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EVALUATION OF ARG-1 SAMPLES PREPARED BY CESIUM CARBONATE DISSOLUTION DURING THE ISOLOK SME ACCEPTABILITY TESTING

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EXECUTIVE SUMMARY

Evaluation of the Defense Waste Processing Facility (DWPF) Chemical Process Cell (CPC) cycle time identified several opportunities to improve the CPC processing time. The Mechanical Systems & Custom Equipment Development (MS&CED) Section of the Savannah River National Laboratory (SRNL) recently completed the evaluation of one of these opportunities — the possibility of using an Isolok sampling valve as an alternative to the Hydragard valve for taking DWPF process samples at the Slurry Mix Evaporator (SME). The use of an Isolok for SME sampling has the potential to improve operability, reduce maintenance time, and decrease CPC cycle time. The SME acceptability testing for the Isolok was requested in Task Technical Request (TTR) HLW-DWPF-TTR-2010-0036 and was conducted as outlined in Task Technical and Quality Assurance Plan (TTQAP) SRNL-RP-2011-00145. RW-0333P QA requirements applied to the task, and the results from the investigation were documented in SRNL-STI-2011-00693.

The objective of that study was to qualify the Isolok for use in sampling the Slurry Mix Evaporator (SME) tank at the DWPF. Measurement of the chemical composition of study samples was a critical component of the SME acceptability testing of the Isolok. A sampling and analytical plan, SRNL-RP-2011-00294, supported the investigation with the analytical plan directing that the study samples be prepared by a cesium carbonate (Cs_2CO_3) fusion dissolution method and analyzed by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP–OES). The use of the cesium carbonate preparation method for the Isolok testing provided an opportunity for an additional assessment of this dissolution method, which is being investigated as a potential replacement for the two methods (i.e., sodium peroxide fusion and mixed acid dissolution) that have been used at the DWPF for the analysis of SME samples. The Cs_2CO_3 testing associated with the Isolok testing does provide additional insight into the performance of the method as conducted by SRNL. The performance is to be investigated by looking to the composition measurement data generated by the samples of a standard glass, the Analytical Reference Glass – 1 (ARG-1), that were prepared by the Cs_2CO_3 method and included in the SME acceptability testing of the Isolok. The measurements of theses samples were presented in SRNL-STI-2011-00693, but no statistical analysis of these measurements was conducted as part of those results.

The ARG-1 measurements, as weight percent (wt%) oxides, generated during the SME acceptability testing for the Isolok are statistically analyzed in this report. Three sources of variation in these measurements were explored: variation due to preparation block effects, variation due to ICP-OES analytical block effects, and within block variation for these measurements, where the within block variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process. These results suggest that differences among the preparation blocks played an insignificant role in the variation seen in the ARG-1 results over the course of the Isolok testing. From this investigation, the components of variation (i.e., the analytical block-to-block effects and the within-block effects) as % relative standard deviations are less than 5% for those oxides at concentrations of 0.1 wt% or greater. A bound on the bias in the Cs_2CO_3 method of its measurements for each oxide of interest value is also provided at a 95% confidence. For those oxides present in ARG-1 at concentrations greater than or equal to 0.1 wt%, only B_2O_3 has a bias bound (its value is 9.84%) that is larger than 4.41% (the value for MnO). Thus, only B_2O_3 has a bias bound that is larger than 5%.

The low bias for boron measurements is almost certainly due to volatilization of boron at the 1050 °C fusion conditions of the Cs_2CO_3 method. Cesium species also can be volatilized under these conditions and it is believed that a carrier distillation effect results in the loss of boron. The relatively high fusion temperature was used in anticipation of needing very rigorous fusion conditions for dissolving a 1-1.5 gram wafer of glass that potentially results from vitrifying a 3 mL SME sample taken with the Isolok sampler. Experiments performed at the DWPF Laboratory and at SRNL indicate that the low boron bias from the Cs_2CO_3 method is essentially eliminated when the method is carried out at 900 °C. The lower fusion temperature is effective for attacking powdered glass and small shards of glass, but may not result in complete attack of a1.5 gram wafer of glass.

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LIST OF ABBREVIATIONS

ARG-1	Analytical Reference Glass - 1
DWPF	Defense Waste Processing Facility
ICP–OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
JMP	Statistical software package from SAS Institute, Inc.
SME	Slurry Mix Evaporator
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation, LLC
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance
wt%	Weight Percent

1.0 Introduction

Evaluation of Defense Waste Processing Facility (DWPF) Chemical Process Cell (CPC) cycle time identified several opportunities to improve the CPC processing time. The Mechanical Systems & Custom Equipment Development (MS&CED) Section of the Savannah River National Laboratory (SRNL) recently completed the evaluation of one of these opportunities — the possibility of using an Isolok sampling valve as an alternative to the Hydragard valve for taking DWPF process samples at the Slurry Mix Evaporator (SME). The use of an Isolok for SME sampling has the potential to improve operability, reduce maintenance time, and decrease CPC cycle time. The SME acceptability testing for the Isolok was requested in Task Technical Request (TTR) HLW-DWPF-TTR-2010-0036 [1] and was conducted as outlined in Task Technical and Quality Assurance Plan (TTQAP) SRNL-RP-2011-00145 [2]. RW-0333P QA requirements applied to the task, and the results from the investigation were documented in SRNL-STI-2011-00693 [3].

Measurement of the chemical composition of study samples was a critical component of the SME acceptability testing of the Isolok. A sampling and analytical plan [4] supported the investigation with the analytical plan directing that the study samples be prepared by a cesium carbonate (Cs_2CO_3) fusion dissolution method and analyzed by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP–OES). The use of the cesium carbonate preparation method for the Isolok testing provided an opportunity for an additional assessment of this dissolution method, which is being investigated as a potential replacement for the two methods (i.e., sodium peroxide fusion and mixed acid dissolution) that have been used at the DWPF for the analysis of SME samples. Earlier testing of the Cs_2CO_3 method yielded promising results [5]-[7] which led to a TTR [8] from Savannah River Remediation, LLC (SRR) to SRNL for additional support and an associated TTQAP [9] to direct the SRNL efforts. A technical report [8] resulting from this work was issued that recommended that the mixed acid method be replaced by the Cs_2CO_3 method for the measurement of magnesium (Mg), sodium (Na), and zirconium (Zr) with additional testing of the method by DWPF Laboratory being needed before further implementation of the Cs_2CO_3 method at that laboratory.

While the SME acceptability testing of the Isolok does not address any of the open issues remaining after the publication of the recommendation for the replacement of the mixed acid method by the Cs_2CO_3 method [10] (since those issues are to be addressed by the DWPF Laboratory), the Cs_2CO_3 testing associated with the Isolok testing does provide additional insight into the performance of the method as conducted by SRNL. The performance is to be investigated by looking to the composition measurement data generated by the samples of a standard glass, the Analytical Reference Glass – 1 (ARG-1), that were prepared by the Cs_2CO_3 method and included in the SME acceptability testing of the Isolok. The measurements of these samples were presented as part of the study results [3], but no statistical analysis of these measurements was conducted as part of those results. It is the purpose of this report to provide that analysis, which was supported using JMP Version 7.0.2 [11].

2.0 Discussion

The ARG-1 measurements, as weight percent (wt%) oxides, that are of interest in this investigation are provided in Table A1 in the Appendix. The values were generated by the analytical plan [4] that was issued to support the SME acceptability testing conducted for the Isolok. Table A2 in the Appendix provides the reference composition of the ARG-1 standard as wt% oxides.

2.1 Initial Plots of the Measurements

Exhibit A1 in the Appendix provides a series of plots by oxide of the measurements of Table A1 grouped by test phase, preparation groupings, and analytical groupings. The sample ID and the targeted value for each oxide from Table A2 are also shown as part of the information on the x-axis of each plot. The average of all of the measurements over all three test phases is shown on each plot as a horizontal line. Included in this exhibit is a plot of the sums of oxides for the measurements.

Some interesting observations can be made for the sum of oxides plot. As seen in this plot, the targeted sum for the ARG-1 oxides of interest in this report was 99.43 wt%. Eight of the 54 sums (i.e., ~ 14.8%) fall outside of the interval from 95 to 105 wt%, where this interval is typically used as one of the metrics in assessing the analytical process that was used to generate the chemical composition measurements. There are two aspects of these results that are worth noting here. One is that those samples showing a low sum of oxides also show low recoveries for many of the other oxides, not just SiO_2. Of even more interest is the fact that 6 of the 8 samples that have a sum that falls below the 95% value are actually measured in two analytical blocks but their sum only falls below the 95% value in one of the two blocks. See Table 1. This suggests that the preparation of the samples was not an issue in the low sums of oxides seen for some of the results, but the likely problem was the performance of the ICP-OES instrumentation or human errors such mislabeling of sample bottles or an error during sample dilution. There is also a suggestion that one of the bigger contributors to the variation seen in the measurements for all of the oxides is the repeatability of the measurements by the ICP-OES instrumentation. The sources of variation are investigated in the next section.

Table 1. A Subset of the Sums of Oxides Values

Test	Preparation Block	Analytical Block	Sample ID	Measurement
Phase 1	2	2	ARG-1B22	99.62
Phase 1	2	5	ARG-1B22	91.63
Phase 1	3	3	ARG-1B33	102.38
Phase 1	3	6	ARG-1B33	94.27
Phase 2	2	2	ARG-1B21	92.86
Phase 2	2	5	ARG-1B21	98.08
Phase 2	2	2	ARG-1B23	98.93
Phase 2	2	5	ARG-1B23	93.79
Phase 2	3	3	ARG-1B31	93.92
Phase 2	3	6	ARG-1B31	98.00
Phase 2	3	3	ARG-1B33	101.05
Phase 2	3	6	ARG-1B33	92.75

2.2 Preparation Block versus ICP-OES Analytical Block Variation

For those ARG-1 samples that were measured twice during the Isolok testing, once in each of two different ICP-OES analytical blocks, there is an opportunity to estimate the variation in these measurements due to preparation block effects and the variation due the ICP-OES analytical block effects. These variations are assessed relative to the within block variation for these measurements, where the within block variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process. The statistical model for the measurement of each of the oxides of interest that facilitates the estimation of the components of variance is given by:

Equation 1.

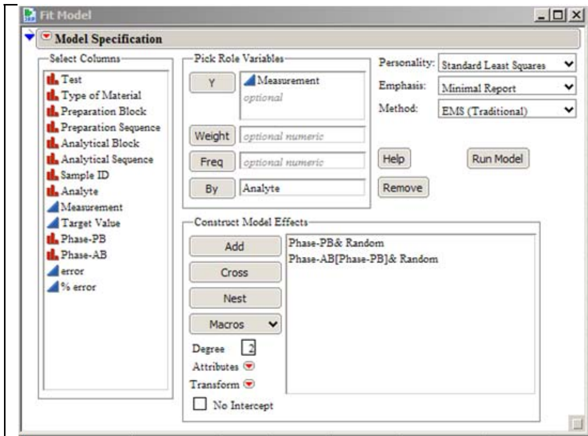
$$y_{ijk} = \mu + p_i + a_{j(i)} + e_{ijk}$$

where

- y_{ijk} is the measurement of the k^{th} sample in analytical block j for a sample prepared in preparation block i,
- μ represents the average measurement for the given oxide,
- p_i represents a random effect for the i^{th} preparation block that captures block-to-block effects,
- $a_{j(i)}$ represents a random effect for the j^{th} analytical block nested within the i^{th} preparation block, this random effect captures block-to-block effects for the analytical process, and
- e_{ijk} represents the within block variation, and as described above this variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process.

The a's in this model are assumed to be normally distributed with a zero mean and with a constant but unknown standard deviation that may be represented by σ_a. The p's in the model are assumed to be normally distributed with a zero mean and with a constant but unknown standard deviation that may be represented by σ_p. The e's in this model are random effects that are assumed to be normally distributed with a zero mean and with a constant but unknown standard deviation that may be represented by σ_e.

To facilitate the analysis of this model using JMP, the preparation blocks and analytical blocks were uniquely tied to the test phases as indicated in Exhibit 1. The results from the JMP analysis are provided in Exhibit A2 in the Appendix, and they include tests for the statistical significance of two components of variation of the model in equation (1) (i.e., σ_p and σ_a) relative to the size of the within block component, i.e., the standard deviation, σ_e. The outcome of one of these tests indicates a statistically significant component of variation by its p-value, which is labeled as "**Prob > F**" in the portion of the results with the heading "**Tests wrt Random Effects**." When the p-value is less than 0.05, then the estimated component of variation is statistically significant at the 5% level. To help with this interpretation, the p-values in Exhibit A2 have been shaded: green if the variance component is not statistically significant and red if it is. The estimated variance for the analytical blocks is statistically significant for several of the oxides (specifically, B_2O_3, Li_2O, MgO, MnO, and NiO), while the estimated variance for the preparation blocks is statistically significant for only CuO and ZnO (two minor oxides for ARG-1). These results suggest that differences among the preparation blocks played an insignificant role in the variation seen in the ARG-1 results over the course of the Isolok testing. This allows for the use of a less complex model with a more complete set of the measurements.



Phase-PB stands for Phase-preparation block and is used to uniquely identify these blocks over all of the Isolok test phases.

Phase-AB stands for Phase analytical block and is used to uniquely identify these blocks over all of the Isolok test phases.

Also, note that the Phase-AB terms are nested within the Phase-PB blocks, as was the case for the data selected for this analysis

Exhibit 1. Nested, Random Effects Model for Preparation and Analytical Block Effects

2.3 ICP-OES Analytical Block Variation

In this section two sources for the variation in the ARG-1 measurements is investigated: an ICP-OES analytical block-to-block effect and the within analytical block variation. For this analysis, all of the ARG-1 measurements were used. Once again, the within block variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process. The statistical model for the measurement of each of the oxides of interest that facilitates the estimation of the components of variance is given by:

Equation 1.

$$y_{ij} = \mu + a_i + e_{ij}$$

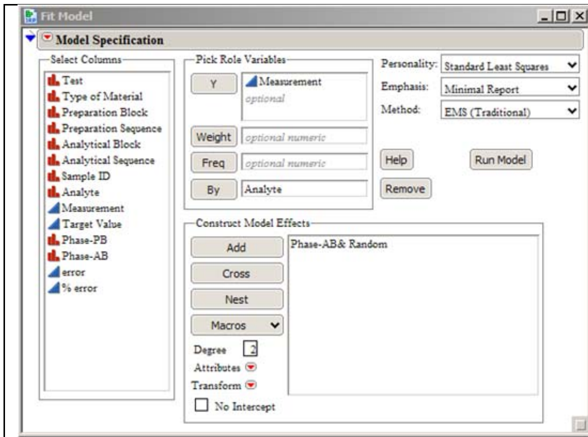
where

- y_{ij} is the measurement of the j^{th} sample in analytical block i for a prepared sample,
- μ represents the average measurement for the given oxide,
- a_i represents a random effect for the i^{th} analytical block that captures block-to-block effects,
- e_{ij} represents the within block variation, and as described above this variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process.

The a's in this model are assumed to be normally distributed with a zero mean and with a constant but unknown standard deviation that may be represented by σ_a. The e's in this model are random effects that are assumed to be normally distributed with a zero mean and with a constant but unknown standard deviation that may be represented by σ_e.

To facilitate the analysis of this model using JMP, the analytical blocks were uniquely tied to the test phases as indicated in Exhibit 2. The results from the JMP analysis are provided in Exhibit A3 in the Appendix, and they include a test for the statistical significance of the σ_a variation of the model in equation (1) relative to the size of the within block variation, i.e., the standard deviation, σ_e. The outcome of one of these tests indicates a statistically significant component of variation by its p-value, which is labeled as "**Prob > F**" in the portion of the results with the heading "**Tests wrt Random**

Effects." When the p-value is less than 0.05, then the estimated block-to-block component of variation is statistically significant at the 5% level. To help with this interpretation, the p-values in Exhibit A3 have been shaded: green if the variance component is not statistically significant and red if it is. The estimated variance for the analytical blocks is statistically significant for several of the oxides (especially, B_2O_3, BaO, CaO, Cr_2O_3, CuO, Li_2O, MgO, MnO, and NiO). These results suggest that differences among the analytical blocks can play a significant role in the variation seen in the ARG-1 results.



Phase-AB stands for Phase analytical block and is used to uniquely identify these blocks over all of the Isolok test phases.

Exhibit 2. Random Effects Model for Analytical Block Effects

In Table 2, the information from Exhibit A3 for each oxide is summarized and used to determine a 95% confidence interval for the mean of the measurements for each oxide. If the confidence interval for an oxide does not contain the reference concentration for that oxide (also provided in Table 2), then there is a statistically significant bias (at the 5% level) in the measurements. The results in Table 2 indicate that there are biases for all of the oxides except CaO, K_2O, Na_2O, SiO_2, and TiO_2. Regardless of the statistical significance of the bias, a bound on its value (at a 95% confidence) is also provided in Table 2. For those oxides present in ARG-1 at concentrations greater than or equal to 0.1 wt%, only B_2O_3 has a bias bound (its value is 9.84%) that is larger than 4.41% (the value for MnO). Thus, only B_2O_3 has a bias bound that is larger than 5%.

The low bias for boron measurements is almost certainly due to volatilization of boron at the 1050 °C fusion conditions of the Cs_2CO_3 method. Cesium species also can be volatilized under these conditions, and it is believed that a carrier distillation effect results in the loss of boron. The relatively high fusion temperature was used in anticipation of needing very rigorous fusion conditions for dissolving a 1-1.5 gram wafer of glass that potentially results from vitrifying a 3 mL SME sample taken with the Isolok sampler. Experiments performed at the DWPF Laboratory and at SRNL indicate that the low boron bias from the Cs_2CO_3 method is essentially eliminated when the method is carried out at 900 °C. The lower fusion temperature is effective for attacking powdered glass and small shards of glass, but may not result in complete attack of a1.5 gram wafer of glass.

Also, note that Table 2 presents the components of variation (i.e., the analytical block-to-block effects and the within-block effects) as % relative standard deviations, and from Table 2, all of these values are less than 5% for those oxides at concentrations of 0.1 wt% or greater.

Table 2. Summary of Results from the Model of Random Analytical Block Effects

Analyte	Reference Concentration	n	Average Measured Value	ICP Block to Block Variance	Residual Variance	Total Variance	% Relative ICP Block Std. Dev.	% Relative Residual Std. Dev.	% Relative Total Std. Dev.	Mean Square Error	DoF	95% Confidence Interval for the Mean: Lower Limit	95% Confidence Interval for the Mean: Upper Limit	% Bias Bound at ≥ 95% Conf
Al_2O_3	4.73	54	4.607	2.854E-03	1.400E-02	1.685E-02	1.2%	2.6%	2.8%	2.26E-02	17	4.5641	4.6504	3.51%
B_2O_3	8.67	54	7.929	2.755E-02	6.891E-02	9.646E-02	2.1%	3.3%	3.9%	1.52E-01	17	7.8169	8.0405	9.84%
BaO	0.09	54	0.092	3.482E-06	1.100E-05	1.448E-05	2.0%	3.6%	4.1%	2.15E-05	17	0.0908	0.0935	3.88%
CaO	1.43	54	1.444	4.600E-04	1.299E-03	1.759E-03	1.5%	2.5%	2.9%	2.68E-03	17	1.4286	1.4584	1.98%
Cr_2O_3	0.09	54	0.098	9.885E-06	1.425E-05	2.414E-05	3.2%	3.9%	5.0%	4.39E-05	17	0.0958	0.0996	10.66%
CuO	0	54	0.010	6.474E-05	6.519E-06	7.126E-05	81.0%	25.7%	85.0%	2.00E-04	17	0.0059	0.0140	na
Fe_2O_3	14	54	13.900	0.000E+00	1.082E-01	1.082E-01	0.0%	2.4%	2.4%	8.74E-02	17	13.8153	13.9850	1.32%
K_2O	2.71	54	2.695	0.000E+00	1.560E-02	1.560E-02	0.0%	4.6%	4.6%	1.53E-02	17	2.6593	2.7302	1.87%
Li_2O	3.21	54	3.156	1.969E-03	5.845E-03	7.814E-03	1.4%	2.4%	2.8%	1.18E-02	17	3.1249	3.1871	2.65%
MgO	0.86	54	0.842	2.020E-04	4.260E-04	6.280E-04	1.7%	2.5%	3.0%	1.03E-03	17	0.8324	0.8508	3.21%
MnO	1.89	54	1.827	9.940E-04	2.087E-03	3.081E-03	1.7%	2.5%	3.0%	5.07E-03	17	1.8066	1.8475	4.41%
Na_2O	11.5	54	11.445	1.559E-02	9.152E-02	1.071E-01	1.1%	2.6%	2.9%	1.38E-01	17	11.3380	11.5515	1.41%
NiO	1.05	54	1.018	3.310E-04	6.990E-04	1.030E-03	1.8%	2.6%	3.2%	1.69E-03	17	1.0057	1.0293	4.22%
SiO_2	47.9	54	48.043	1.908E-01	1.482E+00	1.673E+00	0.9%	2.5%	2.7%	2.05E+00	17	47.6316	48.4547	1.16%
TiO_2	1.15	54	1.148	7.514E-05	8.440E-04	9.191E-04	0.8%	2.5%	2.6%	1.07E-03	17	1.1382	1.1570	1.03%
ZnO	0.02	54	0.024	1.115E-08	1.893E-06	1.904E-06	0.4%	5.6%	5.7%	1.93E-06	17	0.0240	0.0248	24.04%
ZrO_2	0.13	54	0.132	2.322E-06	2.154E-05	2.386E-05	1.2%	3.5%	3.7%	2.85E-05	17	0.1303	0.1334	2.58%
Sum	99.43	54	98.408	5.737E-03	6.169E+00	6.175E+00	0.1%	2.5%	2.5%	6.19E+00	17	97.6938	99.1220	1.75%

significant ICP effect

3.0 Conclusions

The ARG-1 measurements, as wt% oxides, generated during the SME acceptability testing for the Isolok are statistically analyzed in this report. Three sources of variation in these measurements were explored: variation due to preparation block effects, variation due the ICP-OES analytical block effects, and within block variation for these measurements, where the within block variation is due to the repeatability of the preparation process for a given preparation block and to the variation in the measurements during an analytical block of work that is due to the repeatability of the ICP-OES measurement process. These results suggest that differences among the preparation blocks played an insignificant role in the variation seen in the ARG-1 results over the course of the Isolok testing. From this investigation, the components of variation (i.e., the analytical block-to-block effects and the within-block effects) as % relative standard deviations are less than 5% for those oxides at concentrations of 0.1 wt% or greater. A bound on the bias in the Cs_2CO_3 method of its measurements for each oxide of interest value is also provided at a 95% confidence. For those oxides present in ARG-1 at concentrations greater than or equal to 0.1 wt%, only B_2O_3 has a bias bound (its value is 9.84%) that is larger than 4.41% (the value for MnO). Thus, only B_2O_3 has a bias bound that is larger than 5%. The low boron bias results from boron volatilization at the 1050 °C fusion conditions used to enable the Cs_2CO_3 method to completely attack large wafers of glass.

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5.0 Appendix

Supplemental Tables and Exhibits

Table A1. ARG-1 Measured Oxide Concentrations (part 1)

Test	Type of Material	Preparation Block	Preparation Sequence	Analytical Block	Analytical Sequence	Sample ID	Al2O3 (wt%)	B2O3 (wt%)	BaO (wt%)	CaO (wt%)	Cr2O3 (wt%)	CuO (wt%)	Fe2O3 (wt%)	K2O (wt%)	Li2O (wt%)	MgO (wt%)
Phase 1	ARG-1	1	1	1	1	ARG-1B11	4.535	8.211	0.090	1.455	0.097	0.020	13.954	2.734	3.186	0.857
Phase 1	ARG-1	1	15	1	15	ARG-1B12	4.554	8.114	0.090	1.455	0.096	0.020	13.940	2.662	3.186	0.859
Phase 1	ARG-1	1	29	1	29	ARG-1B13	4.705	7.857	0.092	1.483	0.095	0.025	14.011	2.626	3.208	0.864
Phase 1	ARG-1	2	1	2	1	ARG-1B21	4.667	7.760	0.090	1.455	0.100	0.017	14.011	2.686	3.251	0.869
Phase 1	ARG-1	2	12	2	15	ARG-1B22	4.799	7.567	0.092	1.483	0.097	0.018	14.054	2.445	3.251	0.876
Phase 1	ARG-1	2	23	2	29	ARG-1B23	4.743	7.824	0.094	1.483	0.099	0.023	13.997	2.542	3.251	0.869
Phase 1	ARG-1	3	1	3	1	ARG-1B31	4.686	8.468	0.092	1.469	0.104	0.021	14.025	2.662	3.251	0.871
Phase 1	ARG-1	3	12	3	15	ARG-1B32	4.724	7.953	0.091	1.469	0.096	0.022	13.897	2.530	3.208	0.866
Phase 1	ARG-1	3	23	3	29	ARG-1B33	4.856	8.243	0.093	1.497	0.101	0.020	14.297	2.746	3.315	0.887
Phase 1	ARG-1	4	1	4	1	ARG-1B41	4.648	7.985	0.090	1.455	0.096	0.022	14.011	2.746	3.229	0.864
Phase 1	ARG-1	4	7	4	15	ARG-1B42	4.591	8.082	0.090	1.441	0.096	0.021	13.968	2.855	3.208	0.861
Phase 1	ARG-1	4	13	4	29	ARG-1B43	4.667	7.857	0.090	1.427	0.094	0.022	13.997	2.638	3.229	0.859
Phase 1	ARG-1	2	1	5	1	ARG-1B21	4.610	7.760	0.089	1.469	0.097	0.017	13.839	2.746	3.272	0.847
Phase 1	ARG-1	2	12	5	15	ARG-1B22	4.270	7.309	0.083	1.367	0.090	0.018	12.867	2.494	3.036	0.789
Phase 1	ARG-1	2	23	5	29	ARG-1B23	4.629	7.728	0.091	1.441	0.097	0.023	14.011	2.674	3.208	0.854
Phase 1	ARG-1	3	1	6	1	ARG-1B31	4.629	7.953	0.089	1.441	0.096	0.021	13.968	2.698	3.165	0.837
Phase 1	ARG-1	3	12	6	15	ARG-1B32	4.554	7.953	0.087	1.427	0.095	0.022	13.668	2.626	3.100	0.823
Phase 1	ARG-1	3	23	6	29	ARG-1B33	4.384	7.728	0.085	1.387	0.090	0.020	13.196	2.638	3.122	0.794
Phase 2	ARG-1	1	1	1	1	ARG-1B11	4.762	7.985	0.100	1.469	0.100	0.009	14.297	2.783	3.186	0.856
Phase 2	ARG-1	1	15	1	15	ARG-1B12	4.780	8.018	0.099	1.483	0.100	0.002	14.583	2.783	3.229	0.872
Phase 2	ARG-1	1	29	1	29	ARG-1B13	4.346	7.052	0.090	1.364	0.090	0.006	13.153	2.361	2.906	0.784
Phase 2	ARG-1	2	1	2	1	ARG-1B21	4.365	7.631	0.091	1.321	0.092	0.002	13.239	2.674	3.014	0.799
Phase 2	ARG-1	2	12	2	15	ARG-1B22	4.573	8.114	0.098	1.427	0.094	0.003	13.940	2.807	3.186	0.841
Phase 2	ARG-1	2	23	2	29	ARG-1B23	4.610	8.146	0.097	1.441	0.094	0.003	13.954	2.939	3.251	0.842
Phase 2	ARG-1	3	1	3	1	ARG-1B31	4.403	7.985	0.085	1.382	0.094	0.007	13.296	2.614	3.057	0.808
Phase 2	ARG-1	3	12	3	15	ARG-1B32	4.610	8.243	0.098	1.483	0.096	0.009	13.997	2.903	3.208	0.852
Phase 2	ARG-1	3	23	3	29	ARG-1B33	4.743	8.275	0.098	1.483	0.099	0.003	14.226	2.915	3.294	0.867
Phase 2	ARG-1	4	1	4	1	ARG-1B41	4.421	7.696	0.087	1.391	0.094	0.002	13.382	2.518	3.014	0.798
Phase 2	ARG-1	4	7	4	15	ARG-1B42	4.535	7.631	0.097	1.455	0.096	0.003	13.839	2.674	3.100	0.826
Phase 2	ARG-1	4	13	4	29	ARG-1B43	4.648	7.792	0.091	1.455	0.103	0.003	14.140	2.734	3.165	0.842
Phase 2	ARG-1	2	1	5	1	ARG-1B21	4.573	8.082	0.097	1.427	0.096	0.008	13.911	2.674	3.143	0.837
Phase 2	ARG-1	2	12	5	15	ARG-1B22	4.667	8.114	0.099	1.413	0.096	0.003	14.068	2.722	3.165	0.844
Phase 2	ARG-1	2	23	5	29	ARG-1B23	4.440	7.438	0.093	1.349	0.091	0.003	13.353	2.554	3.014	0.801
Phase 2	ARG-1	3	1	6	1	ARG-1B31	4.629	8.018	0.092	1.441	0.097	0.003	13.997	2.759	3.143	0.839
Phase 2	ARG-1	3	12	6	15	ARG-1B32	4.667	7.857	0.097	1.441	0.098	0.003	14.083	2.783	3.165	0.847
Phase 2	ARG-1	3	23	6	29	ARG-1B33	4.421	7.309	0.090	1.381	0.092	0.003	13.253	2.590	3.014	0.798
Phase 3	ARG-1	1	1	1	1	ARG-1B11	4.459	7.760	0.088	1.399	0.095	0.010	13.525	2.855	3.057	0.811
Phase 3	ARG-1	1	15	1	15	ARG-1B12	4.459	7.953	0.090	1.413	0.097	0.003	13.897	2.590	3.057	0.827
Phase 3	ARG-1	1	29	1	29	ARG-1B13	4.535	7.985	0.089	1.427	0.096	0.003	13.854	2.722	3.079	0.824
Phase 3	ARG-1	2	1	2	1	ARG-1B21	4.705	8.307	0.094	1.455	0.101	0.008	14.168	2.783	3.122	0.831
Phase 3	ARG-1	2	12	2	15	ARG-1B22	4.516	8.050	0.091	1.469	0.097	0.003	14.068	2.614	3.057	0.823
Phase 3	ARG-1	2	23	2	29	ARG-1B23	4.629	7.696	0.089	1.469	0.106	0.003	14.025	2.903	3.100	0.823
Phase 3	ARG-1	3	1	3	1	ARG-1B31	4.705	7.953	0.090	1.427	0.096	0.003	14.083	2.710	3.165	0.847
Phase 3	ARG-1	3	12	3	15	ARG-1B32	4.686	7.824	0.092	1.455	0.103	0.003	14.226	2.867	3.143	0.857

Table A1. ARG-1 Measured Oxide Concentrations (part 1)

Test	Type of Material	Preparation Block	Preparation Sequence	Analytical Block	Analytical Sequence	Sample ID	Al2O3 (wt%)	B2O3 (wt%)	BaO (wt%)	CaO (wt%)	Cr2O3 (wt%)	CuO (wt%)	Fe2O3 (wt%)	K2O (wt%)	Li2O (wt%)	MgO (wt%)
Phase 3	ARG-1	3	23	3	29	ARG-1B33	4.573	7.599	0.091	1.441	0.095	0.003	13.868	2.698	3.186	0.839
Phase 3	ARG-1	4	1	4	1	ARG-1B41	4.554	7.921	0.095	1.469	0.098	0.003	14.083	2.771	3.186	0.857
Phase 3	ARG-1	4	7	4	14	ARG-1B42	4.591	7.857	0.095	1.427	0.098	0.003	14.011	2.674	3.143	0.847
Phase 3	ARG-1	4	13	4	26	ARG-1B43	4.535	7.535	0.093	1.427	0.095	0.003	13.968	2.843	3.122	0.842
Phase 3	ARG-1	2	1	5	1	ARG-1B21	4.743	8.050	0.093	1.469	0.103	0.011	14.011	2.554	3.229	0.866
Phase 3	ARG-1	2	12	5	15	ARG-1B22	4.762	8.436	0.098	1.511	0.101	0.006	14.083	2.759	3.337	0.872
Phase 3	ARG-1	2	23	5	29	ARG-1B23	4.762	8.758	0.093	1.539	0.116	0.017	14.054	2.831	3.229	0.866
Phase 3	ARG-1	3	1	6	1	ARG-1B31	4.743	8.146	0.092	1.469	0.102	0.003	14.111	2.602	3.100	0.839
Phase 3	ARG-1	3	12	6	15	ARG-1B32	4.629	8.404	0.093	1.497	0.111	0.003	14.154	2.710	3.079	0.837
Phase 3	ARG-1	3	23	6	29	ARG-1B33	4.762	8.179	0.093	1.469	0.106	0.003	13.997	2.494	3.100	0.836

Table A1. ARG-1 Measured Oxide Concentrations (part 2)

Test	Type of Material	Preparation Block	Preparation Sequence	Analytical Block	Analytical Sequence	Sample ID	MnO (wt%)	Na2O (wt%)	NiO (wt%)	SiO2 (wt%)	TiO2 (wt%)	ZnO (wt%)	ZrO2 (wt%)	Sum of Oxides (wt%)
Phase 1	ARG-1	1	1	1	1	ARG-1B11	1.846	11.525	1.035	48.348	1.154	0.024	0.133	99.205
Phase 1	ARG-1	1	15	1	15	ARG-1B12	1.859	11.606	1.036	48.348	1.151	0.025	0.133	99.135
Phase 1	ARG-1	1	29	1	29	ARG-1B13	1.859	11.687	1.026	48.348	1.158	0.024	0.136	99.204
Phase 1	ARG-1	2	1	2	1	ARG-1B21	1.885	11.633	1.068	48.562	1.158	0.024	0.135	99.372
Phase 1	ARG-1	2	12	2	15	ARG-1B22	1.898	11.701	1.029	48.990	1.159	0.023	0.135	99.618
Phase 1	ARG-1	2	23	2	29	ARG-1B23	1.898	11.755	1.055	48.562	1.161	0.024	0.133	99.511
Phase 1	ARG-1	3	1	3	1	ARG-1B31	1.898	11.674	1.071	48.990	1.168	0.024	0.132	100.608
Phase 1	ARG-1	3	12	3	15	ARG-1B32	1.885	11.579	1.029	48.348	1.149	0.023	0.130	98.999
Phase 1	ARG-1	3	23	3	29	ARG-1B33	1.937	11.916	1.063	50.060	1.183	0.023	0.139	102.375
Phase 1	ARG-1	4	1	4	1	ARG-1B41	1.872	11.458	1.014	48.134	1.153	0.023	0.131	98.934
Phase 1	ARG-1	4	7	4	15	ARG-1B42	1.859	11.458	1.007	48.134	1.156	0.025	0.133	98.985
Phase 1	ARG-1	4	13	4	29	ARG-1B43	1.859	11.525	1.005	48.134	1.156	0.024	0.133	98.717
Phase 1	ARG-1	2	1	5	1	ARG-1B21	1.834	11.458	1.015	47.706	1.141	0.024	0.132	98.059
Phase 1	ARG-1	2	12	5	15	ARG-1B22	1.704	10.744	0.937	44.711	1.063	0.022	0.122	91.626
Phase 1	ARG-1	2	23	5	29	ARG-1B23	1.859	11.714	1.015	48.348	1.154	0.025	0.132	99.004
Phase 1	ARG-1	3	1	6	1	ARG-1B31	1.821	11.512	1.013	47.920	1.128	0.024	0.130	98.445
Phase 1	ARG-1	3	12	6	15	ARG-1B32	1.782	11.418	0.998	47.279	1.104	0.023	0.131	97.090
Phase 1	ARG-1	3	23	6	29	ARG-1B33	1.730	11.121	0.973	45.781	1.071	0.022	0.128	94.270
Phase 2	ARG-1	1	1	1	1	ARG-1B11	1.846	11.822	1.029	49.418	1.158	0.026	0.130	100.977
Phase 2	ARG-1	1	15	1	15	ARG-1B12	1.885	12.038	1.037	50.487	1.174	0.026	0.131	102.729
Phase 2	ARG-1	1	29	1	29	ARG-1B13	1.691	10.878	0.944	45.139	1.058	0.023	0.117	92.003
Phase 2	ARG-1	2	1	2	1	ARG-1B21	1.743	10.744	0.965	44.925	1.096	0.025	0.130	92.856
Phase 2	ARG-1	2	12	2	15	ARG-1B22	1.834	11.458	1.021	47.492	1.156	0.027	0.136	98.206
Phase 2	ARG-1	2	23	2	29	ARG-1B23	1.846	11.647	1.014	47.706	1.173	0.025	0.143	98.933
Phase 2	ARG-1	3	1	3	1	ARG-1B31	1.769	10.851	0.965	45.353	1.101	0.022	0.124	93.916
Phase 2	ARG-1	3	12	3	15	ARG-1B32	1.859	11.606	1.003	48.348	1.164	0.025	0.139	99.644
Phase 2	ARG-1	3	23	3	29	ARG-1B33	1.898	11.795	1.026	48.990	1.181	0.026	0.133	101.051
Phase 2	ARG-1	4	1	4	1	ARG-1B41	1.730	10.851	0.981	45.995	1.108	0.024	0.126	94.217
Phase 2	ARG-1	4	7	4	15	ARG-1B42	1.795	11.337	1.008	47.492	1.149	0.027	0.130	97.194
Phase 2	ARG-1	4	13	4	29	ARG-1B43	1.834	11.593	1.017	48.776	1.166	0.022	0.139	99.520
Phase 2	ARG-1	2	1	5	1	ARG-1B21	1.821	11.377	1.014	47.706	1.153	0.026	0.133	98.078
Phase 2	ARG-1	2	12	5	15	ARG-1B22	1.821	11.458	1.023	48.348	1.156	0.025	0.136	99.158
Phase 2	ARG-1	2	23	5	29	ARG-1B23	1.730	10.932	0.968	45.781	1.093	0.026	0.126	93.793
Phase 2	ARG-1	3	1	6	1	ARG-1B31	1.821	11.350	1.004	47.492	1.156	0.023	0.133	97.996
Phase 2	ARG-1	3	12	6	15	ARG-1B32	1.821	11.377	1.009	47.920	1.158	0.027	0.131	98.482
Phase 2	ARG-1	3	23	6	29	ARG-1B33	1.730	10.744	0.954	45.139	1.089	0.025	0.121	92.754
Phase 3	ARG-1	1	1	1	1	ARG-1B11	1.756	10.824	1.000	48.134	1.149	0.028	0.132	97.084
Phase 3	ARG-1	1	15	1	15	ARG-1B12	1.795	10.986	1.042	49.204	1.169	0.025	0.135	98.743
Phase 3	ARG-1	1	29	1	29	ARG-1B13	1.795	10.986	1.008	48.990	1.176	0.024	0.138	98.731
Phase 3	ARG-1	2	1	2	1	ARG-1B21	1.808	11.701	1.051	49.204	1.184	0.025	0.138	100.683
Phase 3	ARG-1	2	12	2	15	ARG-1B22	1.795	11.647	1.029	48.562	1.168	0.024	0.139	99.151
Phase 3	ARG-1	2	23	2	29	ARG-1B23	1.782	11.782	0.985	48.776	1.173	0.021	0.135	99.497
Phase 3	ARG-1	3	1	3	1	ARG-1B31	1.834	11.525	1.010	49.204	1.169	0.025	0.133	99.980
Phase 3	ARG-1	3	12	3	15	ARG-1B32	1.859	11.593	1.033	49.204	1.168	0.026	0.132	100.272

Table A1. ARG-1 Measured Oxide Concentrations (part 2)

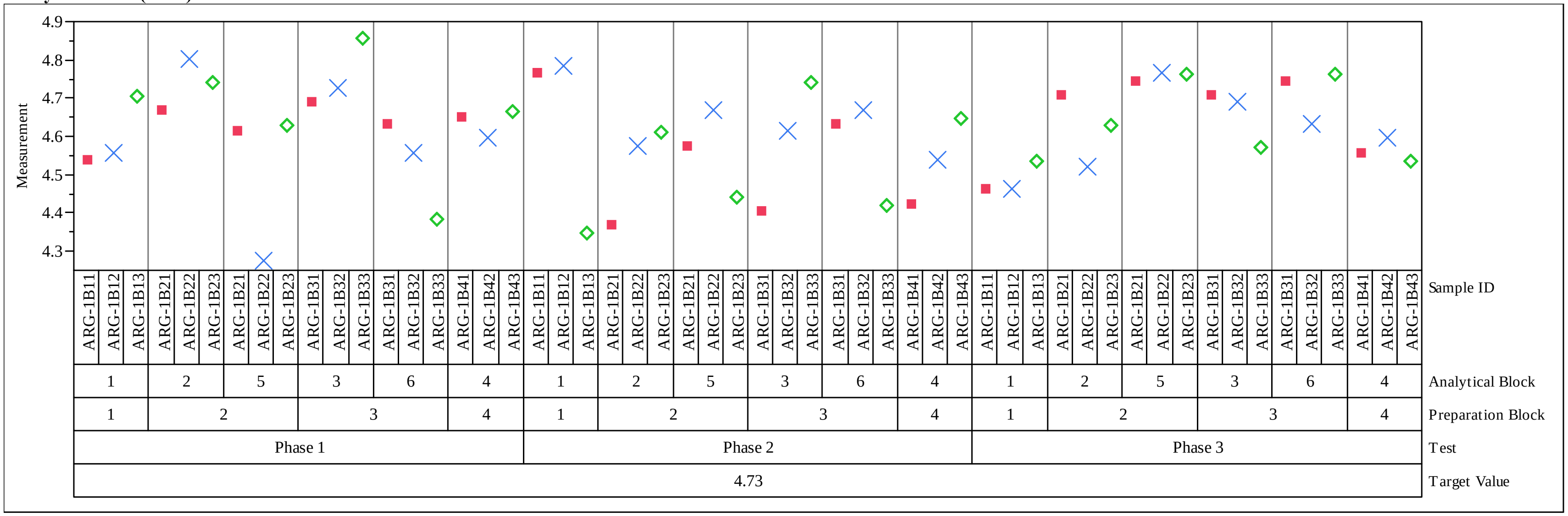
Test	Type of Material	Preparation Block	Preparation Sequence	Analytical Block	Analytical Sequence	Sample ID	MnO (wt%)	Na2O (wt%)	NiO (wt%)	SiO2 (wt%)	TiO2 (wt%)	ZnO (wt%)	ZrO2 (wt%)	Sum of Oxides (wt%)
Phase 3	ARG-1	3	23	3	29	ARG-1B33	1.821	11.593	1.007	48.562	1.156	0.024	0.133	98.688
Phase 3	ARG-1	4	1	4	1	ARG-1B41	1.872	11.593	1.029	48.562	1.161	0.024	0.128	99.406
Phase 3	ARG-1	4	7	4	14	ARG-1B42	1.846	11.579	1.040	48.348	1.144	0.026	0.127	98.857
Phase 3	ARG-1	4	13	4	26	ARG-1B43	1.834	11.458	1.007	48.134	1.136	0.025	0.128	98.184
Phase 3	ARG-1	2	1	5	1	ARG-1B21	1.885	11.391	1.029	48.348	1.151	0.025	0.131	99.089
Phase 3	ARG-1	2	12	5	15	ARG-1B22	1.885	11.876	1.049	48.990	1.156	0.025	0.132	101.077
Phase 3	ARG-1	2	23	5	29	ARG-1B23	1.885	11.377	1.050	48.562	1.163	0.023	0.129	100.452
Phase 3	ARG-1	3	1	6	1	ARG-1B31	1.821	11.539	1.052	48.776	1.164	0.025	0.127	99.712
Phase 3	ARG-1	3	12	6	15	ARG-1B32	1.821	11.579	1.084	48.990	1.169	0.024	0.136	100.321
Phase 3	ARG-1	3	23	6	29	ARG-1B33	1.808	11.647	1.069	48.562	1.163	0.024	0.128	99.437

Table A2. Reference Oxide Composition for ARG-1

Oxide	Targeted wt%
Al2O3 (wt%)	4.73
B2O3 (wt%)	8.67
BaO (wt%)	0.09
CaO (wt%)	1.43
Cr2O3 (wt%)	0.09
CuO (wt%)	0
Fe2O3 (wt%)	14
K2O (wt%)	2.71
Li2O (wt%)	3.21
MgO (wt%)	0.86
MnO (wt%)	1.89
Na2O (wt%)	11.5
NiO (wt%)	1.05
SiO2 (wt%)	47.9
TiO2 (wt%)	1.15
ZnO (wt%)	0.02
ZrO2 (wt%)	0.13
Sum of Oxides (wt%)	99.43

Exhibit A1. Variability Chart for ARG-1 Oxide Measurements

Analyte=Al2O3 (wt%)



Analyte=B2O3 (wt%)

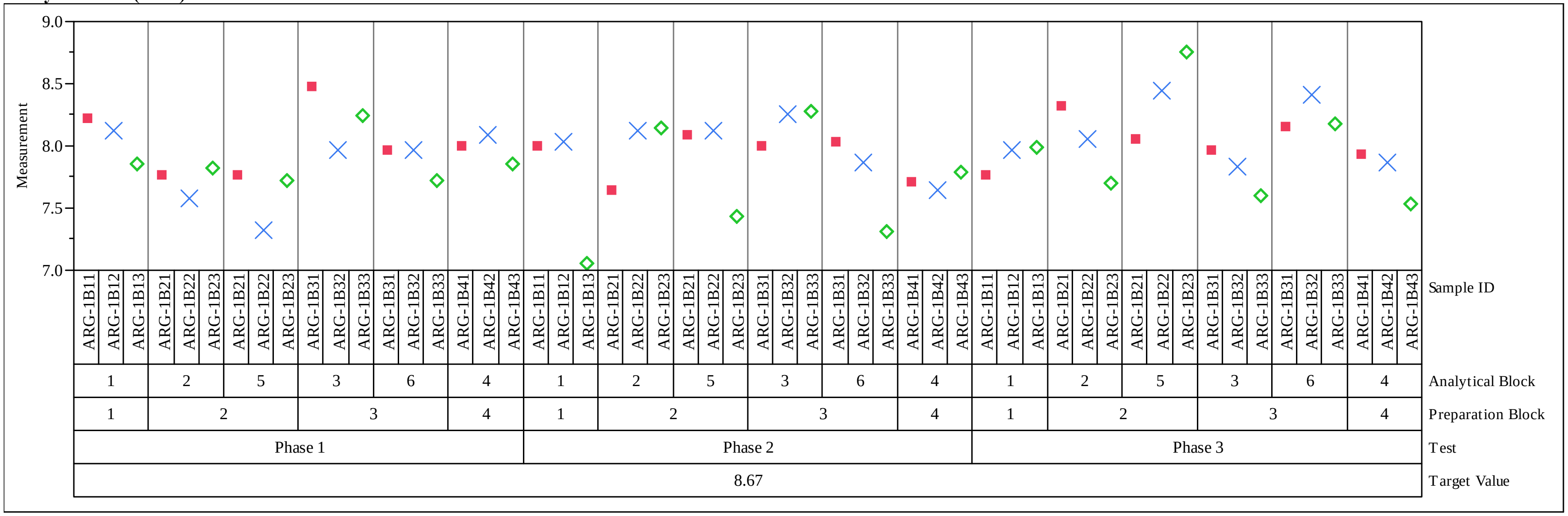
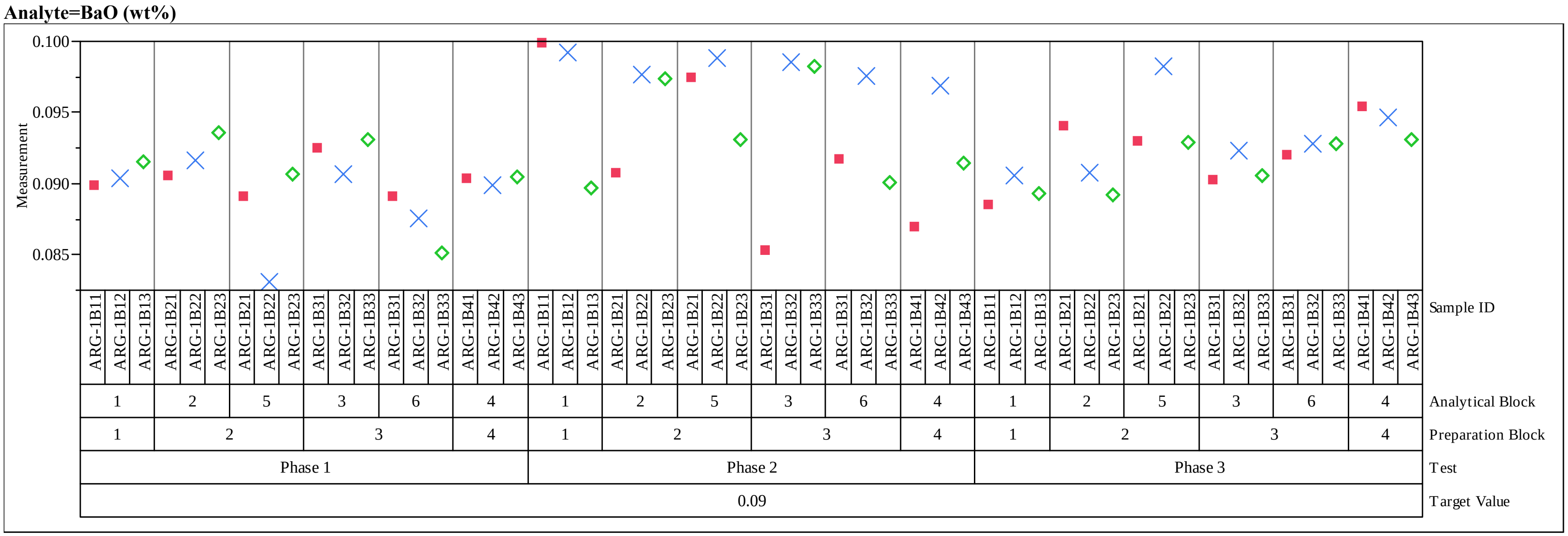


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements



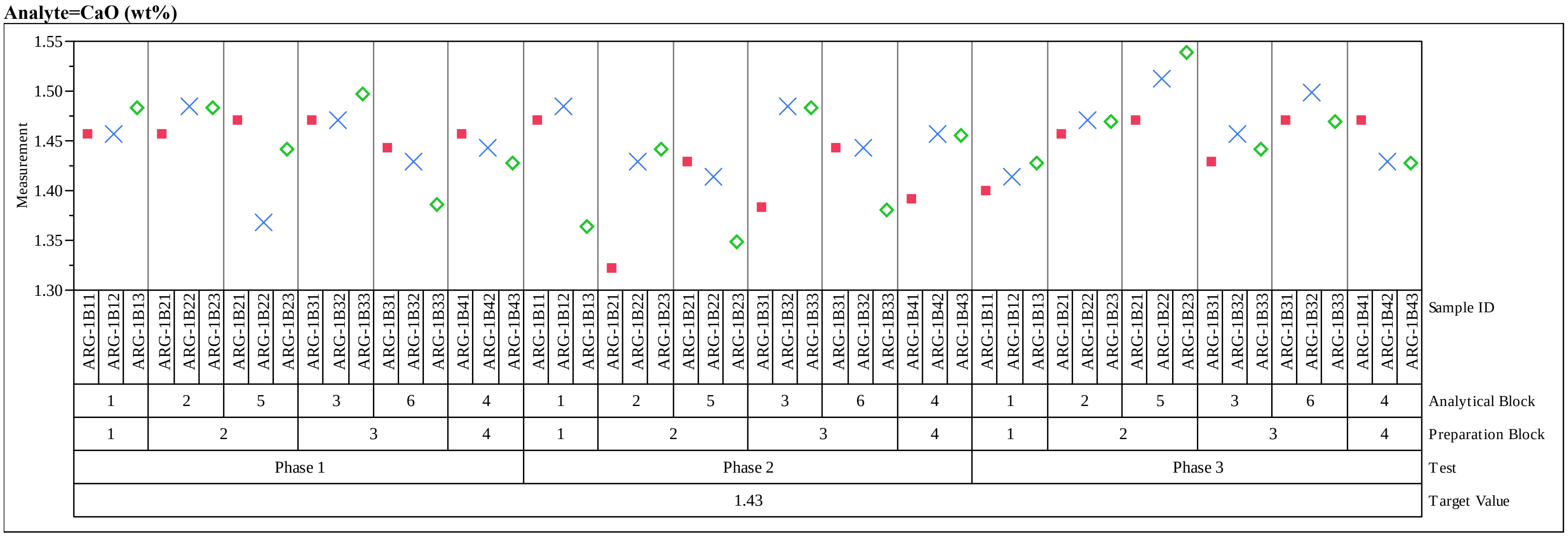
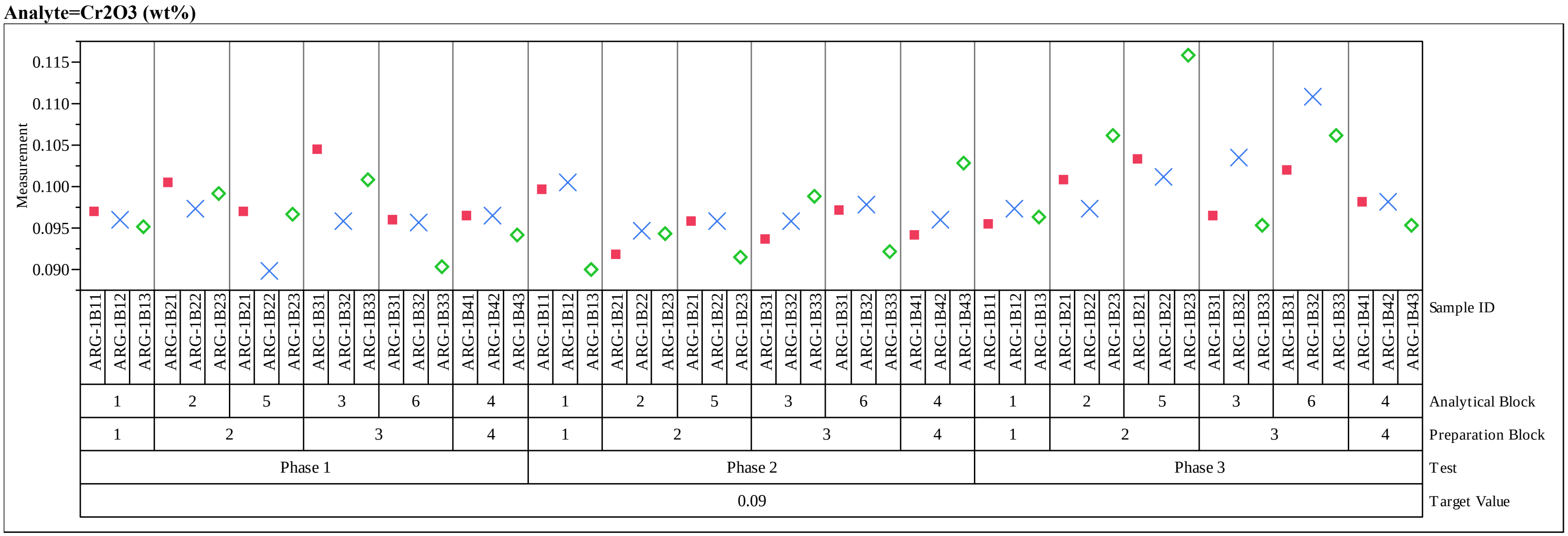


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements



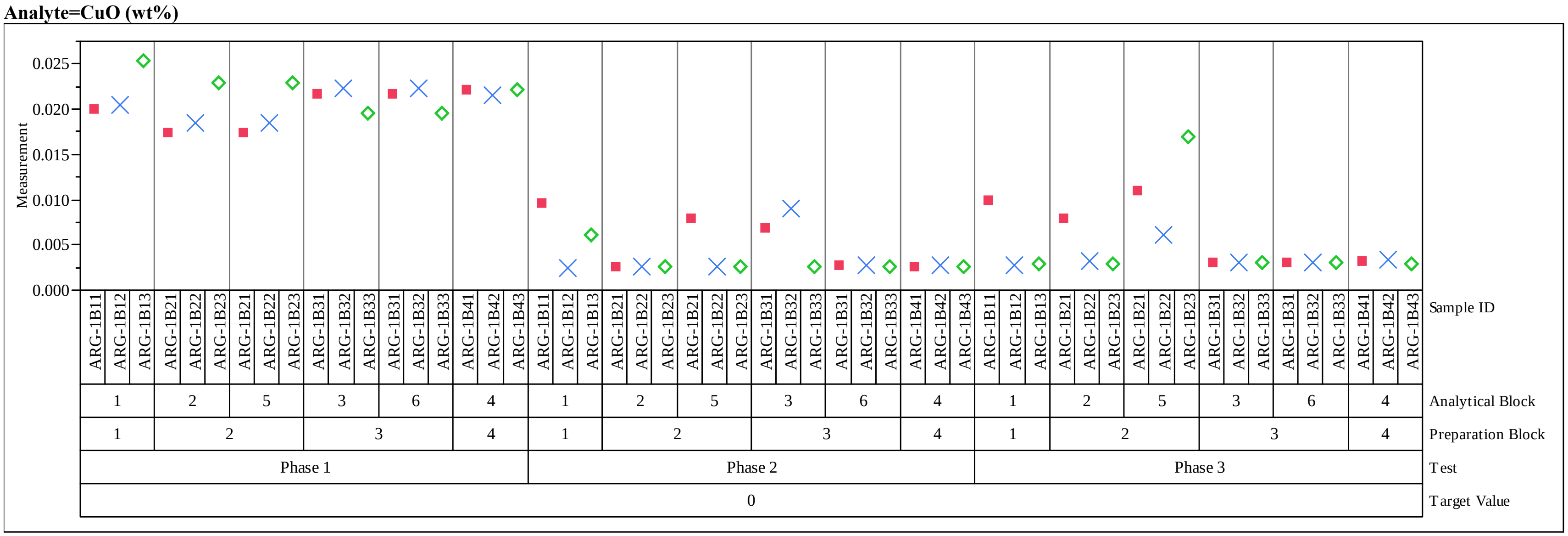
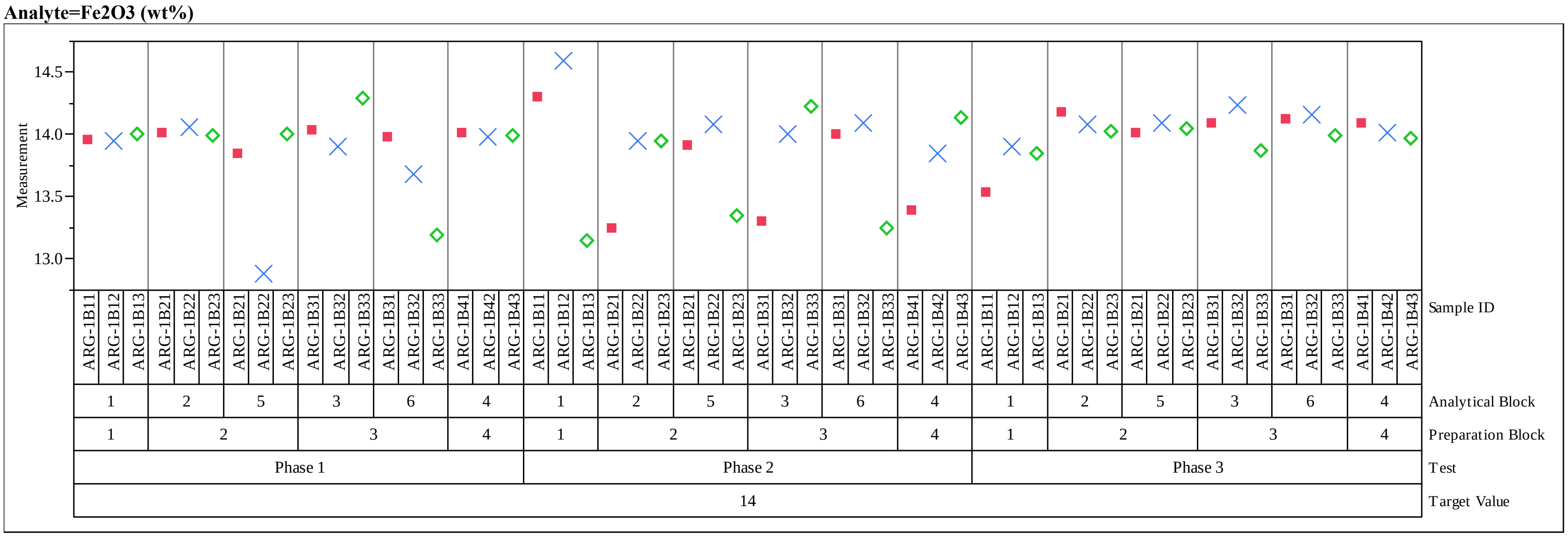


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements



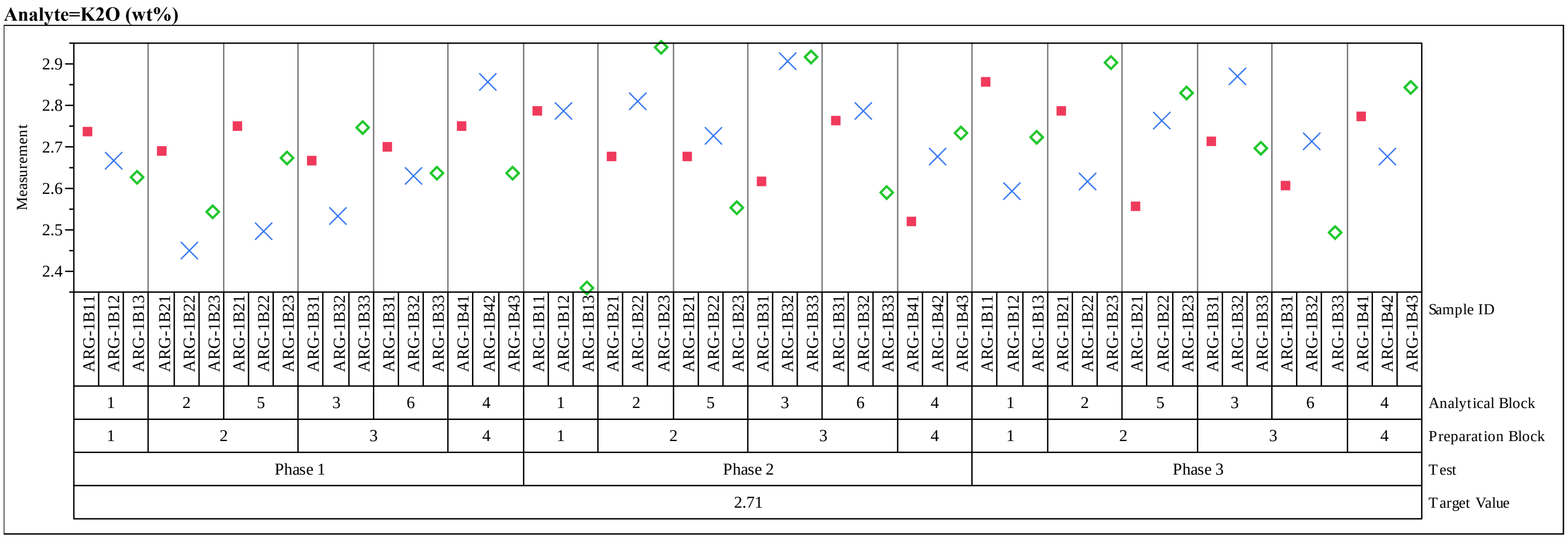
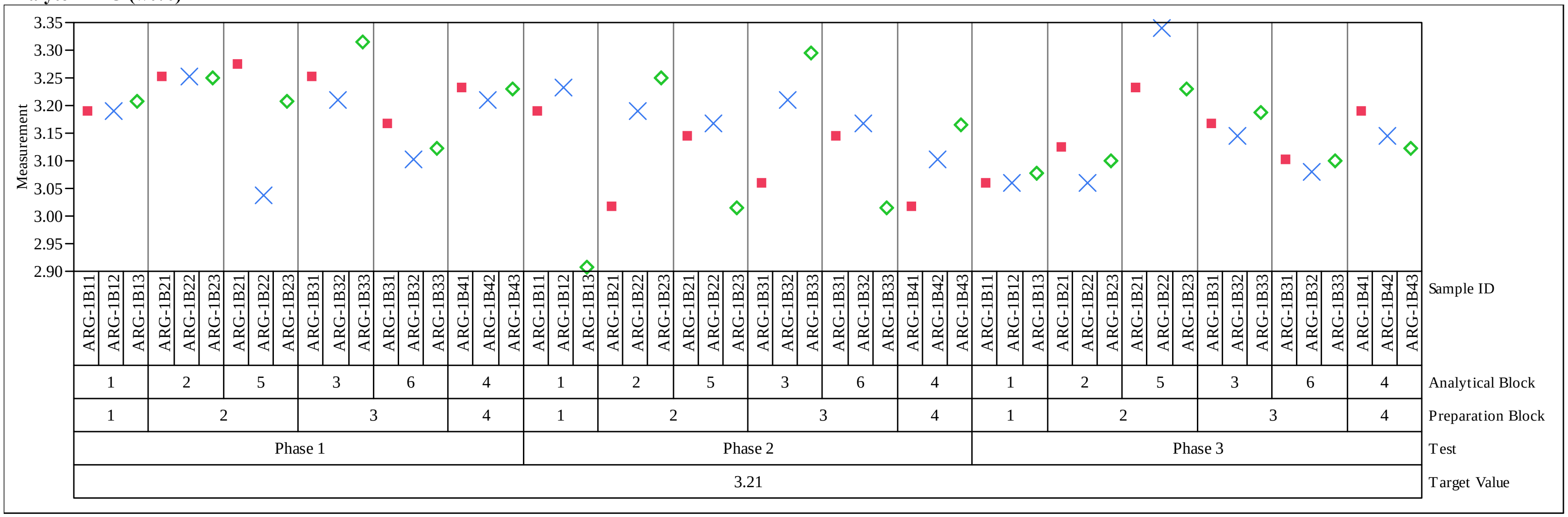


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements

Analyte=Li2O (wt%)



Analyte=MgO (wt%)

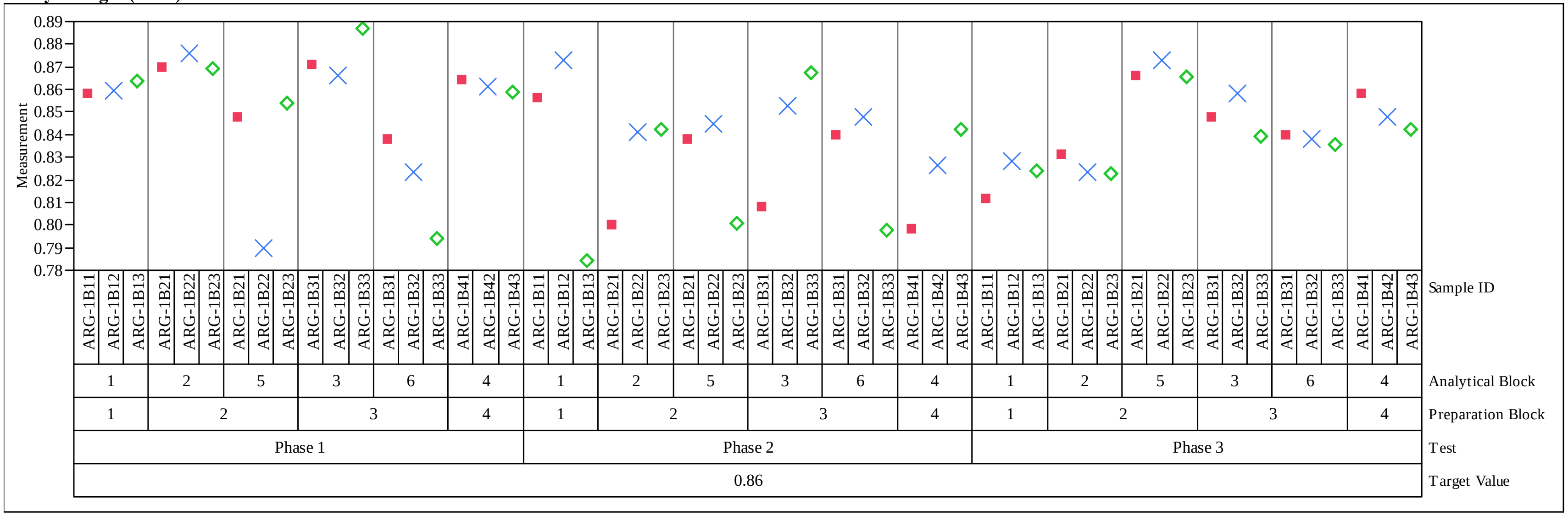
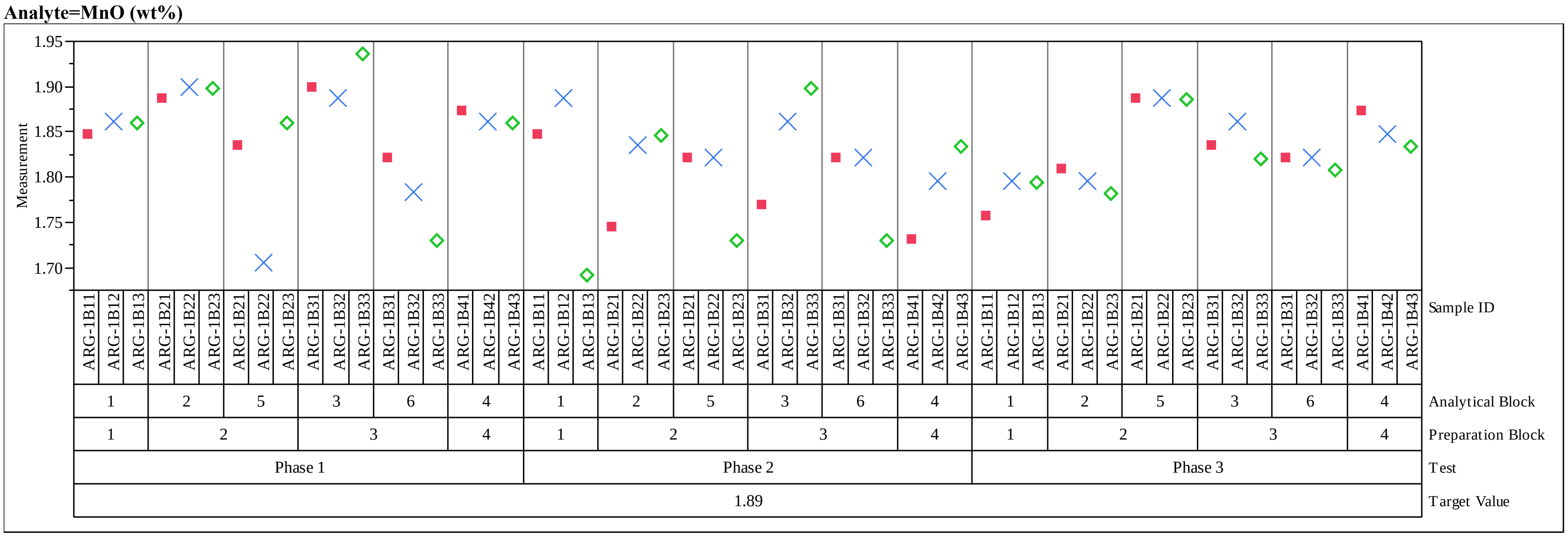


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements



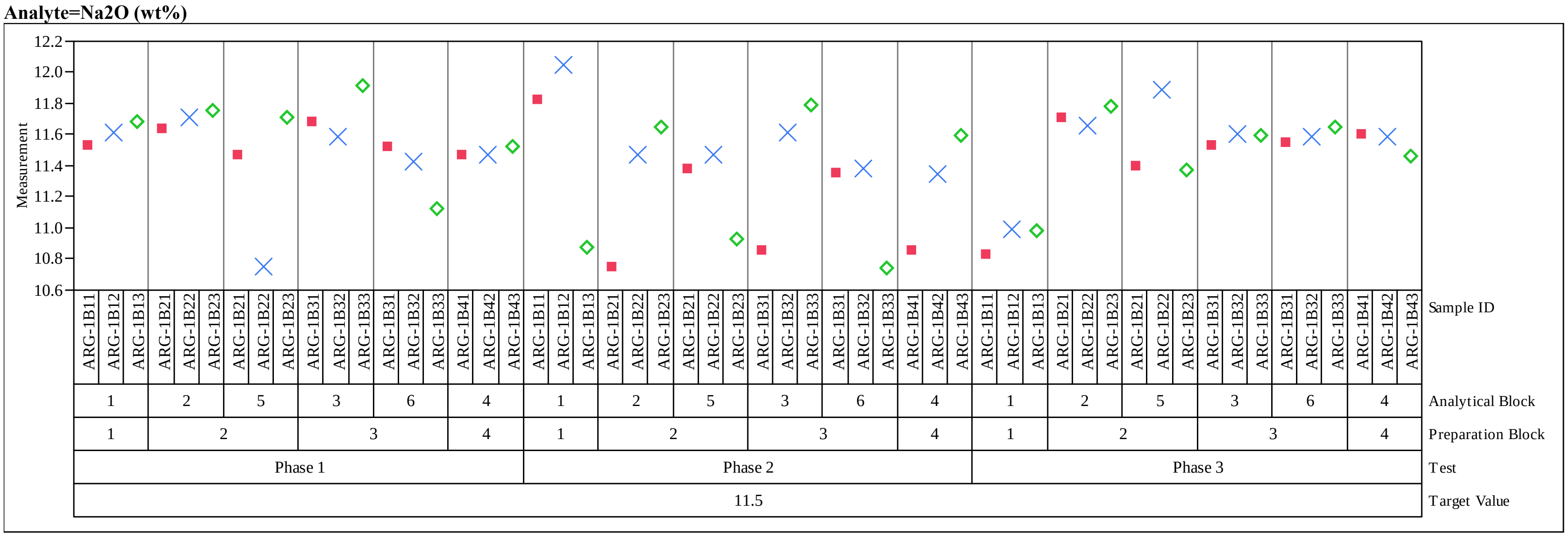
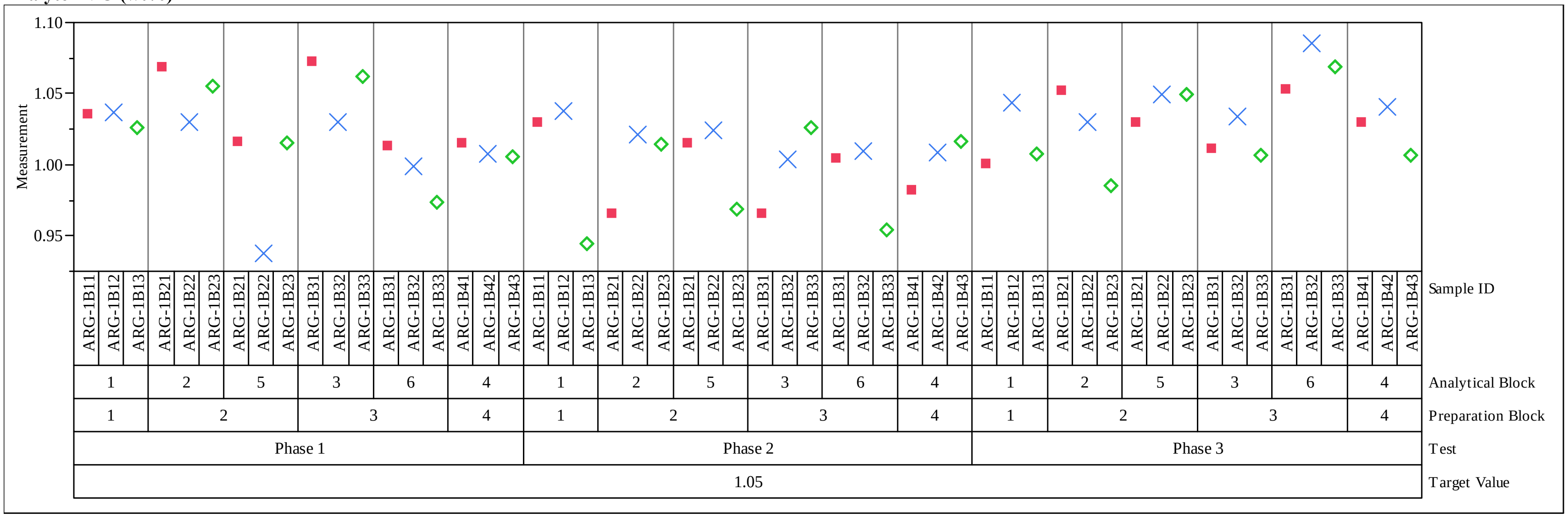


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements

Analyte=NiO (wt%)



Analyte=SiO2 (wt%)

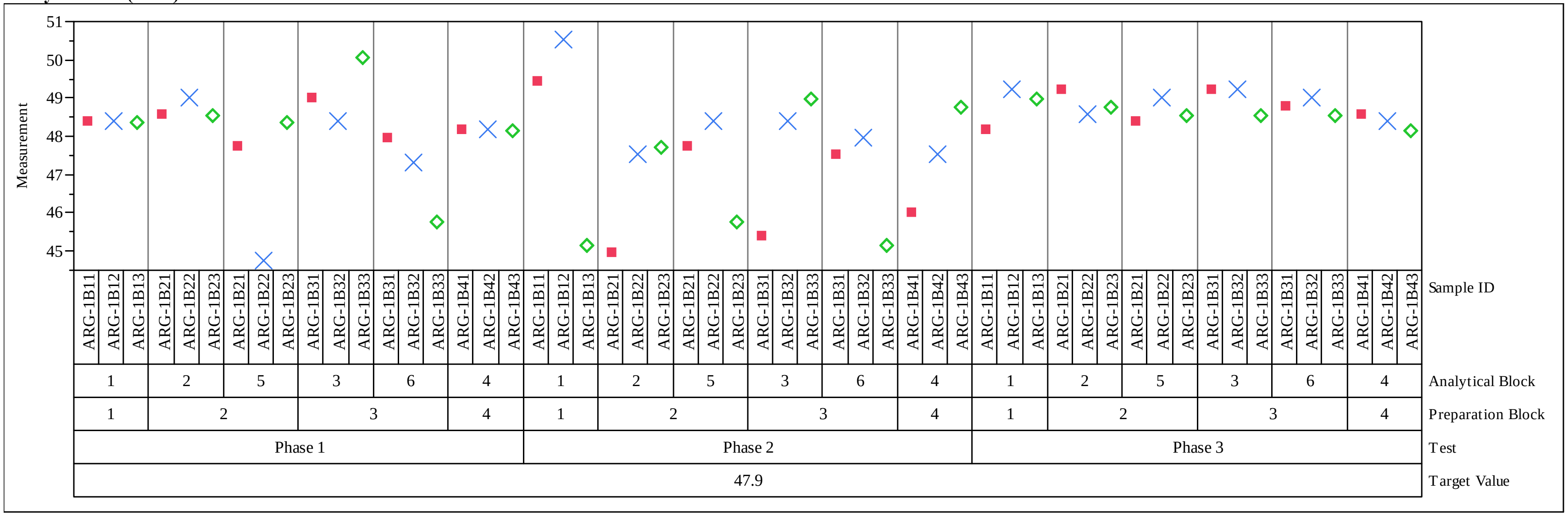
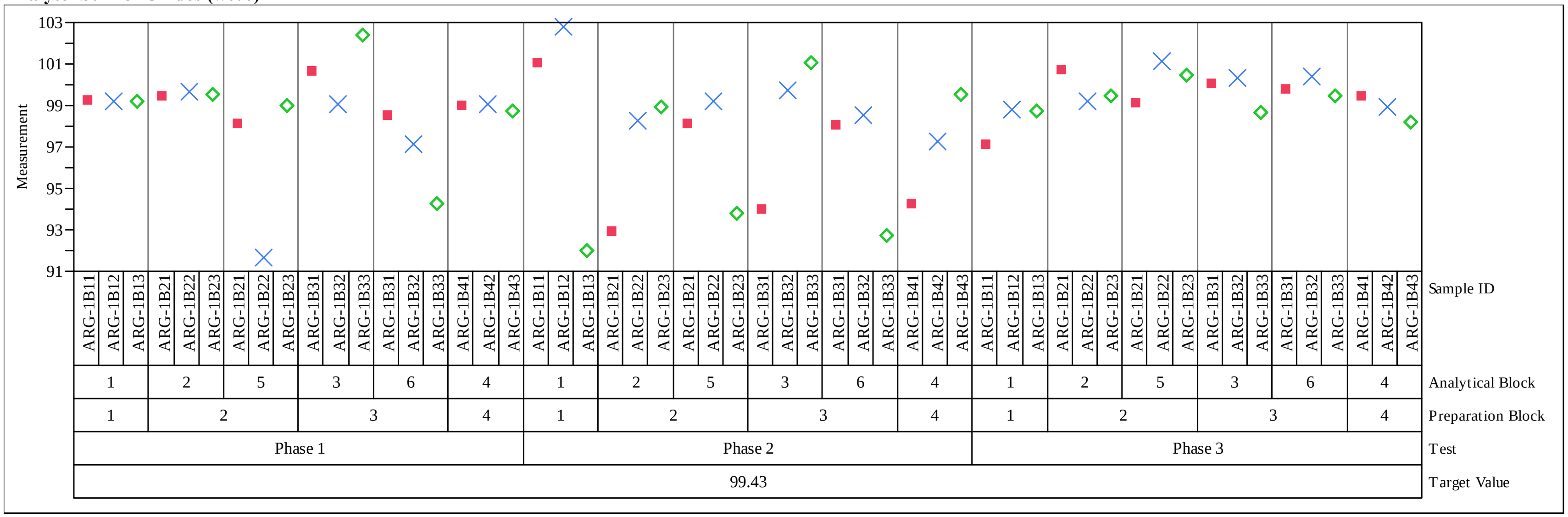


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements

Analyte=Sum of Oxides (wt%)



Analyte=TiO2 (wt%)

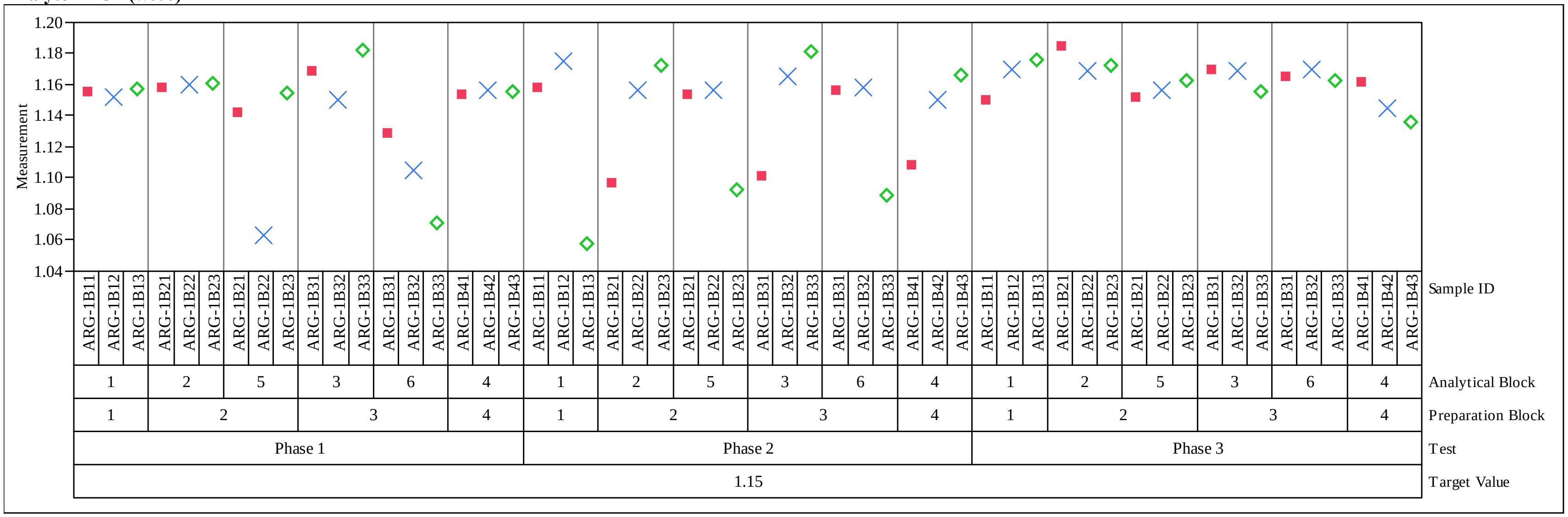
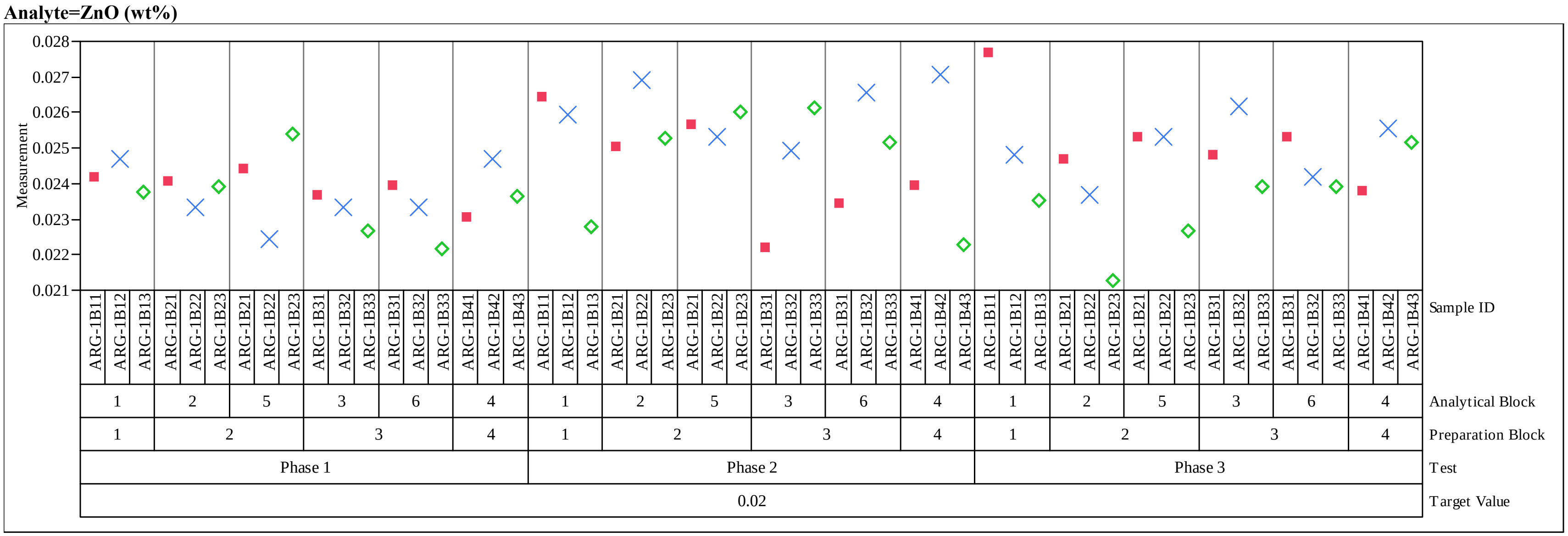


Exhibit A1. Variability Chart for ARG-1 Oxide Measurements



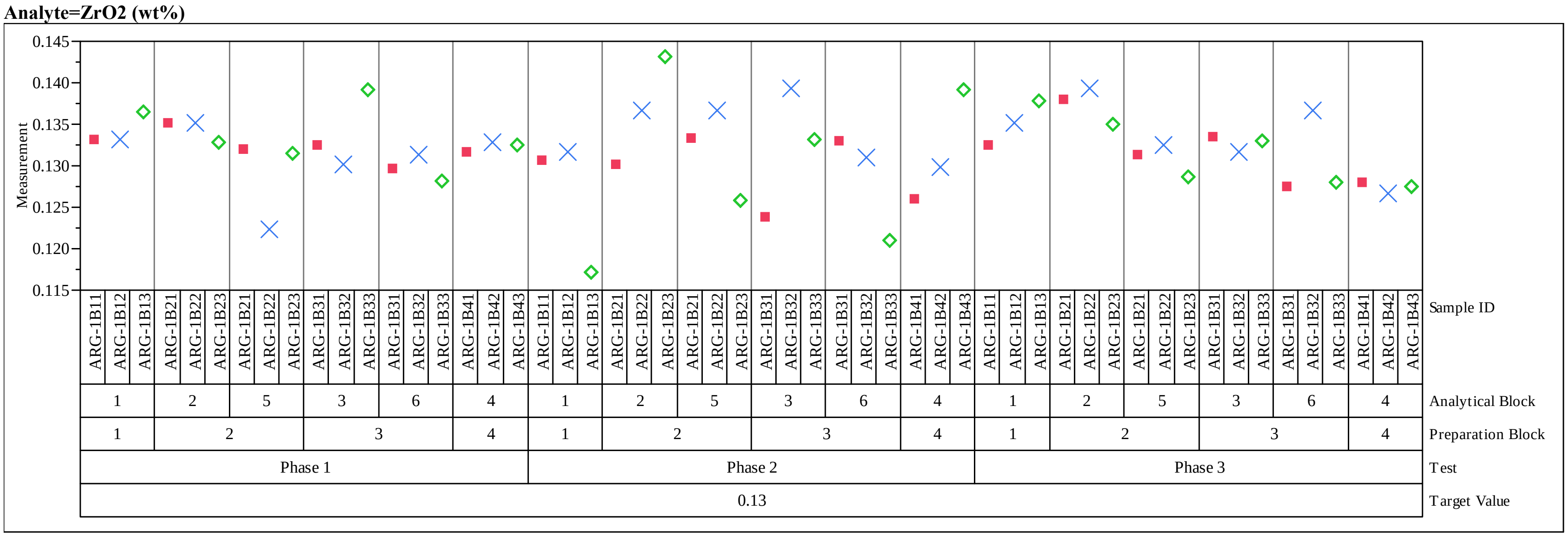


Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=Al2O3 (wt%), Target Value=4.73
Summary of Fit

RSquare	0.476283
RSquare Adj	0.236246
Root Mean Square Error	0.117536
Mean of Response	4.624026
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.30152409	0.027411	1.9842
Error	24	0.33155353	0.013815	**Prob > F**
C. Total	35	0.63307762		0.0778

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-0.00215	-11.860
Phase-AB[Phase-PB]&Random	0.006489	35.751
Residual	0.013815	76.109
Total	0.018151	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.03328	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.01381	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.10183	0.02037	5	0.6119	0.6968
Phase-AB[Phase-PB]&Random	0.19969	0.03328	6	2.4092	0.0577

Response Measurement Analyte=B2O3 (wt%), Target Value=8.67
Summary of Fit

RSquare	0.553816
RSquare Adj	0.349314
Root Mean Square Error	0.262794
Mean of Response	7.968358
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	2.0572796	0.187025	2.7081
Error	24	1.6574586	0.069061	**Prob > F**
C. Total	35	3.7147382		0.0200

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.004975	4.571
Phase-AB[Phase-PB]&Random	0.034799	31.974
Residual	0.069061	63.455
Total	0.108835	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.17346	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.06906	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	1.01653	0.20331	5	1.1721	0.4192
Phase-AB[Phase-PB]&Random	1.04075	0.17346	6	2.5117	0.0497

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=BaO (wt%), Target Value=0.09
Summary of Fit

RSquare	0.483184
RSquare Adj	0.246309
Root Mean Square Error	0.003393
Mean of Response	0.092217
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.00025826	0.000023	2.0398
Error	24	0.00027624	0.000012	**Prob > F**
C. Total	35	0.00053450		0.0700

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	3.297e-6	20.867
Phase-AB[Phase-PB]&Random	0.000001	6.281
Residual	1.151e-5	72.852
Total	1.58e-5	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	1.45e-5	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	1.15e-5	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00017	3.43e-5	5	2.3654	0.1622
Phase-AB[Phase-PB]&Random	8.69e-5	1.45e-5	6	1.2587	0.3129

Response Measurement Analyte=CaO (wt%), Target Value=1.43
Summary of Fit

RSquare	0.55286
RSquare Adj	0.347921
Root Mean Square Error	0.037126
Mean of Response	1.445879
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.04090083	0.003718	2.6977
Error	24	0.03307963	0.001378	**Prob > F**
C. Total	35	0.07398046		0.0204

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.000457	20.759
Phase-AB[Phase-PB]&Random	0.000365	16.585
Residual	0.001378	62.656
Total	0.0022	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.00247	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.00138	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.02606	0.00521	5	2.1080	0.1952
Phase-AB[Phase-PB]&Random	0.01484	0.00247	6	1.7941	0.1429

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=Cr2O3 (wt%), Target Value=0.09
Summary of Fit

RSquare	0.642053
RSquare Adj	0.477994
Root Mean Square Error	0.004037
Mean of Response	0.098276
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.00070166	0.000064	3.9135
Error	24	0.00039118	0.000016	**Prob > F**
C. Total	35	0.00109284		0.0025

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	9.312e-6	28.241
Phase-AB[Phase-PB]&Random	7.364e-6	22.331
Residual	1.63e-5	49.429
Total	0.000033	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	3.84e-5	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	1.63e-5	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00047	9.43e-5	5	2.4554	0.1523
Phase-AB[Phase-PB]&Random	0.00023	3.84e-5	6	2.3553	0.0624

Response Measurement Analyte=CuO (wt%), Target Value=0
Summary of Fit

RSquare	0.931258
RSquare Adj	0.899751
Root Mean Square Error	0.002567
Mean of Response	0.009868
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.00214201	0.000195	29.5574
Error	24	0.00015811	6.588e-6	**Prob > F**
C. Total	35	0.00230012		<.0001

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	6.584e-5	87.456
Phase-AB[Phase-PB]&Random	2.856e-6	3.793
Residual	6.588e-6	8.751
Total	7.529e-5	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	1.52e-5	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	6.59e-6	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00205	0.00041	5	27.0685	0.0005
Phase-AB[Phase-PB]&Random	0.00009	1.52e-5	6	2.3003	0.0677

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=Fe2O3 (wt%), Target Value=14
Summary of Fit

RSquare	0.315326
RSquare Adj	0.001517
Root Mean Square Error	0.337976
Mean of Response	13.88874
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	1.2625821	0.114780	1.0048
Error	24	2.7414693	0.114228	**Prob > F**
C. Total	35	4.0040513		0.4707

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.003134	2.732
Phase-AB[Phase-PB]&Random	-0.00266	-2.323
Residual	0.114228	99.591
Total	0.114697	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.10623	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.11423	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.62518	0.12504	5	1.1770	0.4173
Phase-AB[Phase-PB]&Random	0.6374	0.10623	6	0.9300	0.4915

Response Measurement Analyte=K2O (wt%), Target Value=2.71
Summary of Fit

RSquare	0.386106
RSquare Adj	0.104737
Root Mean Square Error	0.119957
Mean of Response	2.69295
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.21720773	0.019746	1.3722
Error	24	0.34535256	0.014390	**Prob > F**
C. Total	35	0.56256029		0.2481

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.000974	5.988
Phase-AB[Phase-PB]&Random	0.0009	5.535
Residual	0.01439	88.477
Total	0.016264	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.01709	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.01439	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.11467	0.02293	5	1.3419	0.3611
Phase-AB[Phase-PB]&Random	0.10254	0.01709	6	1.1877	0.3460

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=Li2O (wt%), Target Value=3.21
Summary of Fit

RSquare	0.501718
RSquare Adj	0.273339
Root Mean Square Error	0.074837
Mean of Response	3.164763
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.13534137	0.012304	2.1969
Error	24	0.13441437	0.005601	**Prob > F**
C. Total	35	0.26975574		0.0519

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-0.00152	-19.772
Phase-AB[Phase-PB]&Random	0.003618	47.006
Residual	0.005601	72.767
Total	0.007697	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.01645	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.0056	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.03662	0.00732	5	0.4451	0.8039
Phase-AB[Phase-PB]&Random	0.09873	0.01645	6	2.9379	0.0271

Response Measurement Analyte=MgO (wt%), Target Value=0.86
Summary of Fit

RSquare	0.568216
RSquare Adj	0.370315
Root Mean Square Error	0.020047
Mean of Response	0.841495
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.01269259	0.001154	2.8712
Error	24	0.00964502	0.000402	**Prob > F**
C. Total	35	0.02233761		0.0149

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-0.00022	-34.259
Phase-AB[Phase-PB]&Random	0.000448	70.755
Residual	0.000402	63.504
Total	0.000633	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.00175	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.0004	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00222	0.00044	5	0.2546	0.9226
Phase-AB[Phase-PB]&Random	0.01047	0.00175	6	4.3425	0.0042

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=MnO (wt%), Target Value=1.89
Summary of Fit

RSquare	0.584912
RSquare Adj	0.394663
Root Mean Square Error	0.044103
Mean of Response	1.828483
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.06578020	0.005980	3.0745
Error	24	0.04668153	0.001945	**Prob > F**
C. Total	35	0.11246173		0.0104

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-0.00122	-38.390
Phase-AB[Phase-PB]&Random	0.002454	77.207
Residual	0.001945	61.183
Total	0.003179	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.00931	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.00195	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00993	0.00199	5	0.2133	0.9444
Phase-AB[Phase-PB]&Random	0.05585	0.00931	6	4.7857	0.0024

Response Measurement Analyte=Na2O (wt%), Target Value=11.5
Summary of Fit

RSquare	0.366715
RSquare Adj	0.07646
Root Mean Square Error	0.303192
Mean of Response	11.46699
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	1.2775453	0.116140	1.2634
Error	24	2.2062065	0.091925	**Prob > F**
C. Total	35	3.4837518		0.3024

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.007106	7.060
Phase-AB[Phase-PB]&Random	0.001612	1.602
Residual	0.091925	91.338
Total	0.100643	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.09676	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.09193	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.69698	0.1394	5	1.4406	0.3318
Phase-AB[Phase-PB]&Random	0.58056	0.09676	6	1.0526	0.4173

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=NiO (wt%), Target Value=1.05
Summary of Fit

RSquare	0.60098
RSquare Adj	0.418095
Root Mean Square Error	0.026981
Mean of Response	1.018919
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.02631489	0.002392	3.2861
Error	24	0.01747177	0.000728	**Prob > F**
C. Total	35	0.04378667		0.0072

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-0.00009	-7.026
Phase-AB[Phase-PB]&Random	0.000636	49.911
Residual	0.000728	57.115
Total	0.001275	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.00264	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.00073	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.0105	0.0021	5	0.7962	0.5897
Phase-AB[Phase-PB]&Random	0.01582	0.00264	6	3.6216	0.0106

Response Measurement Analyte=SiO2 (wt%), Target Value=47.9
Summary of Fit

RSquare	0.486224
RSquare Adj	0.250743
Root Mean Square Error	1.167939
Mean of Response	47.95003
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	30.982341	2.81658	2.0648
Error	24	32.737977	1.36408	**Prob > F**
C. Total	35	63.720319		0.0667

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.237051	12.678
Phase-AB[Phase-PB]&Random	0.268664	14.369
Residual	1.364082	72.953
Total	1.869797	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	2.17007	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	1.36408	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	17.9619	3.59238	5	1.6554	0.2775
Phase-AB[Phase-PB]&Random	13.0204	2.17007	6	1.5909	0.1930

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=Sum of Oxides (wt%), Target Value=99.43
Summary of Fit

RSquare	0.407881
RSquare Adj	0.136494
Root Mean Square Error	2.407604
Mean of Response	98.39457
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	95.83103	8.71191	1.5029
Error	24	139.11731	5.79655	**Prob > F**
C. Total	35	234.94834		0.1946

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	0.378185	5.559
Phase-AB[Phase-PB]&Random	0.627981	9.231
Residual	5.796555	85.209
Total	6.802721	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	7.6805	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	5.79655	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	49.748	9.94961	5	1.2954	0.3760
Phase-AB[Phase-PB]&Random	46.083	7.6805	6	1.3250	0.2845

Response Measurement Analyte=TiO2 (wt%), Target Value=1.15
Summary of Fit

RSquare	0.444705
RSquare Adj	0.190195
Root Mean Square Error	0.028767
Mean of Response	1.147074
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.01590590	0.001446	1.7473
Error	24	0.01986137	0.000828	**Prob > F**
C. Total	35	0.03576727		0.1224

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-6.7e-5	-6.516
Phase-AB[Phase-PB]&Random	0.000267	25.984
Residual	0.000828	80.532
Total	0.001028	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	0.00163	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	0.00083	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	0.00613	0.00123	5	0.7533	0.6133
Phase-AB[Phase-PB]&Random	0.00977	0.00163	6	1.9680	0.1104

Exhibit A2. Analysis of Variance to Estimate Preparation Block (PB) Versus Analytical Block (AB) Effects

Response Measurement Analyte=ZnO (wt%), Target Value=0.02
Summary of Fit

RSquare	0.424851
RSquare Adj	0.161241
Root Mean Square Error	0.001236
Mean of Response	0.024322
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.00002707	2.4612e-6	1.6117
Error	24	0.00003665	1.5271e-6	**Prob > F**
C. Total	35	0.00006373		0.1585

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	7.001e-7	36.807
Phase-AB[Phase-PB]&Random	-3.25e-7	-17.092
Residual	1.527e-6	80.285
Total	1.902e-6	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	5.52e-7	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	1.53e-6	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	2.38e-5	4.75e-6	5	8.6128	0.0104
Phase-AB[Phase-PB]&Random	3.31e-6	5.52e-7	6	0.3613	0.8961

Response Measurement Analyte=ZrO2 (wt%), Target Value=0.13
Summary of Fit

RSquare	0.345542
RSquare Adj	0.045582
Root Mean Square Error	0.004694
Mean of Response	0.13218
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	11	0.00027920	0.000025	1.1520
Error	24	0.00052881	0.000022	**Prob > F**
C. Total	35	0.00080801		0.3680

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-PB&Random	Phase-AB[Phase-PB]&Random
Intercept	0	0	0
Phase-PB&Random	0	6	3
Phase-AB[Phase-PB]&Random	0	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-PB&Random	-3.13e-6	-13.701
Phase-AB[Phase-PB]&Random	3.964e-6	17.337
Residual	0.000022	96.364
Total	2.287e-5	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-PB&Random	3.39e-5	6	Phase-AB[Phase-PB]&Random
Phase-AB[Phase-PB]&Random	2.2e-5	24	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-PB&Random	7.56e-5	1.51e-5	5	0.4460	0.8033
Phase-AB[Phase-PB]&Random	0.0002	3.39e-5	6	1.5397	0.2081

Exhibit A3. Analysis of Variance to Estimate Analytical Block (AB) Effects

Response Measurement Type of Material=ARG-1, Analyte=Al2O3 (wt%), Target Value=4.73
Summary of Fit

RSquare	0.432173
RSquare Adj	0.164033
Root Mean Square Error	0.118307
Mean of Response	4.607231
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.38350008	0.022559	1.6117
Error	36	0.50387567	0.013997	**Prob > F**
C. Total	53	0.88737576		0.1125

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.002854	16.938
Residual	0.013997	83.062
Total	0.016851	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.014	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.3835	0.02256	17	1.6117	0.1125

Response Measurement Type of Material=ARG-1, Analyte=BaO (wt%), Target Value=0.09
Summary of Fit

RSquare	0.479088
RSquare Adj	0.233102
Root Mean Square Error	0.00332
Mean of Response	0.092165
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.00036496	0.000021	1.9476
Error	36	0.00039682	0.000011	**Prob > F**
C. Total	53	0.00076177		0.0458

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	3.482e-6	24.005
Residual	0.000011	75.995
Total	1.45e-5	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	1.1e-5	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.00036	2.15e-5	17	1.9476	0.0458

Response Measurement Type of Material=ARG-1, Analyte=B2O3 (wt%), Target Value=8.67
Summary of Fit

RSquare	0.509485
RSquare Adj	0.277853
Root Mean Square Error	0.262502
Mean of Response	7.928706
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	2.5765986	0.151565	2.1995
Error	36	2.4806584	0.068907	**Prob > F**
C. Total	53	5.0572570		0.0231

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.027552	28.564
Residual	0.068907	71.436
Total	0.09646	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.06891	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	2.5766	0.15156	17	2.1995	0.0231

Response Measurement Type of Material=ARG-1, Analyte=CaO (wt%), Target Value=1.43
Summary of Fit

RSquare	0.493477
RSquare Adj	0.254285
Root Mean Square Error	0.036045
Mean of Response	1.443508
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.04556753	0.002680	2.0631
Error	36	0.04677221	0.001299	**Prob > F**
C. Total	53	0.09233974		0.0335

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.00046	26.165
Residual	0.001299	73.835
Total	0.00176	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.0013	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.04557	0.00268	17	2.0631	0.0335

Exhibit A3. Analysis of Variance to Estimate Analytical Block (AB) Effects

Response Measurement Type of Material=ARG-1, Analyte=Cr2O3 (wt%), Target Value=0.09
Summary of Fit

RSquare	0.592668
RSquare Adj	0.400316
Root Mean Square Error	0.003775
Mean of Response	0.097694
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.00074638	0.000044	3.0812
Error	36	0.00051298	0.000014	**Prob > F**
C. Total	53	0.00125936		0.0022

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	9.885e-6	40.958
Residual	1.425e-5	59.042
Total	2.413e-5	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	1.42e-5	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.00075	4.39e-5	17	3.0812	0.0022

Response Measurement Type of Material=ARG-1, Analyte=Fe2O3 (wt%), Target Value=14
Summary of Fit

RSquare	0.25807
RSquare Adj	-0.09229
Root Mean Square Error	0.344422
Mean of Response	13.90013
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	1.4854470	0.087379	0.7366
Error	36	4.2705490	0.118626	**Prob > F**
C. Total	53	5.7559960		0.7464

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	-0.01042	-9.625
Residual	0.118626	109.625
Total	0.108211	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.11863	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	1.48545	0.08738	17	0.7366	0.7464

Response Measurement Type of Material=ARG-1, Analyte=CuO (wt%), Target Value=0
Summary of Fit

RSquare	0.935652
RSquare Adj	0.905266
Root Mean Square Error	0.002553
Mean of Response	0.00993
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.00341254	0.000201	30.7918
Error	36	0.00023469	6.519e-6	**Prob > F**
C. Total	53	0.00364723		<.0001

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	6.474e-5	90.851
Residual	6.519e-6	9.149
Total	7.126e-5	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	6.52e-6	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.00341	0.0002	17	30.7918	<.0001

Response Measurement Type of Material=ARG-1, Analyte=K2O (wt%), Target Value=2.71
Summary of Fit

RSquare	0.313485
RSquare Adj	-0.0107
Root Mean Square Error	0.125604
Mean of Response	2.694735
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.25934225	0.015255	0.9670
Error	36	0.56794534	0.015776	**Prob > F**
C. Total	53	0.82728759		0.5118

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	-0.00017	-1.113
Residual	0.015776	101.113
Total	0.015603	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.01578	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.25934	0.01526	17	0.9670	0.5118

Exhibit A3. Analysis of Variance to Estimate Analytical Block (AB) Effects

Response Measurement Type of Material=ARG-1, Analyte=Li2O (wt%), Target Value=3.21
Summary of Fit

RSquare	0.487027
RSquare Adj	0.24479
Root Mean Square Error	0.076454
Mean of Response	3.155992
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.19978474	0.011752	2.0105
Error	36	0.21042802	0.005845	**Prob > F**
C. Total	53	0.41021276		0.0386

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.001969	25.197
Residual	0.005845	74.803
Total	0.007814	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.00585	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.19978	0.01175	17	2.0105	0.0386

Response Measurement Type of Material=ARG-1, Analyte=MnO (wt%), Target Value=1.89
Summary of Fit

RSquare	0.534195
RSquare Adj	0.314231
Root Mean Square Error	0.045685
Mean of Response	1.827048
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.08616632	0.005069	2.4286
Error	36	0.07513503	0.002087	**Prob > F**
C. Total	53	0.16130135		0.0124

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.000994	32.258
Residual	0.002087	67.742
Total	0.003081	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.00209	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.08617	0.00507	17	2.4286	0.0124

Response Measurement Type of Material=ARG-1, Analyte=MgO (wt%), Target Value=0.86
Summary of Fit

RSquare	0.533492
RSquare Adj	0.313197
Root Mean Square Error	0.020641
Mean of Response	0.841587
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.01753970	0.001032	2.4217
Error	36	0.01533744	0.000426	**Prob > F**
C. Total	53	0.03287713		0.0127

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.000202	32.153
Residual	0.000426	67.847
Total	0.000628	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.00043	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.01754	0.00103	17	2.4217	0.0127

Response Measurement Type of Material=ARG-1, Analyte=Na2O (wt%), Target Value=11.5
Summary of Fit

RSquare	0.416417
RSquare Adj	0.140836
Root Mean Square Error	0.30252
Mean of Response	11.44477
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	2.3509052	0.138289	1.5111
Error	36	3.2946518	0.091518	**Prob > F**
C. Total	53	5.6455571		0.1461

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance

Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.01559	14.556
Residual	0.091518	85.444
Total	0.107108	100.000

These estimates based on equating Mean Squares to Expected Value.

Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.09152	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	2.35091	0.13829	17	1.5111	0.1461

Exhibit A3. Analysis of Variance to Estimate Analytical Block (AB) Effects

Response Measurement Type of Material=ARG-1, Analyte=NiO (wt%), Target Value=1.05
Summary of Fit

RSquare	0.533245
RSquare Adj	0.312833
Root Mean Square Error	0.026437
Mean of Response	1.017505
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.02874531	0.001691	2.4193
Error	36	0.02516108	0.000699	**Prob > F**
C. Total	53	0.05390639		0.0128

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.000331	32.116
Residual	0.000699	67.884
Total	0.00103	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.0007	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.02875	0.00169	17	2.4193	0.0128

Response Measurement Type of Material=ARG-1, Analyte=SiO2 (wt%), Target Value=47.9
Summary of Fit

RSquare	0.395606
RSquare Adj	0.110197
Root Mean Square Error	1.217502
Mean of Response	48.04313
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	34.928815	2.05464	1.3861
Error	36	53.363208	1.48231	**Prob > F**
C. Total	53	88.292023		0.2003

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.190775	11.403
Residual	1.482311	88.597
Total	1.673086	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	1.48231	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	34.9288	2.05464	17	1.3861	0.2003

Response Measurement Type of Material=ARG-1, Analyte=Sum of Oxides (wt%), Target Value=99.43
Summary of Fit

RSquare	0.321362
RSquare Adj	0.000894
Root Mean Square Error	2.483708
Mean of Response	98.40794
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	105.16228	6.18602	1.0028
Error	36	222.07700	6.16881	**Prob > F**
C. Total	53	327.23928		0.4774

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	0.005737	0.093
Residual	6.168806	99.907
Total	6.174542	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	6.16881	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	105.162	6.18602	17	1.0028	0.4774

Response Measurement Type of Material=ARG-1, Analyte=TiO2 (wt%), Target Value=1.15
Summary of Fit

RSquare	0.374351
RSquare Adj	0.078905
Root Mean Square Error	0.029052
Mean of Response	1.147584
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.01818091	0.001069	1.2671
Error	36	0.03038560	0.000844	**Prob > F**
C. Total	53	0.04856650		0.2674

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	7.514e-5	8.175
Residual	0.000844	91.825
Total	0.000919	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	0.00084	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.01818	0.00107	17	1.2671	0.2674

Exhibit A3. Analysis of Variance to Estimate Analytical Block (AB) Effects

Response Measurement Type of Material=ARG-1, Analyte=ZnO (wt%), Target Value=0.02
Summary of Fit

RSquare	0.324582
RSquare Adj	0.005635
Root Mean Square Error	0.001376
Mean of Response	0.02441
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.00003274	1.9262e-6	1.0177
Error	36	0.00006814	1.8927e-6	**Prob > F**
C. Total	53	0.00010088		0.4635

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	1.115e-8	0.585
Residual	1.893e-6	99.415
Total	1.904e-6	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	1.89e-6	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	3.27e-5	1.93e-6	17	1.0177	0.4635

Response Measurement Type of Material=ARG-1, Analyte=ZrO2 (wt%), Target Value=0.13
Summary of Fit

RSquare	0.384584
RSquare Adj	0.093971
Root Mean Square Error	0.004641
Mean of Response	0.131821
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	17	0.00048462	0.000029	1.3234
Error	36	0.00077549	0.000022	**Prob > F**
C. Total	53	0.00126011		0.2337

Expected Mean Squares
The Mean Square per row by the Variance Component per column

EMS	Intercept	Phase-AB&Random
Intercept	0	0
Phase-AB&Random	0	3

plus 1.0 times Residual Error Variance
Variance Component Estimates

Component	Var Comp Est	Percent of Total
Phase-AB&Random	2.322e-6	9.730
Residual	2.154e-5	90.270
Total	2.386e-5	100.000

These estimates based on equating Mean Squares to Expected Value.
Test Denominator Synthesis

Source	MS Den	DF Den	Denom MS Synthesis
Phase-AB&Random	2.15e-5	36	Residual

Tests wrt Random Effects

Source	SS	MS Num	DF Num	F Ratio	Prob > F
Phase-AB&Random	0.00048	2.85e-5	17	1.3234	0.2337

Distribution:

Name:	Location:
Sharon Marra	773-A
Connie Herman	999-W
Charles J. Coleman	773-A
Damon Click	773-A
Clint Gregory	773-A
Mark Barnes	773-A
Patricia Lee	703-41A
Richard Walker	703-41A
Michael Stone	999-W
David Peeler	999-W
Kevin Hera	723-A
Tommy Edwards	999-W
John Pareizs	773-A
Kevin Fox	999-W
Fabienne Johnson	999-W
Charles Crawford	773-42A
David Best	999-W
John Occhipinti	704-S
Jonathan Bricker	704-27S
John Iaukea	704-30S
Aaron Staub	704-27S
Jeff Ray	704-S
Perry Bovan	704-27S
Robert Hinds	704-S
Terri Fellinger	704-26S
Ryan McNew	704-S
Michael T. Hart	210-S
Roger N. Mahannah	704-28S
Michael T. Feller	704-28S
Omar Cardona-Quiles	704-24S
Amanda Shafer	704-27S
Mason Clark	704-27S
Helen Boyd	704-27S
Hank Elder	704-24S
Bill Holtzscheiter	704-15S
Pat Vaughan	773-41A