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Characterization of Analytical Reference Glass-1 (ARG-1)

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Summary

High-level radioactive waste may be immobilized in borosilicate glass. Before disposing of this glass in a geologic repository, waste form producers must report the measured chemical composition of the vitrified waste according to the U. S. Department of Energy's Waste Acceptance Product Specifications. To help meet this requirement, the Pacific Northwest Laboratory acquires and distributes appropriate nuclear-waste-related standard analytical reference materials. Use of common analytical reference materials will allow the waste glass producers' analytical laboratories to calibrate instrumentation and check procedures. Analytical Reference Glass - 1 (ARG-1) is one such standard. This document reports the chemical analyses and characterization of ARG-1. When new data on ARG-1 are generated, this document will be revised.

Acknowledgments

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1.0 Introduction

High-level radioactive waste may be immobilized in borosilicate glass at the West Valley Demonstration Project, West Valley, New York, the Defense Waste Processing Facility (DWPF), Aiken, South Carolina, and the Hanford Waste Vitrification Project (HWVP), Richland, Washington. The vitrified waste form will be stored in stainless steel canisters before its eventual transfer to a geologic repository for long-term disposal. Waste Acceptance Product Specifications (WAPS) (DOE 1993), Section 1.1.2 requires that the waste form producers must report the measured chemical composition of the vitrified waste in their production records before disposal.

Chemical analysis of glass waste forms is receiving increased attention due to qualification requirements of vitrified waste forms. The Pacific Northwest Laboratory (PNL)^(a) has been supporting the glass producers' analytical laboratories by a continuing program of multilaboratory analytical testing using interlaboratory "round robin" methods (Smith and Marschman, in press). At the PNL Materials Characterization Center Analytical Round Robin 4 workshop "Analysis of Nuclear Waste Glass and Related Materials," January 16-17, 1990, Pleasanton, California, the meeting attendees decided that simulated nuclear waste analytical reference glasses were needed for use as analytical standards. Use of common standard analytical reference materials would allow the glass producers' analytical laboratories to calibrate procedures and instrumentation, to control laboratory performance and conduct self-appraisals, and to help qualify their various waste forms.

Based on recommendations and discussions from that workshop, it was decided that Analytical Reference Glass - 1 (ARG-1) would consist of approximately 110 kg of Defense Waste Processing Facility (DWPF) simulated waste glass frit. This reference glass would need to be a homogeneous material with accurately known chemical composition, but leaching characteristics would not be relevant since this glass would be used only as an analytical standard and not as a leaching standard. The ARG-1 glass^(b) contains the more commonly measured elements in all proposed nuclear waste glass form compositions.

(a) Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

(b) Available from K. M. Olson, Materials Characterization Center, Pacific Northwest Laboratory.

2.0 Experimental

2.1 Glass Fabrication

The DWPF startup frit was manufactured by the Ferro Corporation for the Savannah River Laboratory (Jantzen 1989). Ferro Corporation was asked to batch the black frit to the Savannah River product oxide specification shown in Table 2.1. The glass was fabricated in a gas fired refractory smelter at a temperature of 1315°C. The glass melt was roll quenched into sheets that were processed through a finger crusher and hammer mill into a "corn flake" size consistency. The "corn flake" sized glass was then processed in a Kennedy Van Saun ceramic-lined continuous ball mill and sieved to a -80 to +200 mesh particle size. Fines were reprocessed in the smelter, and the larger particles were remilled. Approximately 15,000 pounds of black frit was produced, corresponding to Ferro Corporation product number SG-565.

2.2 Glass Sampling

Approximately 110 kg of DWPF startup frit from Lot #10-27-87 was shipped to Corning Incorporated in a single container. Corning ground and sieved the frit to -100 mesh and then blended the entire sample. The crushed and blended frit was then repackaged into three drums. A metal tube of ~ 1/2 in. diameter was used to sample each of the three drums near the bottom and top to make up the six samples used for chemical analyses. Upon receipt of the 110+ kg of powdered ARG-1 glass from Corning, PNL repackaged it into 1-kg lots for shipment. In addition to the sampling and analysis done by CELS - Corning Laboratory Services, PNL used ARG-1 as an unknown in Nuclear Waste Analytical Round Robin 6. For this round robin, each participating laboratory was given two 5-g samples of

Table 2.1. Savannah River Product Specification for Manufacture of the DWPF Startup Frit

Oxide	wt% $\pm$ wt% range
SiO ₂	49.0 $\pm$ 1.0
Fe ₂ O ₃	13.5 $\pm$ 1.0
Na ₂ O	11.5 $\pm$ 1.0
B ₂ O ₃	8.8 $\pm$ 1.0
Li ₂ O	3.5 $\pm$ 0.5
Al ₂ O ₃	4.7 $\pm$ 1.0
MnO ₂	2.3 $\pm$ 0.5
CaO	1.2 $\pm$ 0.5
NiO	1.1 $\pm$ 0.5
MgO	0.7 $\pm$ 0.5
K ₂ O	2.6 $\pm$ 0.5
TiO ₂	1.0 $\pm$ 0.5
Cr ₂ O ₃	0.1 $\pm$ 0.05
Sum	100.00

ARG-1. The first sample was representative of a 1-kg lot of ARG-1. The second sample was representative of a composite sample made up of more than twenty 10-g samples taken from every fifth kg while the 110+ kg of ARG-1 was being repackaged into 1-kg lots.

2.3 Glass Composition Analyses

CELS - Corning Laboratory Services analyzed six samples of ARG-1, which were selected randomly from the ground and blended glass. Each glass sample was dissolved in triplicate (18 replicates) with each replicate read twice independently to ensure that the two values corresponded closely (these duplicate reads are designated a and b in Appendix A). Details of the procedures used by Corning to analyze ARG-1 are given by C. M. Jantzen et al. (1993).

Samples of ARG-1 (see Glass Sampling above) were analyzed by seven different laboratories in Nuclear Waste Analytical Round Robin 6. A number of different sample preparations and analytical methods were used in the chemical analysis of ARG-1. In these cases ARG-1, was sent out to the Round Robin 6 participants as an unknown.

Three 5-g samples of ARG-1 were sent to the United States Geological Survey (USGS) for analysis in conjunction with Nuclear Waste Analytical Round Robin 7. The samples were chemically analyzed using both wavelength-dispersive x-ray fluorescence (WDXRF) and inductively coupled plasma atomic emission spectroscopy (ICP-AES). The WDXRF and ICP-AES data precision and accuracy were excellent and therefore are included in this report to compare with the Corning data. Both Corning and the USGS had nominal composition data before the analyses.

2.4 Glass Homogeneity Analyses

Three techniques were used to determine glass powder sample homogeneity: transmitted light optical microscopy based on color distribution and glass grain characteristics; scanning electron microscopy (SEM) analysis using energy dispersive analysis by x-ray (EDAX) of about 100 individual glass grains; and collection of chemical analysis data on random glass samples (see Glass Sampling above).

3.0 Results And Discussion

3.1 Glass Composition Analyses

The mean wt% and standard deviations of the 18 replicate ARG-1 glass analyses (Appendix A) measured by CELS - Corning Laboratory Services are given in Table 3.1. The nominal composition shown in Table 3.1 is chemical composition data provided by Savannah River Laboratory. These data were given to Corning before their chemical analyses.

Table 3.1 also lists the mean wt% and standard deviations for the USGS analyses obtained using WDXRF and ICP-AES. The USGS WDXRF group prepared working discs for three samples from a single bottle. The discs were counted at three different times over the course of two days. The USGS was provided with the Corning data as the nominal composition before their analyses.

The mean wt% and total standard deviations for the laboratories that participated in Round Robin 6 are also reported in Table 3.1, along with the number of laboratories whose data contributed to the mean wt% value reported for each oxide. Each laboratory analyzed ARG-1 as an unknown. It was found that in almost all cases, the interlaboratory variability is higher than the intralaboratory variability. The total standard deviations reported for Round Robin 6 were calculated from the data reported by each laboratory and are in most cases higher than the standard deviations reported by an individual laboratory. Comparison of the data in Table 3.1 gives a good representation of the quality of data to be expected when the sample being analyzed is an unknown. Standard deviations associated with the interlaboratory data are representative of data reported in other round robins.

The ferrous/ferric ratio in Table 3.1 listed under the Round Robin 6 data is the mean of three analyses performed at Pacific Northwest Laboratory. The redox measurement method used was a calorimetric 1, 10-phenanthroline procedure (Hara 1992). The standard deviation was calculated from three different measurements. The redox value reported by the USGS is also the mean of three analyses and the calculated standard deviation.

3.2 Glass Homogeneity Analyses

Transmitted light optical microscopy observations showed that the entire lot of ARG-1 was homogeneous on the basis of the color distribution and glass grain characteristics. Optical microscopy observation showed that most grains were brown although a few were colorless. Use of index matching fluid showed that the brown grains had an index of refraction, n_D , approximately equal to 1.6. The colorless glass grains had a different index of refraction, but this was not measured. Some glass grains showed striations, very small dark inclusions, or very small bubbles. An extremely small fraction of grains, $\leq 1\%$, appeared to be a crystalline phase.

In conjunction with EDAX, SEM was used to characterize this possible crystalline phase. The SEM observations did not conclusively show that the phase was crystalline, but found the phase high in aluminum. The phase was thought to be alumina from the alumina ball mill used to grind up the initial DWPF frit. However, this phase is an extremely small portion of the ARG-1 powder and is very evenly distributed throughout the sample.

Table 3.1. Comparison of the Target and Analyzed Analytical Reference Glass-1 (ARG-1) Compositions

Oxide	Nominal Composition	Corning Inc. Glass Composition		USGS Glass Composition		MCC Round Robin 6 Consensus Glass Composition		
	wt%	Mean wt%	SD	Mean wt% WDXRF/ICP	SD WDXRF/ICP	# Labs	Mean wt%	Total SD
Al ₂ O ₃	4.53	4.73	0.022	4.60/4.66	0.016/0.031	7	4.66	0.152
B ₂ O ₃	8.39	8.67	0.040	-/8.41	-/0.069	7	8.54	0.344
BaO	0.04	0.088	0.001	-/0.087	-/0.001	6	0.09	0.01
BeO				-/0.006	-/ < 0.0005			
CaO	1.53	1.43	0.009	1.43/1.43	0.003/0.015	7	1.42	0.055
CeO ₂				-/0.004	-/ < 0.0005			
CoO				-/0.005	-/0.001			
Cr ₂ O ₃	0.096	0.093	0.001	0.09/0.106	< 0.005/0.001	7	0.10	0.010
CuO	0.008	0.004	0.000	-/0.004	-/ < 0.0005	5	0.01	0.009
Fe ₂ O ₃	13.85	14.0	0.073	13.9/14.2	0.021/0.047	7	14.02	0.439
K ₂ O	2.59	2.71	0.016	2.66/2.47	0.007/0.085	7	2.68	0.182
Li ₂ O	3.26	3.21	0.015	-/3.49	-/0.073	7	3.18	0.237
MgO	0.85	0.86	0.005	0.88/0.86	0.009/0.005	7	0.87	0.034
MnO ₂	2.39	2.31	0.012	2.23/2.39	0.003/0.007	7	2.32	0.057
Na ₂ O	11.36	11.5	0.023	11.1/-	0.038/-	7	11.20	0.791
NiO	1.03	1.05	0.006	-/1.05	-/0.004	7	1.04	0.104
P ₂ O ₅	0.25	0.22	0.011	0.286/0.29	0.003/0.011	6	0.27	0.070
SiO ₂	48.6	47.9	0.157	47.2/-	0.059/-	7	47.75	1.531
SrO	0.006	0.0037	0.000	-/0.004	-/ < 0.0005	6	0.00	0.001
TiO ₂	1.12	1.15	0.007	1.14/1.18	0.005/0.005	7	1.17	0.063
V ₂ O ₅				-/0.021	-/ < 0.0005			
Y ₂ O ₃				-/0.001	-/ < 0.005			
ZnO	0.014	0.02	0.000	-/0.02	-/ < 0.0005	6	0.02	0.010
ZrO ₂	0.128	0.13	0.005			7	0.14	0.019
Sum	100.04	100.08		N/A			99.48	
Fe ²⁺ /Fe ³⁺	-	-	-	0.057	0.001		0.0581	0.0002

From SEM observations, it was also found that all grains showed about the same suite of elements, with variations in the Fe/Si ratio. Differences in this ratio could reflect differences in the orientation of the surface being analyzed relative to the detector or in lack of compositional homogeneity in the original glass melt or both.

Statistical analysis of the different splits of ARG-1 glass used in Round Robin 6 showed that powdered and mixed -100 mesh samples are homogeneous to well within analytical precision. As shown above, ARG-1 is a homogeneous glass powder and can be used as an analytical standard.

4.0 Conclusions

The chemical analysis values for ARG-1 measured by CELS - Corning Laboratory Services, the USGS, and the MCC's Round Robin 6 participants are all within the total standard deviation values from the round robin data. The nominal composition for ARG-1, to be used until further notice, will be the CELS - Corning Laboratory Services analysis. The $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio measured at PNL is 0.058 and 0.057 measured at the USGS.

The chemical composition of the approximately 110 kg of -100 mesh ARG-1 powder and the individually packaged ~1-kg lots were determined to be homogeneous. Although glass grain characteristics varied slightly on a microscopic level, the grains were homogeneously distributed. The ARG-1 glass fabricated by Ferro Corporation and ground, sieved, and blended by CELS - Corning Laboratory Services is suitable as an analytical standard.

5.0 References

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Appendix A

CELS - Corning Laboratory Services Chemical Analysis of Analytical Reference Glass-1 (ARG-1)

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 1

Lab I.D. No.: 34048

Element	1a	1b	2a	2b	3a	3b
Al2O3	4.71	4.73	4.73	4.73	4.75	4.76
BaO	0.088	0.087	0.087	0.087	0.087	0.086
B2O3	8.62	8.67	8.66	8.68	8.70	8.72
CaO	1.43	1.43	1.43	1.43	1.43	1.43
Cr2O3	0.095	0.095	0.092	0.092	0.092	0.093
CuO	0.0035	0.0035	0.0035	0.0035	0.0035	0.0034
Fe2O3	14.0	14.0	14.0	14.0	14.0	14.0
K2O	2.67	2.72	2.68	2.72	2.71	2.72
Li2O	3.20	3.23	3.20	3.23	3.20	3.23
MgO	0.85	0.85	0.85	0.85	0.85	0.85
MnO2	2.30	2.30	2.30	2.30	2.30	2.30
Na2O	11.5	11.4	11.5	11.5	11.5	11.5
NiO	1.05	1.04	1.04	1.04	1.04	1.04
P2O5	0.22	0.22	0.24	0.23	0.23	0.21
SiO2	47.9	47.9	48.0	47.9	48.0	48.2
SrO	0.0036	0.0035	0.0036	0.0036	0.0036	0.0036
TiO2	1.15	1.15	1.15	1.15	1.16	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.12	0.13	0.12	0.13	0.13	0.13
TOTAL	99.97	99.96	100.10	100.11	100.17	100.44

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 2

Lab I.D. No.: 34048

Element	4a	4b	5a	5b	6a	6b
Al2O3	4.72	4.76	4.71	4.72	4.70	4.75
BaO	0.087	0.088	0.087	0.088	0.088	0.088
B2O3	8.67	8.65	8.71	8.67	8.59	8.63
CaO	1.43	1.43	1.42	1.43	1.44	1.43
Cr2O3	0.092	0.093	0.092	0.092	0.093	0.093
CuO	0.0035	0.0036	0.0035	0.0035	0.0035	0.0035
Fe2O3	13.9	14.0	14.0	14.0	14.0	14.0
K2O	2.68	2.72	2.72	2.72	2.68	2.70
Li2O	3.20	3.23	3.22	3.23	3.20	3.20
MgO	0.85	0.85	0.85	0.86	0.85	0.85
MnO2	2.29	2.30	2.29	2.31	2.30	2.29
Na2O	11.5	11.5	11.5	11.5	11.5	11.5
NiO	1.04	1.04	1.04	1.05	1.04	1.04
P2O5	0.23	0.22	0.23	0.22	0.23	0.23
SiO2	47.8	48.2	47.7	47.9	47.6	48.0
SiO	0.0036	0.0036	0.0036	0.0036	0.0036	0.0036
TiO2	1.15	1.16	1.14	1.15	1.14	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.13	0.12	0.13	0.13	0.13	0.13
TOTAL	99.88	100.34	99.78	100.11	99.63	100.10

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 3

Lab I.D. No.: 34048

Element	7a	7b	8a	8b	9a	9b
Al2O3	4.76	4.72	4.71	4.71	4.75	4.77
BaO	0.088	0.088	0.087	0.087	0.087	0.087
B2O3	8.69	8.72	8.69	8.65	8.71	8.66
CaO	1.43	1.43	1.42	1.43	1.42	1.42
Cr2O3	0.094	0.093	0.093	0.093	0.093	0.093
CuO	0.0035	0.0034	0.0035	0.0035	0.0035	0.0036
Fe2O3	14.0	14.0	13.9	14.0	13.9	13.9
K2O	2.72	2.70	2.70	2.70	2.71	2.70
Li2O	3.22	3.20	3.23	3.23	3.20	3.20
MgO	0.85	0.85	0.85	0.85	0.85	0.85
MnO2	2.30	2.30	2.29	2.30	2.29	2.29
Na2O	11.5	11.5	11.5	11.5	11.5	11.5
NiO	1.04	1.04	1.04	1.04	1.04	1.04
P2O5	0.22	0.23	0.22	0.23	0.22	0.22
SiO2	48.1	47.8	47.7	47.8	48.1	48.2
StO	0.0035	0.0035	0.0035	0.0035	0.0035	0.0036
TiO2	1.16	1.15	1.15	1.15	1.16	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.13	0.13	0.13	0.13	0.13	0.13
TOTAL	100.31	99.99	99.76	99.94	100.21	100.26

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 4

Lab I.D. No.: 34048

Element	10a	10b	11a	11b	12a	12b
Al2O3	4.72	4.74	4.76	4.74	4.75	4.70
BaO	0.088	0.089	0.088	0.089	0.088	0.089
B2O3	8.71	8.69	8.60	8.59	8.73	8.66
CaO	1.44	1.45	1.44	1.45	1.45	1.44
Cr2O3	0.093	0.092	0.094	0.093	0.093	0.093
CuO	0.0035	0.0035	0.0034	0.0035	0.0035	0.0036
Fe2O3	14.1	14.1	14.1	14.2	14.1	14.2
K2O	2.71	2.76	2.71	2.70	2.71	2.72
Li2O	3.20	3.20	3.23	3.20	3.23	3.23
MgO	0.86	0.86	0.86	0.86	0.86	0.86
MnO2	2.32	2.32	2.32	2.33	2.32	2.33
Na2O	11.5	11.5	11.5	11.5	11.5	11.5
NiO	1.05	1.05	1.05	1.06	1.05	1.06
P2O5	0.23	0.22	0.20	0.22	0.23	0.22
SiO2	48.0	48.0	48.0	48.0	47.8	47.9
StO	0.0037	0.0037	0.0036	0.0037	0.0036	0.0037
TiO2	1.15	1.15	1.17	1.14	1.16	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.13	0.13	0.14	0.13	0.14	0.13
TOTAL	100.33	100.41	100.33	100.29	100.24	100.29

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 5

Lab I.D. No.: 34048

Element	13a	13b	14a	14b	15a	15b
Al2O3	4.76	4.72	4.71	4.75	4.71	4.69
BaO	0.088	0.088	0.088	0.088	0.088	0.088
B2O3	8.67	8.68	8.70	8.66	8.70	8.67
CaO	1.45	1.43	1.44	1.44	1.43	1.44
Cr2O3	0.093	0.093	0.093	0.093	0.093	0.092
CuO	0.0035	0.0036	0.0034	0.0039	0.0035	0.0034
Fe2O3	14.1	14.1	14.1	14.1	14.0	14.0
K2O	2.70	2.70	2.71	2.72	2.71	2.70
Li2O	3.20	3.20	3.20	3.20	3.23	3.20
MgO	0.86	0.86	0.86	0.86	0.86	0.86
MnO2	2.32	2.31	2.31	2.31	2.30	2.31
Na2O	11.4	11.5	11.5	11.5	11.5	11.5
NiO	1.05	1.05	1.05	1.05	1.05	1.05
P2O5	0.21	0.22	0.23	0.23	0.22	0.22
SiO2	48.1	48.0	48.0	48.1	47.8	47.7
StO	0.0039	0.0039	0.0039	0.0040	0.0039	0.0039
TiO2	1.16	1.15	1.15	1.14	1.15	1.14
ZnO	0.023	0.023	0.023	0.024	0.023	0.023
ZrO2	0.13	0.13	0.13	0.13	0.13	0.13
TOTAL	100.33	100.21	100.31	100.35	100.08	99.89

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Sample 6

Lab I.D. No.: 34048

Element	16a	16b	17a	17b	18a	18b
Al2O3	4.76	4.72	4.70	4.73	4.74	4.71
BaO	0.088	0.088	0.088	0.088	0.088	0.089
B2O3	8.67	8.56	8.64	8.69	8.64	8.70
CaO	1.43	1.44	1.44	1.45	1.44	1.43
Cr2O3	0.093	0.092	0.092	0.093	0.093	0.092
CuO	0.0036	0.0036	0.0035	0.0034	0.0036	0.0036
Fe2O3	14.1	14.1	14.1	14.1	14.1	14.0
K2O	2.72	2.70	2.73	2.72	2.72	2.70
Li2O	3.23	3.23	3.20	3.20	3.20	3.20
MgO	0.86	0.86	0.86	0.86	0.86	0.86
MnO2	2.30	2.32	2.31	2.31	2.32	2.31
Na2O	11.5	11.5	11.5	11.5	11.5	11.5
NiO	1.05	1.05	1.05	1.05	1.05	1.04
P2O5	0.24	0.23	0.22	0.22	0.20	0.26
SiO2	48.1	47.8	47.8	48.2	48.0	47.6
SrO	0.0039	0.0040	0.0038	0.0039	0.0039	0.0039
TiO2	1.16	1.14	1.15	1.16	1.15	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.14	0.13	0.14	0.14	0.13	0.13
TOTAL	100.37	100.03	100.07	100.55	100.25	99.87

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Averages of Samples 1 through 6

Lab I.D. No.: 34048

Element	1 avg	2 avg	3 avg	4avg	5 avg	6 avg
Al2O3	4.74	4.73	4.74	4.74	4.72	4.73
BaO	0.087	0.088	0.087	0.089	0.088	0.088
B2O3	8.68	8.65	8.69	8.66	8.68	8.65
CaO	1.43	1.43	1.43	1.45	1.44	1.44
Cr2O3	0.093	0.093	0.093	0.093	0.093	0.093
CuO	0.0035	0.0035	0.0035	0.0035	0.0035	0.0035
Fe2O3	14.0	14.0	14.0	14.1	14.1	14.1
K2O	2.70	2.70	2.71	2.72	2.71	2.72
Li2O	3.22	3.21	3.21	3.22	3.21	3.21
MgO	0.85	0.85	0.85	0.86	0.86	0.86
MnO2	2.30	2.30	2.30	2.32	2.31	2.31
Na2O	11.5	11.5	11.5	11.5	11.5	11.5
NiO	1.04	1.04	1.04	1.05	1.05	1.05
P2O5	0.23	0.23	0.22	0.22	0.22	0.23
SiO2	48.0	47.9	48.0	47.9	47.9	47.9
StO	0.0036	0.0036	0.0035	0.0037	0.0039	0.0039
TiO2	1.15	1.15	1.15	1.15	1.15	1.15
ZnO	0.023	0.023	0.023	0.023	0.023	0.023
ZrO2	0.13	0.13	0.13	0.13	0.13	0.14
TOTAL	100.13	99.97	100.08	100.32	100.20	100.19

LABORATORY ANALYSIS REPORT

CELS Client No.: 14594-004

Exhibit B : Quantitative Chemical Analysis

Sample Description: ARG-1 Stastical Data

Lab I.D. No.: 34048

Element	Target	Mean	Std Dev	% RSD
Al2O3	4.53	4.73	0.022	0.5
BaO	0.04	0.088	0.001	0.8
B2O3	8.39	8.67	0.040	0.5
CaO	1.53	1.43	0.009	0.6
Cr2O3	0.096	0.093	0.001	0.8
CuO	0.008	0.004	0.000	2.6
Fe2O3	13.85	14.0	0.073	0.5
K2O	2.59	2.71	0.016	0.6
Li2O	3.26	3.21	0.015	0.5
MgO	0.85	0.86	0.005	0.6
MnO2	2.39	2.31	0.012	0.5
Na2O	11.36	11.5	0.023	0.2
NiO	1.03	1.05	0.006	0.6
P2O5	0.25	0.22	0.011	4.8
SiO2	48.6	47.9	0.157	0.3
SrO	0.006	0.0037	0.000	4.4
TiO2	1.12	1.15	0.007	0.6
ZnO	0.014	0.02	0.000	0.7
ZrO2	0.128	0.13	0.005	3.6
TOTAL	100.00	100.15		

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