ratio	-1.95877
M-13	0.69405	DF	36
Mean Difference	-0.0157	Prob >  t	0.0579
Std Error	0.008	Prob > t	0.9710
Upper95%	0.00055	Prob < t	0.0290
Lower95%	-0.0319		
N	37		
Correlation	0.76727		

Difference: Screened Prototypic-M-14

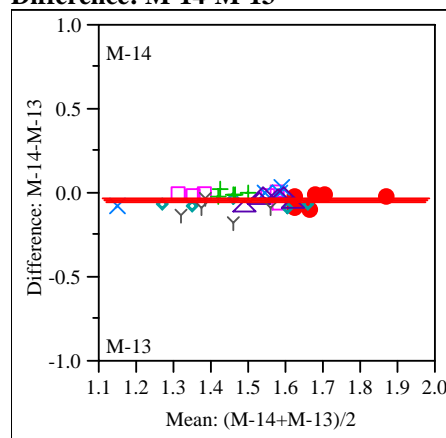


Screened Prototypic	0.67838	t-Ratio	1.07e-15
M-14	0.67838	DF	36
Mean Difference	9e-18	Prob >  t	1.0000
Std Error	0.00838	Prob > t	0.5000
Upper95%	0.017	Prob < t	0.5000
Lower95%	-0.017		
N	37		
Correlation	0.73083		

Type/ Analyte/UoM=SME/Mn/wt%

Matched Pairs

Difference: M-14-M-13

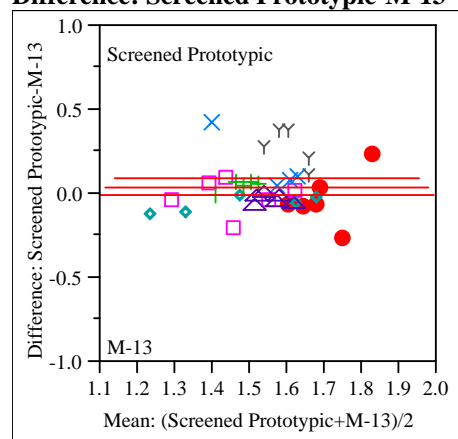


M-14	1.49189	t-Ratio	-5.1381
M-13	1.52892	DF	36
Mean Difference	-0.037	Prob >  t	<.0001
Std Error	0.00721	Prob > t	1.0000
Upper95%	-0.0224	Prob < t	<.0001
Lower95%	-0.0516		
N	37		
Correlation	0.9542		



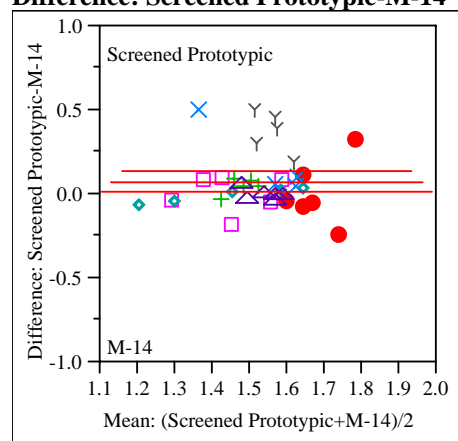
### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

#### Difference: Screened Prototypic-M-13



Screened Prototypic	1.56811	t-Ratio	1.583652
M-13	1.52892	DF	36
Mean Difference	0.03919	Prob >  t	0.1220
Std Error	0.02475	Prob > t	0.0610
Upper95%	0.08938	Prob < t	0.9390
Lower95%	-0.011		
N	37		
Correlation	0.46826		

#### Difference: Screened Prototypic-M-14

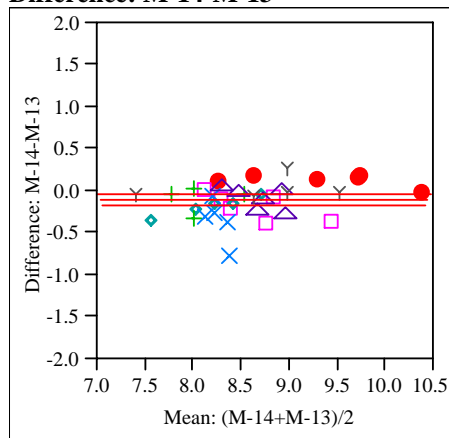


Screened Prototypic	1.56811	t-Ratio	2.663012
M-14	1.49189	DF	36
Mean Difference	0.07622	Prob >  t	0.0115
Std Error	0.02862	Prob > t	0.0058
Upper95%	0.13426	Prob < t	0.9942
Lower95%	0.01817		
N	37		
Correlation	0.32118		

#### Type/ Analyte/UoM=SME/Na/wt%

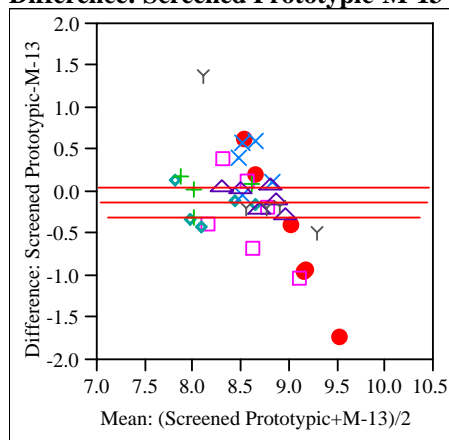
#### Matched Pairs

#### Difference: M-14-M-13



M-14	8.54784	t-Ratio	-3.02295
M-13	8.65135	DF	36
Mean Difference	-0.1035	Prob >  t	0.0046
Std Error	0.03424	Prob > t	0.9977
Upper95%	-0.0341	Prob < t	0.0023
Lower95%	-0.173		
N	37		
Correlation	0.95122		

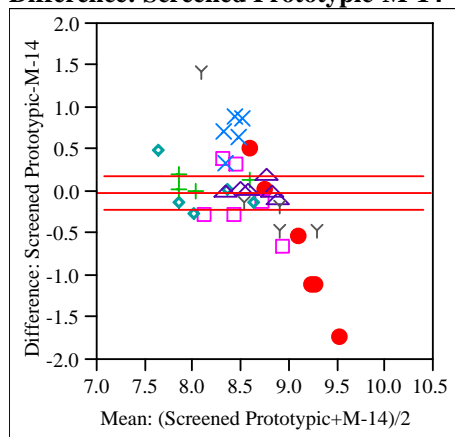
#### Difference: Screened Prototypic-M-13



Screened Prototypic	8.53784	t-Ratio	-1.2854
M-13	8.65135	DF	36
Mean Difference	-0.1135	Prob >  t	0.2069
Std Error	0.08831	Prob > t	0.8966
Upper95%	0.06559	Prob < t	0.1034
Lower95%	-0.2926		
N	37		
Correlation	0.46972		

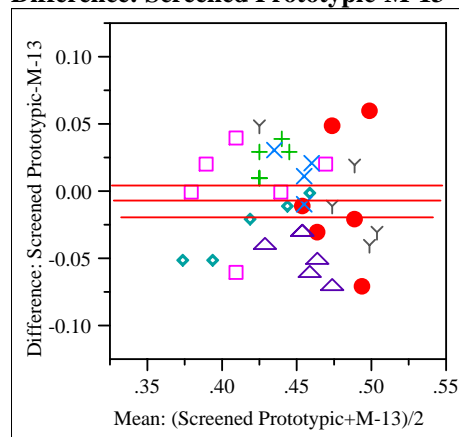
**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening**

**Difference: Screened Prototypic-M-14**



Screened Prototypic	8.53784	t-Ratio	-0.104
M-14	8.54784	DF	36
Mean Difference	-0.01	Prob >  t	0.9177
Std Error	0.09616	Prob > t	0.5411
Upper95%	0.18502	Prob < t	0.4589
Lower95%	-0.205		
N	37		
Correlation	0.48363		

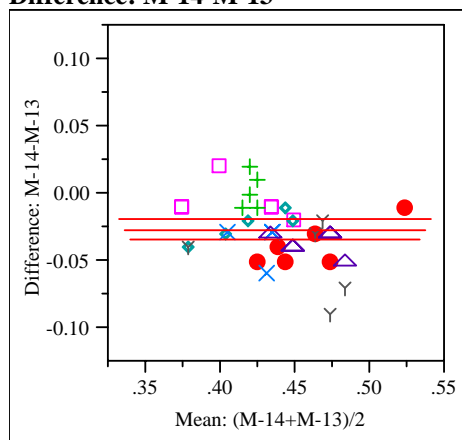
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.44459	t-Ratio	-1.13493
M-13	0.45135	DF	36
Mean Difference	-0.0068	Prob >  t	0.2639
Std Error	0.00595	Prob > t	0.8680
Upper95%	0.00532	Prob < t	0.1320
Lower95%	-0.0188		
N	37		
Correlation	0.55252		

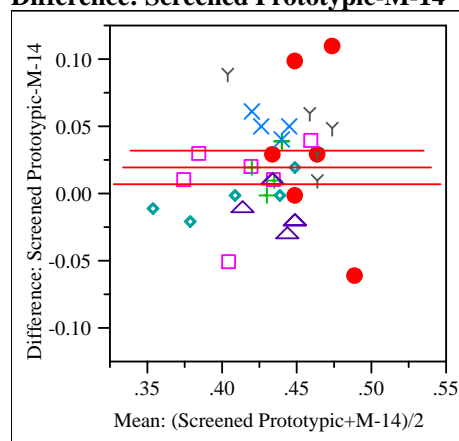
**Type/ Analyte/UoM=SME/Ni/wt%  
Matched Pairs**

**Difference: M-14-M-13**



M-14	0.42432	t-Ratio	-7.16115
M-13	0.45135	DF	36
Mean Difference	-0.027	Prob >  t	<.0001
Std Error	0.00377	Prob > t	1.0000
Upper95%	-0.0194	Prob < t	<.0001
Lower95%	-0.0347		
N	37		
Correlation	0.81024		

**Difference: Screened Prototypic-M-14**



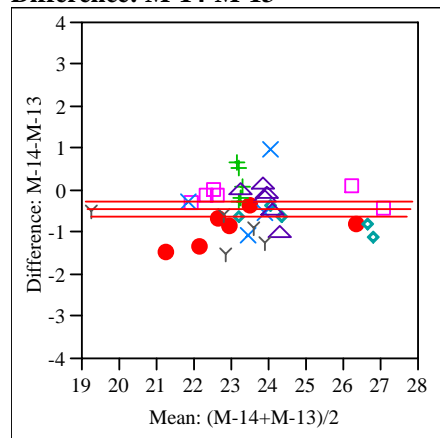
Screened Prototypic	0.44459	t-Ratio	3.311869
M-14	0.42432	DF	36
Mean Difference	0.02027	Prob >  t	0.0021
Std Error	0.00612	Prob > t	0.0011
Upper95%	0.03268	Prob < t	0.9989
Lower95%	0.00786		
N	37		
Correlation	0.42817		

### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

Type/ Analyte/UoM=SME/Si/wt%

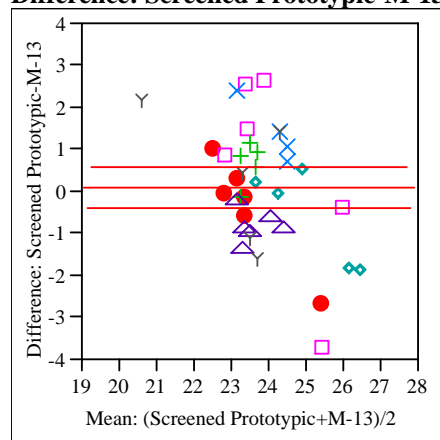
Matched Pairs

Difference: M-14-M-13



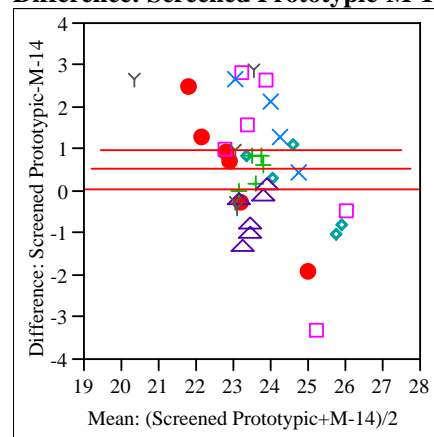
M-14	23.3603	t-Ratio	-4.5691
M-13	23.7832	DF	36
Mean Difference	-0.423	Prob >  t	<.0001
Std Error	0.09257	Prob > t	1.0000
Upper95%	-0.2352	Prob < t	<.0001
Lower95%	-0.6107		
N	37		
Correlation	0.9371		

Difference: Screened Prototypic-M-13



Screened Prototypic	23.8995	t-Ratio	0.494498
M-13	23.7832	DF	36
Mean Difference	0.11622	Prob >  t	0.6240
Std Error	0.23502	Prob > t	0.3120
Upper95%	0.59286	Prob < t	0.6880
Lower95%	-0.3604		
N	37		
Correlation	0.45891		

Difference: Screened Prototypic-M-14

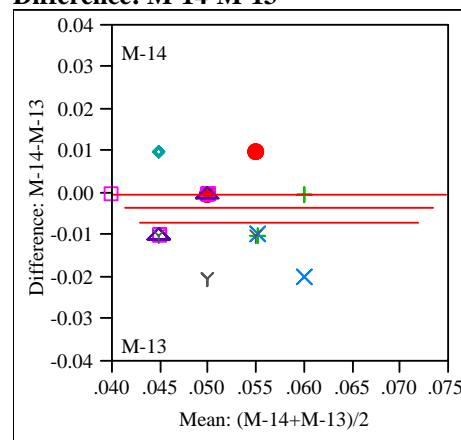


Screened Prototypic	23.8995	t-Ratio	2.336089
M-14	23.3603	DF	36
Mean Difference	0.53919	Prob >  t	0.0252
Std Error	0.23081	Prob > t	0.0126
Upper95%	1.00729	Prob < t	0.9874
Lower95%	0.07109		
N	37		
Correlation	0.47716		

Type/ Analyte/UoM=SME/Ti/wt%

Matched Pairs

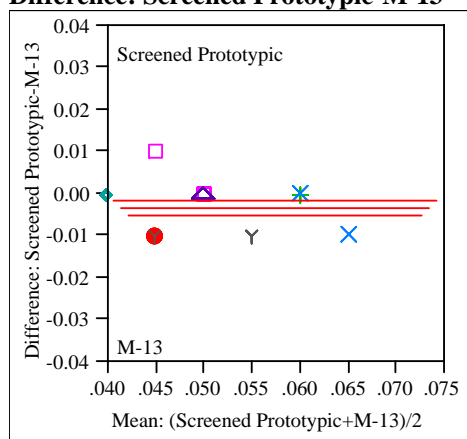
Difference: M-14-M-13



M-14	0.04892	t-Ratio	-2.12447
M-13	0.05243	DF	36
Mean Difference	-0.0035	Prob >  t	0.0406
Std Error	0.00165	Prob > t	0.9797
Upper95%	-0.0002	Prob < t	0.0203
Lower95%	-0.0069		
N	37		
Correlation	0.09856		

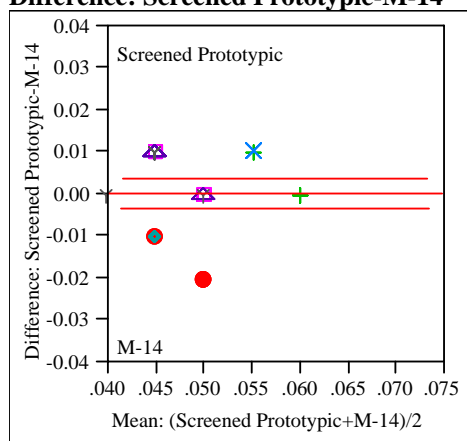
### Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening

#### Difference: Screened Prototypic-M-13



Screened Prototypic	0.04919	t-Ratio	-3.72303
M-13	0.05243	DF	36
Mean Difference	-0.0032	Prob >  t	0.0007
Std Error	0.00087	Prob > t	0.9997
Upper95%	-0.0015	Prob < t	0.0003
Lower95%	-0.005		
N	37		
Correlation	0.76885		

#### Difference: Screened Prototypic-M-14

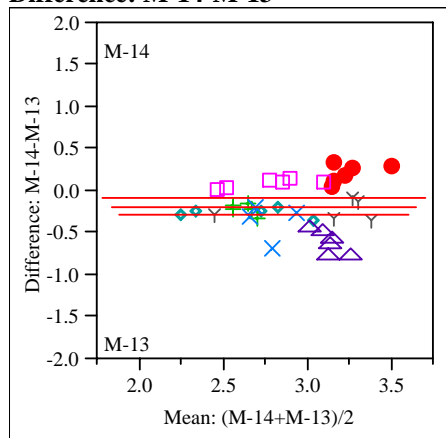


Screened Prototypic	0.04919	t-Ratio	0.158004
M-14	0.04892	DF	36
Mean Difference	0.00027	Prob >  t	0.8753
Std Error	0.00171	Prob > t	0.4377
Upper95%	0.00374	Prob < t	0.5623
Lower95%	-0.0032		
N	37		
Correlation	-0.017		

#### Type/ Analyte/UoM=SME/U/wt%

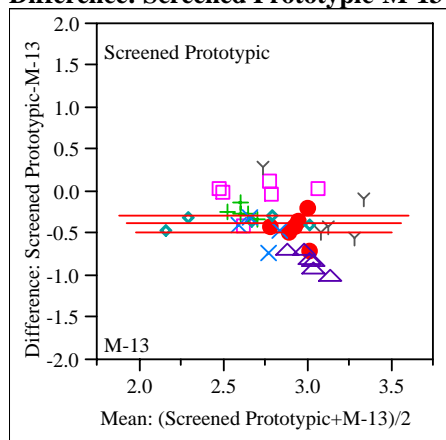
#### Matched Pairs

#### Difference: M-14-M-13



M-14	2.82568	t-Ratio	-3.86492
M-13	3.01027	DF	36
Mean Difference	-0.1846	Prob >  t	0.0004
Std Error	0.04776	Prob > t	0.9998
Upper95%	-0.0877	Prob < t	0.0002
Lower95%	-0.2815		
N	37		
Correlation	0.66046		

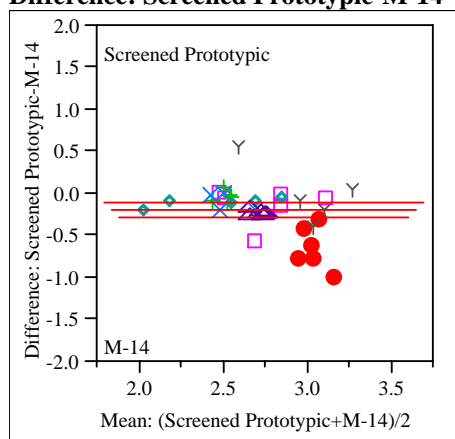
#### Difference: Screened Prototypic-M-13



Screened Prototypic	2.63486	t-Ratio	-7.70947
M-13	3.01027	DF	36
Mean Difference	-0.3754	Prob >  t	<.0001
Std Error	0.04869	Prob > t	1.0000
Upper95%	-0.2766	Prob < t	<.0001
Lower95%	-0.4742		
N	37		
Correlation	0.53863		

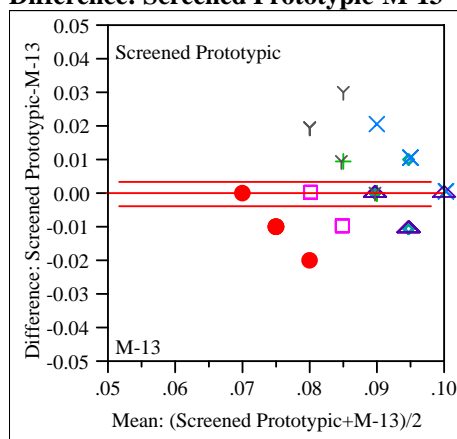
**Exhibit A3. Initial Comparisons by SME Batch among M-13, M-14, and Prototypic Measurements for Each Element by Type of Sample after Screening**

**Difference: Screened Prototypic-M-14**



Screened Prototypic	2.63486	t-Ratio	-4.22214
M-14	2.82568	DF	36
Mean Difference	-0.1908	Prob >  t	0.0002
Std Error	0.04519	Prob > t	0.9999
Upper95%	-0.0992	Prob < t	<.0001
Lower95%	-0.2825		
N	37		
Correlation	0.65249		

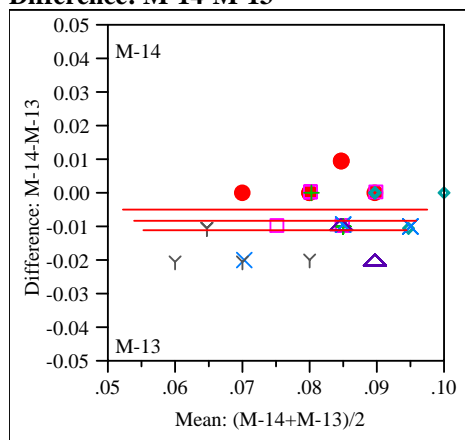
**Difference: Screened Prototypic-M-13**



Screened Prototypic	0.0873	t-Ratio	0
M-13	0.0873	DF	36
Mean Difference	0	Prob >  t	1.0000
Std Error	0.00178	Prob > t	0.5000
Upper95%	0.0036	Prob < t	0.5000
Lower95%	-0.0036		
N	37		
Correlation	0.36999		

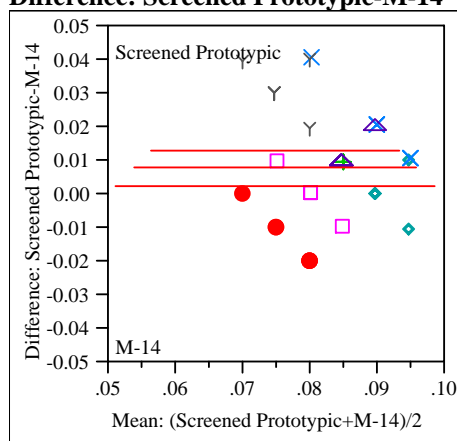
**Type/ Analyte/UoM=SME/Zr/wt%  
Matched Pairs**

**Difference: M-14-M-13**



M-14	0.07946	t-Ratio	-5.58105
M-13	0.0873	DF	36
Mean Difference	-0.0078	Prob >  t	<.0001
Std Error	0.0014	Prob > t	1.0000
Upper95%	-0.005	Prob < t	<.0001
Lower95%	-0.0107		
N	37		
Correlation	0.64789		

**Difference: Screened Prototypic-M-14**



Screened Prototypic	0.0873	t-Ratio	3.044677
M-14	0.07946	DF	36
Mean Difference	0.00784	Prob >  t	0.0043
Std Error	0.00257	Prob > t	0.0022
Upper95%	0.01306	Prob < t	0.9978
Lower95%	0.00262		
N	37		
Correlation	-0.1441		

**Distribution:**

C.J. Bannochie, 773-42A	S.P. Harris, 773-42A
M.J. Barnes, SRNL	C.C. Herman, 773-42A
N.E. Bibler, SRNL	C.M. Jantzen, SRNL
D.R. Best, 786-1A	R.N. Mahannah, 704-28S
D.B. Burns, 786-5A	J.E. Marra, SRNL
T.B. Calloway, 999-W	W.L. Melton III, 704-28S
L.M. Chandler, SRNL	M. S. Miller, 704-S
D.R. Click, SRNL	J.E. Occhipinti, 704-S
C.J. Coleman, SRNL	P.M. Patel, 704-27S
A.J. Cross, 704-71S	F.M. Pennebaker, SRNL
D.A. Crowley, SRNL	D.K. Peeler, 999-W
B.H. Culbertson, 704-27S	J.W. Ray, 704-S
B.A. Davis, 704-27S	M.E. Smith, 773-42A
R.E. Edwards, SRNL	M.E. Stone, 999-W
T.B. Edwards, 773-42A	R.C. Tuckfield, 773-42A
M.T. Feller, 704-28S	J.P. Vaughan, 773-41A
T.L. Fellingner, 704-27S	S.W. Wilkerson, 704-S