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PNNL-31372 Rev 0
EWG-RPT-034 Rev 0

Low-Activity Waste Glass Standards Preparation and Characterization for Calibration of Analytical Instruments

July 2021

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U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
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Pacific Northwest National Laboratory
Richland, Washington 99354

Summary

Seven standard glasses were fabricated and characterized for use as equipment/procedure standards for analysis of low-activity waste (LAW) glasses at the Hanford Waste Treatment and Immobilization Plant (WTP). The samples fabricated include monolithic bars and ≤ 70 mesh powdered glass. The bars could be used as standards for monolithic sample tests such as laser ablation inductively coupled plasma mass spectroscopy (LA-ICP-MS), X-ray fluorescence, and electron probe microanalysis (EPMA). Powdered glass samples could be used as standards for powder analyses techniques such as ICP of fused and/or dissolved samples. The glasses were formulated to represent a range of compositions of LAW glasses forecast to be produced at the WTP.

Glasses were fabricated in 3-kg batches at Pacific Northwest National Laboratory (PNNL) using an induction-heated tilt-pour furnace to provide sufficient material for testing homogeneity and use as standards. Each glass batch produced 12 glass bars and approximately 1 kg of crushed glass. The glass bars were sectioned and characterized using EPMA at PNNL. The 1 kg of crushed glass was analyzed at Southwest Research Institute (SwRI) using fusion/dissolution process followed by ICP-MS and ion chromatography (IC) analyses of the solutions.

The compositions of these standard glasses were verified to be on target with the batched compositions after fabrication using EPMA, IC, and ICP-MS analyses of each of the seven glasses. Glass bars were systematically analyzed using EPMA to determine the variability across the batch. For each batch, 5 of the 12 glass bars were analyzed in three locations in the bar (close to each end and the middle), with nine spots at each location. Calculated percent differences and relative standard deviations increased as batched concentration decreased, for a given component. This trend has been observed in past studies conducted to characterize waste glass standards such as Approved Reference Material-1 (ARM-1), Analytical Reference Glass-1 (ARG-1), Environmental Assessment (EA) Glass, and Low-Activity Test Reference Material (LRM).[1,2,3,4] All but four components showed analyzed values within ±10% relative difference from batch concentrations. The four components with higher relative differences were Cl, F, SO_3, and Y_2O_3, three of which (Cl, F, and SO_3) can be volatile in silicate melts. For these three components, the analyzed concentrations were biased low (consistent with volatile loss). The fourth component (Y_2O_3) was higher than targeted and had a concentration correlated with ZrO_2 in glass, suggesting an impurity in ZrO_2 source chemicals. All four components were also minor components (< 1 mass %) in the glass. It is recommended that the mean analyzed concentration of each of these four components be used for nominal composition of the standards.

Statistical plots were made from EPMA data to evaluate homogeneity within single bars and between bars for each of the seven glasses. Some visual indications of systematic variation at the bar-to-bar level were observed from these plots for components such as Al_2O_3 in glass LGS-19-01; however, overall, the variability from bar-to-bar within each of the seven glasses was determined to be statistically random. Further statistical analyses determined that most of the variability of the major components such as Al_2O_3,

[1] Mellinger GB and JL Daniel. 1984. *Materials Characterization Center (MCC) Approved Reference and Testing Materials for Use in Nuclear Waste Management Research and Development Programs*. PNL-4955-2, Pacific Northwest National Laboratory, Richland, WA.
[2] Smith GL. 1993. *Characterization of Analytical Reference Glass-1 (ARG-1)*. PNL-8992, Pacific Northwest National Laboratory, Richland, WA.
[3] Jantzen CM, NE Bibler, and DC Beam. 1992. *Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material (U)*. WSRC-TR-92-346, Westinghouse Savannah River Company, Aiken, SC.
[4] Ebert WL and SF Wolf. 1999. *Round-Robin Testing of a Reference Glass for Low-Activity Waste Forms*. ANL-99/22, Argonne National Laboratory, Argonne, IL.

SiO_2, and Na_2O occurred at the bar-to-bar level. Because the EPMA instrument was calibrated prior to each bar analysis, the variability associated with repeated calibrations was correlated and likely contributed to the observed bar-to-bar variability. Therefore, the EPMA analyses from all locations (bars, coupons, and spots) were pooled together to calculate a mean analyzed concentration and standard deviation (SD) for each glass component that encompasses the variability of the glass analysis, see Table S.1. Also shown in Table S.1 are the mean analyzed concentration and SD from SwRI's triplicate analysis of the crushed glass using ICP-MS and IC.

We recommend using the overall mean composition for each glass and SD, given in Table S.1, to estimate variance for each glass by EPMA and ICP-MS when using these standards to verify that calibrated instruments perform within specification. The SwRI-analyzed composition and SD most appropriately match up with calibration of the fusion/dissolution ICP methods of crushed glass. The EPMA composition and SD show that the variability of robust localized analysis may be more appropriate for calibration of any method(s) that analyzes glass at localized spots within monolithic glass samples.

Table S.1. Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-01 and -02 glass standards, in mass %.

		LGS19-01						LGS19-02					
Oxide	SwRI Method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	8.124	0.008	7.96	0.047	7.87	0.114	8.991	0.009	8.99	0.029	8.66	0.106
B_2O_3	ICP	9.748	0.010	9.66	0.256	9.91	0.372	6.162	0.006	5.99	0.340	6.37	0.398
CaO	ICP	7.311	0.007	7.08	0.035	7.13	0.072	10.203	0.010	10.02	0.061	10.19	0.109
Cl	IC	0.102	0.000	UD	NA	0.07	0.006	0.202	0.000	UD	NA	0.09	0.010
Cr_2O_3	ICP	0.102	0.000	0.10	0.000	0.10	0.014	0.202	0.000	0.19	0.002	0.19	0.017
F	IC	0.102	0.000	0.10	0.001	0.04	0.021	0.707	0.001	0.83	0.018	0.88	0.028
Fe_2O_3	ICP	0.203	0.000	0.21	0.010	0.20	0.019	3.839	0.004	3.82	0.022	3.82	0.052
K_2O	ICP	0.914	0.001	0.88	0.014	0.86	0.014	2.02	0.002	1.90	0.018	1.85	0.015
Li_2O	ICP	4.976	0.005	5.04	0.065	NM	NA	2.425	0.002	2.48	0.022	NM	NA
MgO	ICP	0.102	0.000	0.10	0.001	0.06	0.029	2.93	0.003	2.86	0.019	2.86	0.025
Na_2O	ICP	9.932	0.010	9.88	0.125	9.74	0.132	11.975	0.012	11.8	0.089	11.59	0.164
P_2O_5	ICP	0.811	0.001	0.88	0.062	0.83	0.029	0.538	0.001	0.63	0.106	0.55	0.027
SiO_2	ICP	44.375	0.044	45.71	0.539	44.14	0.529	38.389	0.038	39.65	0.539	39.21	0.377
SnO_2	ICP	0.000	0.000	0.00	0.000	0.00	0.011	1.818	0.002	1.74	0.038	1.69	0.023
SO_3	ICP	1.625	0.002	1.27	0.055	1.36	0.057	1.111	0.001	0.72	0.005	0.75	0.052
SO_3	IC	1.625	0.002	1.18	0.039	NA	NA	1.111	0.001	0.78	0.014	NA	NA
TiO_2	ICP	1.929	0.002	1.87	0.029	1.88	0.053	1.111	0.001	1.12	0.019	1.11	0.042
V_2O_5	ICP	3.960	0.004	3.93	0.036	3.85	0.094	3.031	0.003	3.05	0.036	2.92	0.070
Y_2O_3	ICP	0.782	0.001	0.68	0.006	0.63	0.045	0.347	0.000	0.31	0.001	0.28	0.039
ZnO	ICP	0.000	0.000	0.00	0.000	0.00	0.025	1.818	0.002	1.81	0.007	1.81	0.066
ZrO_2	ICP	4.905	0.005	4.58	0.024	4.73	0.101	2.178	0.002	2.04	0.039	2.15	0.084
Sum		100.00		99.8[(b)]		98.4[(c)]		100.00		100.0[(b)]		99.4[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table S.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-03 and -04 glass standards, in mass %.

		LGS19-03						LGS19-04					
Oxide	SwRI method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	6.966	0.007	6.96	0.011	6.92	0.102	5.708	0.006	5.60	0.076	5.66	0.074
B_2O_3	ICP	13.527	0.014	13.15	0.565	13.24	0.384	8.913	0.009	8.84	0.226	8.81	0.372
CaO	ICP	2.019	0.002	1.99	0.021	2.02	0.039	3.505	0.004	3.43	0.000	3.50	0.046
Cl	IC	0.303	0.000	0.03	0.007	0.18	0.013	0.401	0.000	0.14	0.018	0.34	0.011
Cr_2O_3	ICP	0.505	0.001	0.47	0.005	0.49	0.017	0.601	0.001	0.57	0.005	0.60	0.026
F	IC	0.404	0.000	0.45	0.015	0.40	0.027	0.3	0.000	0.35	0.013	0.32	0.023
Fe_2O_3	ICP	1.413	0.001	1.41	0.015	1.41	0.038	0.701	0.001	0.68	0.009	0.71	0.034
K_2O	ICP	5.047	0.005	4.76	0.000	4.75	0.042	0.100	0.000	0.11	0.000	0.11	0.009
Li_2O	ICP	0.808	0.001	0.80	0.005	NM	NA	0.000	0.000	0.00	0.000	NM	NA
MgO	ICP	0.505	0.001	0.50	0.005	0.49	0.019	1.803	0.002	1.73	0.019	1.84	0.027
Na_2O	ICP	16.015	0.016	15.59	0.078	16.22	0.230	23.713	0.024	23.28	0.280	23.71	0.173
P_2O_5	ICP	0.499	0.001	0.59	0.013	0.52	0.023	0.076	0.000	0.08	0.003	0.08	0.015
SiO_2	ICP	37.15	0.037	38.43	0.445	37.48	0.350	41.06	0.041	41.71	0.428	41.25	0.488
SnO_2	ICP	1.009	0.001	0.95	0.015	0.93	0.020	3.104	0.003	2.94	0.039	2.89	0.057
SO_3	ICP	0.808	0.001	0.64	0.024	0.60	0.036	0.501	0.001	0.45	0.004	0.44	0.027
SO_3	IC	0.808	0.001	0.49	0.012	NA	NA	0.501	0.001	0.50	0.008	NA	NA
TiO_2	ICP	0.505	0.001	0.51	0.003	0.50	0.032	1.502	0.002	1.50	0.014	1.50	0.072
V_2O_5	ICP	2.322	0.002	2.34	0.010	2.27	0.058	2.003	0.002	1.98	0.027	1.97	0.082
Y_2O_3	ICP	0.916	0.001	0.81	0.004	0.76	0.050	0.578	0.001	0.50	0.005	0.50	0.044
ZnO	ICP	3.533	0.004	3.49	0.045	3.57	0.088	1.803	0.002	1.77	0.026	1.85	0.063
ZrO_2	ICP	5.747	0.006	5.46	0.061	5.72	0.138	3.628	0.004	3.37	0.039	3.68	0.122
Sum		100.00		99.2[(b)]		99.3[(c)]		100.00		99.1[(b)]		99.8[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table S.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-05 and -06 glass standards, in mass %.

		LGS19 -05						LGS19-06					
Oxide	SwRI method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	10.035	0.010	10.09	0.332	9.92	0.096	6.337	0.006	6.24	0.029	6.15	0.057
B_2O_3	ICP	8.045	0.008	8.00	0.049	7.65	0.346	11.064	0.011	10.91	0.555	10.92	0.381
CaO	ICP	5.419	0.005	5.36	0.032	5.51	0.069	8.348	0.008	8.22	0.021	8.32	0.082
Cl	IC	0.201	0.000	0.02[(a)]	NA	0.14	0.008	0.603	0.001	0.09	0.016	0.26	0.012
Cr_2O_3	ICP	0.301	0.000	0.29	0.001	0.30	0.016	0.402	0.000	0.38	0.004	0.39	0.018
F	IC	1.104	0.001	1.31	0.681	1.39	0.059	0.201	0.000	0.22	0.003	0.16	0.025
Fe_2O_3	ICP	2.609	0.003	2.54	0.030	2.65	0.047	5.532	0.006	5.43	0.044	5.47	0.063
K_2O	ICP	2.408	0.002	2.27	0.014	2.26	0.018	3.319	0.003	3.08	0.007	3.00	0.023
Li_2O	ICP	0.702	0.001	0.70	0.004	NA	NA	4.224	0.004	4.26	0.022	NA	NA
MgO	ICP	1.104	0.001	1.07	0.008	1.05	0.034	2.515	0.003	2.42	0.017	2.37	0.022
Na_2O	ICP	19.415	0.019	18.83	0.078	19.26	0.258	5.347	0.005	5.20	0.034	5.16	0.092
P_2O_5	ICP	0.191	0.000	0.17	0.001	0.20	0.019	0.306	0.000	0.40	0.017	0.32	0.020
SiO_2	ICP	38.132	0.038	39.22	0.988	38.49	0.320	46.268	0.046	47.85	0.247	46.37	0.571
SnO_2	ICP	4.516	0.005	3.64	0.516	4.25	0.037	0.503	0.001	0.44	0.005	0.47	0.015
SO_3	ICP	0.201	0.000	0.15	0.001	0.19	0.023	0.402	0.000	0.21	0.009	0.24	0.024
SO_3	IC	0.201	0.000	0.20	0.011	NA	NA	0.402	0.000	0.13	0.004	NA	NA
TiO_2	ICP	0.100	0.000	0.09	0.009	0.10	0.022	0.201	0.000	0.20	0.001	0.20	0.025
V_2O_5	ICP	0.100	0.000	0.20	0.202	0.10	0.020	0.905	0.001	0.90	0.005	0.88	0.038
Y_2O_3	ICP	0.428	0.000	0.39	0.032	0.35	0.042	0.401	0.000	0.35	0.001	0.32	0.036
ZnO	ICP	2.308	0.002	2.26	0.026	2.37	0.075	0.603	0.001	0.60	0.005	0.60	0.043
ZrO_2	ICP	2.683	0.003	2.59	0.020	2.68	0.100	2.516	0.003	2.38	0.020	2.46	0.088
Sum		100.00		99.2[(b)]		99.6[(c)]		100.00		99.7[(b)]		98.3[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table S.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-07 glass standards, in mass %.

Oxide	SwRI method	LGS19-07					
		Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	8.106	0.008	7.92	0.033	7.94	0.091
B_2O_3	ICP	6.586	0.007	6.58	0.081	6.44	0.400
CaO	ICP	5.775	0.006	5.68	0.043	5.74	0.050
Cl	IC	0.203	0.000	0.02[(a)]	NA	0.14	0.015
Cr_2O_3	ICP	0.507	0.001	0.48	0.002	0.50	0.018
F	IC	0.304	0.000	0.35	0.006	0.33	0.021
Fe_2O_3	ICP	1.013	0.001	0.98	0.011	1.01	0.034
K_2O	ICP	1.013	0.001	0.94	0.007	0.96	0.013
Li_2O	ICP	1.52	0.002	1.50	0.003	NM	NA
MgO	ICP	2.026	0.002	1.91	0.020	1.99	0.023
Na_2O	ICP	21.087	0.021	20.62	0.135	20.94	0.162
P_2O_5	ICP	0.694	0.001	0.75	0.035	0.72	0.030
SiO_2	ICP	40.022	0.040	41.22	0.124	40.03	0.518
SnO_2	ICP	2.026	0.002	1.86	0.032	1.89	0.023
SO_3	ICP	0.709	0.001	0.60	0.001	0.59	0.036
SO_3	IC	0.709	0.001	0.65	0.052	NA	NA
TiO_2	ICP	1.52	0.002	1.48	0.023	1.51	0.045
V_2O_5	ICP	1.52	0.002	1.49	0.018	1.49	0.046
Y_2O_3	ICP	0.571	0.001	0.49	0.000	0.47	0.047
ZnO	ICP	1.216	0.001	1.18	0.018	1.23	0.059
ZrO_2	ICP	3.583	0.004	3.25	0.096	3.56	0.118
Sum		100.00		99.3[(b)]		99.0[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Acknowledgments

The authors would like to thank Albert Kruger and the Office of River Protection for supporting the development and characterization of this set of LAW glass standards. We would also like to thank Southwest Research Institute for the expert analysis of the glasses, Matt Wilburn for editing the report, and Jeff Bonnett is acknowledged for help preparing the glass batches. Peter Benson and David Aubry (WTCC) assisted in determining the appropriate glass composition ranges and test matrix design to support WTP laboratory requirements. Brad Johnson (ORNL) and Greg Piepel (retired) were instrumental in the planning of this testing. PNNL is operated for the U.S. Department of Energy by Battelle under contract DE-AC05-76RL01830

Acronyms and Abbreviations

ARG-1	Analytical Reference Glass-1
ARM-1	Approved Reference Material-1
DOE	U.S. Department of Energy
EA	Environmental Assessment
EDS	energy dispersive spectroscopy
EPMA	electron probe microanalysis
FEG	field-emission gun
IC	ion chromatography
ICP	inductively coupled plasma
ILAW	immobilized low-activity waste
LA	laser ablation
LAW	low-activity waste
LGS-19	LAW Glass Standard 2019
LRM	Low-Activity Reference Material
MS	mass spectroscopy
NQAP	Nuclear Quality Assurance Program
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
RSD	relative standard deviation
SD	standard deviation
SEM	scanning electron microscopy
SwRI	Southwest Research Institute
WTP	Hanford Waste Treatment and Immobilization Plant

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1.0 Introduction

The U.S. Department of Energy (DOE) Office of River Protection is constructing and commissioning the Hanford Waste Treatment and Immobilization Plant (WTP) to vitrify low-activity waste (LAW) that is currently stored in underground tanks on the Hanford Site to form borosilicate glass. LAW glass melt will be poured into stainless steel containers, welded shut, and disposed of as immobilized low-activity waste (ILAW) glass at the Integrated Disposal Facility, located at the Hanford Site. ILAW glass will be produced using a feed forward process, whereby a glass composition must be pre-qualified, with a series of glass property-composition models that demonstrate that each batch will produce a melt and a glass that satisfy processing, performance, and contract constraints (Vienna et al. 2020). Properties that are dependent on composition include (but are not limited to) melt viscosity, melt electrical conductivity, crystallinity, sulfate solubility, product consistency test response, vapor hydration test response, and melter refractory corrosion (Vienna et al. 2020).

The compositions of the ILAW glass produced by the WTP will be verified at the WTP analytical laboratory using techniques such as laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) and fusion/dissolution ICP. The LA-ICP-MS technique uses a focused laser beam to vaporize a spot(s) on the glass surface, which is then analyzed by the coupled ICP-MS instrument. When measuring glass samples, LA-ICP-MS offers advantages of reduced handling procedures and localized measurements. However, the LA-ICP-MS technique suffers from elemental fractionation effects within the instrument, which requires matrix matching standards, like those produced in this work, to accurately measure each component of the LAW glass (Günther et al., 2005). Traditionally, bulk analysis of glass has been performed by grinding a glass sample, dissolving it into solution, using microwave digestion or fusion techniques, and analyzing the solution by ICP coupled with optical emission spectroscopy or MS, following a procedure such as ASTM C1463-19. Fusion/dissolution ICP techniques have been used to analyze glasses for decades and to characterize several reference waste glasses in the past, such as Approved Reference Material-1 ARM-1), Environmental Assessment (EA) Glass, Analytical Reference Glass-1 (ARG-1), and Low-Activity Test Reference Material (LRM) (Mellinger et al. 1984, Jantzen et al. 1992, Smith 1993, Ebert et al. 1999). Before these analytical instruments can be used to measure ILAW glass samples, they require calibration using primary standards and verification of the instrument calibration using secondary standards. Therefore, this set of glass standards (secondary) was synthesized to be used for analysis of ILAW glasses produced at the WTP.

Traceable reference material of known composition is fundamental for accurate ICP analyses of complex chemistries for samples like dissolved LAW glasses. Ideally, standards should also resemble the chemistry of analyzed samples to account for matrix effects. To satisfy the requirement from WTP for standard material, a series of seven LAW glass standards that varied key components over the applicable composition space was designed, produced, and analyzed.

In this work, these seven glasses were analyzed using two different techniques: (1) electron probe microanalysis (EPMA) and (2) fusion ICP to provide a robust measured composition of each glass, on target with the batched compositions.

EPMA was used to systematically examine the glasses for changes in glass composition versus location on a glass sample. A total of 135 discrete measurements for each glass standard were made by EPMA, as follows:

- The EPMA measurements were performed on 5 bars evenly distributed within the 12 glass bars produced for each glass batch.

- These five bars (1, 4, 6, 8, and 12) were cross sectioned at three locations (each end and the middle) and polished to produce sample coupons.
- Coupons were analyzed using a three-by-three grid, resulting in nine analysis spots.

Fusion/dissolution ICP analysis was performed to measure glass compositions of three representative samples of ground glass powder for each glass.

Following analysis of the glasses, the measured data was statistically evaluated to provide the means, standard deviations (SDs), and relative standard deviations (RSDs) of each glass composition and individual glass bar. Variabilities among glass bars and coupon location within bars, and variability within coupons, were statistically evaluated. This was done to determine if systematic variability exists within the glass batches or if the variability should be treated as random variation. The analysis provided measured compositions and estimated uncertainties for each glass component in each standard glass that can be used for calibration of analytical instruments or verification of analytical results.

1.1 Quality Assurance

This work was performed in accordance with the Pacific Northwest National Laboratory (PNNL) Nuclear Quality Assurance Program (NQAP). The NQAP complies with DOE Order 414.1D, *Quality Assurance*, and 10 CFR 830 Subpart A, Quality Assurance Requirements. The NQAP uses NQA-1-2012, *Quality Assurance Requirements for Nuclear Facility Application*, as its consensus standard and NQA-1-2012, Subpart 4.2.1, as the basis for its graded approach to quality.

The NQAP works in conjunction with PNNL's laboratory-level Quality Management Program, which is based upon the requirements as defined in DOE Order 414.1D, *Quality Assurance*, and 10 CFR 830, *Nuclear Safety Management*, Subpart A, Quality Assurance Requirements.

The work of this report was performed to the quality assurance (QA) level of applied research with a technology readiness level of 6. This work was performed to support technology development. Data obtained may be used to support design input.

2.0 Experimental

2.1 Glass Formulation and Fabrication

The glass formulations included a suite of components found in typical LAW glasses. The compositions for each sample are given in Table 2.1. Samples are named LGS-19-0A where A ranges from 1-7 and LGS-19 is short for "LAW Glass Standard 2019." The seven glasses were formulated so that, for each component, the series of glasses would represent a distribution of major component concentrations across the anticipated range of glasses that could be produced (Vienna et al. 2020). Additionally, glasses were required to have compositions that produced acceptable values of viscosity, electrical conductivity, durability, SO_3 solubility, and K-3 refractory corrosion such that the ratios of components would be representative of LAW glasses likely to be produced. Three kilograms of each glass composition was fabricated so that enough standard material would be generated for multiple characterization methods.

Table 2.1. Target composition of LAW glass standards given in mass %

	LGS19-01	LGS19-02	LGS19-03	LGS19-04	LGS19-05	LGS19-06	LGS19-07
Al_2O_3	8.00	8.90	6.90	5.70	10.00	6.30	8.00
B_2O_3	9.60	6.10	13.40	8.90	8.00	11.00	6.50
CaO	7.20	10.10	2.00	3.50	5.40	8.30	5.70
Cl	0.10	0.20	0.30	0.40	0.20	0.60	0.20
Cr_2O_3	0.10	0.20	0.50	0.60	0.30	0.40	0.50
F	0.10	0.70	0.40	0.30	1.10	0.20	0.30
Fe_2O_3	0.20	3.80	1.40	0.70	2.60	5.50	1.00
K_2O	0.90	2.00	5.00	0.10	2.40	3.30	1.00
Li_2O	4.90	2.40	0.80	0.00	0.70	4.20	1.50
MgO	0.10	2.90	0.50	1.80	1.10	2.50	2.00
Na_2O	10.00	12.00	16.00	23.70	19.40	5.40	21.00
P_2O_5	2.10	1.40	1.30	0.20	0.50	0.80	1.80
SiO_2	43.70	38.00	36.80	41.00	38.00	46.00	39.50
SnO_2	0.00	1.80	1.00	3.10	4.50	0.50	2.00
SO_3	1.60	1.10	0.80	0.50	0.20	0.40	0.70
TiO_2	1.90	1.10	0.50	1.50	0.10	0.20	1.50
V_2O_5	3.90	3.00	2.30	2.00	0.10	0.90	1.50
ZnO	0.00	1.80	3.50	1.80	2.30	0.60	1.20
ZrO_2	5.60	2.50	6.60	4.20	3.10	2.90	4.10
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Target compositions were adjusted to account for impurities in raw materials identified on the certificates of analysis, as shown in Table 2.2. Components primarily affected were P_2O_5 and ZrO_2. Y_2O_3 was also included in the batched compositions due to its presence in the ZrO_2 source chemical. The batched compositions were used throughout the report to compare to the measured compositions.

Table 2.2. Batched composition of LAW glass standards given in mass % oxides

Batched	LGS19-01	LGS19-02	LGS19-03	LGS19-04	LGS19-05	LGS19-06	LGS19-07
Al_2O_3	8.124	8.991	6.966	5.708	10.035	6.337	8.106
B_2O_3	9.748	6.162	13.527	8.913	8.045	11.064	6.586
CaO	7.311	10.203	2.019	3.505	5.419	8.348	5.775
Cl	0.102	0.202	0.303	0.401	0.201	0.603	0.203
Cr_2O_3	0.102	0.202	0.505	0.601	0.301	0.402	0.507
F	0.102	0.707	0.404	0.300	1.104	0.201	0.304
Fe_2O_3	0.203	3.839	1.413	0.701	2.609	5.532	1.013
K_2O	0.914	2.020	5.047	0.100	2.408	3.319	1.013
Li_2O	4.976	2.425	0.808	0.000	0.702	4.224	1.520
MgO	0.102	2.930	0.505	1.803	1.104	2.515	2.026
Na_2O	9.932	11.975	16.015	23.713	19.415	5.347	21.087
P_2O_5	0.811	0.538	0.499	0.076	0.191	0.306	0.694
SiO_2	44.375	38.389	37.150	41.060	38.132	46.268	40.022
SnO_2	0.000	1.818	1.009	3.104	4.516	0.503	2.026
SO_3	1.625	1.111	0.808	0.501	0.201	0.402	0.709
TiO_2	1.929	1.111	0.505	1.502	0.100	0.201	1.520
V_2O_5	3.960	3.031	2.322	2.003	0.100	0.905	1.520
Y_2O_3	0.782	0.347	0.916	0.578	0.428	0.401	0.571
ZnO	0.000	1.818	3.533	1.803	2.308	0.603	1.216
ZrO_2	4.905	2.178	5.747	3.628	2.683	2.516	3.583
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Multiple balances with variable sensitivity were used to batch chemicals with ±0.1% accuracy. The balance types used for different mass ranges and associated accuracies are given in Table 2.3. Measuring equipment was calibrated according to the project QA implementing procedure. The balances were verified to operate properly and within batching specification with calibrated balance check weights (see Table 2.3) of known mass, before each glass batch was prepared.

Table 2.3. List of balances (2-, 3-, or 4-decimal place) used for glass batching to ±0.1% of the target masses, along with acceptable accuracy of each mass range and range of check masses used to verify balance performance.

Balance	Target Mass, X	Balance Acceptable Accuracy	Balance Check Masses
2-place	$X \geq 50$ g	± 0.05g	2kg, 50g
3-place	50 g$\geq X \geq 5$ g	± 0.005g	50g, 5g
4-place	$X \leq 5$ g	± 0.0005g	5g, 0.5g

Glasses were batched using reagent grade chemicals. Special precautions in terms of batch order and weighing technique were adopted for certain chemicals such as H_3BO_3 and NaF due to problems such as electro-static attraction between the chemical and measuring equipment, and loss of mass while weighing, respectively. For example, H_3BO_3 was weighed on a plastic weigh boat and added to the tared container (bag + support container) first, and then the container plus the H_3BO_3 was reweighed after the chemical was added. Any amount lost during transfer from the weigh boat due to static attraction was compensated for by weighing out and adding more of said chemical to the batch.

Scoping studies were performed before glasses were produced in large quantity, by melting small batches of each composition. During scoping studies, SnO_2 was batched as the oxide, which led to undissolved SnO_2 in the glass. However, when batched using the source chemical sodium stannate, no undissolved SnO_2 was observed in the glasses. Therefore, sodium stannate was used in all of the glasses.

Chemicals were batched into a large, custom-made bag and then shaken by hand to mix. Each batch was then further homogenized for 15 minutes inside a V-blender equipped with an intensifier bar. Batches were then placed into a 90% platinum/10% rhodium crucible with a lid and nominally melted at 1150 °C for 1 hour with an induction-heated tilt-pour furnace (Ultra-MELT, TLT-2P). The melt temperature was monitored using an optical pyrometer.

To minimize melt segregation, each melt was stirred with a Pt rod. Glasses were examined visually for signs of segregated salts or undissolved materials during the melting process (top of melt) and after pouring. Each melt was poured into twelve 2.5-cm x 2.5-cm x 9.6-cm steel molds on a heated stainless-steel plate. An exception was LGS19-01, which was poured into 11 bars. The glass bars were annealed for 5 hours at 500 °C and cooled at 1 °C/min to room temperature. In between melts of the different glass compositions, this crucible was cleaned with an alkali borate cleaning glass, followed by a concentrated HF acid leach and water rinse.

2.2 Glass Characterization Methodology

2.2.1 Electron Microprobe Analysis

Glass bars were cross-sectioned and cut into coupons. Three coupons were cut for each bar. Each coupon was embedded into epoxy and polished to a finish of 1 µm using a series of diamond abrasives: 15-um diamond-impregnated disk, followed by progressively smaller diamond suspensions on polishing pads (9, 3, and 1 µm). Samples were cleaned with soap and water and coated with 20 nm of carbon to dissipate charging in the EPMA instrument and match the coating applied to analytical standards.

The elemental composition of each LAW glass was measured by EPMA using a JEOL 8530F HyperProbe, made by JEOL USA (Peabody, MA). The EPMA instrument uses a field-emission gun (FEG) equipped with five wavelength dispersive spectrometers, each with a take-off angle of 40°. The microprobe data was collected at an accelerating voltage of 15 kV, beam current of 20 nA, and beam size of 100 µm. These scan parameters were chosen based on results of samples from preliminary test measurements that were performed to determine element mobility under the electron beam. The microprobe data was collected and analyzed with “Probe for EPMA” software, version 12.6.1, from Probe Software Inc. (Eugene, OR). The standards used, collection times, on/off peak locations, and background fit functions are provided in Table 2.4 for each element analyzed. Standard assignments for certain elements differed in some cases between different glass compositions (e.g., Al, Si), but for coupons of the same glass composition, standards remained the same. Interferences were identified and corrected for by applying interference standards for each element. Wavescans were collected using arbitrarily chosen coupons to fit background functions for each glass composition. Application of time-dependent intensity corrections, using a method described by Nielsen et al. (1981), was limited to the first element analyzed by each spectrometer (B, Zn, Si, Ca, and Na), which is a restriction built into the Probe for EPMA software. Oxygen and lithium were not directly analyzed. Instead, oxygen was calculated based on stoichiometry of the oxides analyzed. Lithium was added based on the “as-batched” composition of each standard glass, which was confirmed to be appropriate based on ICP measurements of Li. Each coupon was measured in nine different locations in an approximate square grid with the intention of maximizing representation of the sample. Spots where charging was observed or where the polish was inadequate were avoided.

Table 2.4. EPMA data collection conditions: primary standard, crystal type, on/off peak locations and collection times, and background function type.

Element	Standard[a]	Spectrometer Crystal[b]	Spectrometer	Run Order	On Peak Location, L-value / Time (s)	High Off-Peak Location, Offset / Time (s)	Low Off-Peak Location, Offset / Time (s)	Background Function Type
Al	#4006, Albite, #1002 Albite	TAP	5	2	90.3030 / 25	4.3125 / 6	-2.8083 / 6	Linear
B	#7001, EA glass	LDE-B	1	1	129.102 / 90	94.353 / 25	-20.003 / 25	Exponential
Ca	#4010, Wollastonite	PET	4	1	107.513 / 30	6.01 / 7	-3.925 / 7	Linear
Cl	#5027 Pyromorphite	PET	4	4	151.413 / 40	3.97701 / 10	-6.913 / 10	Linear
Cr	#4024, Cr2O3	LiF	3	3	159.219 / 40	5.843 / 10	-4.345 / 10	Linear
F	#4016, Fluorite	LDE-1	1	2	86.0170 / 40	2.5827 / 10	-11.61 / 10	Exponential
Fe	#4011, Hematite	LiF	2	2	134.601 / 25	5.24701 / 6	-3.126 / 6	Linear
K	#1008, Orthoclase	PET	4	3	119.801 / 40	8.7001 / 10	-2.081 / 10	Exponential
Mg	#1003, Periclase	TAP	5	3	107.203 / 35	12.497 / 7	-9.1222 / 7	Exponential
Na	#4006, Albite	TAP	5	1	129.202 / 25	9.104 / 6	-11.19 / 6	Linear
P	#4005, Apatite(natural)	PET	3	4	196.844 / 40	5.304 / 10	-6.142 / 10	Linear
S	# 4008, Barite $BaSO_4$	PET	3	3	171.717 / 40	7.10501 / 10	-6.845 / 10	Linear
Si	#4010, Wollastonite, #1008 Orthoclase	PET	3	1	77.4460 / 25	9.6005 / 6	-9.6004 / 6	Linear
Sn	#1026, Cassiterite	PET	4	2	115.229 / 40	2.53101 / 10	-1.971 / 10	Linear
Ti	#4007 TiO2 Rutile	LiF	2	5	191.185 / 25	4.782 / 6	-1.171 / 6	Linear
V	#1033, V metal	LiF	2	4	174.106 / 25	5.72 / 7	-2.028 / 7	Linear
Zn	#2016, Zn metal	LiF	2	1	99.6820 / 25	6.288 / 6	-6.0722 / 6	Exponential
Zr	#4001, Zircon (ZrSiO4)	PET	3	5	194.136 / 25	6.83901 / 6	-5.325 / 6	Linear
Y	#5008, Yttrium Aluminum Garnet	TAP	3	2	70.057 / 30	10.0921 / 7	-8.7919 / 7	Linear
O	Calculated stoichiometrically using other analyzed elemental oxides based on target oxidation states							

(a) The 1000s place in the standard number denotes the standard block that each standard is from, where 1000s refers to GP40 standard block produced by P&H, 2000s refers to standard block SPI #02751-AB produced by SPI Supplies, 3000s refers to borosilicate glass standards assembled at PNNL, and 4000s refers to standard block Geo Mk II produced by P&H.

(b) Crystal abbreviations: thallium acid phthalate (TAP), layered dispersive element (LDE-1, LDE-B), lithium fluoride (LiF), pentaerythritol (PET)

2.2.2 EDS Analysis

Qualitative elemental glass composition analysis by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) was performed using a JEOL JSM-7001F FEG SEM instrument (Peabody, MA) equipped with a Bruker XFlash 6|60 EDS detector (Madison, WI). EDS analysis was conducted using the following microscope parameters: 15 kV accelerating voltage, 10 mm working distance, and 5 nA probe current, resulting in an average of 100 kcps through the detector. EDS analysis was performed on the same set of samples analyzed by EPMA. Three sample coupons, or 1 bar (each end and middle), were loaded into the SEM instrument at a time and analyzed using a square analysis grid (1.5 mm^2), resulting in 81 analysis spots per coupon.

Note that Li cannot be measured as an oxide with this EDS detector. Boron, which can be detected by this detector, was not measured because of interferences by other elements in the glass, the most significant being Ca and Zr, and because of the low signal generated by B from these glass samples.

2.2.3 Fusion ICP/IC Analysis

Approximately 1 kg of each standard glass was crushed and sieved to -70 mesh. Three representative samples of each glass were analyzed using ion chromatography (IC) and ICP-MS at SwRI. Samples were analyzed for Br^-, F^-, Cl^-, and SO_4^{2-} using IC. In sample preparation for IC, approximately 0.200 g of each sample was first fused with Na_2CO_3. Fusions were diluted to 50 mL with deionized water. Samples were pretreated with OnGuard II H to eliminate the matrix interference caused at the fluoride retention time. Samples were prepared for ICP-MS using three digestion techniques. First, a closed vessel digestion using nitric, hydrochloric, and hydrofluoric acids was used to prepare samples for measurement of B (for all samples) and P (for samples LGS-10-04-end1, mid, and end2, and LGS-19-05-end1, mid, and end2). Second, a lithium metaborate/tetraborate fusion was used to prepare samples for measurement of Ca, Cr, Si, and Zr. Third, a nitric, perchloric, hydrofluoric, and hydrochloric digestion in an open vessel was used to prepare samples for measurement of the remaining metals and the remaining samples for P. Loss on ignition was measured and applied for all samples to normalize out any absorbed moisture in the ground glass samples.

3.0 Results and Discussion

3.1 Glass Melting Observations

Large convection cells were observed during melting in the tilt-pour crucible. These cells cycled molten salts, which are slow to incorporate into the melt, to the top of the melt, and in certain samples a gall layer was formed. For one glass (LGS19-02), the gall layer that formed in sample could not be integrated with normal stirring. Milling and remelting is used in smaller and less-sensitive batches to help integrate segregated components into the glass; however, in this case, milling would have likely contributed to batch contamination, and therefore was avoided. Instead, an extended melt time of 4 hours and continued stirring were used to integrate the molten salt layer into the melt (and potentially volatilize salt components) to produce a homogenous glass. As the gall layer contains volatile components, the loss of these components was considered a risk during this extended melting time; however, since the glass was intended to be a standard, obtaining a homogenous glass was deemed most important. The melting times, furnace conditions, and notable melting observations for each sample are given in Table 3.1.

Failures associated with the tilt-pour's electrical controller resulted in some melts being interrupted or receiving different melt histories. It was discovered when first using the tilt-pour that higher power levels resulted in furnace shutoffs. The melting of sample LGS19-04 was most affected by lower power levels (see Table 3.1). To maximize the heating capability of the furnace for subsequent heat treatments, patches were applied to the heating coil, and coolant flow to the tilt-pour was increased. A heating program was also adopted that employed lower power levels at the beginning and gradually increased power levels toward the end of the melt. Temperatures recorded in Table 3.1 were taken from the top of the melt with an optical pyrometer while stirring. The limit of the optical pyrometer is 1200 °C, and therefore, material in the middle of all melts exceeded that temperature, except for LGS19-04. During hold times when the melt was not being stirred, temperatures at the top of the melts were between 1050 and 1175 °C, except for LGS19-04.

Table 3.1. Melting conditions and observations

Batch	Total Time (hr:min:sec)	Hold Time (hr:min:sec)	Max Temp. (when stirred) (°C)	Power	Furnace Failure	Notes
LGS19-01	2:50:00	1:30:00	≥1200 °C	75-90%	Yes	Poured 11 bars instead of normal 12
LGS19-02	5:40:00	4:00:00	≥1200 °C	70-80%	No	Gall layer, extended melting time
LGS19-03	3:15:00	1:25:00	≥1200 °C	80-85%	No	Refractory removed from melt
LGS19-04	4:45:00	1:00:00	900 °C	55-75%	Yes	High foam, crack in one bar from mold
LGS19-05	3:00:00	1:10:00	≥1200 °C	80-85%	No	
LGS19-06	3:20:00	1:30:00	≥1200 °C	75-85%	No	High volatility and segregation
LGS19-07	2:50:00	1:30:00	≥1200 °C	80-90%	No	Small amount of precipitated sulfur

3.2 EPMA Analysis Results

The batched compositions and the measured overall mean glass compositions by EPMA are provided in Table 3.2. The measured overall mean glass composition is the mean of all 135 measurements for each glass composition. These 135 discrete analyses for each glass are broken down as follows: 5 bars per glass × 3 coupons per bar × 9 positions per coupon. The totals for measured means range from 98.3 to 99.8 mass %, with Li_2O included as batched. Li_2O was given as an as-batched composition because of the inability of EPMA to detect Li. Totals approximated 100 mass %, which indicated the analyses accounted for all the expected components in the glass. Specific component measurements were also on target with the batched compositions.

Table 3.3 lists the percent relative difference between the measured mean and batched compositions for each element analyzed. The percent difference between the batched and measured mean compositions was also plotted versus the batched mass fraction (Log_{10}-Log_{10} plot, Figure 3.1), which showed that the relative difference decreased with batched component concentration. Similarly, the RSDs of the measurements, given in Table 3.4, decrease with concentrations of batched components, as shown on the Log_{10}-Log_{10} scale in Figure 3.2. In contrast to Figure 3.1, Figure 3.2 also shows independent trends for three major components (B_2O_3, SiO_2, and Na_2O) and one minor component (K_2O). The RSD of B_2O_3 is shifted above other glass components of similar batch concentration because of its low atomic mass. Atomic mass of the analyzed component affects signal strength by EPMA, so light components, especially B, have low signal strength while K has higher-than-average signal strength. In contrast, the RSDs of SiO_2 and Na_2O are nearly concentration independent. The RSD for SiO_2 was independent of concentration because it was collected on a PET-J crystal, which is the most sensitive to environmental temperature changes in the lab. The changes in RSD for Na_2O were likely due to alkali mobility under the electron beam that required the TDI correction to determine the Na_2O concentration before Na_2O migrates. This TDI fit adds an additional source of error that was not dependent on Na_2O concentration but was most likely linked to differences in the Na bonding environment in the seven different glasses. While the data collection process was optimized for the seven glasses analyzed, it was not optimized for each glass composition and therefore the RSD of Na_2O is independent of concentration.

Figure 3.1 and Figure 3.2 both indicate a general trend that measurements were less precise as concentrations decreased. This is in line with basic principles of EPMA, where component concentration drives signal strength and, as signal strength decreases, the relative variability contributed by counting electronics in the EPMA instrument increases. To improve the counting statistics at low concentrations, the counting times would need to be increased significantly based on the Log_{10}-Log_{10} relationship shown in Figure 3.1. However, increasing the counting time per analysis spot for the minor and trace components would have been impractical for the number of measurements required in this study. In addition, increased counting times may have introduced more environmental variability because the analyzing crystals in an EPMA instrument expand and contract slightly with changes in lab temperature, which impacts the counts.

Some components of the glass (Cl, K, F, S) can be subject to volatility during melting, and in some cases the measured means were significantly different from target compositions. These differences are reflected in Table 3.4. Additionally, difficulties in weighing the fluoride source chemical, NaF, may have contributed to the difference in F between the target and the measured compositions. Yttrium oxide, another component with a high calculated percent difference, was a component used to stabilize the ZrO_2 in the cubic structure in source chemicals, and therefore is not important to LAW glass. The target amount is based on the given fraction (2.00 wt%) of Y_2O_3 present in the source chemical. The accuracy of EPMA measurements for the highlighted high difference components is also represented graphically in 1:1 plots in Figure 3.3. These plots help to illustrate the differences between measured and targeted values of volatile and hard-to-incorporate components.

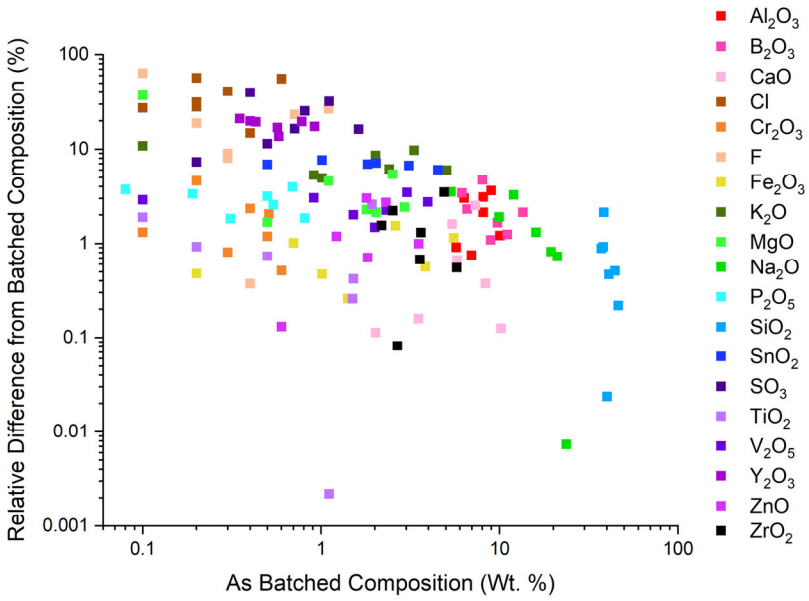

Figure 3.1. Log_{10}-Log_{10} plot of the relative percent difference of batched and mean EPMA-measured compositions versus the batched concentration in wt% oxide for each component. All seven glasses were plotted together. Plot shown for information only (FIO).

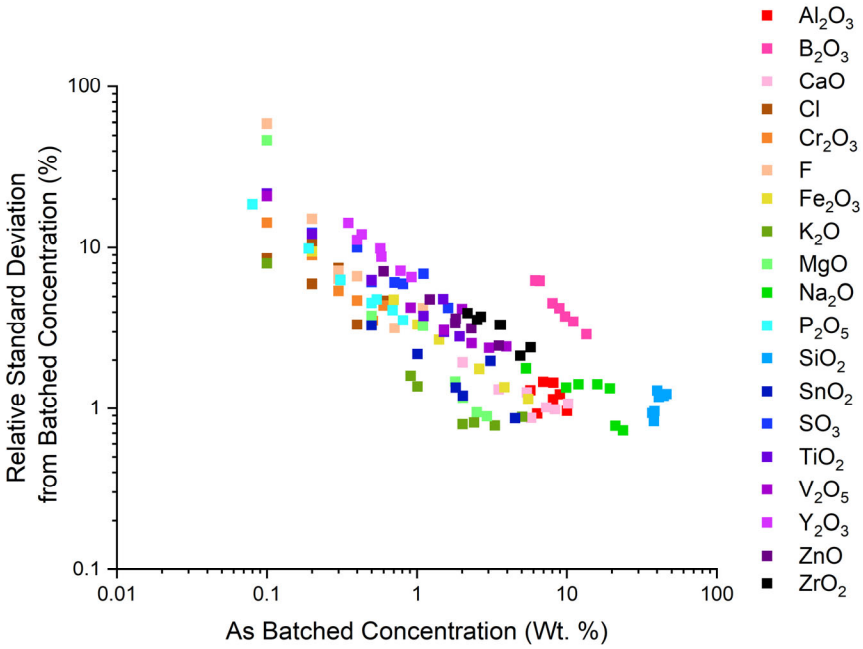

Figure 3.2. Log_{10}-Log_{10} plot of the percent relative standard deviation of the EPMA measurements versus the batched concentration in wt% oxide for each component. All seven glasses were plotted together. Plot shown FIO.

Table 3.2. Batched and average measured compositions from EPMA analysis in mass %. Average means the average of measurements from all bars for a given glass standard.[a]

	LGS19-01		LGS19-02		LGS19-03		LGS19-04		LGS19-05		LGS19-06		LGS19-07	
	Meas.	Batched	Meas.	Batched	Meas.	Batched	Meas.	Batched	Meas.	Batched	Meas.	Batched	Meas.	Batched
Al_2O_3	7.87	8.124	8.66	8.991	6.92	6.966	5.66	5.708	9.92	10.035	6.15	6.337	7.94	8.106
B_2O_3	9.91	9.748	6.37	6.162	13.24	13.527	8.81	8.913	7.65	8.045	10.92	11.064	6.44	6.586
CaO	7.13	7.311	10.19	10.203	2.02	2.019	3.5	3.505	5.51	5.419	8.32	8.348	5.74	5.775
Cl	0.07	0.102	0.09	0.202	0.18	0.303	0.34	0.401	0.14	0.201	0.26	0.603	0.14	0.203
Cr_2O_3	0.1	0.102	0.19	0.202	0.49	0.505	0.6	0.601	0.3	0.301	0.39	0.402	0.5	0.507
F	0.04	0.102	0.88	0.707	0.4	0.404	0.32	0.3	1.39	1.104	0.16	0.201	0.33	0.304
Fe_2O_3	0.2	0.203	3.82	3.839	1.41	1.413	0.71	0.701	2.65	2.609	5.47	5.532	1.01	1.013
K_2O	0.86	0.914	1.85	2.02	4.75	5.047	0.11	0.1	2.26	2.408	3	3.319	0.96	1.013
Li_2O[a]	*4.98*	4.976	*2.43*	2.425	*0.81*	0.808	*0*	0	*0.7*	0.702	*4.22*	4.224	*1.52*	1.52
MgO	0.06	0.102	2.86	2.93	0.49	0.505	1.84	1.803	1.05	1.104	2.37	2.515	1.99	2.026
Na_2O	9.74	9.932	11.59	11.975	16.22	16.015	23.71	23.713	19.26	19.415	5.16	5.347	20.94	21.087
P_2O_5	0.83	0.811	0.55	0.538	0.52	0.499	0.08	0.076	0.2	0.191	0.32	0.306	0.72	0.694
SiO_2	44.14	44.375	39.21	38.389	37.49	37.15	41.25	41.06	38.49	38.132	46.37	46.268	40.03	40.022
SnO_2	0	0	1.69	1.818	0.93	1.009	2.89	3.104	4.25	4.516	0.47	0.503	1.89	2.026
SO_3	1.36	1.625	0.75	1.111	0.6	0.808	0.44	0.501	0.19	0.201	0.24	0.402	0.59	0.709
TiO_2	1.88	1.929	1.11	1.111	0.5	0.505	1.5	1.502	0.1	0.1	0.2	0.201	1.51	1.52
V_2O_5	3.85	3.96	2.92	3.031	2.27	2.322	1.97	2.003	0.1	0.1	0.88	0.905	1.49	1.52
Y_2O_3	0.63	0.782	0.28	0.347	0.76	0.916	0.5	0.578	0.35	0.428	0.32	0.401	0.47	0.571
ZnO	0	0	1.81	1.818	3.57	3.533	1.85	1.803	2.37	2.308	0.6	0.603	1.23	1.216
ZrO_2	4.73	4.905	2.15	2.178	5.72	5.747	3.68	3.628	2.68	2.683	2.46	2.516	3.56	3.583
Sum	98.4	100.00	99.4	100.00	99.3	100.00	99.8	100.00	99.6	100.00	98.3	100.00	99.0	100.00

(a) Li_2O was not measured but was given at the batched values to evaluate sum.

High relative differences generally correlated with low batched concentrations of elements in the glasses, as shown in Table 3.3. Minor components close to 0.1 wt% approached the limit of detection for EPMA, and therefore had proportionally more variable data when measured. Limits of detection vary by element for EPMA, but generally are within the range 0.05-0.20 oxide mass % when using counting times selected for this work.

Table 3.3. Absolute relative difference between measured mean and batched values by EPMA, in percent.

Component	LGS19-01	LGS19-02	LGS19-03	LGS19-04	LGS19-05	LGS19-06	LGS19-07
Al_2O_3	3.14	3.66	0.71	0.88	1.17	2.97	2.08
B_2O_3	1.68	3.38	2.11	1.12	4.71	1.28	2.25
CaO	2.55	0.16	0.08	0.02	1.61	0.36	0.58
Cl	28.47	57.28	41.66	14.93	31.60	56.10	29.03
Cr_2O_3	2.81	5.61	2.09	0.66	1.17	2.91	1.39
F	64.46	23.75	0.60	7.87	26.17	19.36	7.58
Fe_2O_3	1.05	0.54	0.03	0.86	1.57	1.18	0.15
K_2O	5.71	8.59	5.97	10.69	6.12	9.74	5.24
Li_2O	NM	NM	NM	NM	NM	NM	NM
MgO	38.00	2.42	2.61	2.14	4.93	5.59	1.96
Na_2O	1.92	3.23	1.26	0.01	0.81	3.47	0.71
P_2O_5	1.72	2.97	3.37	8.95	2.89	3.14	3.42
SiO_2	0.52	2.14	0.92	0.47	0.93	0.22	0.02
SnO_2	0.00	6.88	7.59	6.85	5.96	7.42	6.96
SO_3	16.47	32.26	25.18	11.53	7.64	40.37	16.42
TiO_2	2.56	0.11	0.20	0.40	1.53	0.34	0.41
V_2O_5	2.76	3.49	2.34	1.62	3.25	2.53	2.00
Y_2O_3	19.76	20.48	16.93	13.39	19.00	19.99	17.12
ZnO	0.00	0.62	0.91	2.87	2.78	0.45	1.53
ZrO_2	3.60	1.49	0.45	1.36	0.22	2.06	0.76

NM = not measured

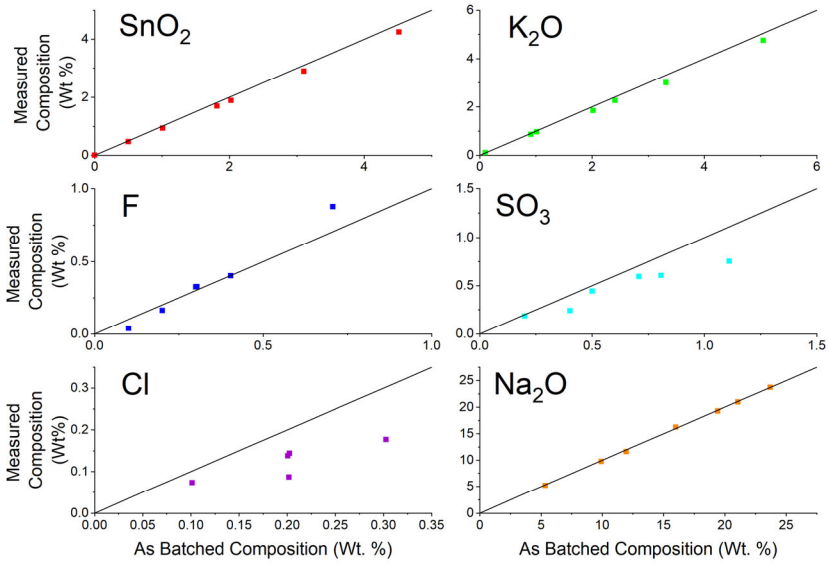

Figure 3.3. EPMA-measured versus batched composition plots for components with high percent difference with trendlines with y-intercepts set to 0. Plot shown FIO.

Table 3.4. Percent relative standard deviation of measurements of each glass composition (on oxide mass basis) using EPMA.

Component	LGS19-01	LGS19-02	LGS19-03	LGS19-04	LGS19-05	LGS19-06	LGS19-07
Al_2O_3	1.45	1.23	1.45	1.30	0.96	0.93	1.14
B_2O_3	3.76	6.25	2.91	4.22	4.53	3.49	6.22
CaO	1.01	1.07	1.93	1.32	1.26	0.99	0.87
Cl	8.55	11.38	7.46	3.33	5.96	4.69	10.08
Cr_2O_3	14.29	8.93	3.35	4.39	5.40	4.72	3.52
F	58.88	3.17	6.63	7.16	4.23	15.11	6.39
Fe_2O_3	9.41	1.36	2.69	4.76	1.77	1.15	3.34
K_2O	1.60	0.80	0.89	7.96	0.82	0.78	1.37
Li_2O	NM	NM	NM	NM	NM	NM	NM
MgO	46.55	0.89	3.81	1.47	3.27	0.94	1.17
Na_2O	1.35	1.42	1.40	0.73	1.34	1.78	0.78
P_2O_5	3.56	4.79	4.54	18.63	9.82	6.31	4.12
SiO_2	1.20	0.96	0.86	1.18	0.83	1.23	1.29
SnO_2	0.00	1.35	2.18	1.98	0.87	3.30	1.21
SO_3	4.23	6.85	5.99	6.11	12.36	9.99	6.09
TiO_2	2.81	3.79	6.29	4.81	21.62	12.15	3.00
V_2O_5	2.43	2.39	2.55	4.18	17.13	4.26	3.08
Y_2O_3	7.15	14.23	6.51	8.75	12.07	11.15	9.84
ZnO	0.00	3.65	2.47	3.40	3.17	7.12	4.78
ZrO_2	2.13	3.93	2.35	3.33	3.74	3.57	3.32

3.3 Qualitative EDS Analysis

Qualitative analysis of the glass samples was performed to look for relative differences in component concentrations with the same systematic sampling approach as EPMA but with significantly more analysis spots, 81 by EDS versus 9 by EPMA. EDS was a faster qualitative analysis process used to screen for inhomogeneous regions. Qualitative EDS analysis did not reveal any systematic changes within coupons. SEM images of the samples showed glass that was devoid of unique features, which also indirectly supports a homogenous sample.

In some coupons, the variability of Na_2O and SiO_2 between analysis spots increased relative to other coupons of the same glass batch analyzed. Unlike EPMA, this analysis technique does not collect TDI data and therefore it's impossible to correct for Na_2O migration caused by the electron beam. Some of the coupons were repolished and reanalyzed at a lower (by one magnitude) beam current to reduce possible Na_2O migration and the increased variability still occurred, but on a different coupon that previously showed smaller, more typical variability from spot-to-spot. This small reanalysis set confirmed that the original measurements were not affected by the electron beam conditions and that the effect appeared to be random. Another possible reason for the increased variance within a sample could be a poorly grounded sample, which would allow the sample to periodically charge and discharge under the electron beam. However, charging was not directly observed while running the samples.

3.4 IC and ICP Analysis

The measured mean bulk glass compositions (mean of triplicate samples) along with the batched compositions are given in Table 3.5. The measured totals range from 100.01 to 99.08 mass %, which on average is roughly 1% higher than the totals determined by EPMA. As with EPMA, these results indicate that ICP and IC bulk analysis of the glasses captures all the components present in the glasses. Li_2O was detected using ICP and was found to be present and on target with the batched composition.

As was done with EPMA data, the percent difference between batched and measured mass % from IC/ICP was plotted versus batch concentration (Figure 3.4). As was observed with EPMA, the errors decrease with increasing component concentrations. In addition, the magnitude of relative errors at small concentrations is comparable between the two methods. RSDs of IC/ICP components were also plotted versus concentration (Figure 3.5), and a trend similar to the one from EPMA was observed. In contrast to the data from EPMA, however, variation in IC/ICP data at low concentrations was nearly twice as large. The components that occurred in smaller concentrations, and thus had larger error and variability, have also been identified as volatile components. Therefore, errors in components such as Cl, F, P_2O_5, and SO_3 may also be due in part to their volatility.

Table 3.5. Batched and measured mean glass compositions by IC and ICP analyses, in mass %.

		LGS19-01		LGS19-02		LGS19-03		LGS19-04		LGS19 -05		LGS19-06		LGS19-07	
Oxide	Method	Batch	Meas.	Batch	Meas.	Batch	Meas.	Batch	Meas.	Batch	Meas.	Batch	Meas.	Batch	Meas.
Al_2O_3	ICP	8.124	7.96	8.991	8.99	6.966	6.96	5.708	5.6	10.035	10.09	6.337	6.24	8.106	7.92
B_2O_3	ICP	9.748	9.66	6.162	5.99	13.527	13.15	8.913	8.84	8.045	8	11.064	10.91	6.586	6.58
CaO	ICP	7.311	7.08	10.203	10.02	2.019	1.99	3.505	3.43	5.419	5.36	8.348	8.22	5.775	5.68
Cl	IC	0.102	[a]	0.202	[a]	0.303	0.03	0.401	0.14	0.201	0.02[b]	0.603	0.09	0.203	0.02[b]
Cr_2O_3	ICP	0.102	0.1	0.202	0.19	0.505	0.47	0.601	0.57	0.301	0.29	0.402	0.38	0.507	0.48
F	IC	0.102	0.1	0.707	0.83	0.404	0.45	0.300	0.35	1.104	1.31	0.201	0.22	0.304	0.35
Fe_2O_3	ICP	0.203	0.21	3.839	3.82	1.413	1.41	0.701	0.68	2.609	2.54	5.532	5.43	1.013	0.98
K_2O	ICP	0.914	0.88	2.020	1.9	5.047	4.76	0.100	0.11	2.408	2.27	3.319	3.08	1.013	0.94
Li_2O	ICP	4.976	5.04	2.425	2.48	0.808	0.8	0.000	0	0.702	0.7	4.224	4.26	1.520	1.5
MgO	ICP	0.102	0.1	2.930	2.86	0.505	0.5	1.803	1.73	1.104	1.07	2.515	2.42	2.026	1.91
Na_2O	ICP	9.932	9.88	11.975	11.8	16.015	15.59	23.713	23.28	19.415	18.83	5.347	5.2	21.087	20.62
P_2O_5	ICP	0.811	0.88	0.538	0.63	0.499	0.59	0.076	0.08	0.191	0.17	0.306	0.4	0.694	0.75
SiO_2	ICP	44.375	45.71	38.389	39.65	37.150	38.43	41.060	41.71	38.132	39.22	46.268	47.85	40.022	41.22
SnO_2	ICP	0.000	0	1.818	1.74	1.009	0.95	3.104	2.94	4.516	3.64	0.503	0.44	2.026	1.86
SO_3	IC	1.625	1.27	1.111	0.72	0.808	0.64	0.501	0.45	0.201	0.15	0.402	0.21	0.709	0.6
SO_3[c]	ICP	1.625	1.18	1.111	0.78	0.808	0.49	0.501	0.5	0.201	0.2	0.402	0.13	0.709	0.65
TiO_2	ICP	1.929	1.87	1.111	1.12	0.505	0.51	1.502	1.5	0.100	0.09	0.201	0.2	1.520	1.48
V_2O_5	ICP	3.960	3.93	3.031	3.05	2.322	2.34	2.003	1.98	0.100	0.2	0.905	0.9	1.520	1.49
Y_2O_3	ICP	0.782	0.68	0.347	0.31	0.916	0.81	0.578	0.5	0.428	0.39	0.401	0.35	0.571	0.49
ZnO	ICP	0.000	0	1.818	1.81	3.533	3.49	1.803	1.77	2.308	2.26	0.603	0.6	1.216	1.18
ZrO_2	ICP	4.905	4.58	2.178	2.04	5.747	5.46	3.628	3.37	2.683	2.59	2.516	2.38	3.583	3.25
Sum		100.00	99.84	100.0	100.01	100.00	99.18	100.00	99.08	100.00	99.22	100.00	99.70	100.00	99.33

(a) One or more analytes from this sample were not detected above SwRI's limit of detection (LOD).
(b) Analyte was detected at or above SwRI's LOD but below SwRI's limit of quantitation.
(c) SO_3 measured by IC was used for sum calculations. SO_3 measured by ICP not used for sum calculations

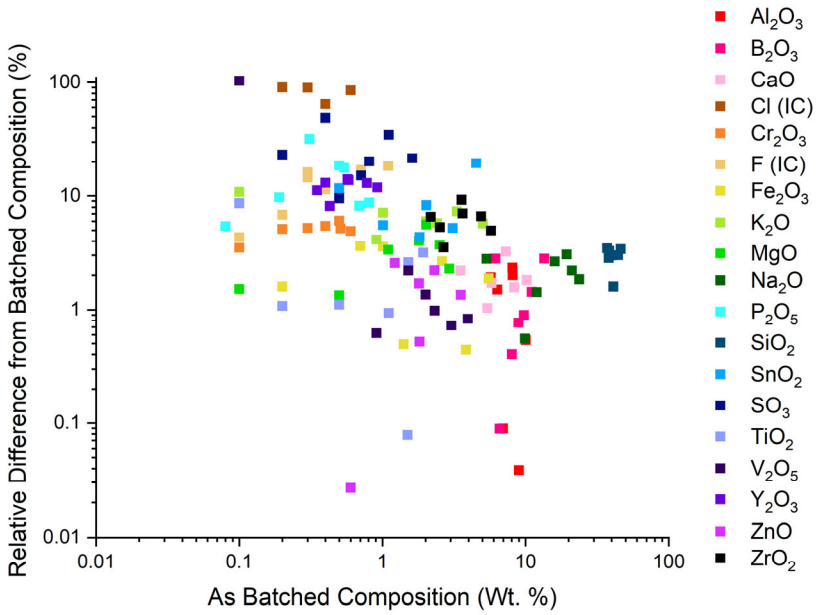

Figure 3.4. Log_{10}-Log_{10} plot of the percent relative difference (absolute values) of measured mean by ICP and IC versus the batched concentration in mass % oxide for each component. All seven glasses were plotted together. Plot shown FIO.

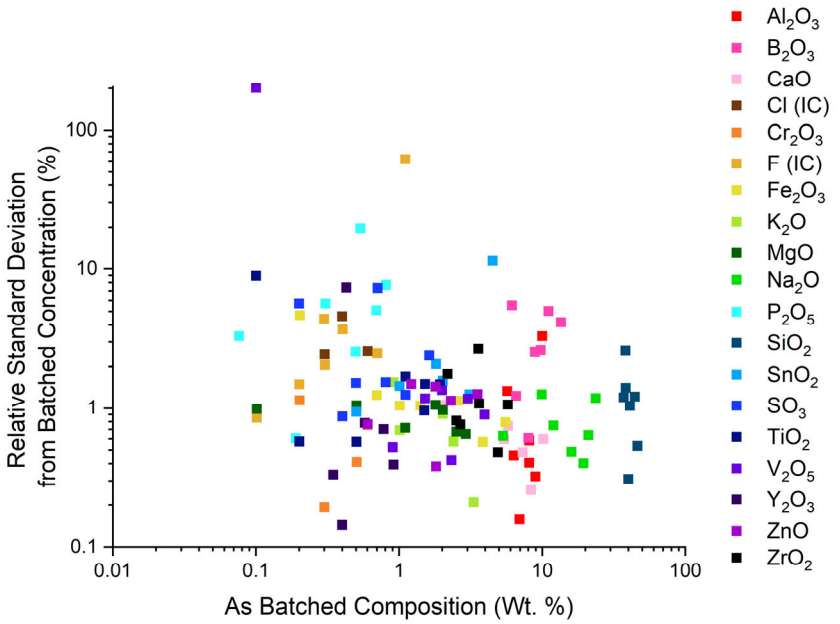

Figure 3.5. Log_{10}-Log_{10} plot of the relative standard deviation of compositions measured by ICP and IC, versus the batched concentration in mass % oxide for each component. All seven glasses were plotted together. Plot shown FIO.

3.5 Statistical Analysis

The EPMA data was collected systematically (see Section 2.0) to determine the following for each glass standard:

1. Is the standard glass composition on target with the as-batched composition?
2. Do systematic differences exist from bar-to-bar within the glass standard batch?
3. Do systematic differences exist from spot-to-spot within a sample coupon?

Four EPMA data points were removed as outliers from the dataset:

- LGS-19-03-bar12-end2-spot1 was removed because the sum of all components was 41 mass %, indicating that this analysis spot likely had a physical defect that drastically reduced total counts for all components. This was the only low total recovery in the dataset.
- LGS-19-03-bar10-middle-spot6 and LGS-19-03-bar12-middle-spot6 were both removed because measured concentrations were significantly shifted (low Na_2O and high K_2O) from other measurements of LGS19-03.
- LGS-19-04-1-bar12-middle-spot 3 was removed because the concentrations of CaO, Cl, K_2O, and SnO_2 were all significantly lower than other measurements on this glass.

Additional outliers were observed in the EPMA data set, where single components were outside the variability of their data set. An example is shown in Figure 3.7, where LGS-19-06 has one low Cl measurement that is an outlier. However, they were not removed from the data set as an outlier unless multiple components were collectively outliers, as discussed above for a given analysis spot. The decision to keep spots when individual components were outliers was made to preserve the full variability of each component analyzed in the EPMA data sets.

The analysis of each glass batch by EPMA and fusion/dissolution ICP showed that the major components were all within ± 10 % (absolute relative difference) of the batch concentrations. Only five components (Cl, F, SO_3, MgO, and Y_2O_3) had absolute relative differences between the measured and batched concentrations that exceeded ± 10 mass % on an oxide basis by ICP and EPMA, see Figure 3.1 and Figure 3.4. Three of these components (Cl, F, and SO_3) may be volatile in borosilicate waste glass melts. LGS-19-1 had a high absolute relative difference for MgO (38.8 %) that was attributed to the low batched concentration (0.102 wt%) in this glass. Higher concentrations of MgO in other glasses had absolute relative differences ≤ 5.6 %. Like MgO, The increased difference in measured Y_2O_3 was attributed to the small concentration in the glasses (0.35 to 0.92 mass %).

From a practical point of view, the analyzed compositions show relative standard deviations of the analyzed concentrations similar to those that have been shown during round robin studies conducted on past standard glasses such as LRM, ARG-1, EA, and ARM-1 glasses, which provides confidence in the glass preparation and analysis of these seven new LAW glass standards (Mellinger et al., 1984, Jantzen et al., 1992, Smith, 1993, Ebert et al., 1999). The RSDs of the measured values for LRM, ARG-1, EA, and ARM-1 glasses, shown in Figure 3.6, are also similar in magnitude and trend to that observed in this work done by ICP and EPMA, shown in Figure 3.2 and Figure 3.5 (Mellinger et al. 1984, Jantzen et al. 1992, Smith 1993, Ebert et al. 1999).

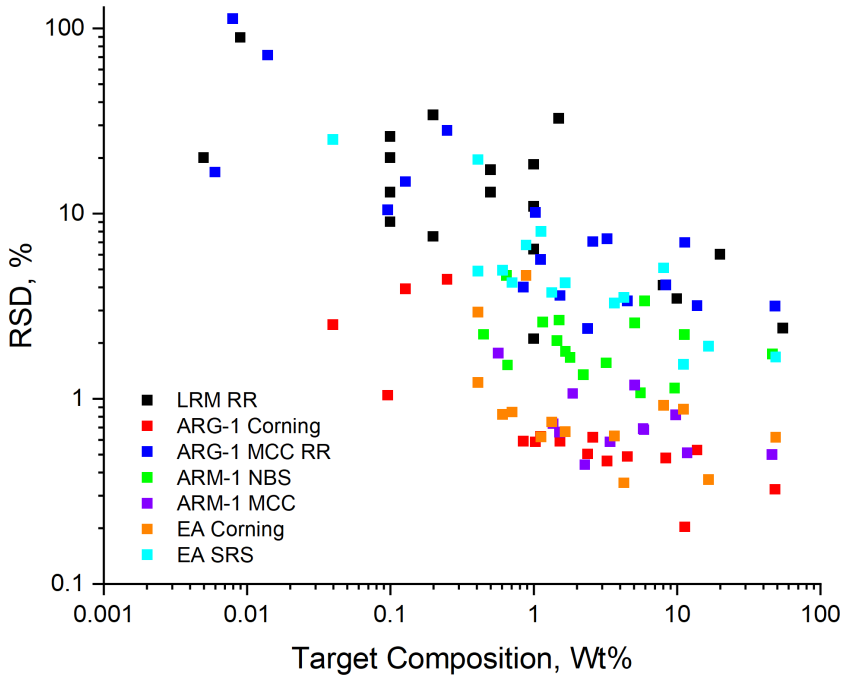

Figure 3.6. Percent relative standard deviation of measured values versus target concentration for existing waste glass standards: LRM, ARG-1, ARM-1, and EA glasses (Mellinger et al., 1984, Jantzen et al., 1992, Smith, 1993, Ebert et al., 1999). RR is round robin, MCC is Materials Characterization Center, SRS is Savannah River Site, and NBS is National Bureau of Standards. Plot shown FIO.

To visualize the variability in the analysis data collected by EPMA for each component, all analysis spots were first plotted versus the measured concentration for the seven standard glass compositions produced (Figure 3.7). The y-axis shows the analysis number, which sequentially increases, beginning with bar 1, spot 1 and ending with bar 12, spot 9 for each glass. Glasses are all uniquely colored. This figure shows there are shifts in the variability that are linked to row number.

In similar fashion, the analyzed concentrations, by EPMA and ICP, were plotted (Figure 3.8 through Figure 3.14) vertically by row number to visualize the differences in the measured values between techniques relative to the as-batched values (vertical lines). The EPMA data in these plots is colored by bar number and the ICP is colored pink.

Next, to better visualize where the variability occurs, the data was plotted as mass fraction versus the coupon position within each bar and bar position within each standard glass for each glass standard (Figure 3.15 through Figure 3.21). These plots show that the shift generally occurs at the bar-to-bar level. This shift at the bar-to-bar level was mostly random, meaning the shift occurred randomly anywhere between the five bars analyzed. In some cases, such as for Al_2O_3 measurements from glasses LSG19-01, -03, and -07, the shift in measured concentration of a component happened to appear more systematic from the first to last bar. Calculations were made using component analysis to determine what fraction of the variance in the data was linked to bar-to-bar versus coupon-to-coupon location in a bar versus random error. The percent variance due to each component analyzed is plotted for each glass composition in Figure 3.22. The contribution to variance linked to the bar-to-bar variable fluctuates widely among the glasses but was mostly present in the major components: Al_2O_3, Na_2O, SiO_2, B_2O_3, and CaO. SnO_2 and Fe_2O_3 were also examined, but the variability of those two components was mainly due to error.

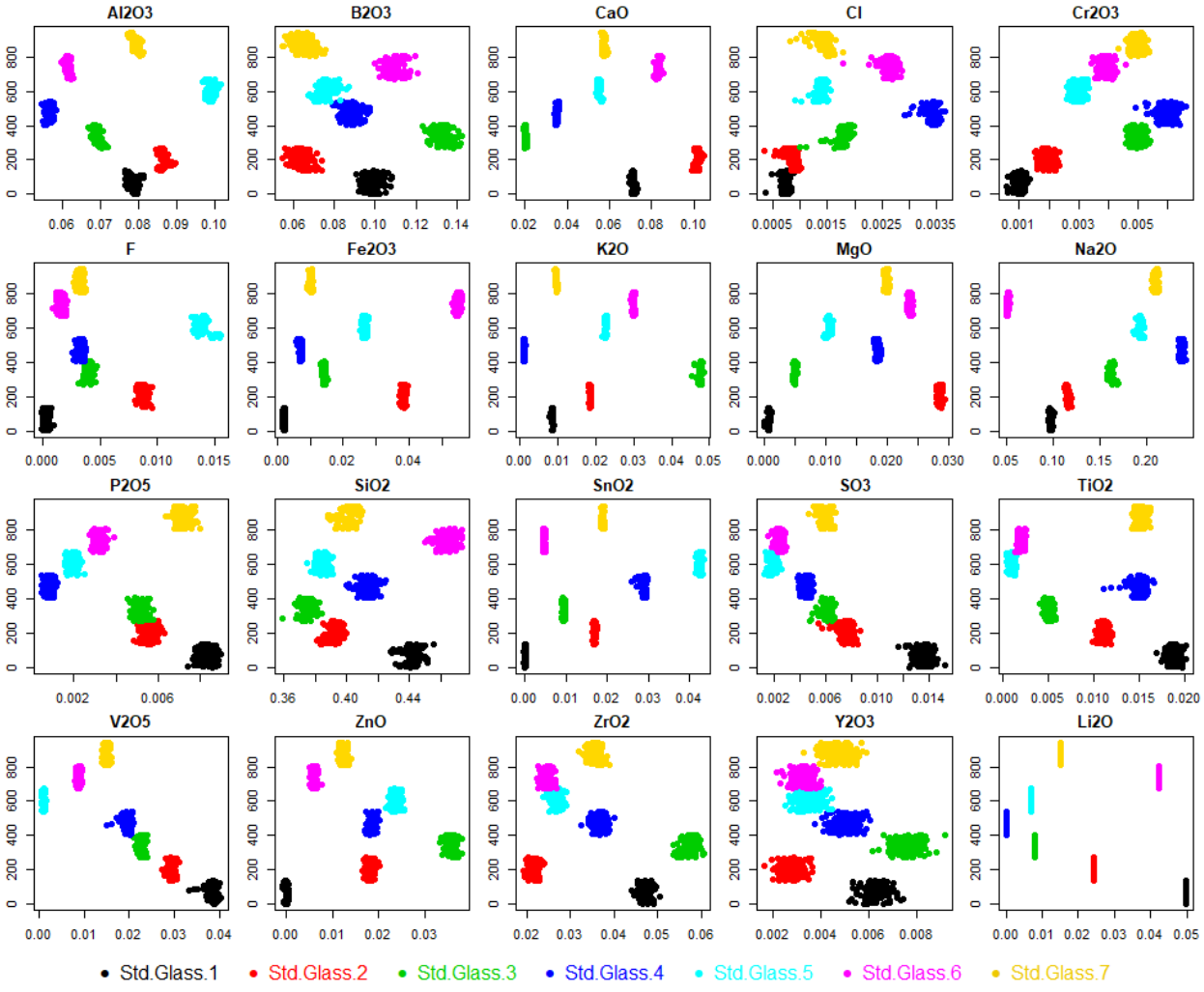

Figure 3.7. All analysis points plotted by row number on the y-axis versus EPMA-measured concentration (mass fraction) on the x-axis. Row numbers were sequentially increased by analysis spot and bar number analyzed. Nominally 9 analysis spots per coupon, 3 coupons per bar, 12 bars per glass, leading to 135 spots/rows per glass.

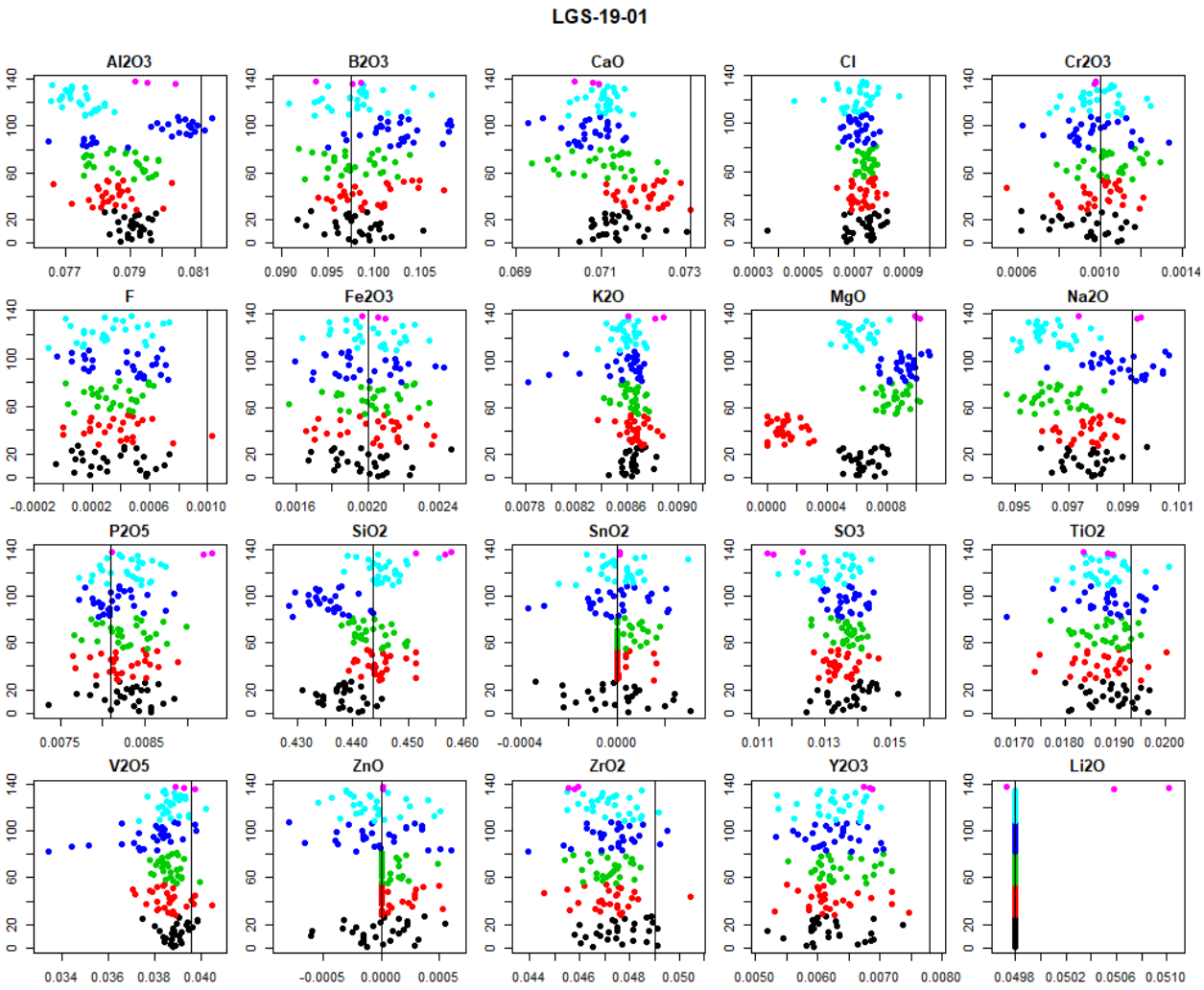

Figure 3.8. Sequential spot analysis of EPMA data plotted versus measured concentration (mass fraction) for LGS19-01 glass with each bar uniquely colored: bar 1 (black), bar 4 (red), bar 6 (green), bar 8 (blue), and bar 11 (cyan). ICP measurements of bulk glass in triplicate are shown in pink. The black line is the batched concentration.

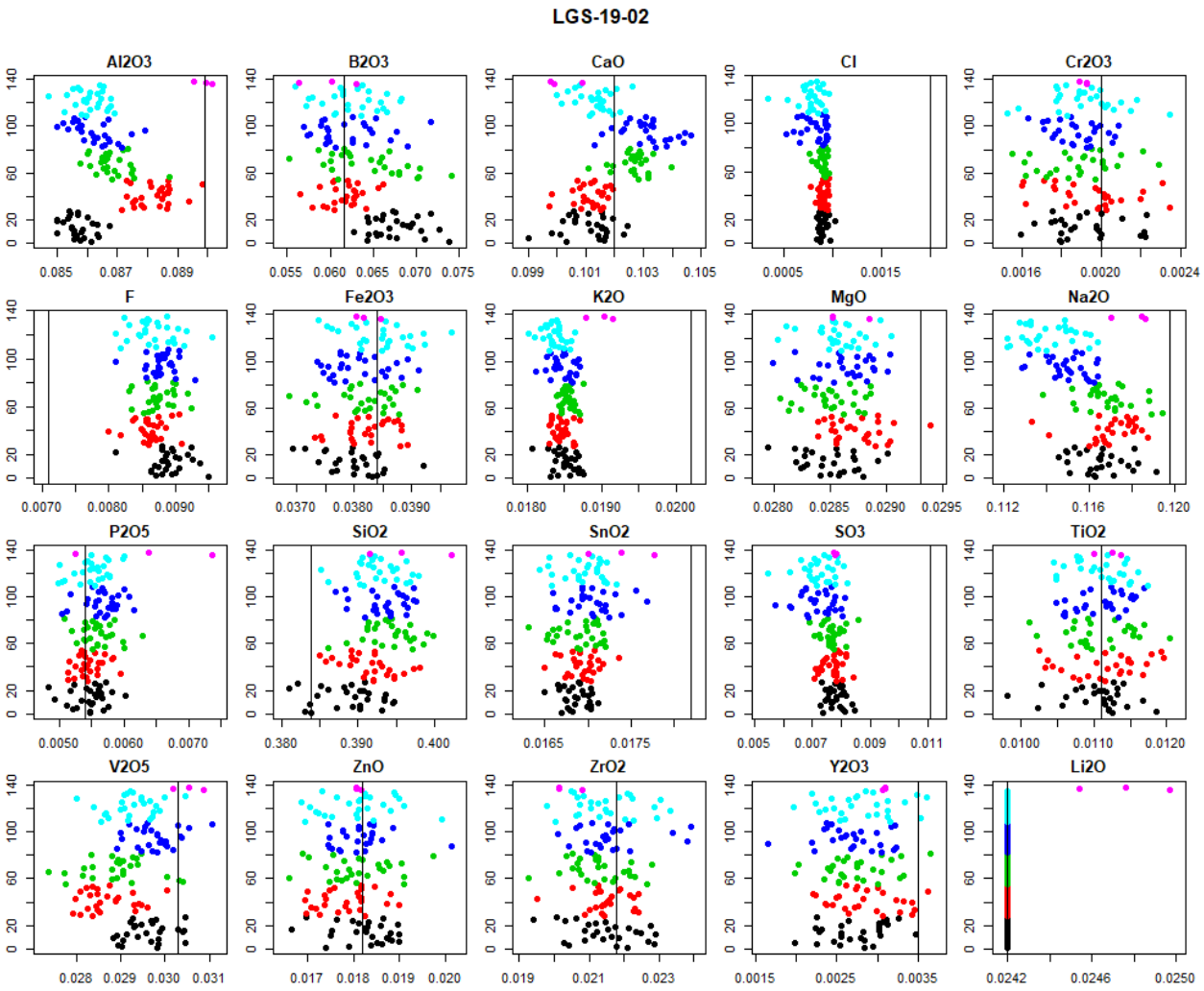

Figure 3.9. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-02 glass, with each bar uniquely colored: bar 1(black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink.

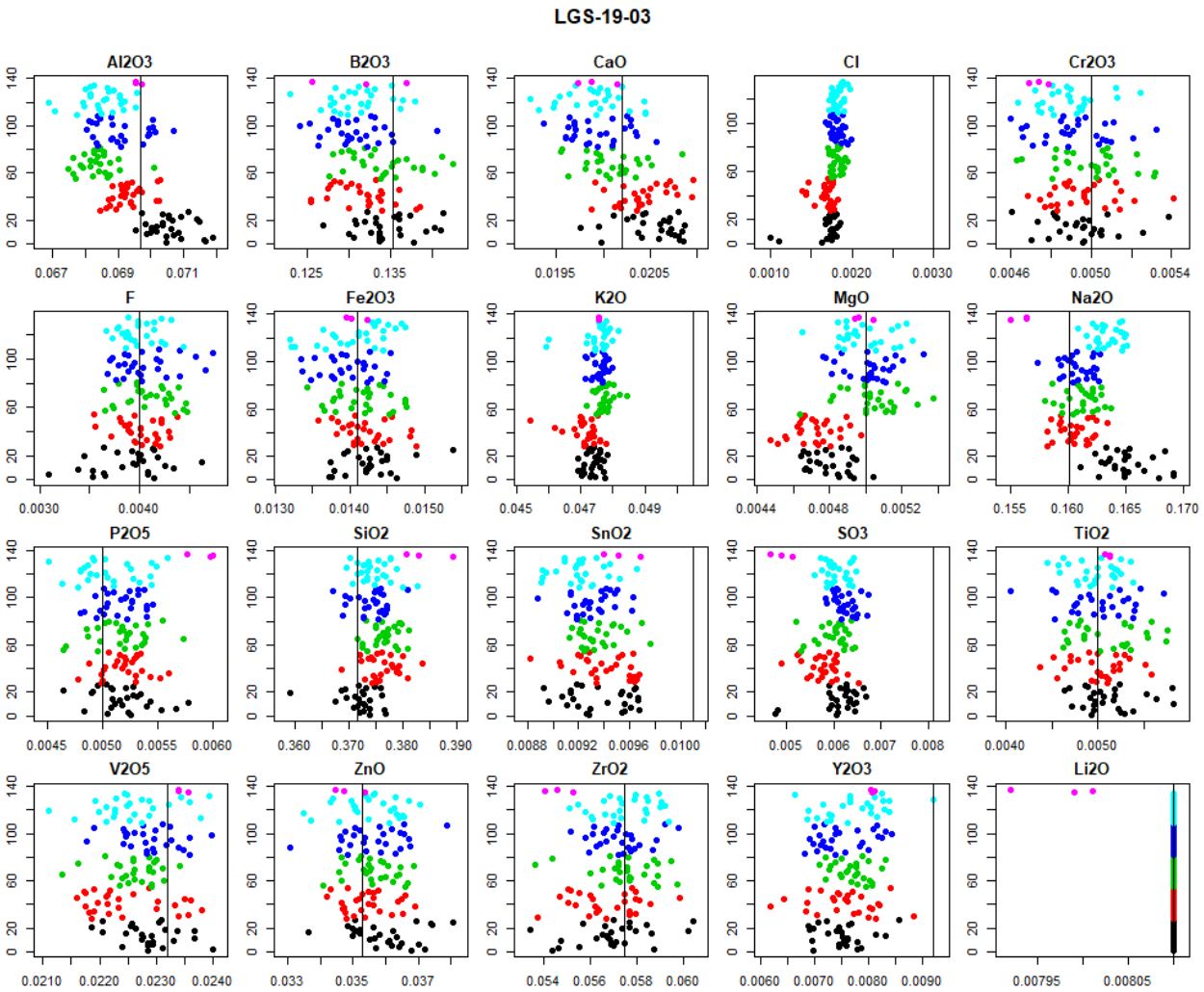

Figure 3.10. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-03 glass, with each bar uniquely colored: bar 1 (black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink.

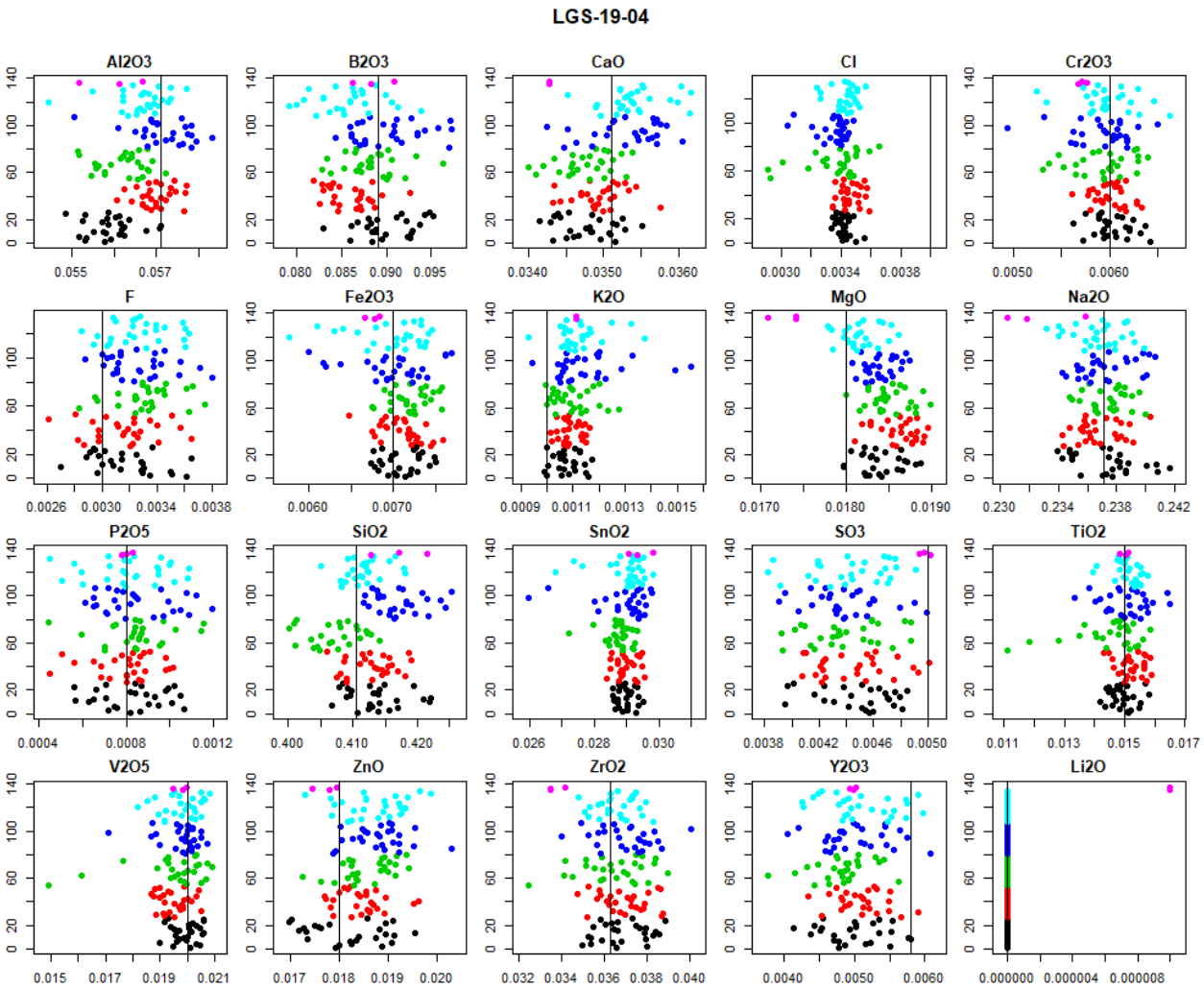

Figure 3.11. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-04 glass, with each bar uniquely colored: bar 1 (black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink

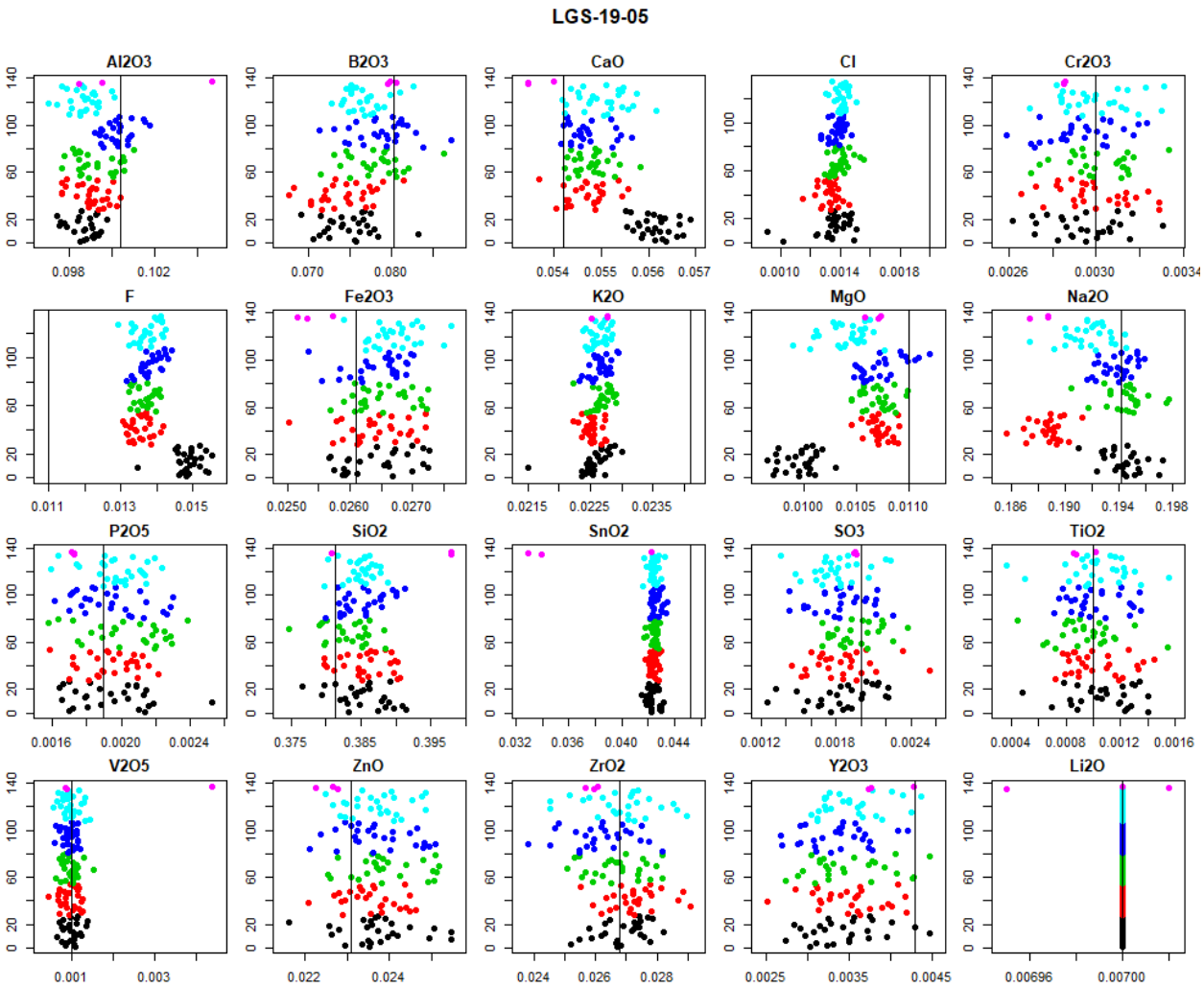

Figure 3.12. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-05 glass, with each bar uniquely colored: bar 1 (black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink.

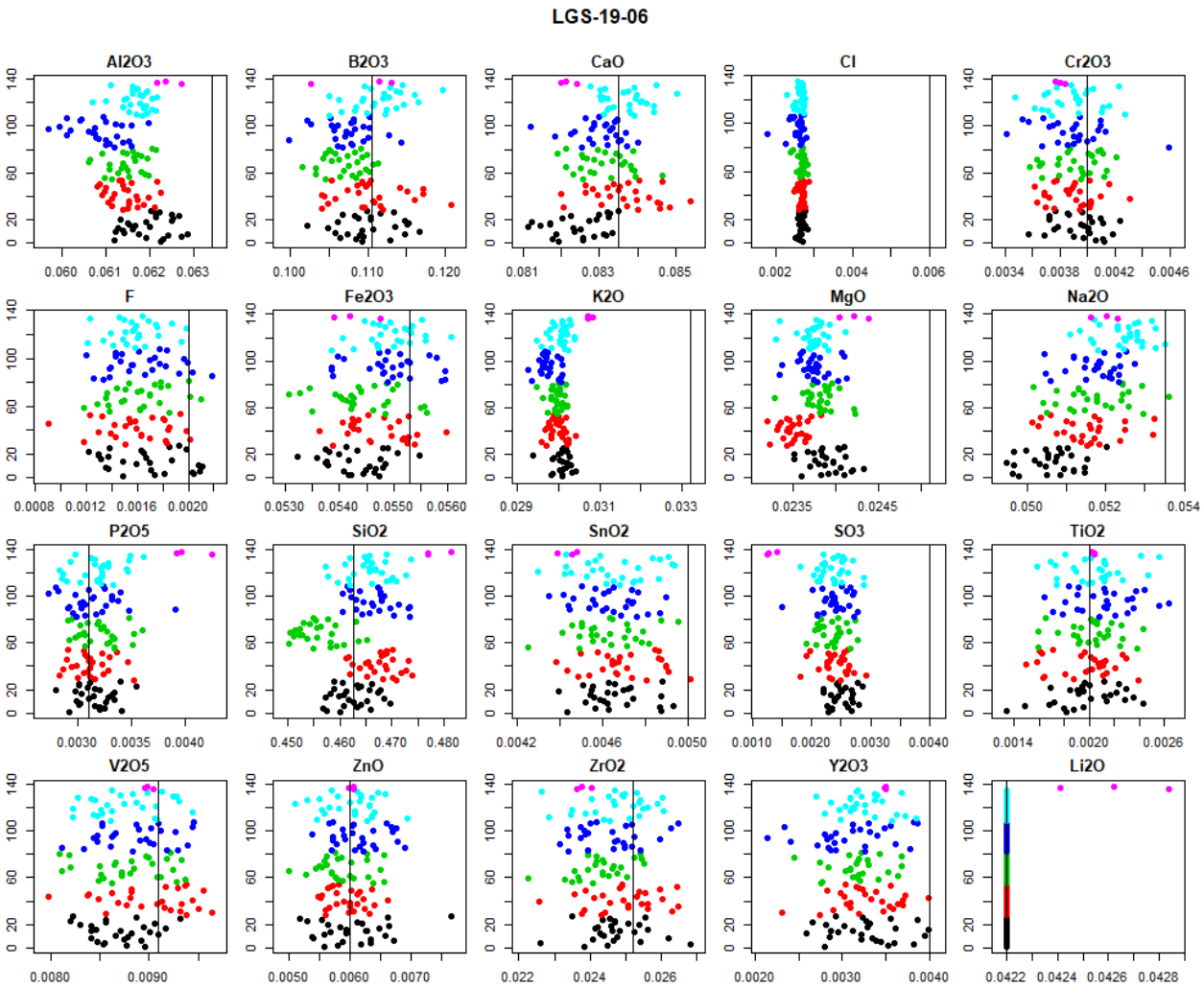

Figure 3.13. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-06 glass, with each bar uniquely colored: bar 1 (black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink.

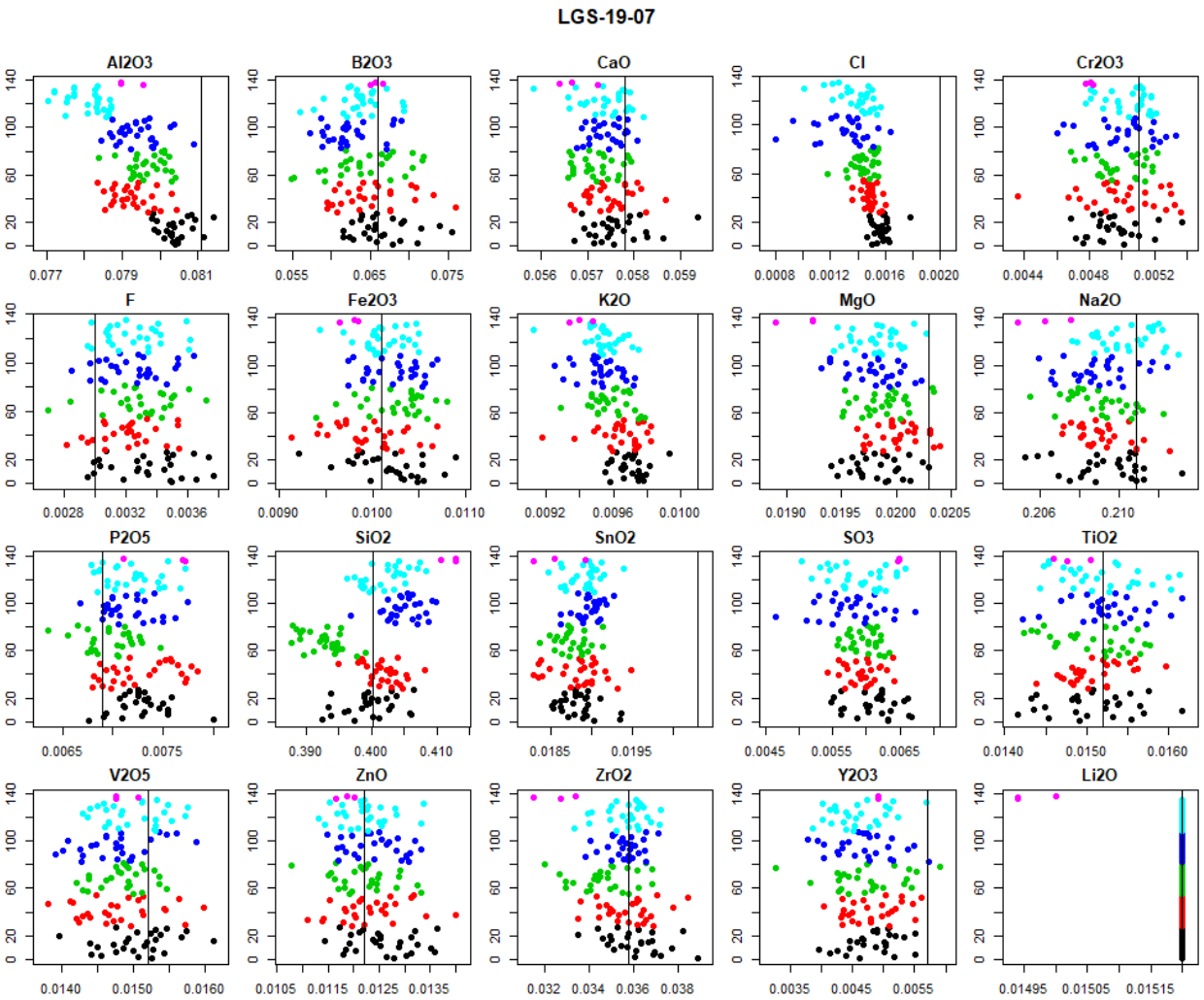

Figure 3.14. Sequential spot analysis by EPMA plotted versus measured concentration (mass fraction) for LGS19-07 glass, with each bar uniquely colored: bar 1(black), bar 4 (red), bar 7 (green), bar 10 (blue), and bar 12 (cyan). ICP measurements of bulk glass in triplicate are shown in pink.

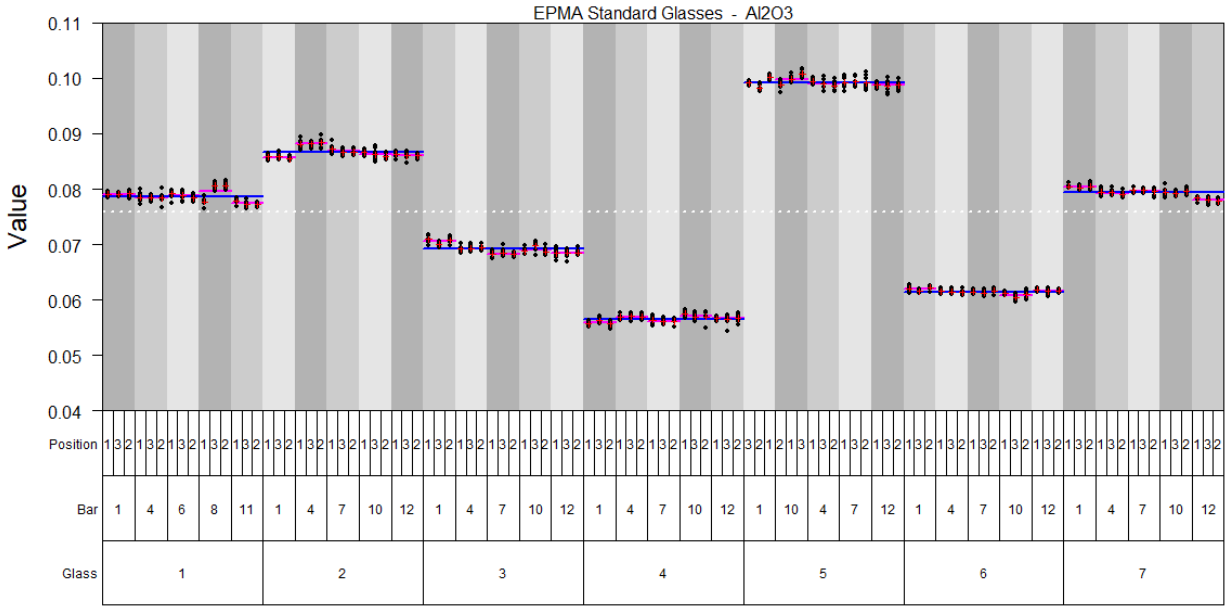

Figure 3.15. Al_2O_3 mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

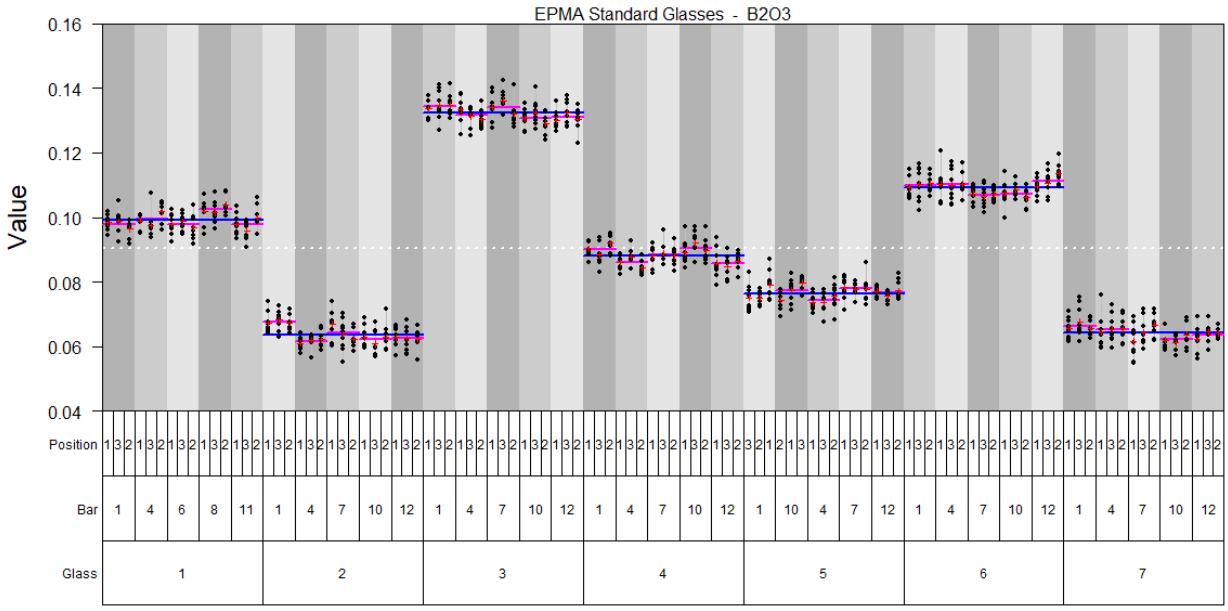

Figure 3.16. B_2O_3 mass fraction measured by EPMA versus coupon position(1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

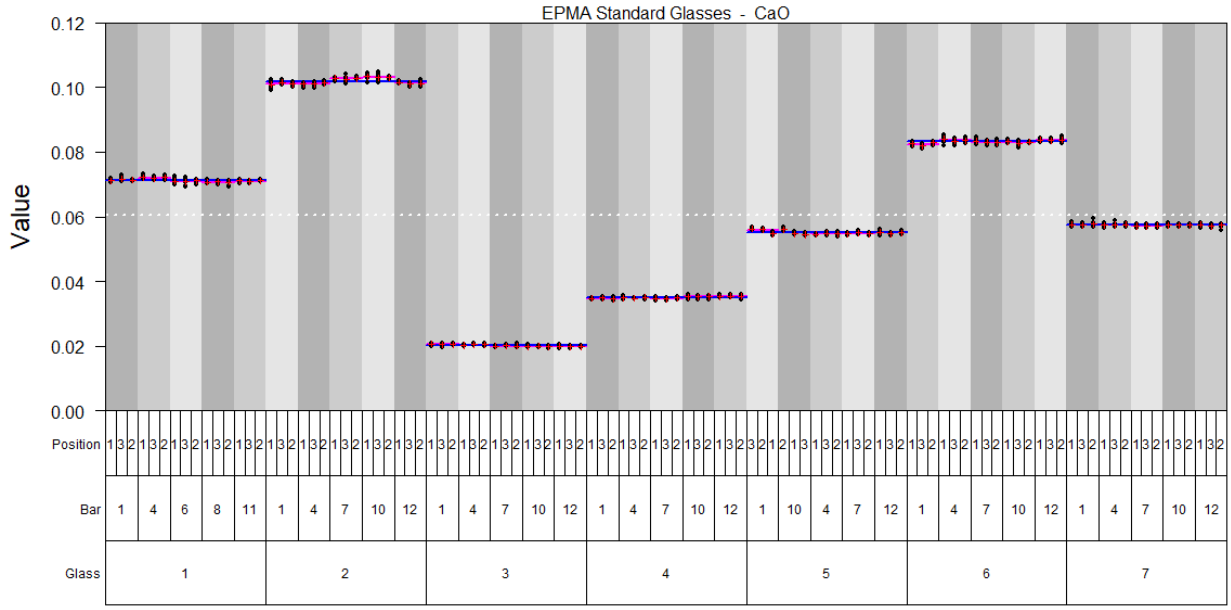

Figure 3.17. CaO mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

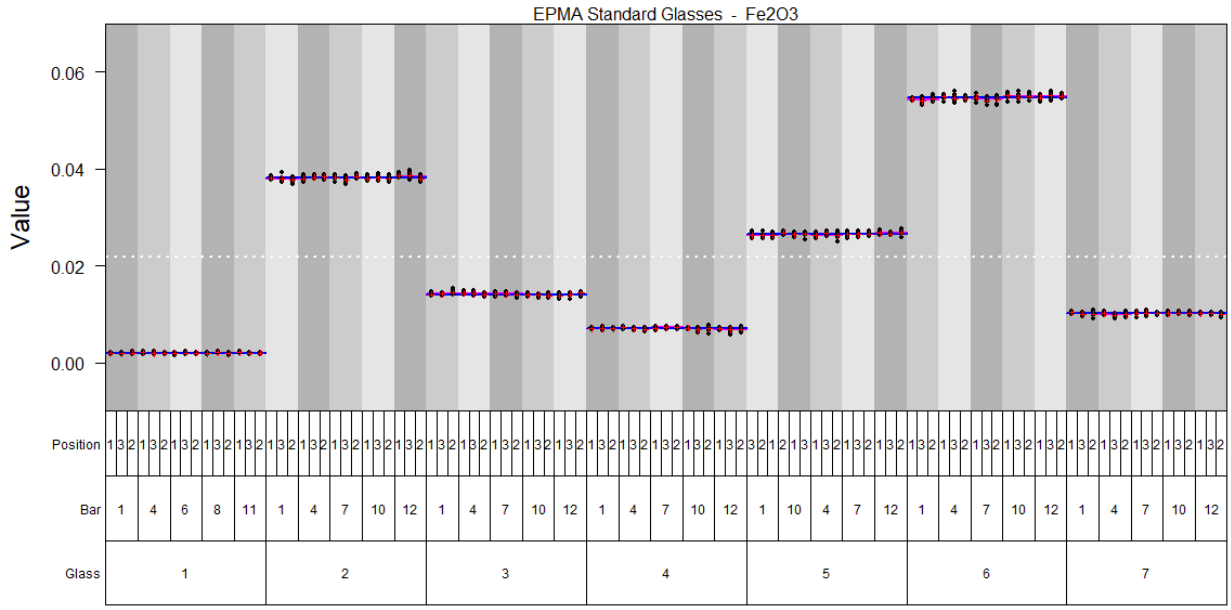

Figure 3.18. Fe_2O_3 mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

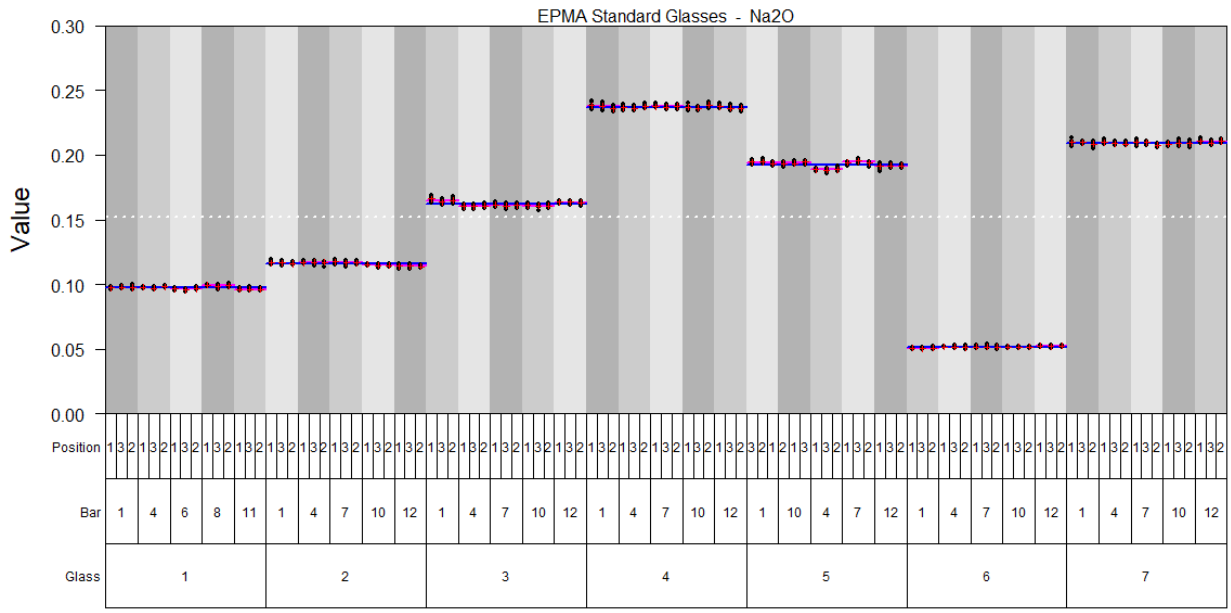

Figure 3.19. Na_2O mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

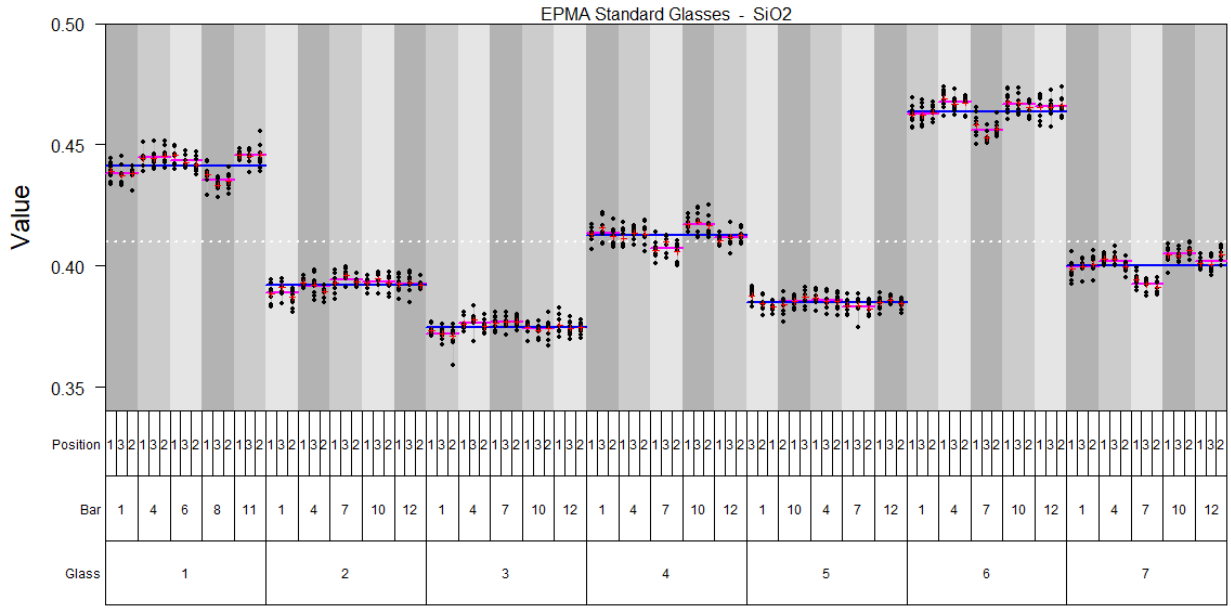

Figure 3.20. SiO_2 mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

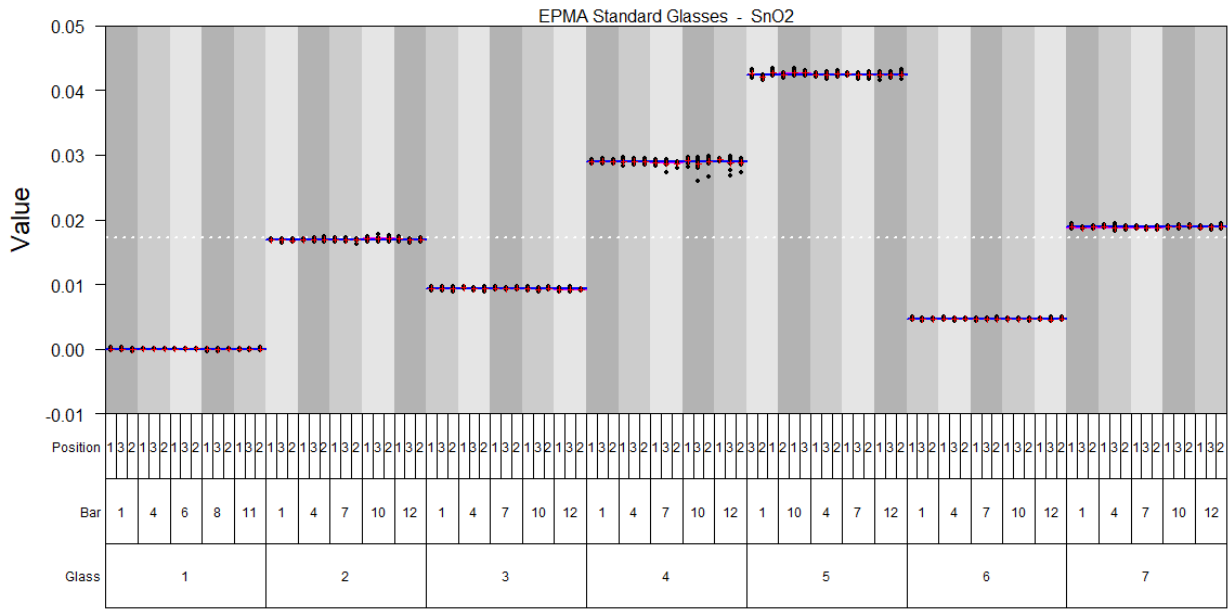

Figure 3.21. SnO_2 mass fraction measured by EPMA versus coupon position (1 = end 1, 2 = end 2, 3 = middle), bar number for each glass standard. Individual data points shown as black dots, means of analysis denoted at each level of analysis: red cross for coupon, pink line for bar, and blue line for each glass. Glass ID shortened from LGS-19-0X to the last digit for space.

Figure 3.22. Percent of variance in EPMA measurement values attributed to bar-to-bar (red), coupon-to-coupon position in bar (black), or random error spot-to-spot (gold), calculated by component analysis of the variance. Glass ID simplified due to space limits from LSG19-01 to -07 down to last digit of the name. Plot shown FIO.

4.0 Summary and Conclusions

Seven LAW standard glasses were fabricated and characterized to be used by the WTP as traceable standards for analytical instrument calibration and method verification during measurement of LAW glass compositions. The glasses were formulated to represent the compositions of LAW glass that are anticipated be generated by the WTP. Large (3-kg) batches were made using an induction-heated tilt-pour furnace to provide sufficient material for verifying composition and homogeneity of the glass standards and for use as standards.

To verify that the compositions of these standard glasses were on target with the batched compositions after melting, samples from each of the seven compositions were measured using EPMA, IC, and ICP-MS. Glasses were systematically analyzed by EPMA to determine the variability across each batch by analyzing 5 of the 12 glass bars produced. Within each bar, three coupons were taken, and nine spots were analyzed on each coupon. Percent differences and RSDs were calculated to evaluate the accuracy and homogeneity of the samples. These values decrease with increasing component concentrations. IC/ICP measurements also showed decreasing RSDs with increasing concentrations. This same trend was reported in previously developed waste glass standards. In comparison to EPMA, variability for components at low concentrations was slightly higher using IC/ICP measurements. Values for Li were exclusively obtained by ICP and were close to those of batched compositions. Components that generally occurred in small concentrations in the glass matrix, and as a result had higher error (>10%) and variability, included Cl, F, SO_3, and Y_2O_3.

In most cases, the same components that occurred in minor fractions in the glass standards were also components that can volatilize during melting. Therefore, relative differences from the batched compositions or measurement variability may have also been influenced by volatile behavior. Two glasses where the most volatilization was observed, LGS-19-02 and LGS-19-06, also had higher percent differences and SDs in volatile components, such as Cl and F, compared to other glasses. Therefore, it is possible that volatilization contributed to differences in glass compositions compared to batched compositions.

Statistical plots were made from EPMA data to evaluate homogeneity within single bars and whole compositions for each of the seven glasses. For some isolated components, such as Al_2O_3 in glass LGS-19-03, there appeared to be small systematic trends across bars. Furthermore, for major components such as Al, Si, and Na, it was determined that a high proportion of the variability in glass samples occurs at the bar-to-bar level. However, these trends occur at a range in concentrations (approximately 0.2 wt% difference in Al_2O_3 in LGS-19-03) that make them inconclusive based on the sensitivity of the EPMA. Since the EPMA instrument was calibrated when each bar was analyzed, the variability caused by repeated calibrations is correlated and likely contributed to the observed variability from bar-to-bar. This bar-to-bar variability occurred randomly and doesn't show a clear trend with bar order.

Based on these analyses, we recommend using the overall mean analyzed composition for each glass and the overall (pooled) variance for each glass by EPMA and ICP (given in Table 4.1) when using these standards to verify that a calibrated instrument/procedure is performing within specification or calibrating instruments.

Table 4.1. Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-01 and -02 glass standards.

		LGS19-01						LGS19-02					
Oxide	SwRI Method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	8.124	0.008	7.96	0.047	7.87	0.114	8.991	0.009	8.99	0.029	8.66	0.106
B_2O_3	ICP	9.748	0.010	9.66	0.256	9.91	0.372	6.162	0.006	5.99	0.340	6.37	0.398
CaO	ICP	7.311	0.007	7.08	0.035	7.13	0.072	10.203	0.010	10.02	0.061	10.19	0.109
Cl	IC	0.102	0.000	UD	NA	0.07	0.006	0.202	0.000	UD	NA	0.09	0.010
Cr_2O_3	ICP	0.102	0.000	0.10	0.000	0.10	0.014	0.202	0.000	0.19	0.002	0.19	0.017
F	IC	0.102	0.000	0.10	0.001	0.04	0.021	0.707	0.001	0.83	0.018	0.88	0.028
Fe_2O_3	ICP	0.203	0.000	0.21	0.010	0.20	0.019	3.839	0.004	3.82	0.022	3.82	0.052
K_2O	ICP	0.914	0.001	0.88	0.014	0.86	0.014	2.02	0.002	1.90	0.018	1.85	0.015
Li_2O	ICP	4.976	0.005	5.04	0.065	NM	NA	2.425	0.002	2.48	0.022	NM	NA
MgO	ICP	0.102	0.000	0.10	0.001	0.06	0.029	2.93	0.003	2.86	0.019	2.86	0.025
Na_2O	ICP	9.932	0.010	9.88	0.125	9.74	0.132	11.975	0.012	11.8	0.089	11.59	0.164
P_2O_5	ICP	0.811	0.001	0.88	0.062	0.83	0.029	0.538	0.001	0.63	0.106	0.55	0.027
SiO_2	ICP	44.375	0.044	45.71	0.539	44.14	0.529	38.389	0.038	39.65	0.539	39.21	0.377
SnO_2	ICP	0.000	0.000	0.00	0.000	0.00	0.011	1.818	0.002	1.74	0.038	1.69	0.023
SO_3	ICP	1.625	0.002	1.27	0.055	1.36	0.057	1.111	0.001	0.72	0.005	0.75	0.052
SO_3	IC	1.625	0.002	1.18	0.039	NA	NA	1.111	0.001	0.78	0.014	NA	NA
TiO_2	ICP	1.929	0.002	1.87	0.029	1.88	0.053	1.111	0.001	1.12	0.019	1.11	0.042
V_2O_5	ICP	3.960	0.004	3.93	0.036	3.85	0.094	3.031	0.003	3.05	0.036	2.92	0.070
Y_2O_3	ICP	0.782	0.001	0.68	0.006	0.63	0.045	0.347	0.000	0.31	0.001	0.28	0.039
ZnO	ICP	0.000	0.000	0.00	0.000	0.00	0.025	1.818	0.002	1.81	0.007	1.81	0.066
ZrO_2	ICP	4.905	0.005	4.58	0.024	4.73	0.101	2.178	0.002	2.04	0.039	2.15	0.084
Sum		100.00		99.8[(b)]		98.4[(c)]		100.00		100.0[(b)]		99.4[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table 4.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-01 and -02 glass standards.

		LGS19-03						LGS19-04					
Oxide	SwRI method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	6.966	0.007	6.96	0.011	6.92	0.102	5.708	0.006	5.60	0.076	5.66	0.074
B_2O_3	ICP	13.527	0.014	13.15	0.565	13.24	0.384	8.913	0.009	8.84	0.226	8.81	0.372
CaO	ICP	2.019	0.002	1.99	0.021	2.02	0.039	3.505	0.004	3.43	0.000	3.50	0.046
Cl	IC	0.303	0.000	0.03	0.007	0.18	0.013	0.401	0.000	0.14	0.018	0.34	0.011
Cr_2O_3	ICP	0.505	0.001	0.47	0.005	0.49	0.017	0.601	0.001	0.57	0.005	0.60	0.026
F	IC	0.404	0.000	0.45	0.015	0.40	0.027	0.300	0.000	0.35	0.013	0.32	0.023
Fe_2O_3	ICP	1.413	0.001	1.41	0.015	1.41	0.038	0.701	0.001	0.68	0.009	0.71	0.034
K_2O	ICP	5.047	0.005	4.76	0.000	4.75	0.042	0.100	0.000	0.11	0.000	0.11	0.009
Li_2O	ICP	0.808	0.001	0.80	0.005	NM	NA	0.000	0.000	0.00	0.000	NM	NA
MgO	ICP	0.505	0.001	0.50	0.005	0.49	0.019	1.803	0.002	1.73	0.019	1.84	0.027
Na_2O	ICP	16.015	0.016	15.59	0.078	16.22	0.230	23.713	0.024	23.28	0.280	23.71	0.173
P_2O_5	ICP	0.499	0.001	0.59	0.013	0.52	0.023	0.076	0.000	0.08	0.003	0.08	0.015
SiO_2	ICP	37.15	0.037	38.43	0.445	37.48	0.350	41.06	0.041	41.71	0.428	41.25	0.488
SnO_2	ICP	1.009	0.001	0.95	0.015	0.93	0.020	3.104	0.003	2.94	0.039	2.89	0.057
SO_3	ICP	0.808	0.001	0.64	0.024	0.60	0.036	0.501	0.001	0.45	0.004	0.44	0.027
SO_3	IC	0.808	0.001	0.49	0.012	NA	NA	0.501	0.001	0.50	0.008	NA	NA
TiO_2	ICP	0.505	0.001	0.51	0.003	0.50	0.032	1.502	0.002	1.50	0.014	1.50	0.072
V_2O_5	ICP	2.322	0.002	2.34	0.010	2.27	0.058	2.003	0.002	1.98	0.027	1.97	0.082
Y_2O_3	ICP	0.916	0.001	0.81	0.004	0.76	0.050	0.578	0.001	0.50	0.005	0.50	0.044
ZnO	ICP	3.533	0.004	3.49	0.045	3.57	0.088	1.803	0.002	1.77	0.026	1.85	0.063
ZrO_2	ICP	5.747	0.006	5.46	0.061	5.72	0.138	3.628	0.004	3.37	0.039	3.68	0.122
Sum		100.00		99.2[(b)]		99.3[(c)]		100.00		99.1[(b)]		99.8[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table 4.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-01 and -02 glass standards.

		LGS19 -05						LGS19-06					
Oxide	SwRI method	Batched	Balance Error	SwRI	SD	EPMA	SD	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	10.035	0.010	10.09	0.332	9.92	0.096	6.337	0.006	6.24	0.029	6.15	0.057
B_2O_3	ICP	8.045	0.008	8.00	0.049	7.65	0.346	11.064	0.011	10.91	0.555	10.92	0.381
CaO	ICP	5.419	0.005	5.36	0.032	5.51	0.069	8.348	0.008	8.22	0.021	8.32	0.082
Cl	IC	0.201	0.000	0.02[(a)]	NA	0.14	0.008	0.603	0.001	0.09	0.016	0.26	0.012
Cr_2O_3	ICP	0.301	0.000	0.29	0.001	0.30	0.016	0.402	0.000	0.38	0.004	0.39	0.018
F	IC	1.104	0.001	1.31	0.681	1.39	0.059	0.201	0.000	0.22	0.003	0.16	0.025
Fe_2O_3	ICP	2.609	0.003	2.54	0.030	2.65	0.047	5.532	0.006	5.43	0.044	5.47	0.063
K_2O	ICP	2.408	0.002	2.27	0.014	2.26	0.018	3.319	0.003	3.08	0.007	3.00	0.023
Li_2O	ICP	0.702	0.001	0.70	0.004	NA	NA	4.224	0.004	4.26	0.022	NA	NA
MgO	ICP	1.104	0.001	1.07	0.008	*1.05*	*0.034*	2.515	0.003	2.42	0.017	*2.37*	*0.022*
Na_2O	ICP	19.415	0.019	18.83	0.078	19.26	0.258	5.347	0.005	5.20	0.034	5.16	0.092
P_2O_5	ICP	0.191	0.000	0.17	0.001	0.20	0.019	0.306	0.000	0.40	0.017	0.32	0.020
SiO_2	ICP	38.132	0.038	39.22	0.988	38.49	0.320	46.268	0.046	47.85	0.247	46.37	0.571
SnO_2	ICP	4.516	0.005	3.64	0.516	4.25	0.037	0.503	0.001	0.44	0.005	0.47	0.015
SO_3	ICP	0.201	0.000	0.15	0.001	0.19	0.023	0.402	0.000	0.21	0.009	0.24	0.024
SO_3	IC	0.201	0.000	0.20	0.011	NA	NA	0.402	0.000	0.13	0.004	NA	NA
TiO_2	ICP	0.100	0.000	0.09	0.009	0.10	0.022	0.201	0.000	0.20	0.001	0.20	0.025
V_2O_5	ICP	0.100	0.000	0.20	0.202	0.10	0.020	0.905	0.001	0.90	0.005	0.88	0.038
Y_2O_3	ICP	0.428	0.000	0.39	0.032	0.35	0.042	0.401	0.000	0.35	0.001	0.32	0.036
ZnO	ICP	2.308	0.002	2.26	0.026	2.37	0.075	0.603	0.001	0.60	0.005	0.60	0.043
ZrO_2	ICP	2.683	0.003	2.59	0.020	2.68	0.100	2.516	0.003	2.38	0.020	2.46	0.088
Sum		100.00		99.2[(b)]		99.6[(c)]		100.00		99.7[(b)]		98.3[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

Table 4.1 (continued). Summary table of batched, analyzed mean compositions (SwRI and EPMA), and standard deviations (SD) of analyses for LGS19-01 and -02 glass standards.

	SwRI	LGS19-07					
Oxide	method	Batched	Balance Error	SwRI	SD	EPMA	SD
Al_2O_3	ICP	8.106	0.008	7.92	0.033	7.94	0.091
B_2O_3	ICP	6.586	0.007	6.58	0.081	6.44	0.400
CaO	ICP	5.775	0.006	5.68	0.043	5.74	0.050
Cl	IC	0.203	0.000	0.02[(a)]	NA	0.14	0.015
Cr_2O_3	ICP	0.507	0.001	0.48	0.002	0.50	0.018
F	IC	0.304	0.000	0.35	0.006	0.33	0.021
Fe_2O_3	ICP	1.013	0.001	0.98	0.011	1.01	0.034
K_2O	ICP	1.013	0.001	0.94	0.007	0.96	0.013
Li_2O	ICP	1.520	0.002	1.50	0.003	NM	NA
MgO	ICP	2.026	0.002	1.91	0.020	1.99	0.023
Na_2O	ICP	21.087	0.021	20.62	0.135	20.94	0.162
P_2O_5	ICP	0.694	0.001	0.75	0.035	0.72	0.030
SiO_2	ICP	40.022	0.040	41.22	0.124	40.03	0.518
SnO_2	ICP	2.026	0.002	1.86	0.032	1.89	0.023
SO_3	ICP	0.709	0.001	0.60	0.001	0.59	0.036
SO_3	IC	0.709	0.001	0.65	0.052	NA	NA
TiO_2	ICP	1.520	0.002	1.48	0.023	1.51	0.045
V_2O_5	ICP	1.520	0.002	1.49	0.018	1.49	0.046
Y_2O_3	ICP	0.571	0.001	0.49	0.000	0.47	0.047
ZnO	ICP	1.216	0.001	1.18	0.018	1.23	0.059
ZrO_2	ICP	3.583	0.004	3.25	0.096	3.56	0.118
Sum		100.00		99.3[(b)]		99.0[(c)]	

UD = below detection limit, NM = not measured, NA = not applicable, a) below quantification limit, b) sum doesn't include SO_3 by ICP, and c) sum includes batched Li_2O

5.0 References

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Appendix A – Statistical Analysis of EPMA Data

This appendix provides additional statistical plots for each standard glass and component analysis of variance for measurements made by electron probe microanalysis (EPMA). The plots and component analysis are arranged by increasing glass number identification. The plots were generated to show the variation in component distribution from bar-to-bar for a given glass (e.g., Figure A.1), and the variation in component distribution from coupon-to-coupon location within a bar was plotted (e.g., Figure A.2).

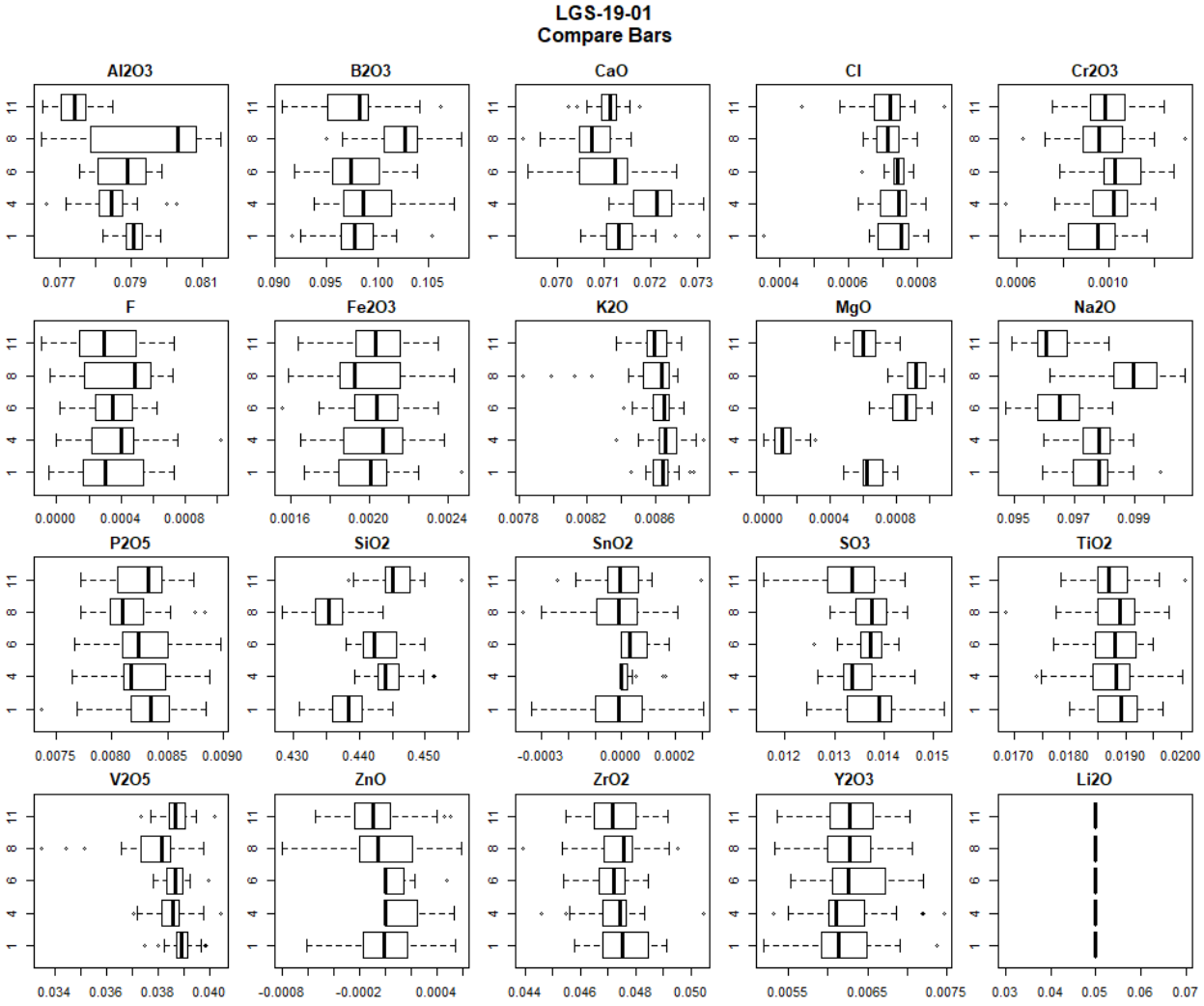

Figure A.1. Component distribution plots for LGS19-01 glass measured by EPMA, plotted by bar number on the y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

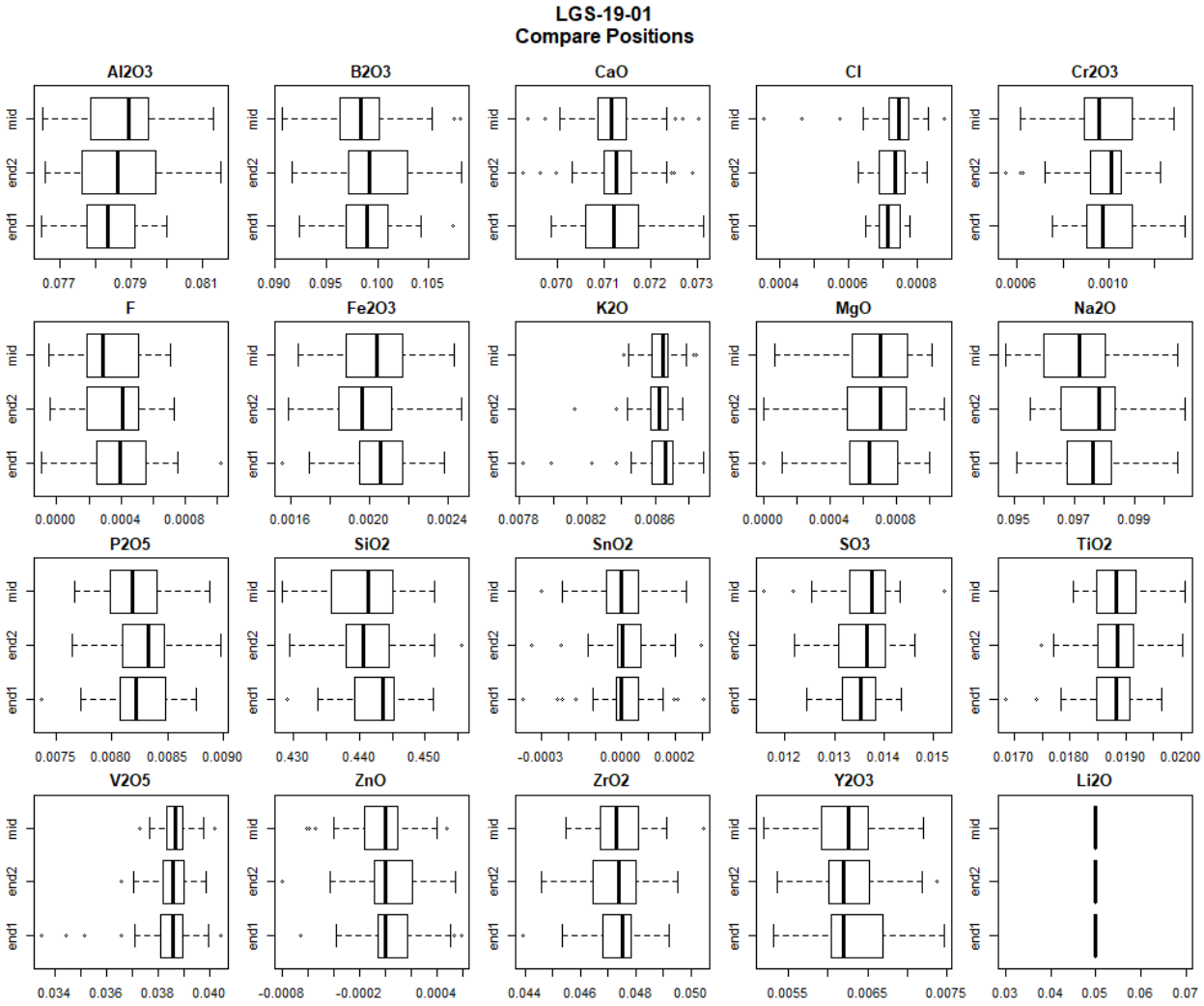

Figure A.2. Component distribution plots for LGS19-01 glass measured by EPMA, plotted by coupon location within bars (end1, middle and end2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-01] "Al2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	14.8973	0	100	0.0012	1.5241		
2 Bar	4	0.0001	0	0	33.4262	0.0007	0.8812
3 Bar:Position	10	0.0001	0	0	41.4449	0.0008	0.9812
4 error	120	0	0	0	25.1289	0.0006	0.764

Mean: 0.0787 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-01] "B2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	43.3203	0	100	0.0038	3.8483		
2 Bar	4	0.0004	0.0001	0	21.5697	0.0018	1.7873
3 Bar:Position	10	0.0002	0	0	9.5647	0.0012	1.1902
4 error	120	0.0012	0	0	68.8656	0.0032	3.1935

Mean: 0.0991 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-01] "CaO"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	18.8876	0	100	0.0008	1.0611		
2 Bar	4	0	0	0	42.8812	0.0005	0.6948
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	120	0	0	0	57.1188	0.0006	0.8019

Mean: 0.0713 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

[LGS-19-01] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	116.9995	0	100	0.0002	9.6797		
2 Bar	4	0	0	0*	0*	0*	0*
3 Bar:Position	10	0	0	0	12.1386	0.0001	3.3724
4 error	120	0	0	0	87.8614	0.0002	9.0732

Mean: 0.002 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-01] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	10.5863	0	100	0.0014	1.4385		
2 Bar	4	0.0001	0	0	58.1546	0.0011	1.097
3 Bar:Position	10	0	0	0	4.5764	0.0003	0.3077
4 error	120	0.0001	0	0	37.269	0.0009	0.8782

Mean: 0.0974 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-01] "SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	10.1244	0	100	0.0056	1.2757		
2 Bar	4	0.0021	0.0005	0	60.3954	0.0044	0.9914
3 Bar:Position	10	0.0002	0	0	2.1679	0.0008	0.1878
4 error	120	0.0014	0	0	37.4368	0.0034	0.7805

Mean: 0.4414 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-01] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	130.7406	0	100	0.0001	950.4152		
2	Bar	4	0	0	0	2.044	0	135.8805
3	Bar:Position	10	0	0	0*	0*	0*	0*
4	error	120	0	0	0	97.956	0.0001	940.6516

Mean: 0 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

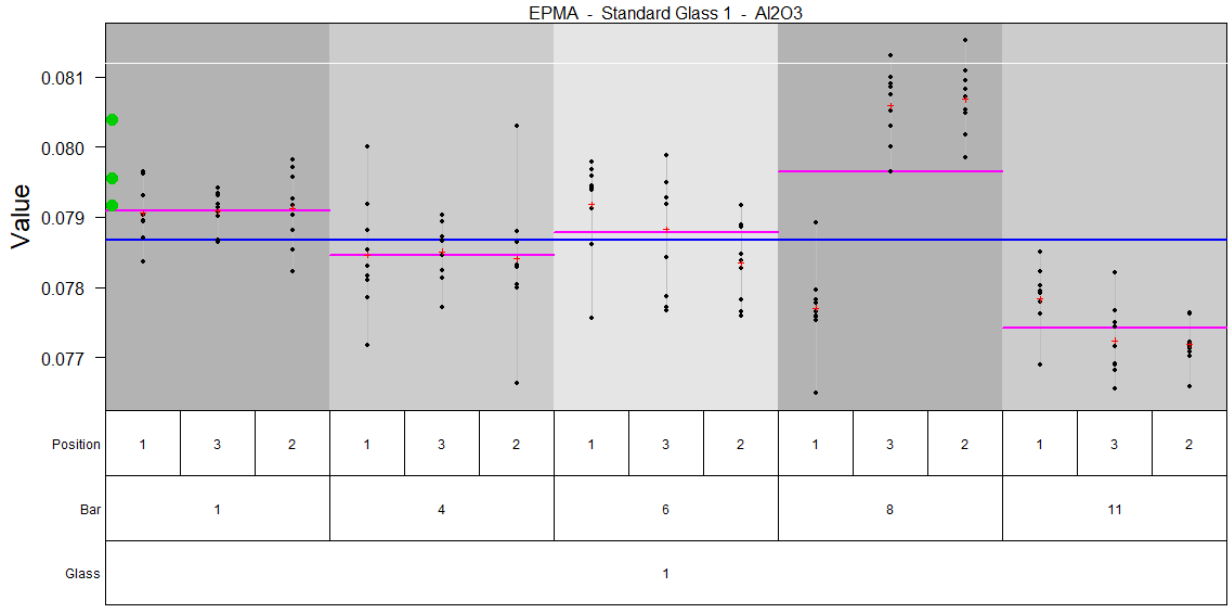

Figure A.3. Measured Al_2O_3 concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points on the far left are inductively coupled plasma (ICP). Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white line is the batched concentration.

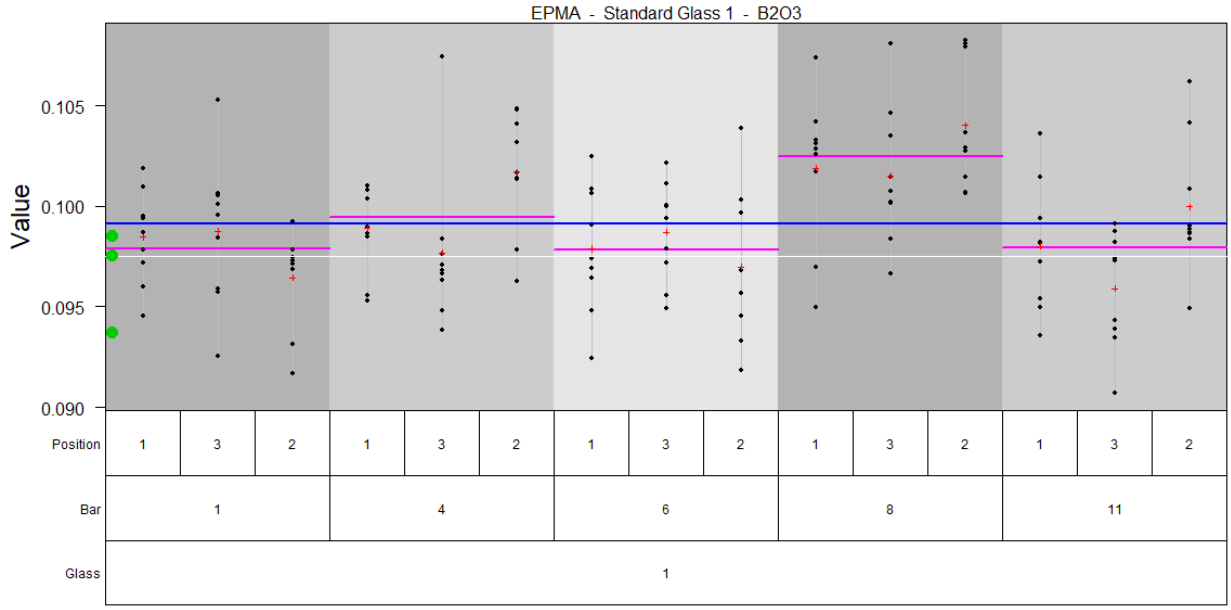

Figure A.4. Measured B_2O_3 concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

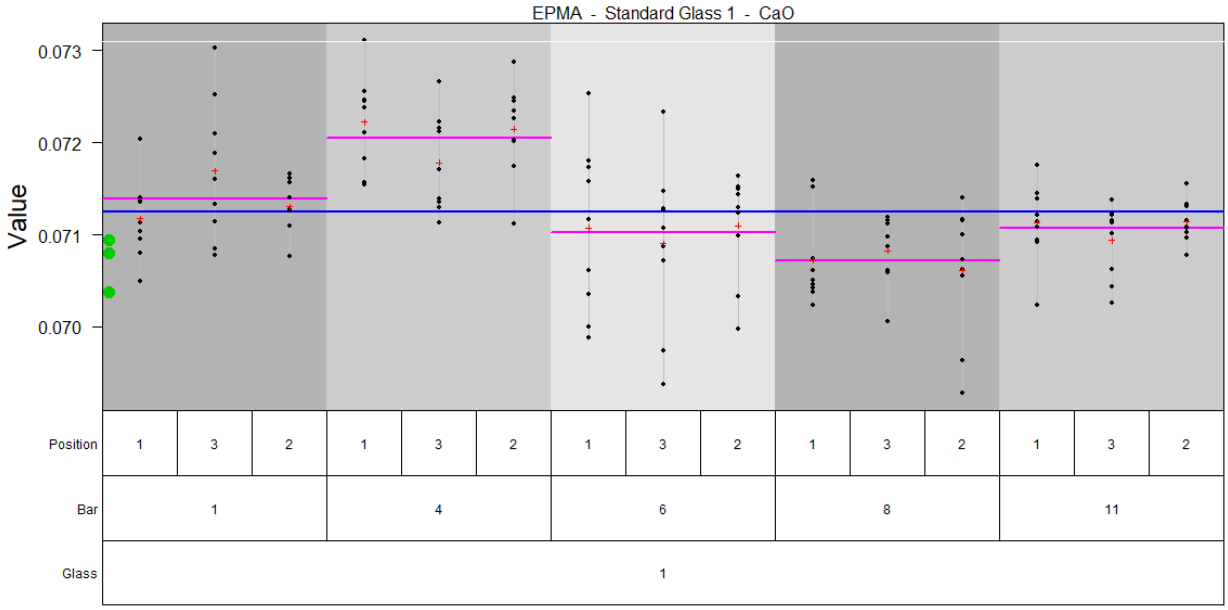

Figure A.5. Measured CaO concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where position 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

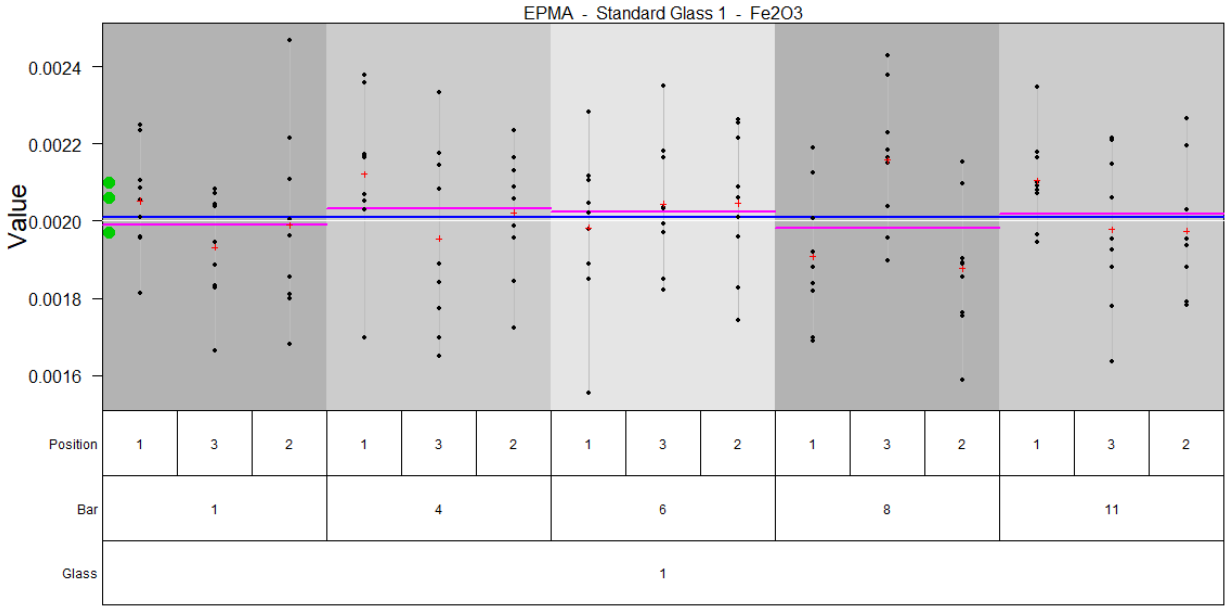

Figure A.6. Measured Fe_2O_3 concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

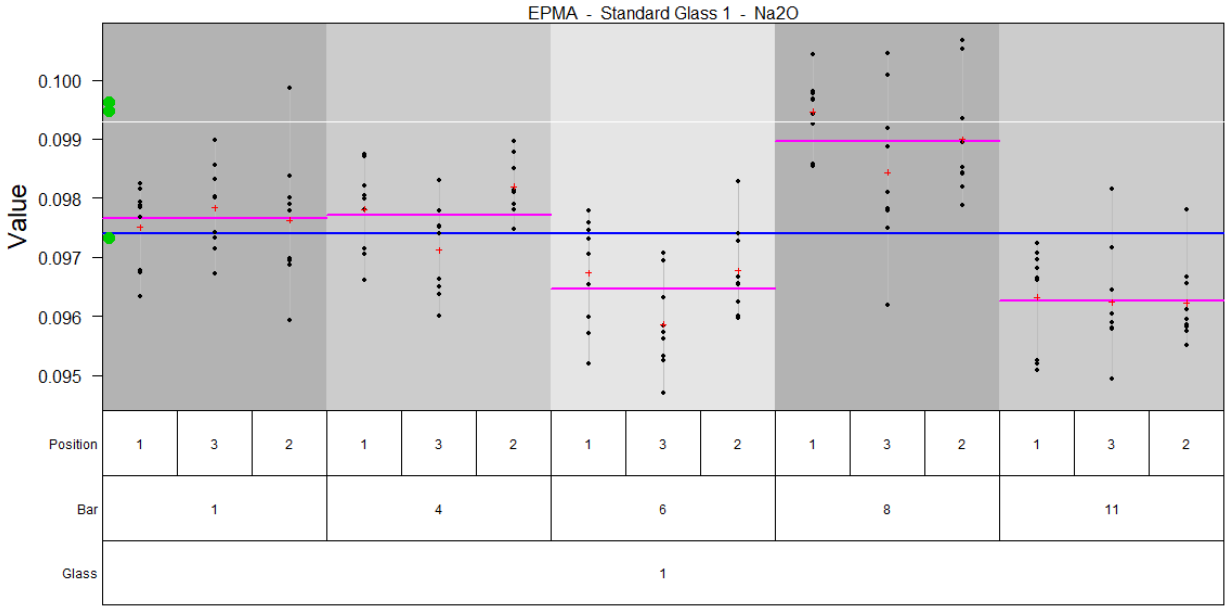

Figure A.7. Measured Na_2O concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

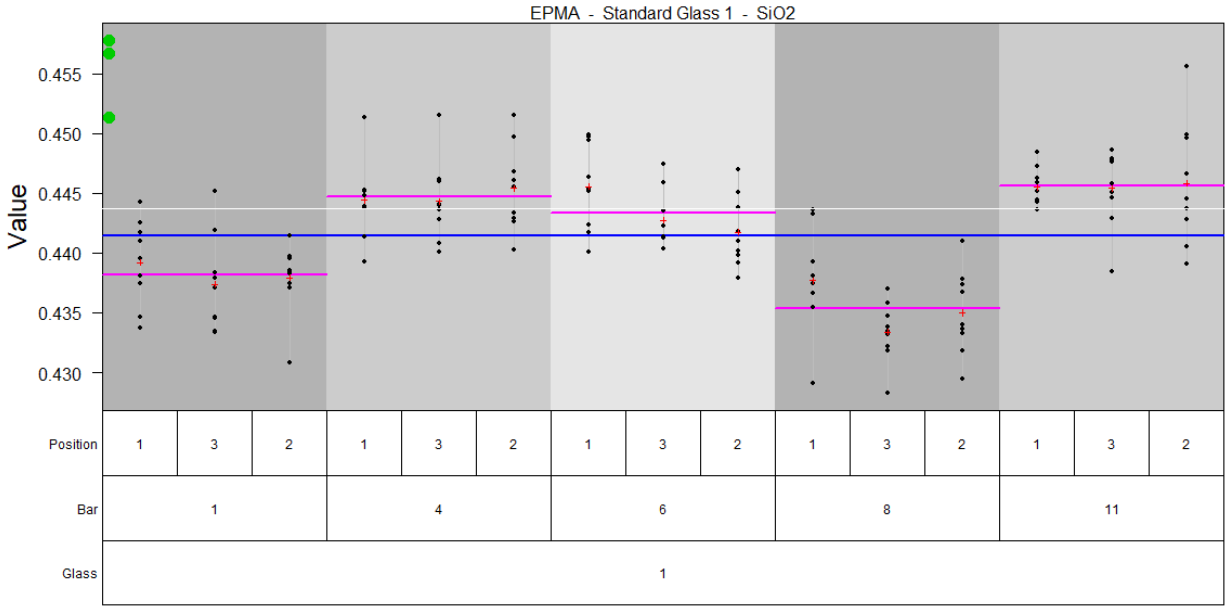

Figure A.8. Measured SiO_2 concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

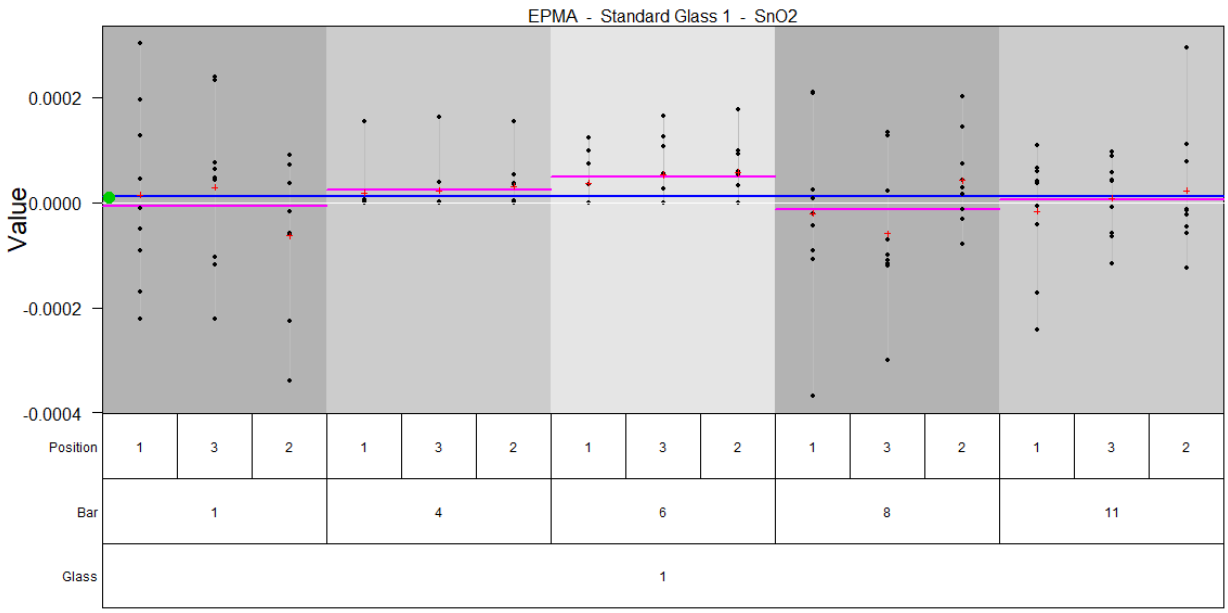

Figure A.9. Measured SnO_2 concentrations of LGS-19-01 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

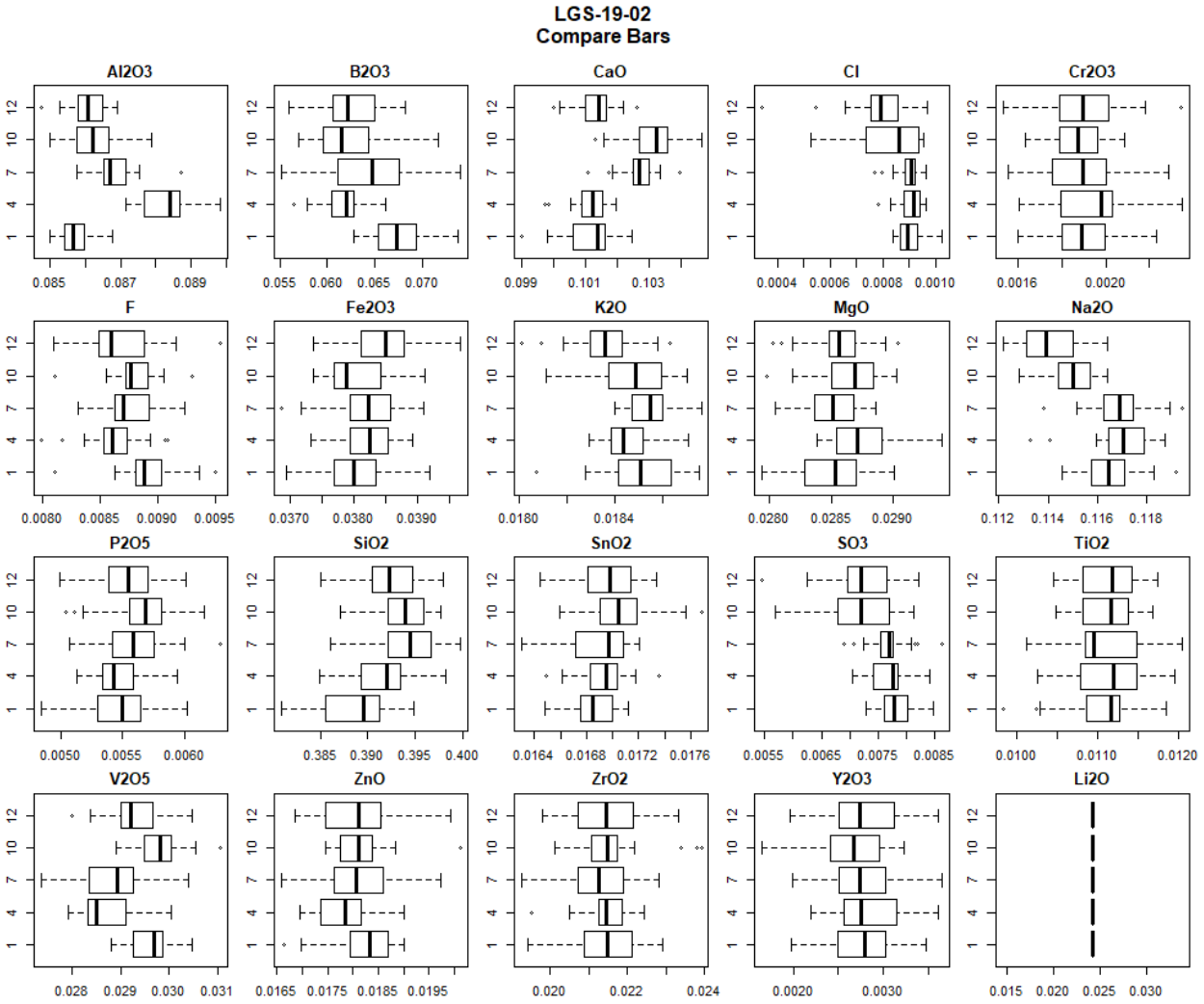

Figure A.10. Component distribution plots for LGS19-02 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

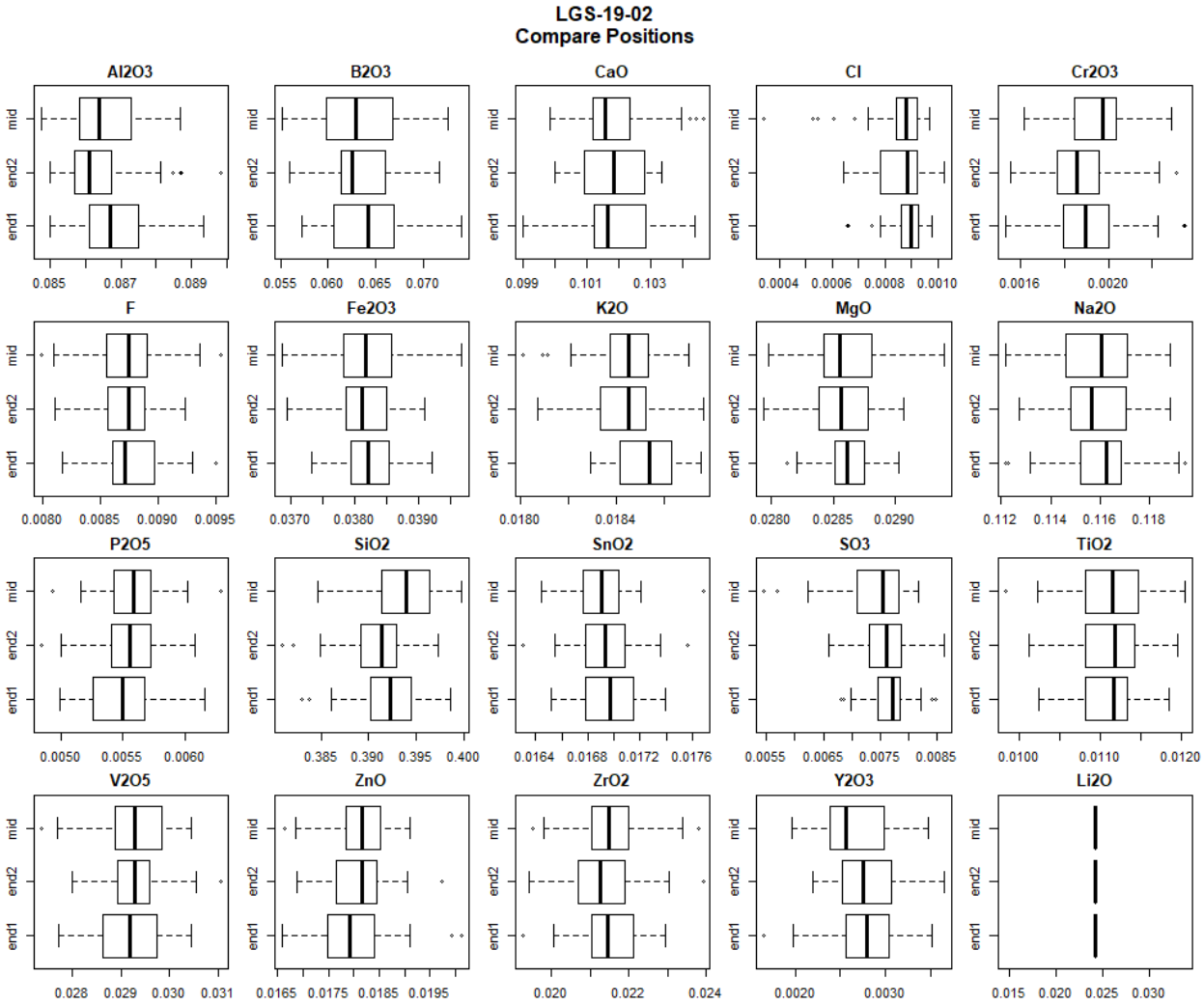

Figure A.11. Component distribution plots for LGS19-02 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-02] "Al2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	7.2357	0	100	0.0011	1.3233		
2 Bar	4	0.0001	0	0	72.4667	0.001	1.1265
3 Bar:Position	10	0	0	0	2.4328	0.0002	0.2064
4 error	120	0	0	0	25.1005	0.0006	0.663

Mean: 0.0866 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-02] "B2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	30.7394	0	100	0.0041	6.4556		
2 Bar	4	0.0006	0.0002	0	30.9947	0.0023	3.594
3 Bar:Position	10	0.0001	0	0	2.0715	0.0006	0.9291
4 error	120	0.0014	0	0	66.9338	0.0034	5.2816

Mean: 0.0637 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-02] "CaO"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	8.7557	0	100	0.0012	1.1475		
2 Bar	4	0.0001	0	0	66.0914	0.001	0.9329
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	120	0.0001	0	0	33.9086	0.0007	0.6682

Mean: 0.1019 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

[LGS-19-02] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	105.6219	0	100	0.0005	1.3694		
2 Bar	4	0	0	0	3.4198	0.0001	0.2532
3 Bar:Position	10	0	0	0	11.7108	0.0002	0.4686
4 error	120	0	0	0	84.8695	0.0005	1.2615

Mean: 0.0382 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-02] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	12.9982	0	100	0.0017	1.5081		
2 Bar	4	0.0002	0	0	53.1727	0.0013	1.0997
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	120	0.0002	0	0	46.8273	0.0012	1.032

Mean: 0.1159 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-02] "SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	39.7602	0	100	0.0039	0.9866		
2 Bar	4	0.0005	0.0001	0	23.3361	0.0019	0.4766
3 Bar:Position	10	0.0002	0	0	9.2695	0.0012	0.3004
4 error	120	0.0012	0	0	67.3944	0.0032	0.81

Mean: 0.3921 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-02] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	116.0041	0	100	0.0002	1.3917		
2	Bar	4	0	0	0	6.2142	0.0001	0.3469
3	Bar:Position	10	0	0	0*	0*	0*	0*
4	error	120	0	0	0	93.7858	0.0002	1.3478

Mean: 0.0169 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

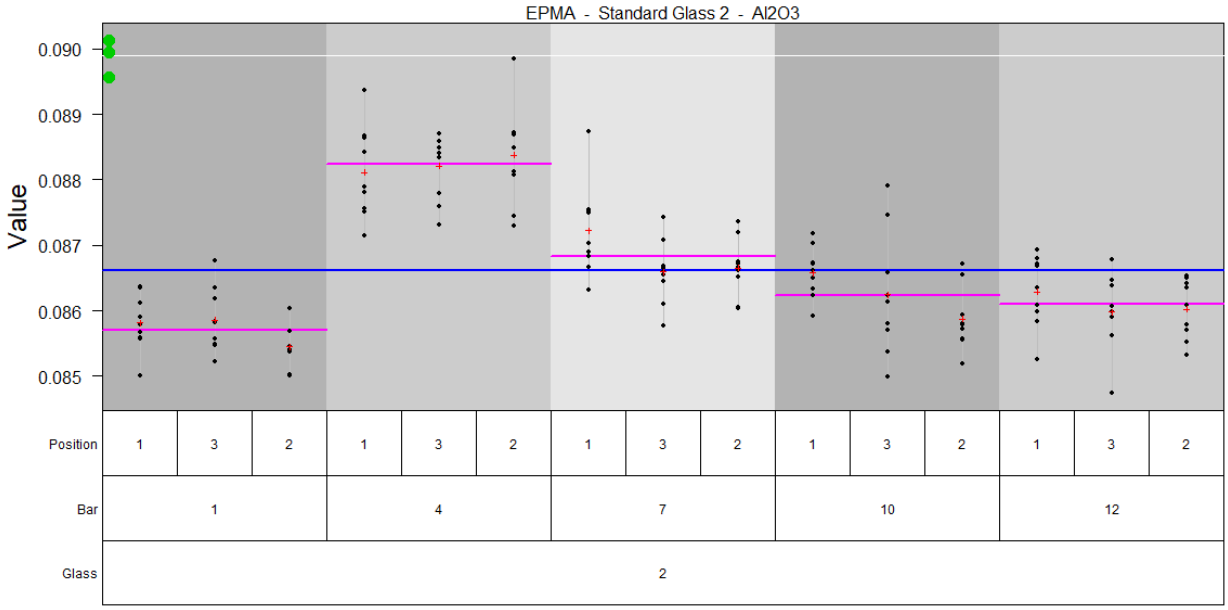

Figure A.12. Measured Al_2O_3 concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

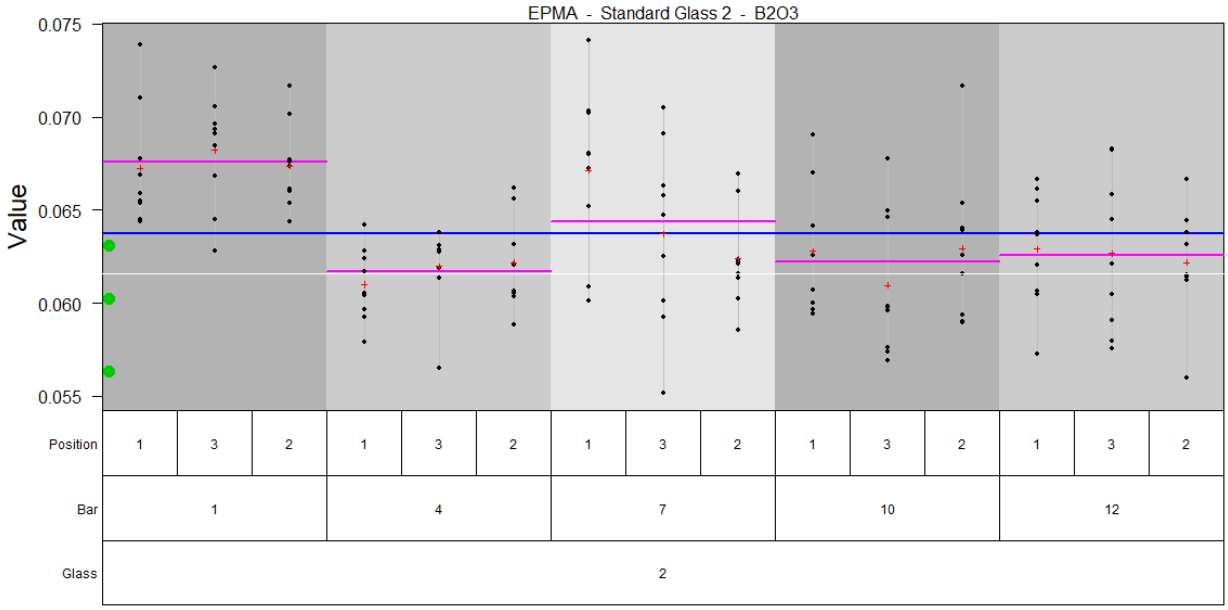

Figure A.13. Measured B_2O_3 concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

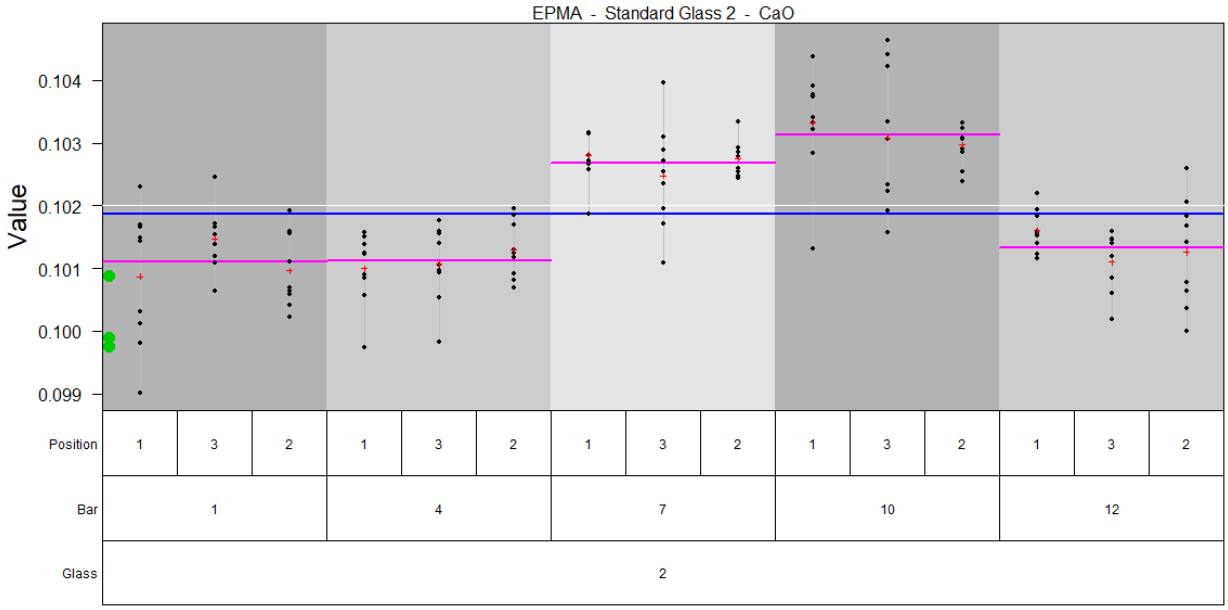

Figure A.14. Measured CaO concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

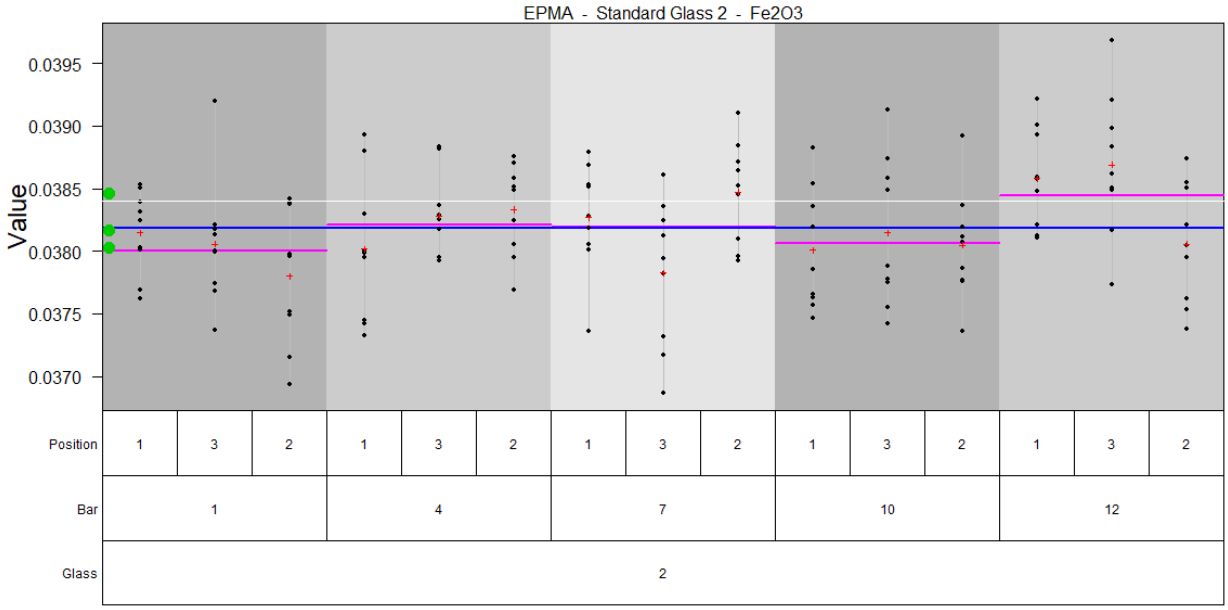

Figure A.15. Measured Fe_2O_3 concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

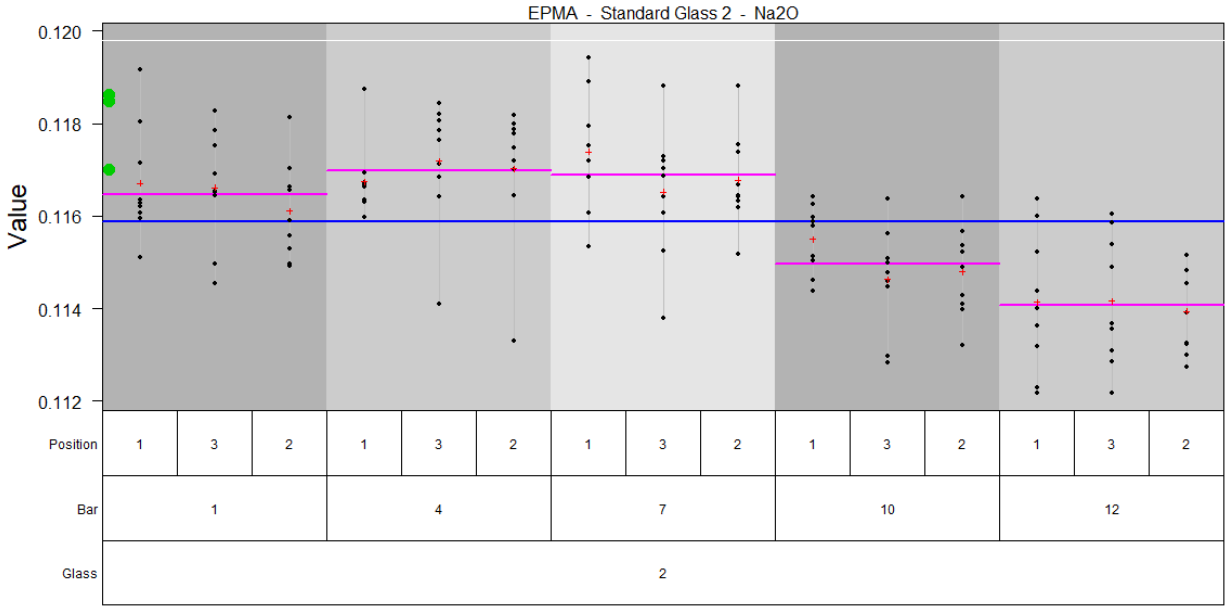

Figure A.16. Measured Na_2O concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

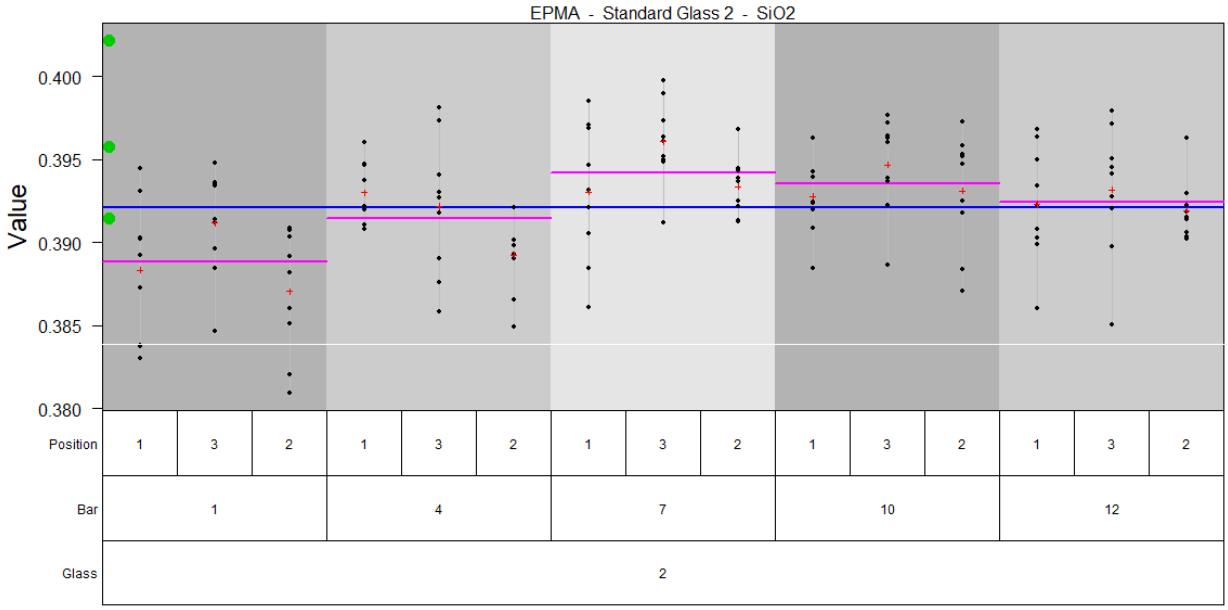

Figure A.17. Measured SiO_2 concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

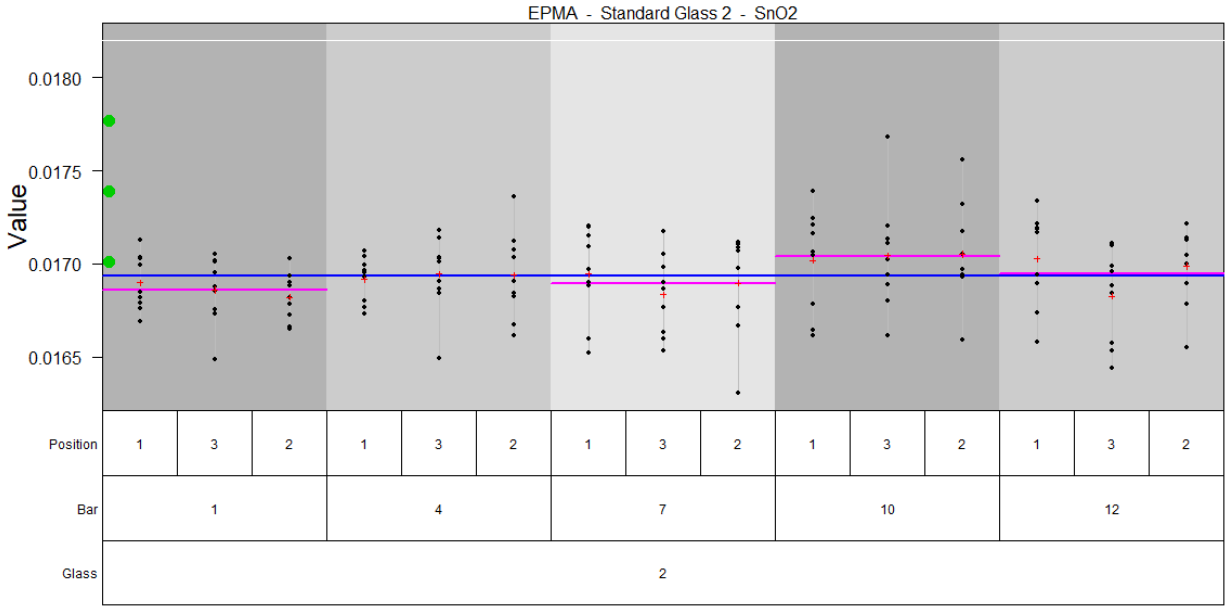

Figure A.18. Measured SnO_2 concentrations of LGS-19-02 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

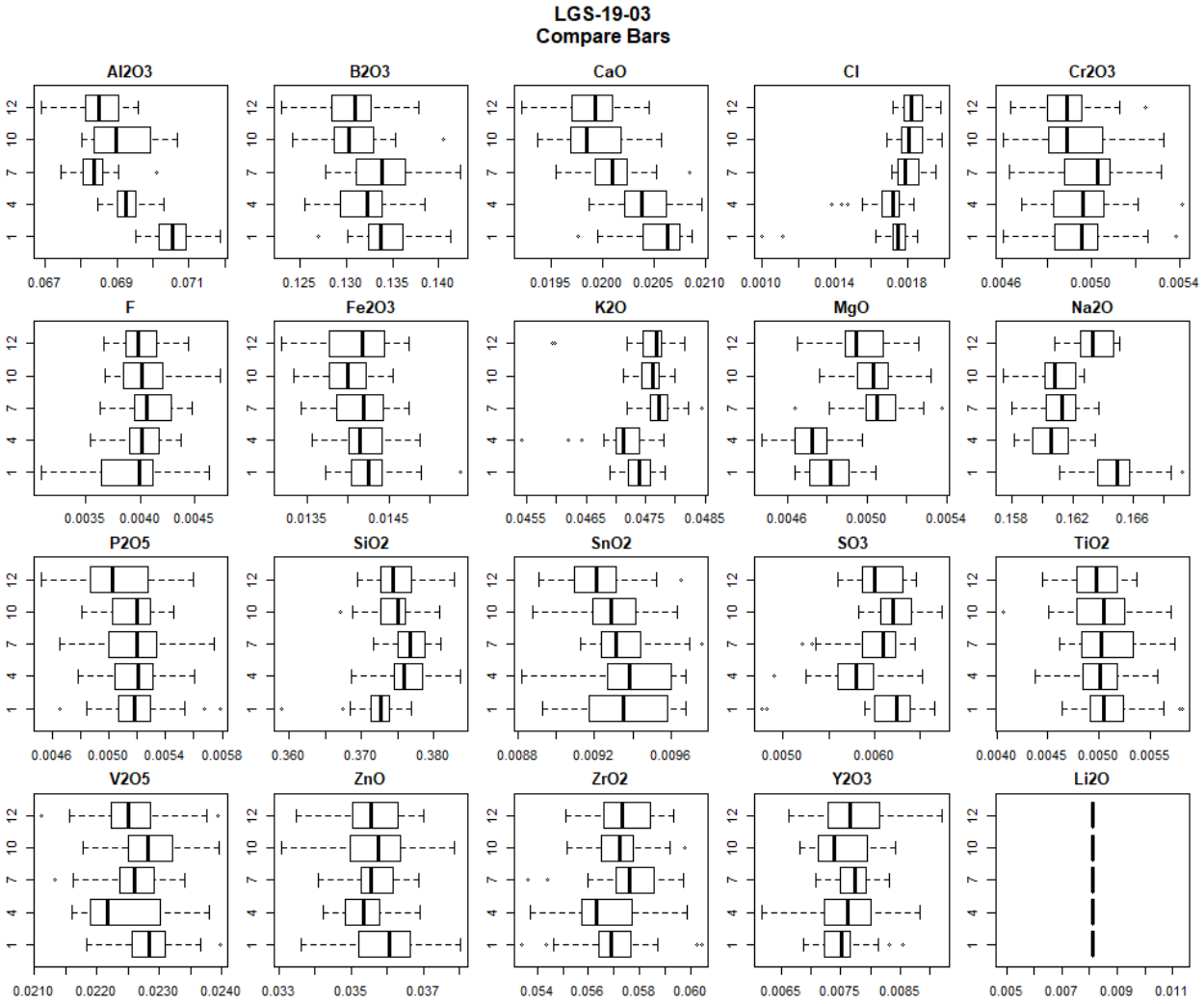

Figure A.19. Component distribution plots for LGS19-03 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

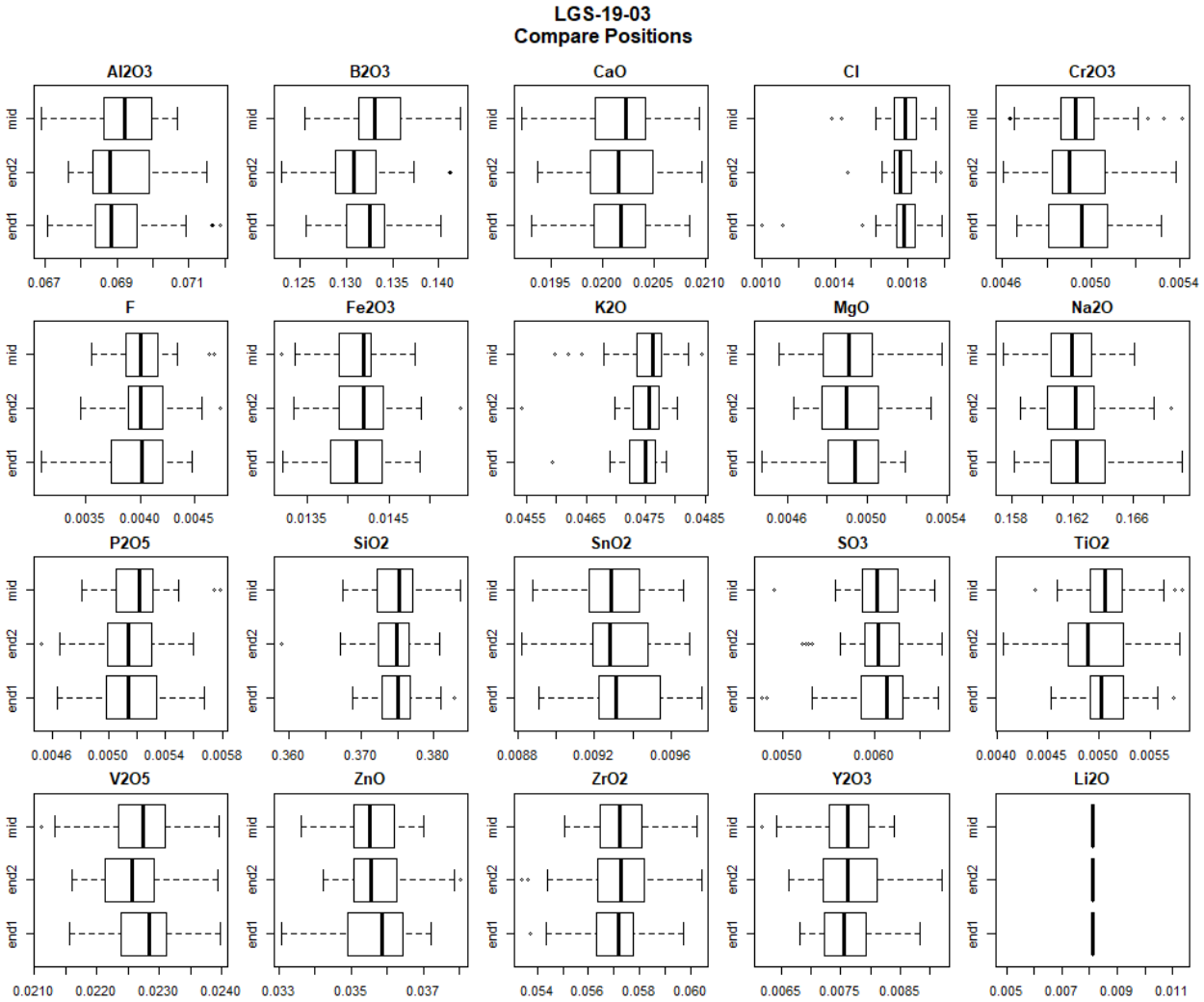

Figure A.20. Component distribution plots for LGS19-03 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration ion the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-03] "Al2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	8.5592	0	100	0.0011	1.5767		
2 Bar	4	0.0001	0	0	64.9075	0.0009	1.2702
3 Bar:Position	10	0	0	0	6.2981	0.0003	0.3957
4 error	119	0	0	0	28.7945	0.0006	0.846

Mean: 0.0692 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-03] "B2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	64.9003	0	100	0.0039	2.9508		
2 Bar	4	0.0003	0.0001	0	15.123	0.0015	1.1475
3 Bar:Position	10	0.0002	0	0	6.764	0.001	0.7674
4 error	119	0.0014	0	0	78.1131	0.0035	2.608

Mean: 0.1324 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-03] "CaO"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	17.4668	0	100	0.0004	2.0499		
2 Bar	4	0	0	0	44.8764	0.0003	1.3732
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	119	0	0	0	55.1236	0.0003	1.5219

Mean: 0.0202 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-03] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	115.9158	0	100	0.0004	2.6919		
2 Bar	4	0	0	0	2.6928	0.0001	0.4417
3 Bar:Position	10	0	0	0	8.0823	0.0001	0.7653
4 error	119	0	0	0	89.225	0.0004	2.5427

Mean: 0.0141 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-03] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	10.0318	0	100	0.0025	1.5122		
2 Bar	4	0.0004	0.0001	0	60.9197	0.0019	1.1803
3 Bar:Position	10	0	0	0	1.4314	0.0003	0.1809
4 error	119	0.0003	0	0	37.6489	0.0015	0.9279

Mean: 0.1622 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-03] "SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	47.6139	0	100	0.0036	0.9639		
2 Bar	4	0.0004	0.0001	0	23.0062	0.0017	0.4623
3 Bar:Position	10	0.0001	0	0*	0*	0*	0*
4 error	119	0.0012	0	0	76.9938	0.0032	0.8458

Mean: 0.3748 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-03] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	107.2251	0	100	0.0002	2.1991		
2	Bar	4	0	0	0	7.1327	0.0001	0.5873
3	Bar:Position	10	0	0	0	2.451	0	0.3443
4	error	119	0	0	0	90.4163	0.0002	2.0911

Mean: 0.0093 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

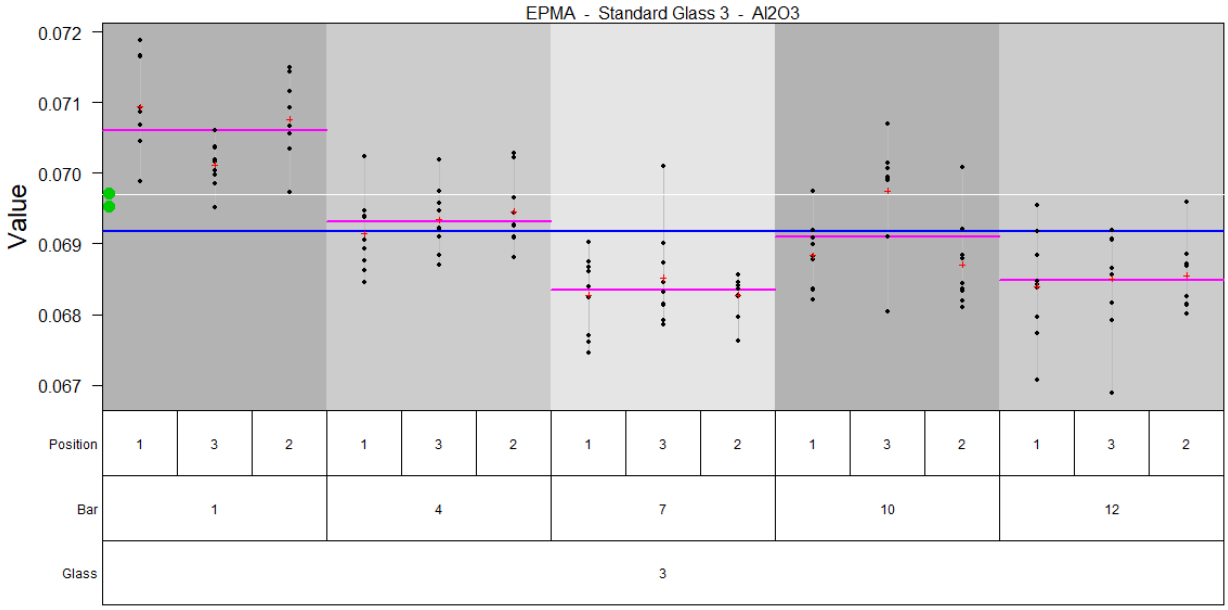

Figure A.21. Measured Al_2O_3 concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

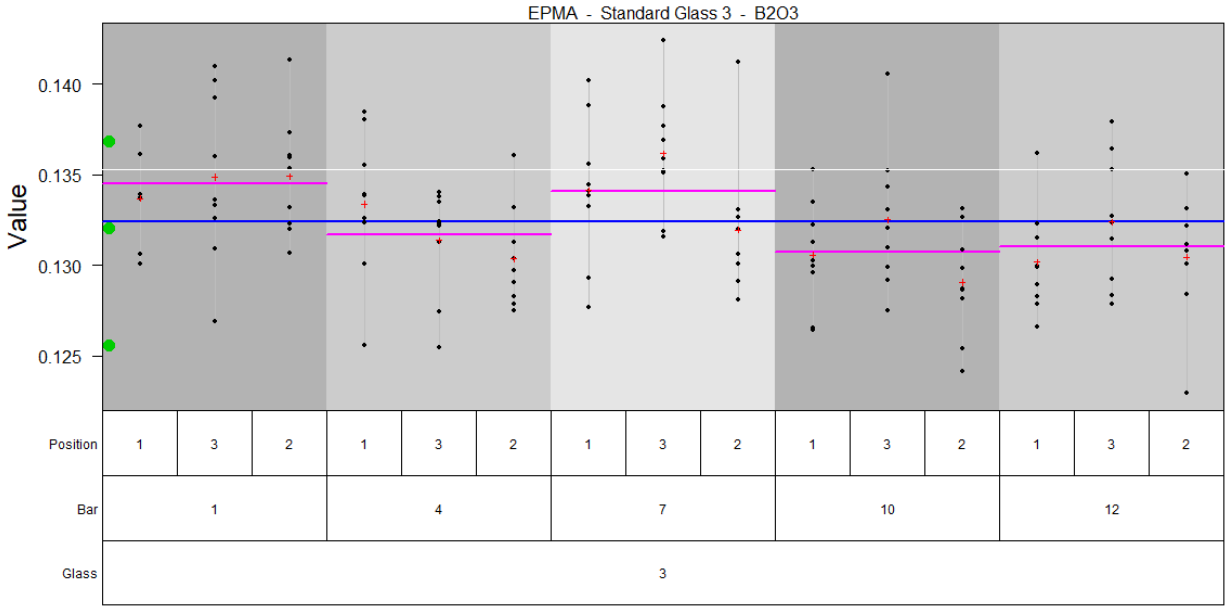

Figure A.22. Measured B_2O_3 concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

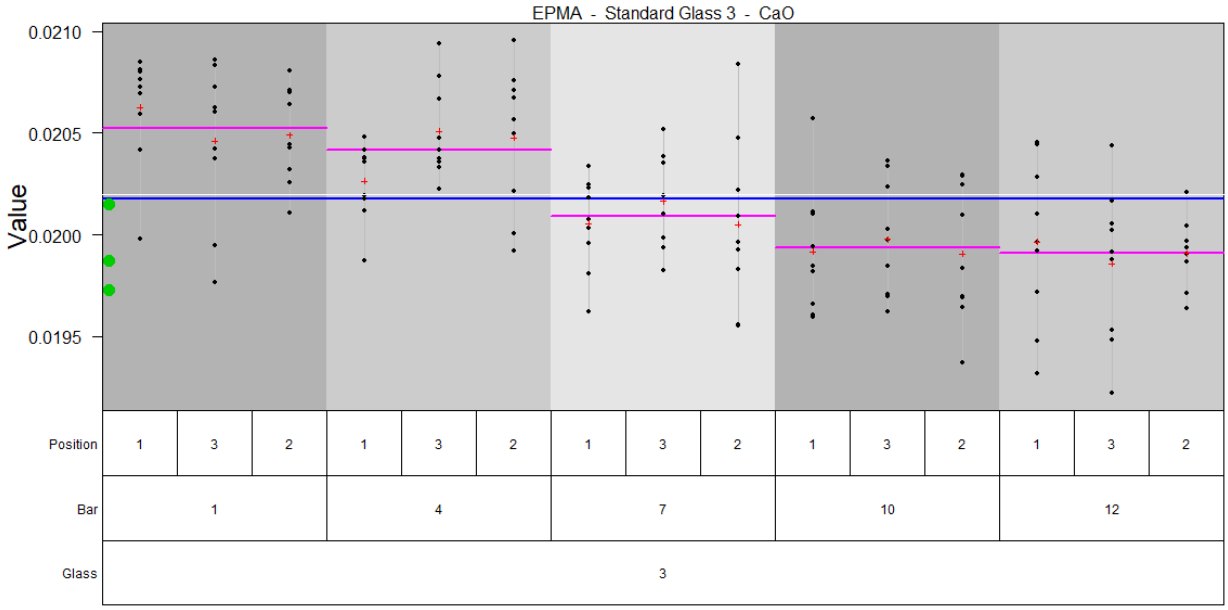

Figure A.23. Measured CaO concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

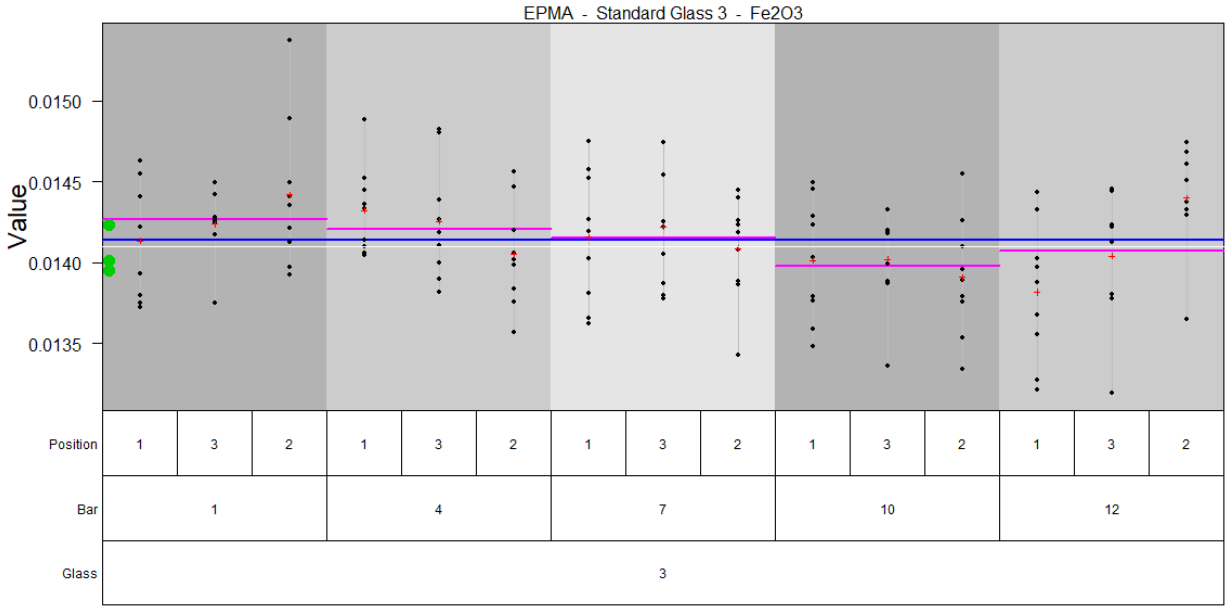

Figure A.24. Measured Fe_2O_3 concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

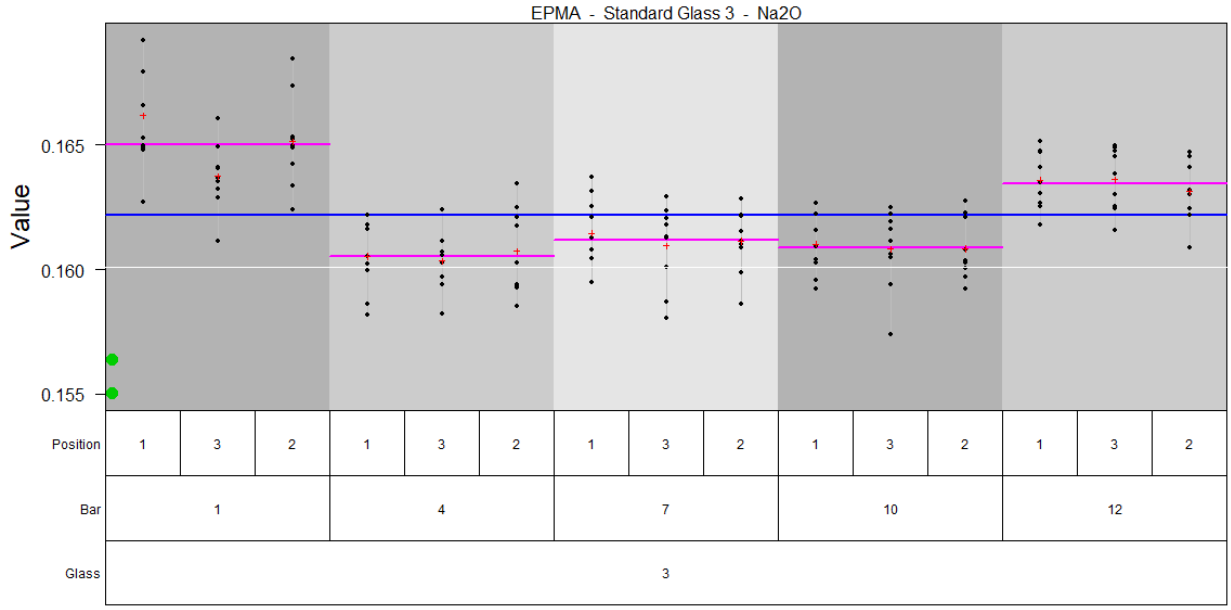

Figure A.25. Measured Na_2O concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

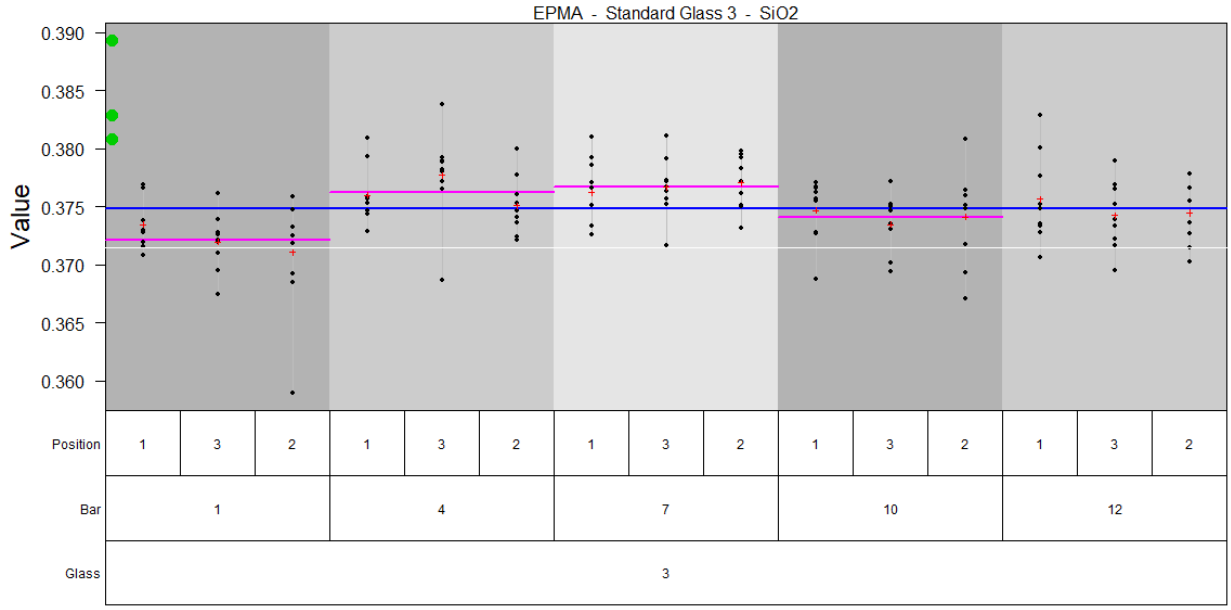

Figure A.26. Measured SiO_2 concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

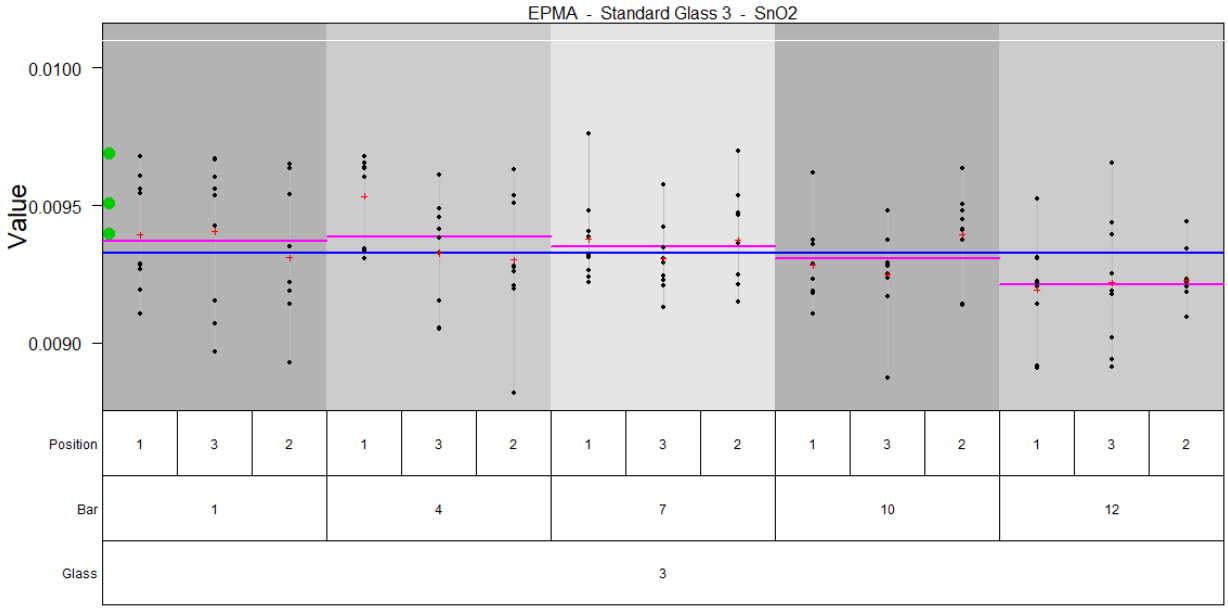

Figure A.27. Measured SnO_2 concentrations of LGS-19-03 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

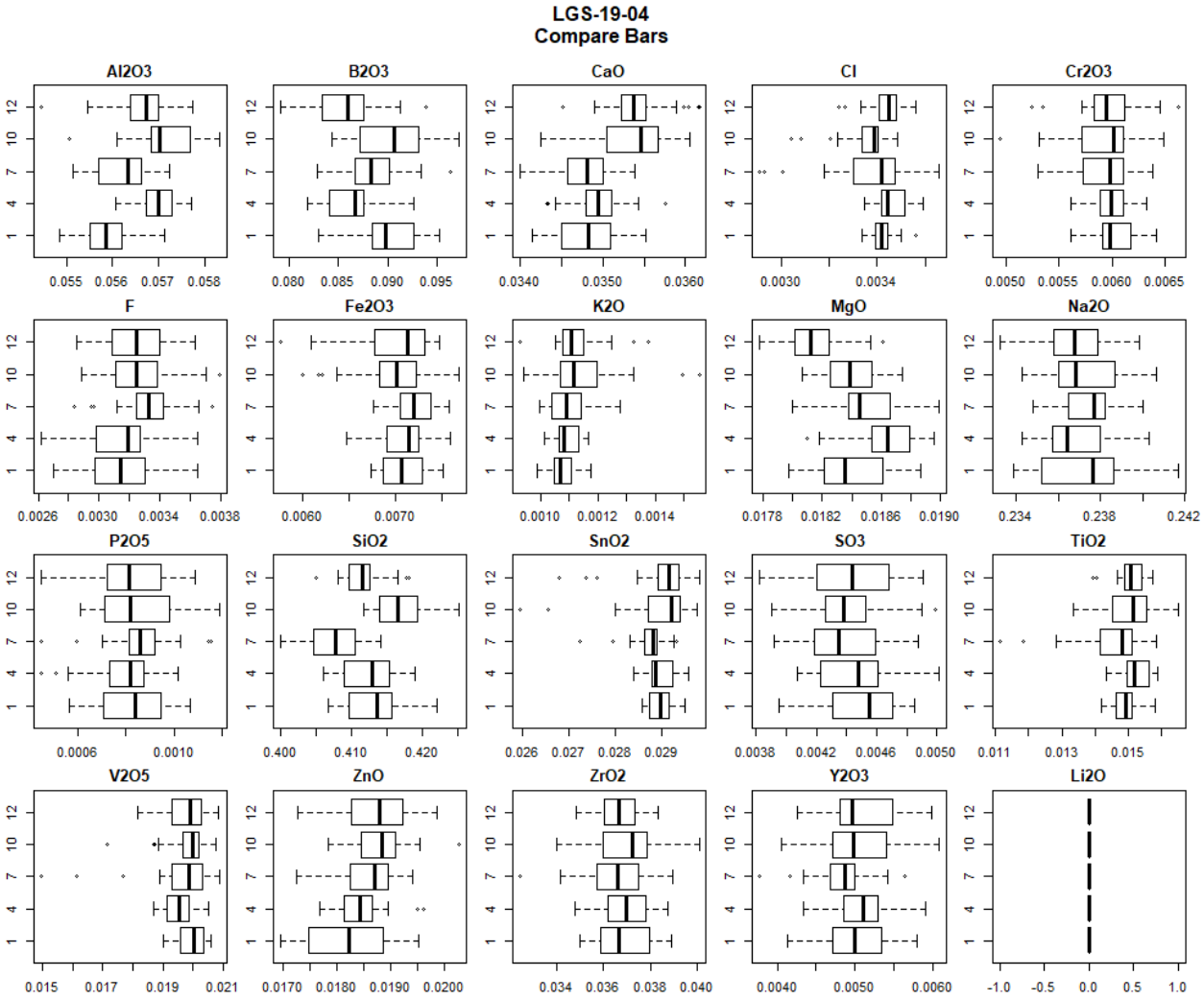

Figure A.28. Component distribution plots for LGS19-04 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

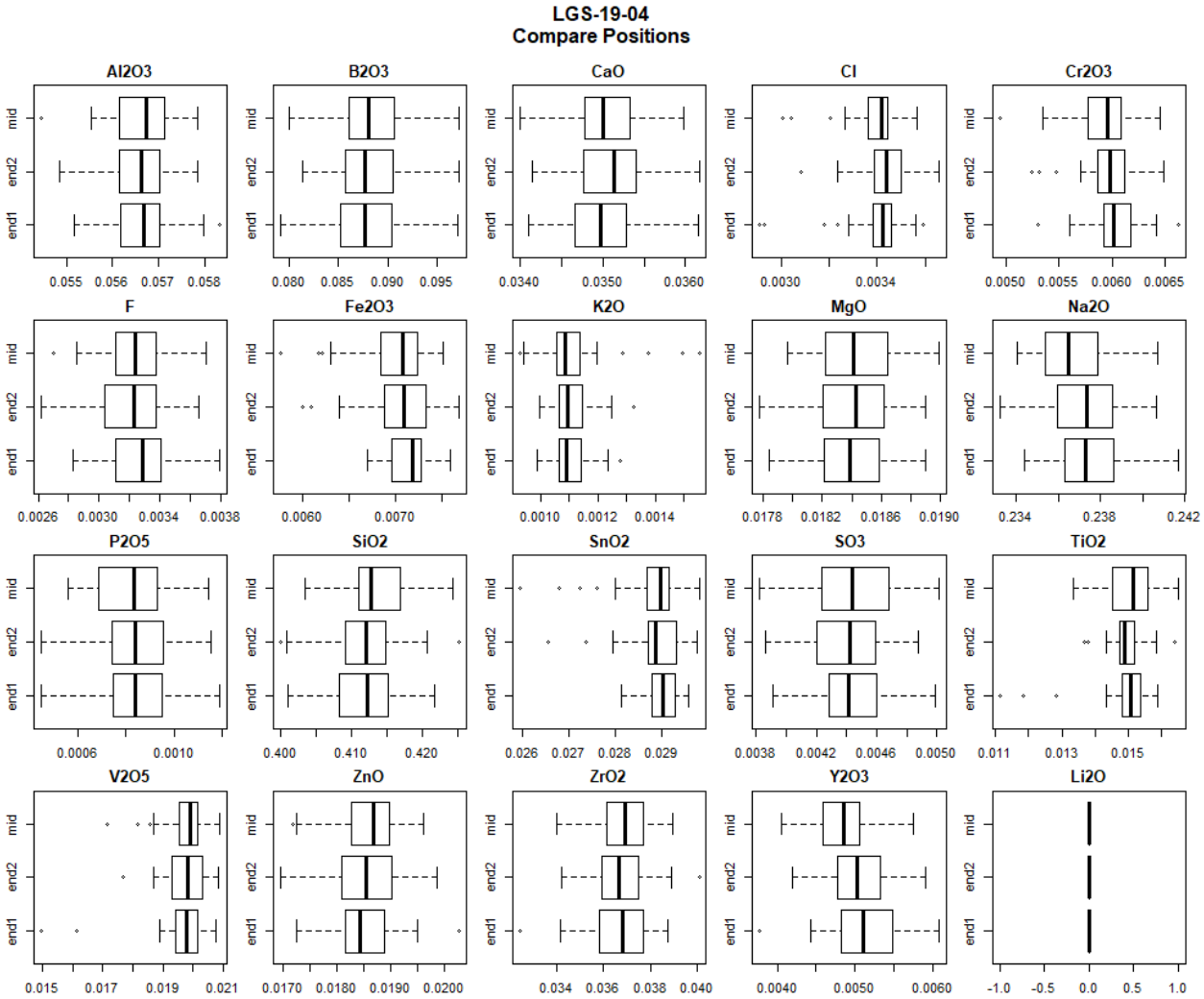

Figure A.29. Component distribution plots for LGS19-04 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-04] "Al2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	18.257	0	100	0.0008	1.3613		
2 Bar	4	0	0	0	42.8482	0.0005	0.8911
3 Bar:Position	10	0	0	0	2.8008	0.0001	0.2278
4 error	119	0	0	0	54.351	0.0006	1.0036

Mean: 0.0566 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-04] "B2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	33.077	0	100	0.0038	4.3439		
2 Bar	4	0.0005	0.0001	0	28.37	0.002	2.3137
3 Bar:Position	10	0.0002	0	0	5.3146	0.0009	1.0014
4 error	119	0.0012	0	0	66.3154	0.0031	3.5374

Mean: 0.0881 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-04] "CaO"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	25.0065	0	100	0.0005	1.3788		
2 Bar	4	0	0	0	36.1169	0.0003	0.8286
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	119	0	0	0	63.8831	0.0004	1.102

Mean: 0.035 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-04] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	121.952	0	100	0.0003	4.7802		
2 Bar	4	0	0	0	3.6383	0.0001	0.9118
3 Bar:Position	10	0	0	0	2.701	0.0001	0.7856
4 error	119	0	0	0	93.6607	0.0003	4.6262

Mean: 0.0071 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-04] "Na2O"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 97.9197 0 100 0.0018 0.7512
2 Bar 4 0 0 0* 0* 0* 0*
3 Bar:Position 10 0.0001 0 0 19.6788 0.0008 0.3332
4 error 119 0.0003 0 0 80.3212 0.0016 0.6732

Mean: 0.2371 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

[LGS-19-04] "SiO2"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 16.5442 0 100 0.0051 1.2397
2 Bar 4 0.0014 0.0003 0 45.4259 0.0034 0.8355
3 Bar:Position 10 0.0002 0 0 2.8516 0.0009 0.2093
4 error 119 0.0016 0 0 51.7225 0.0037 0.8916

Mean: 0.4125 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-04] "SnO2"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 132.7614 0 100 0.0006 1.9971
2 Bar 4 0 0 0* 0* 0* 0*
3 Bar:Position 10 0 0 0* 0* 0* 0*
4 error 119 0 0 0 100 0.0006 1.9971

Mean: 0.0289 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

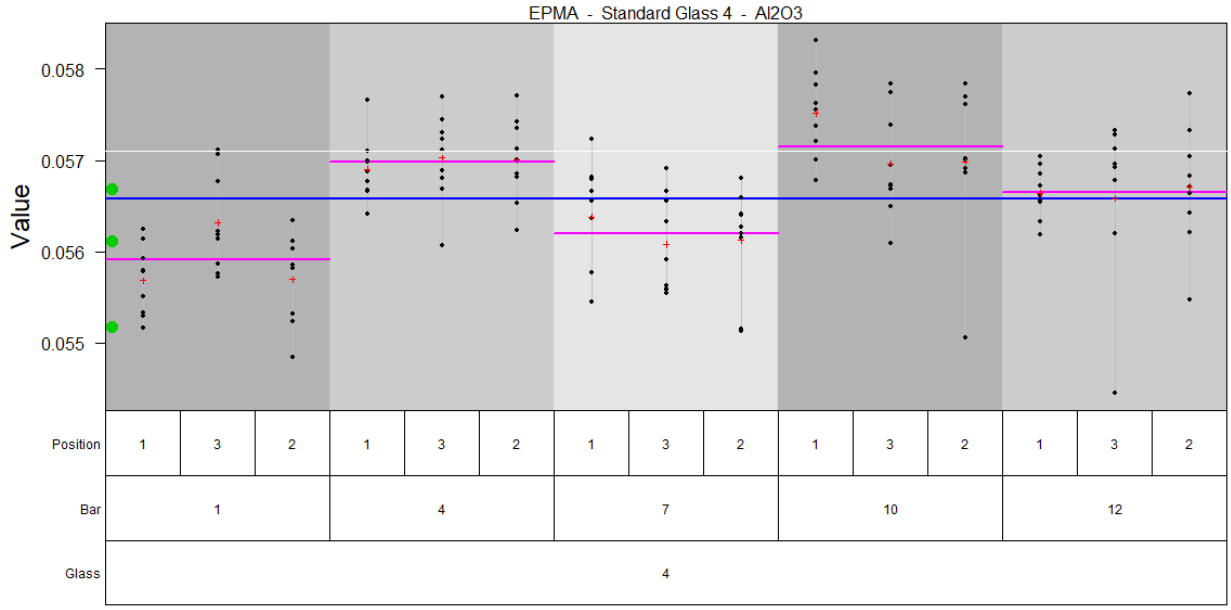

Figure A.30. Measured Al_2O_3 concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

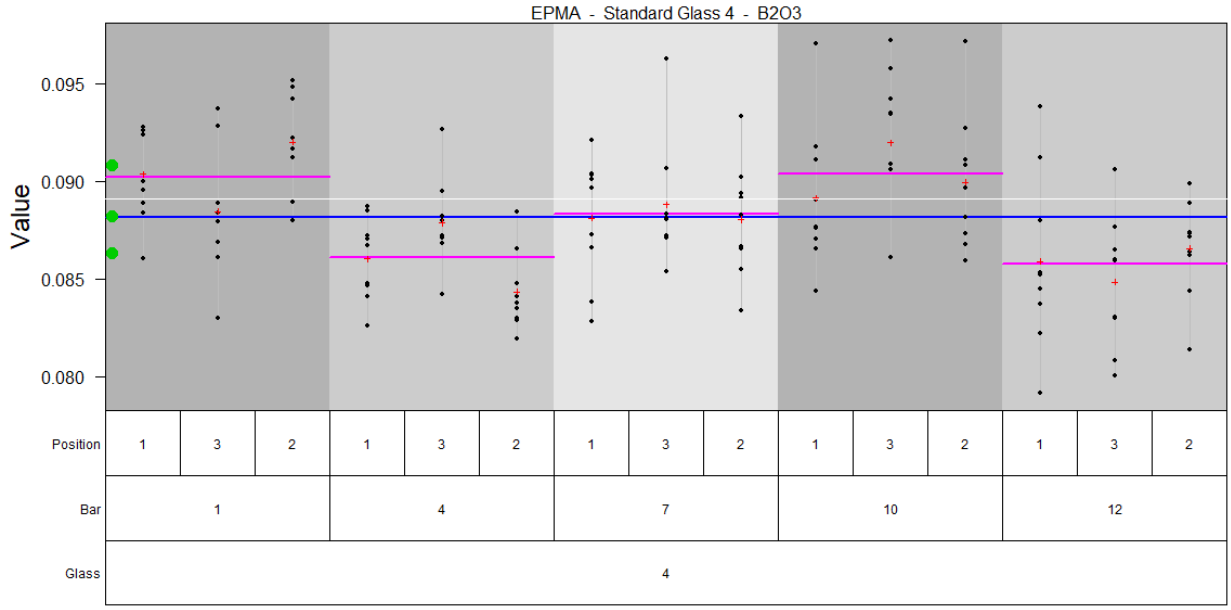

Figure A.31. Measured B_2O_3 concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

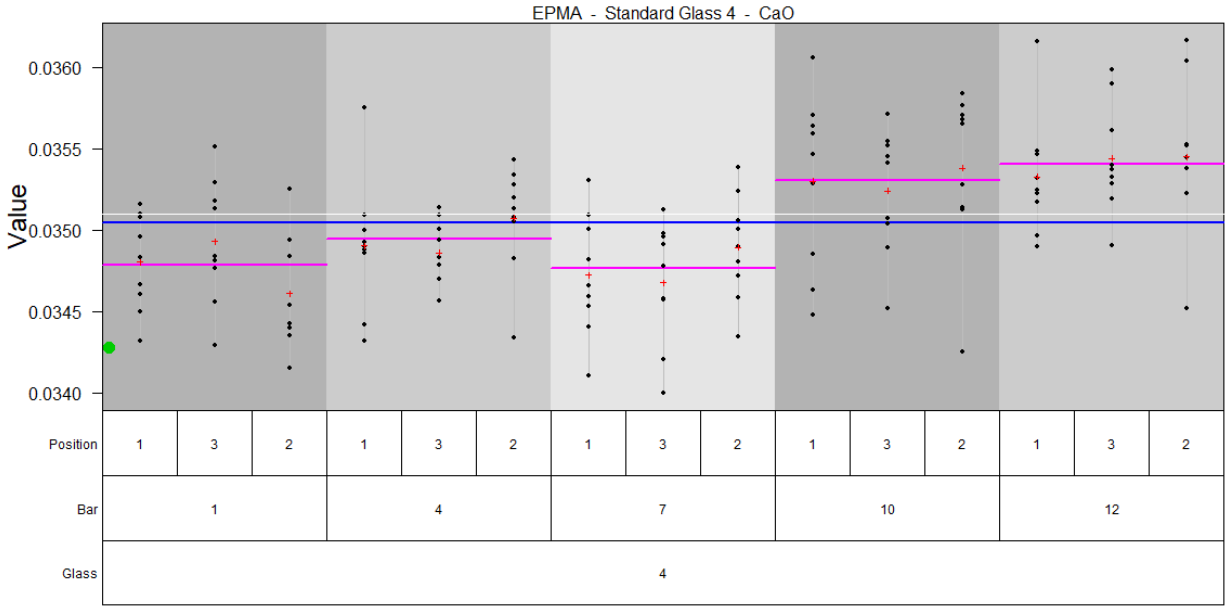

Figure A.32. Measured CaO concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

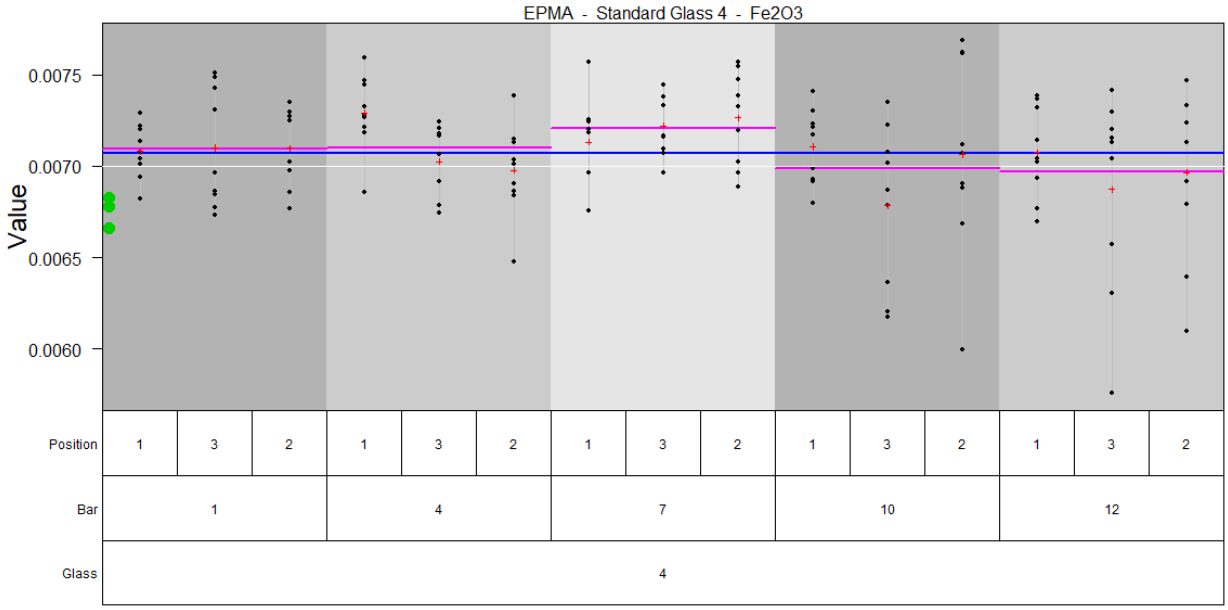

Figure A.33. Measured Fe_2O_3 concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

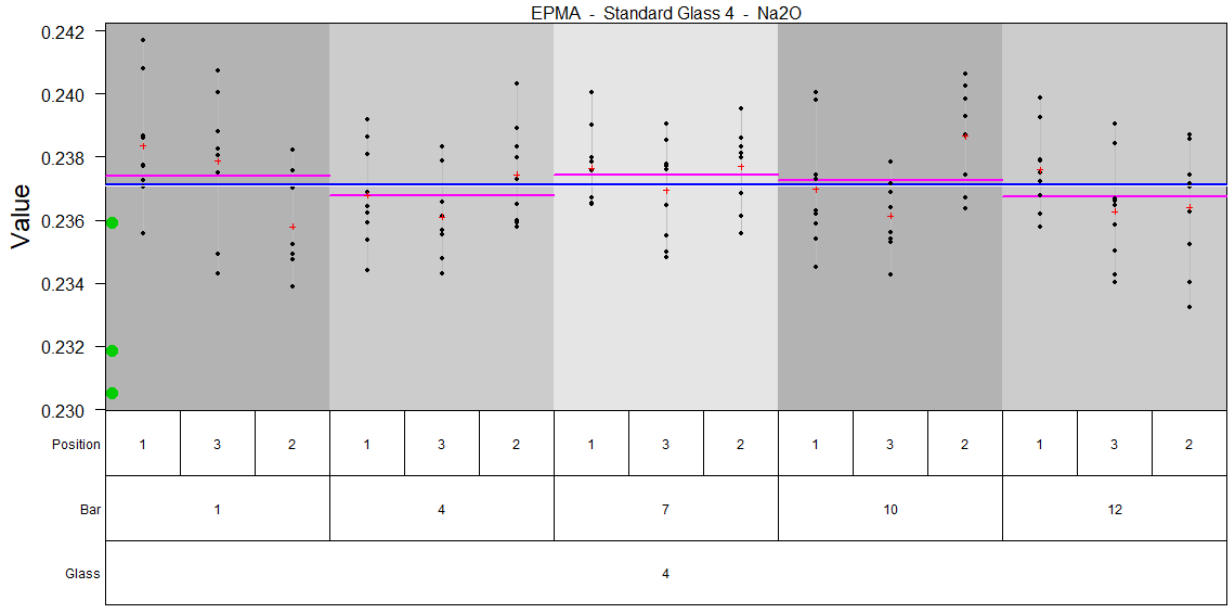

Figure A.34. Measured Na_2O concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

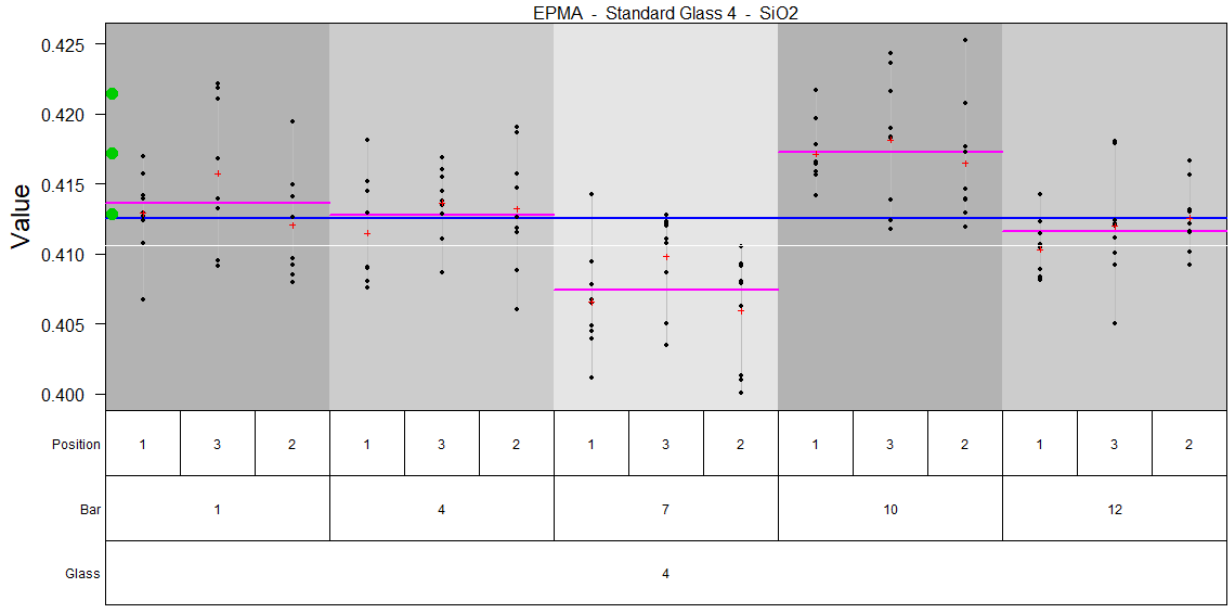

Figure A.35. Measured SiO_2 concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

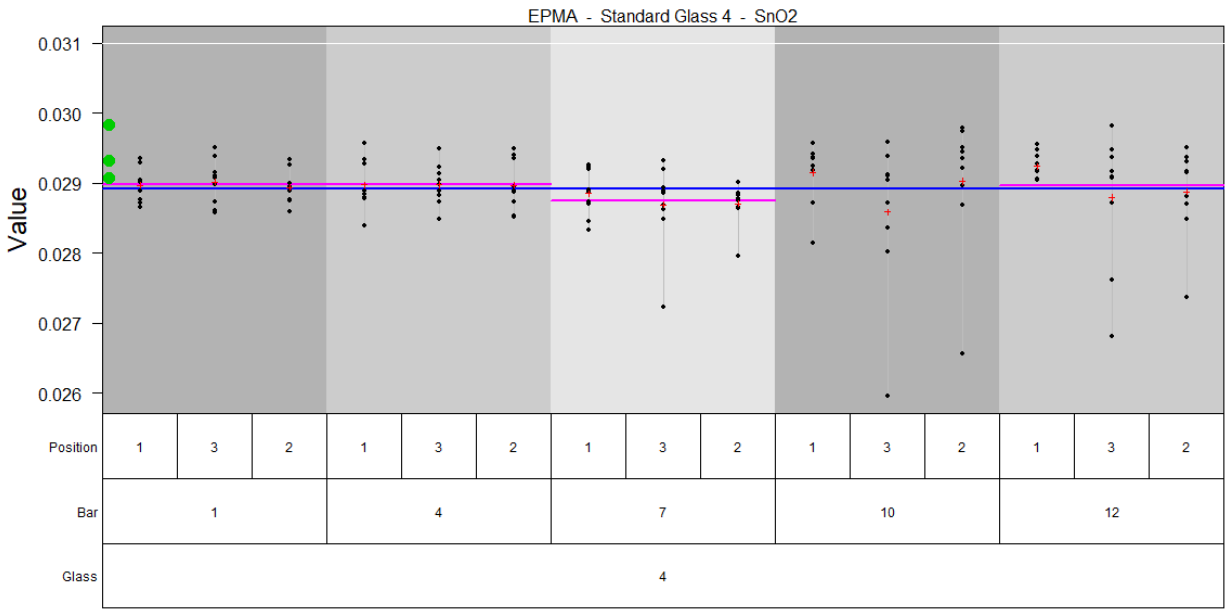

Figure A.36. Measured SnO_2 concentrations of LGS-19-04 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

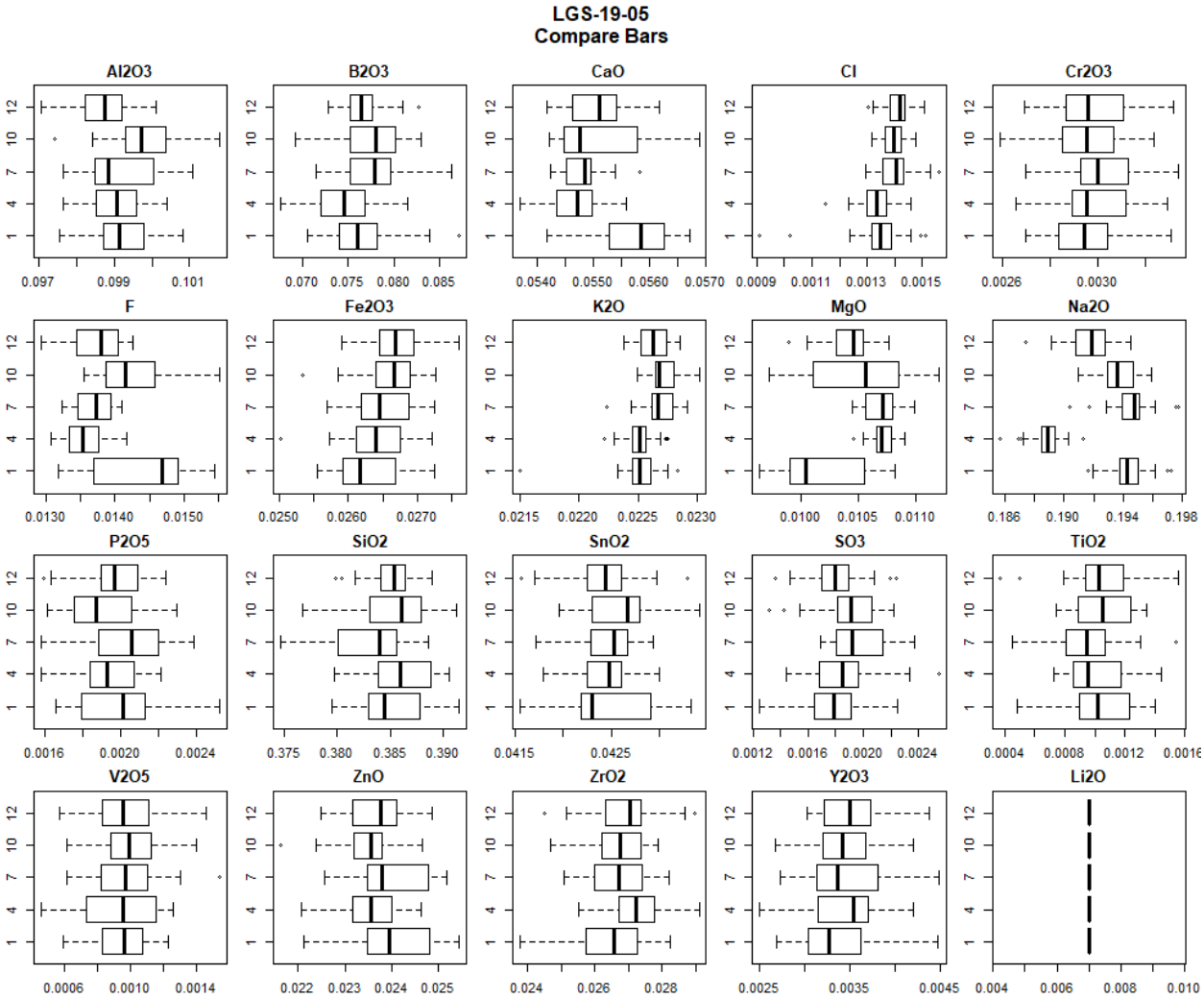

Figure A.37. Component distribution plots for LGS19-05 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

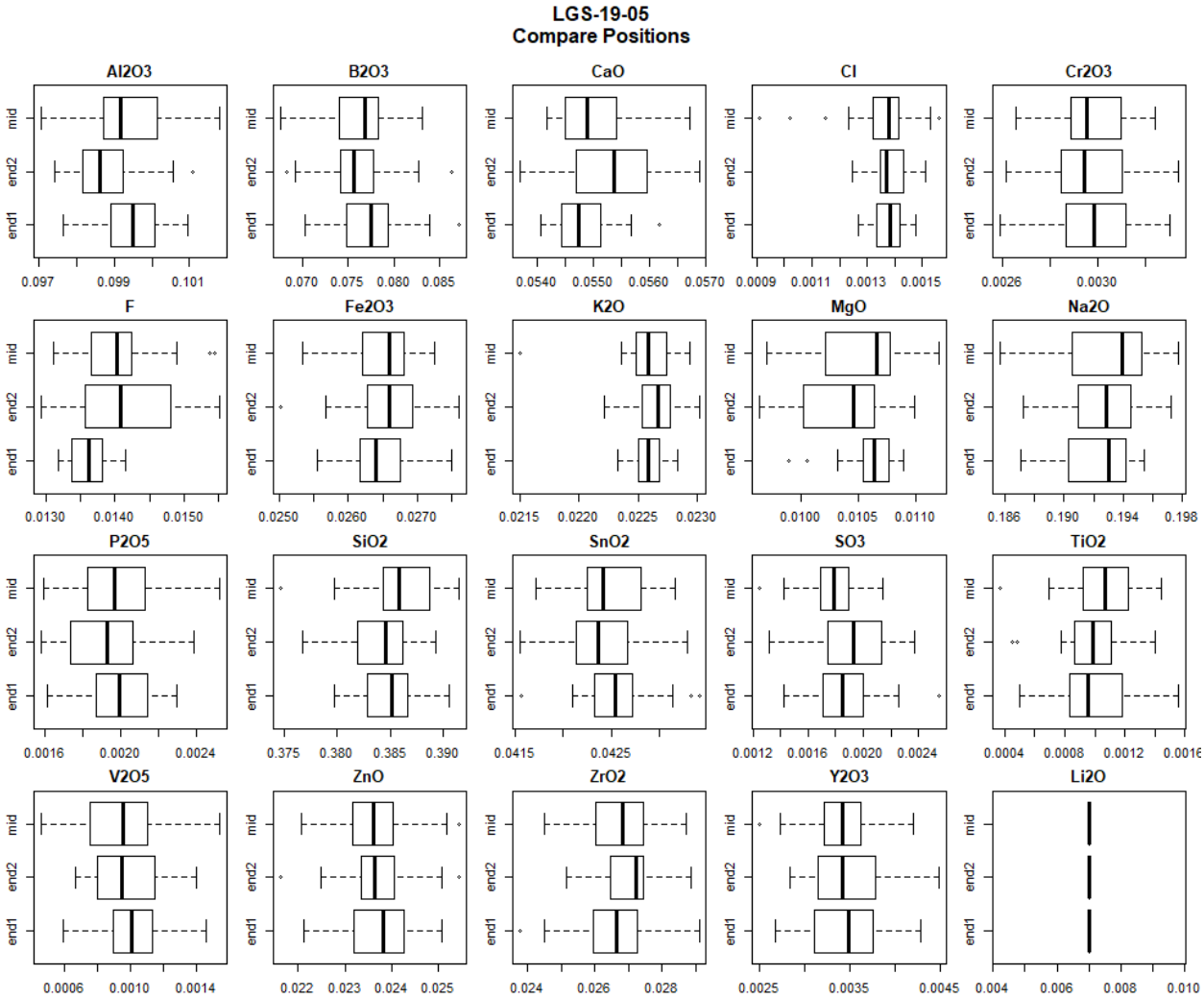

Figure A.38. Component distribution plots for LGS19-05 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-05] "Al2O3"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 53.7052 0 100 0.001 0.9771
2 Bar 4 0 0 0 2.9386 0.0002 0.1675
3 Bar:Position 10 0 0 0 37.12 0.0006 0.5953
4 error 119 0.0001 0 0 59.9415 0.0008 0.7565

Mean: 0.0992 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-05] "B2O3"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 82.0655 0 100 0.0035 4.5799
2 Bar 4 0.0002 0 0 6.8928 0.0009 1.2024
3 Bar:Position 10 0.0003 0 0 15.5027 0.0014 1.8033
4 error 119 0.0011 0 0 77.6045 0.0031 4.0346

Mean: 0.0765 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-05] "CaO"

Result Variance Component Analysis:

 Name DF SS MS VC %Total SD CV[%]
1 total 25.9437 0 100 0.0007 1.2925
2 Bar 4 0 0 0 8.5431 0.0002 0.3778
3 Bar:Position 10 0 0 0 57.5931 0.0005 0.9809
4 error 119 0 0 0 33.8639 0.0004 0.7522

Mean: 0.0551 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-05] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	100.046	0	100	0.0005	1.7954		
2 Bar	4	0	0	0	9.4301	0.0001	0.5514
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	119	0	0	0	90.5699	0.0005	1.7087

Mean: 0.0265 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-05] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	6.8827	0	100	0.0028	1.4481		
2 Bar	4	0.0006	0.0002	0	74.2304	0.0024	1.2476
3 Bar:Position	10	0	0	0	3.0915	0.0005	0.2546
4 error	119	0.0002	0	0	22.6781	0.0013	0.6896

Mean: 0.1926 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-05]"SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	111.8291	0	100	0.0032	0.8348		
2 Bar	4	0.0001	0	0	2.8444	0.0005	0.1408
3 Bar:Position	10	0.0002	0	0	9.7187	0.001	0.2603
4 error	119	0.0011	0	0	87.4369	0.003	0.7806

Mean: 0.3849 (N = 134)
Experimental Design: unbalanced | Method: ANOVA

[LGS-19-05] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	113.1141	0	100	0.0004	0.8845		
2	Bar	4	0	0*	0*	0*	0*	
3	Bar:Position	10	0	0	0	13.5007	0.0001	0.325
4	error	119	0	0	0	86.4993	0.0003	0.8226

Mean: 0.0425 (N = 134)
Experimental Design: unbalanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

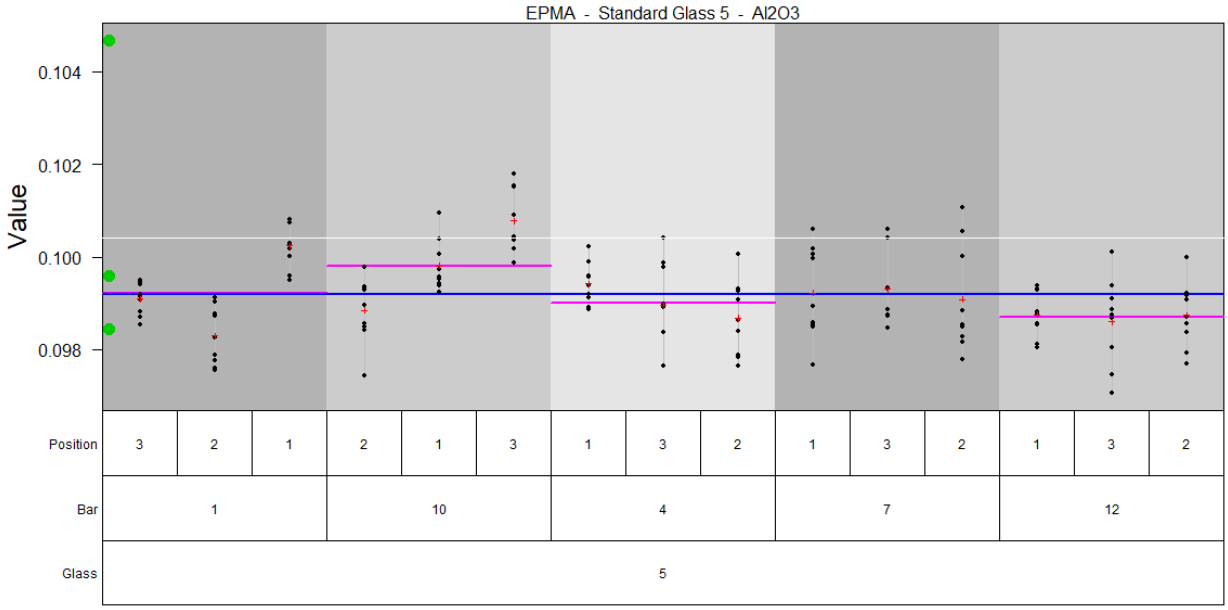

Figure A.39. Measured Al_2O_3 concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

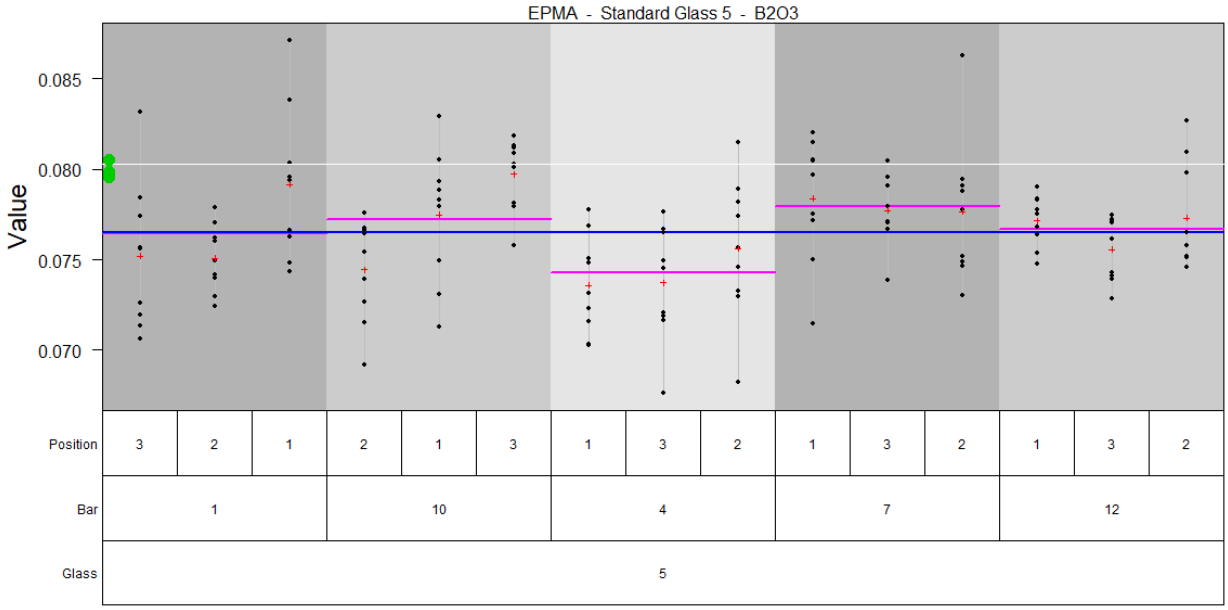

Figure A.40. Measured B_2O_3 concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

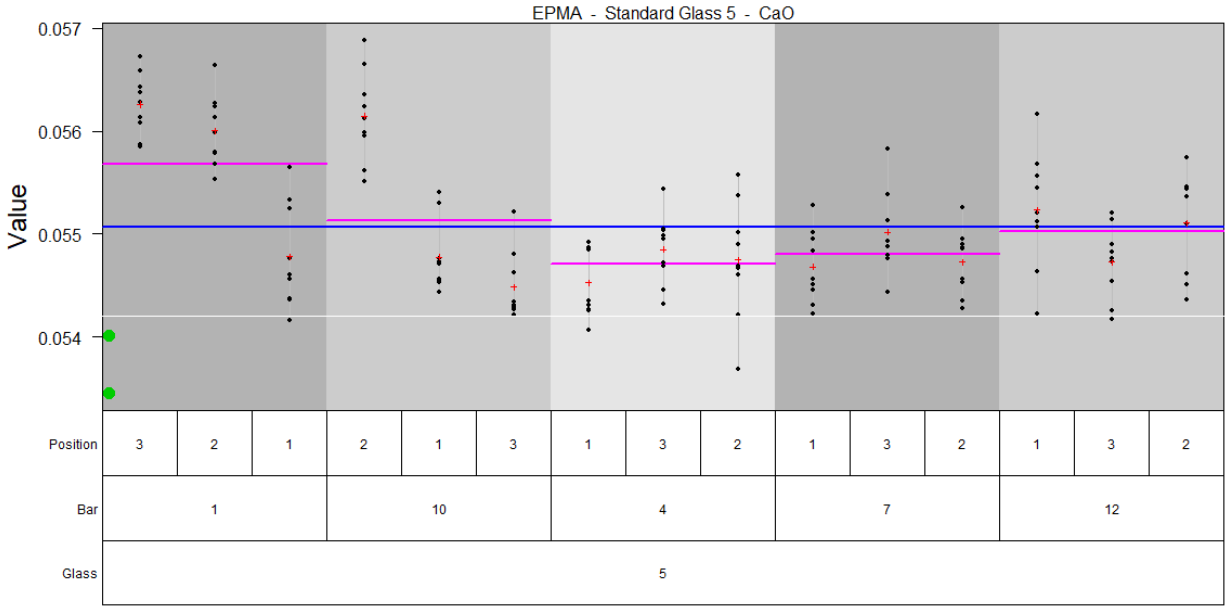

Figure A.41. Measured CaO concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

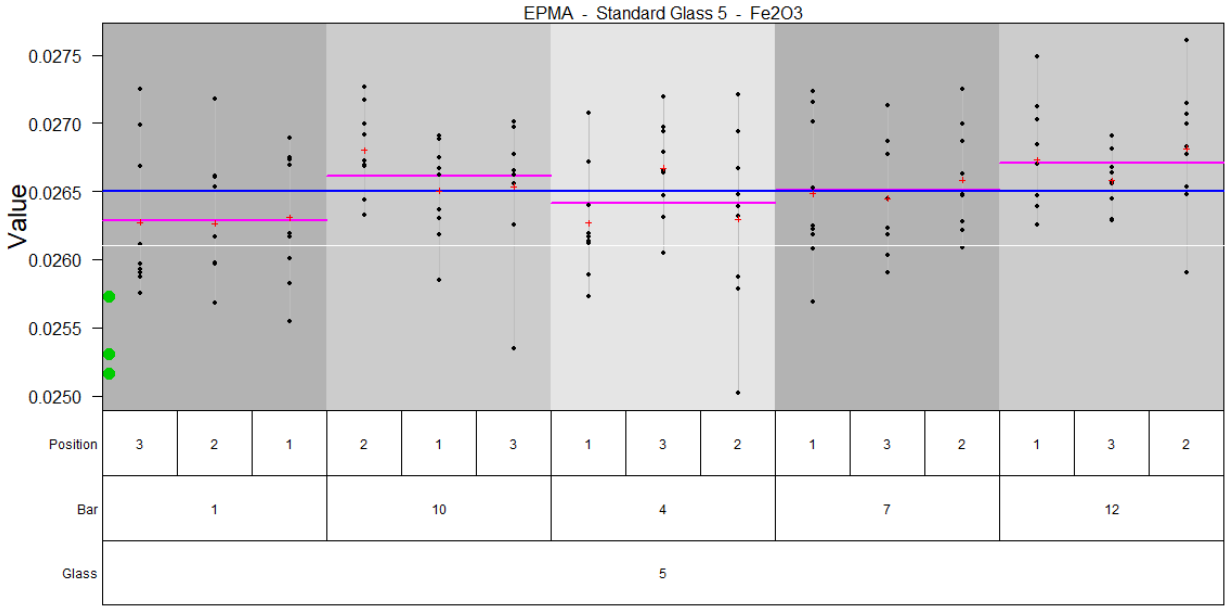

Figure A.42. Measured Fe_2O_3 concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

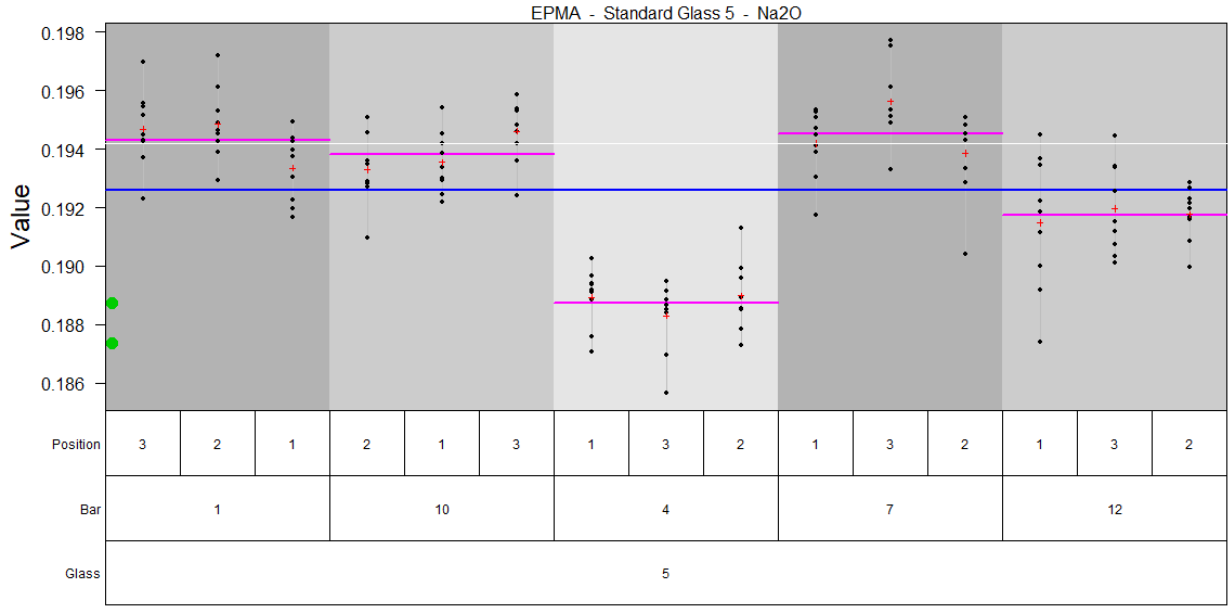

Figure A.43. Measured Na_2O concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

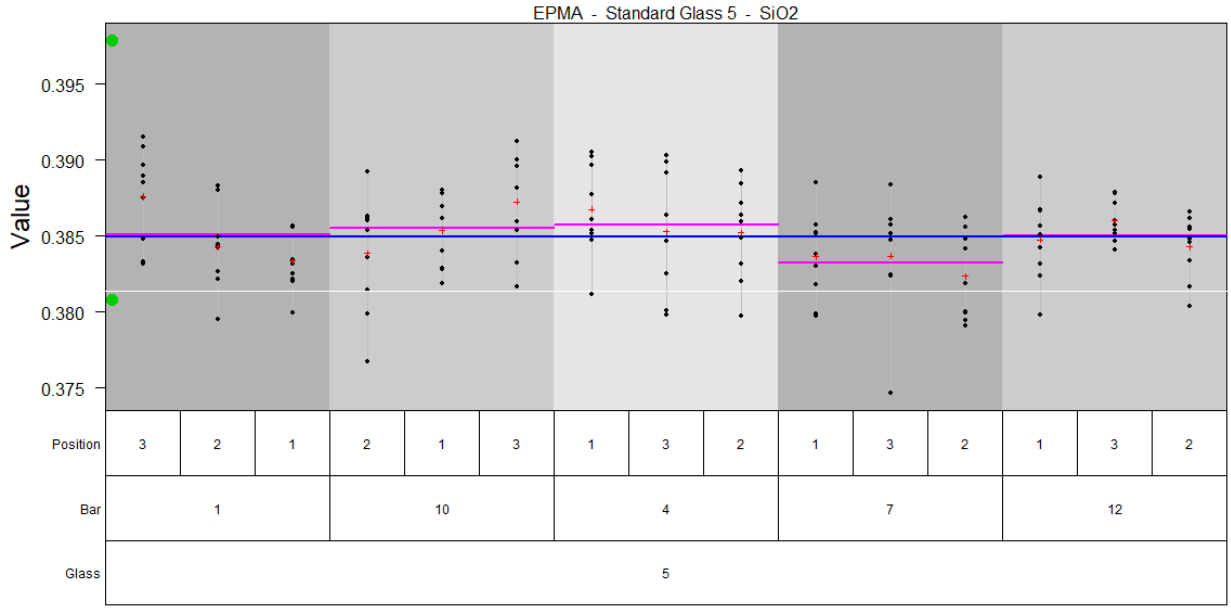

Figure A.44. Measured SiO_2 concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

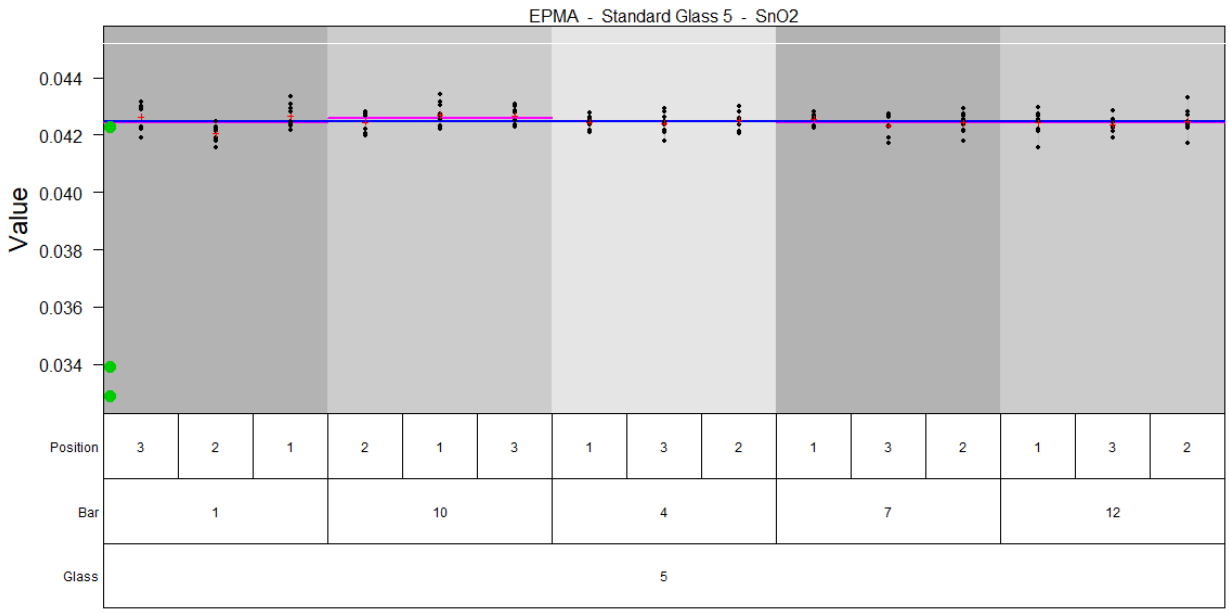

Figure A.45. Measured SnO_2 concentrations of LGS-19-05 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

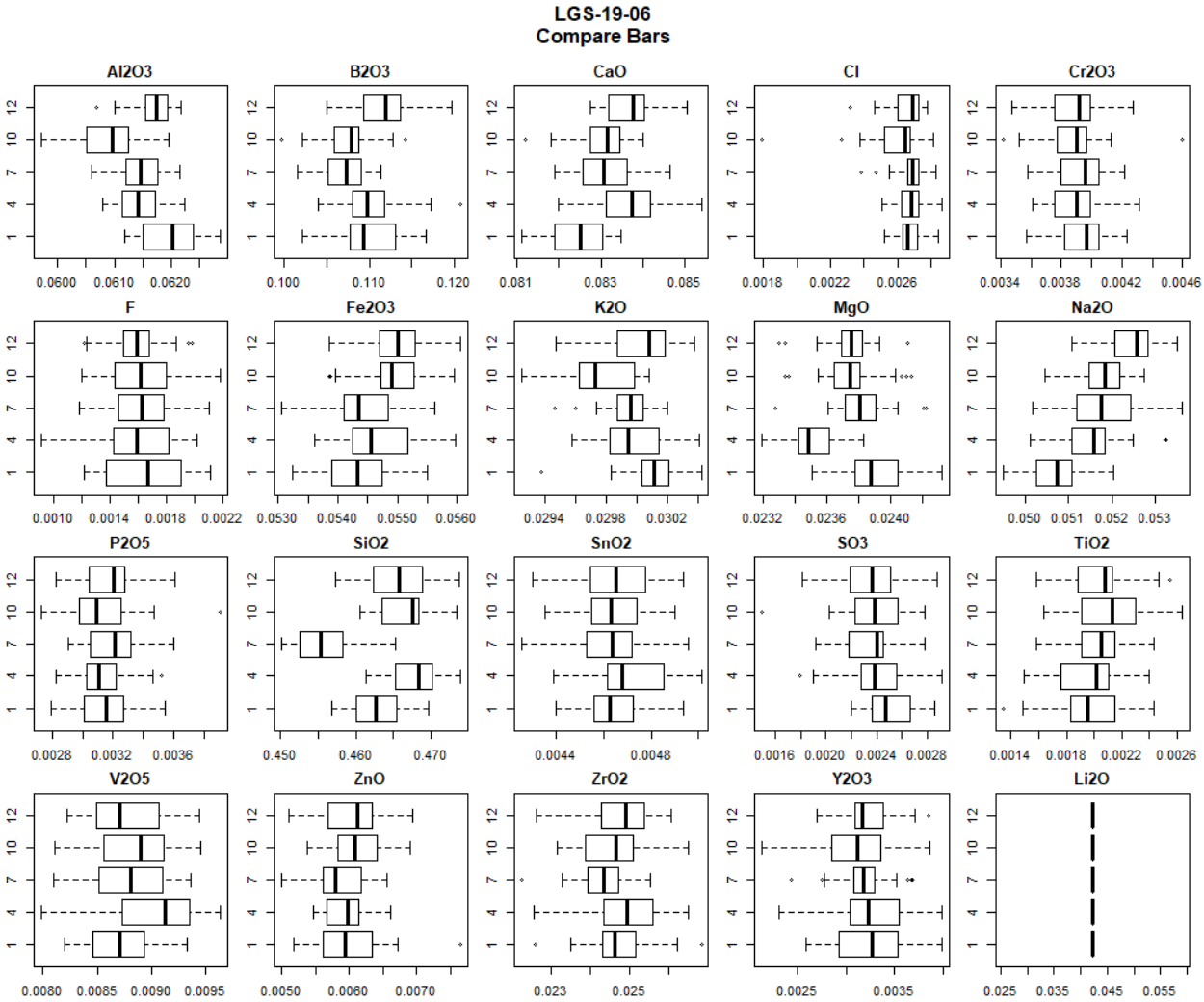

Figure A.46. Component distribution plots for LGS19-06 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

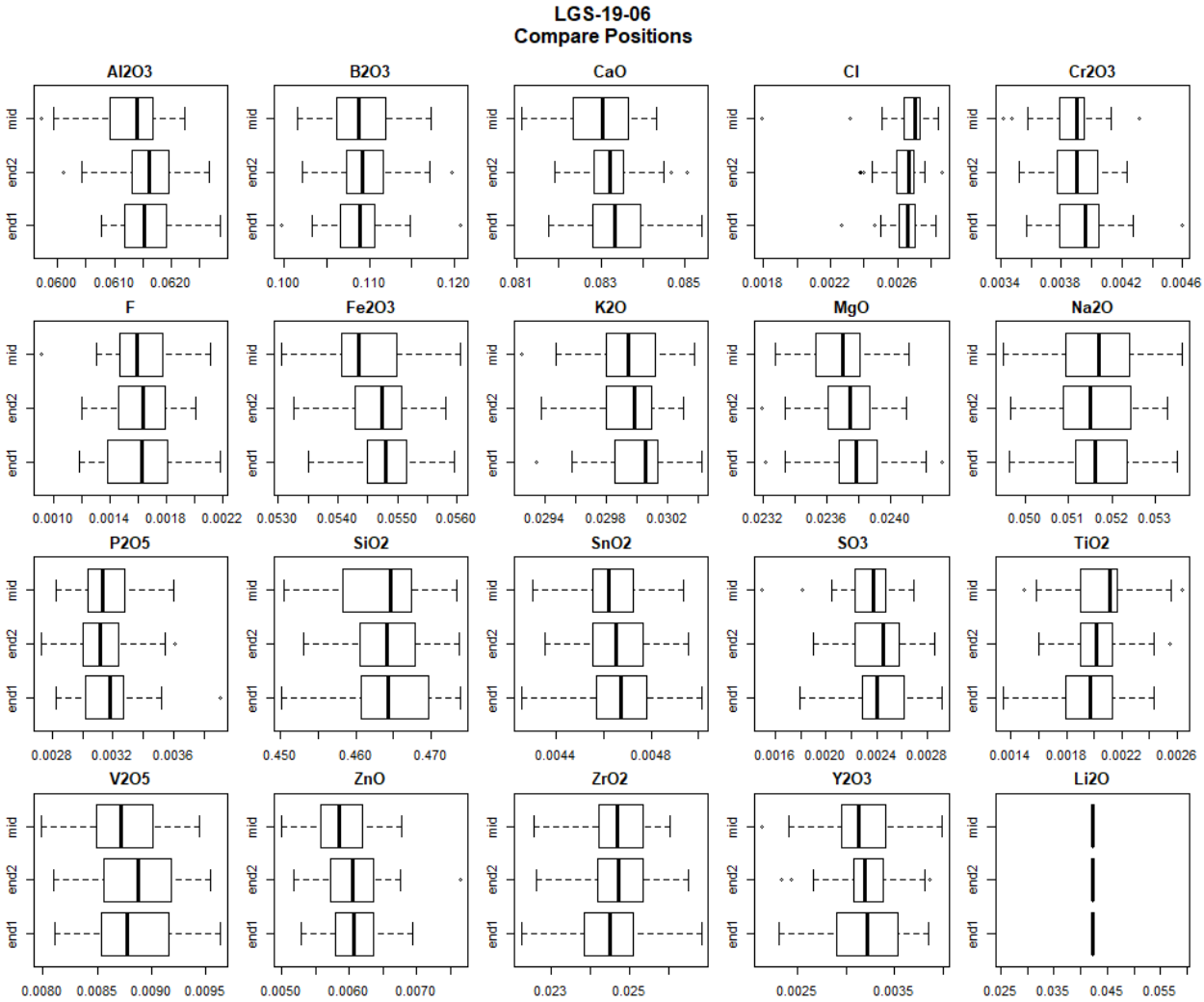

Figure A.47. Component distribution plots for LGS19-06 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-06] "Al2O3"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	18.2461	0	100	0.0006	0.9683		
2	Bar	4	0	0	0	39.4724	0.0004	0.6084
3	Bar:Position	10	0	0	0	12.9036	0.0002	0.3478
4	error	120	0	0	0	47.624	0.0004	0.6683

Mean: 0.0615 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-06] "B2O3"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	52.6963	0	100	0.0039	3.5727		
2	Bar	4	0.0004	0.0001	0	21.2046	0.0018	1.6452
3	Bar:Position	10	0.0001	0	0*	0*	0*	0*
4	error	120	0.0014	0	0	78.7954	0.0035	3.1714

Mean: 0.1092 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-06] "CaO"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	26.389	0	100	0.0009	1.0225		
2	Bar	4	0	0	0	31.8566	0.0005	0.5771
3	Bar:Position	10	0	0	0	9.3383	0.0003	0.3125
4	error	120	0.0001	0	0	58.8051	0.0007	0.7841

Mean: 0.0832 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-06] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	63.8082	0	100	0.0006	1.1665		
2 Bar	4	0	0	0	16.1258	0.0003	0.4684
3 Bar:Position	10	0	0	0	5.0234	0.0001	0.2615
4 error	120	0	0	0	78.8508	0.0006	1.0359

Mean: 0.0547 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-06] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	18.2468	0	100	0.001	1.8933		
2 Bar	4	0	0	0	43.764	0.0006	1.2525
3 Bar:Position	10	0	0	0*	0*	0*	0*
4 error	120	0.0001	0	0	56.236	0.0007	1.4198

Mean: 0.0516 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 | adapted MS used for total DF

[LGS-19-06] "SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	10.5756	0	100	0.0061	1.3106		
2 Bar	4	0.0024	0.0006	0	59.1232	0.0047	1.0077
3 Bar:Position	10	0.0002	0	0	1.6214	0.0008	0.1669
4 error	120	0.0017	0	0	39.2555	0.0038	0.8211

Mean: 0.4637 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-06] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	133.0493	0	100	0.0002	3.3374		
2	Bar	4	0	0	0.7475	0	0.2885	
3	Bar:Position	10	0	0	0*	0*	0*	0*
4	error	120	0	0	0	99.2525	0.0002	3.3249

Mean: 0.0047 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

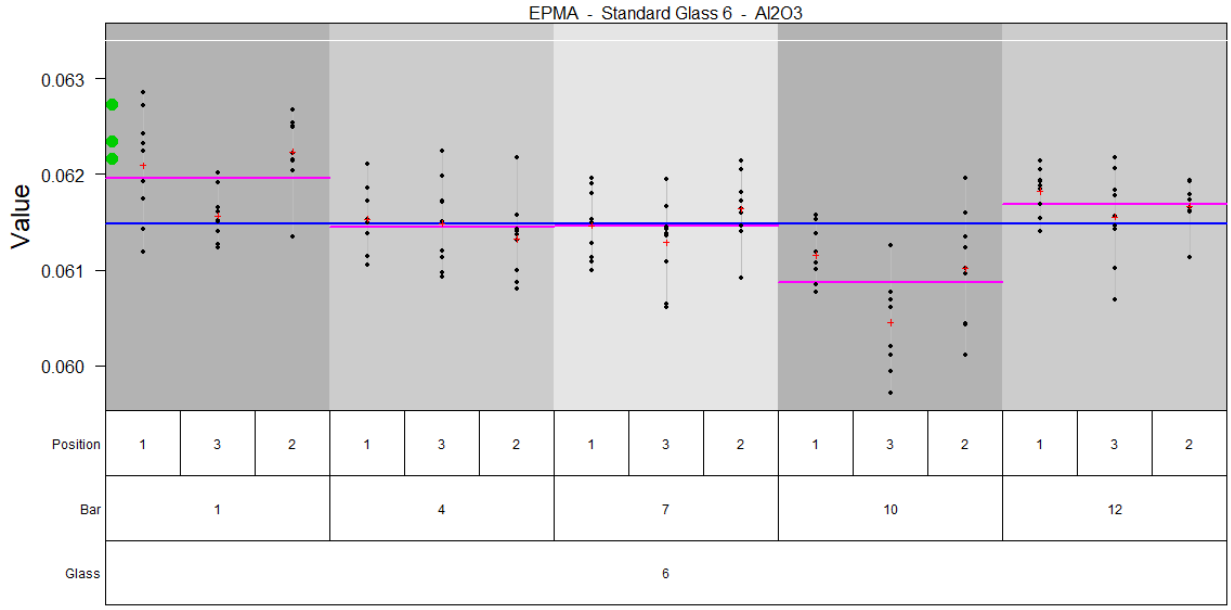

Figure A.48. Measured Al_2O_3 concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

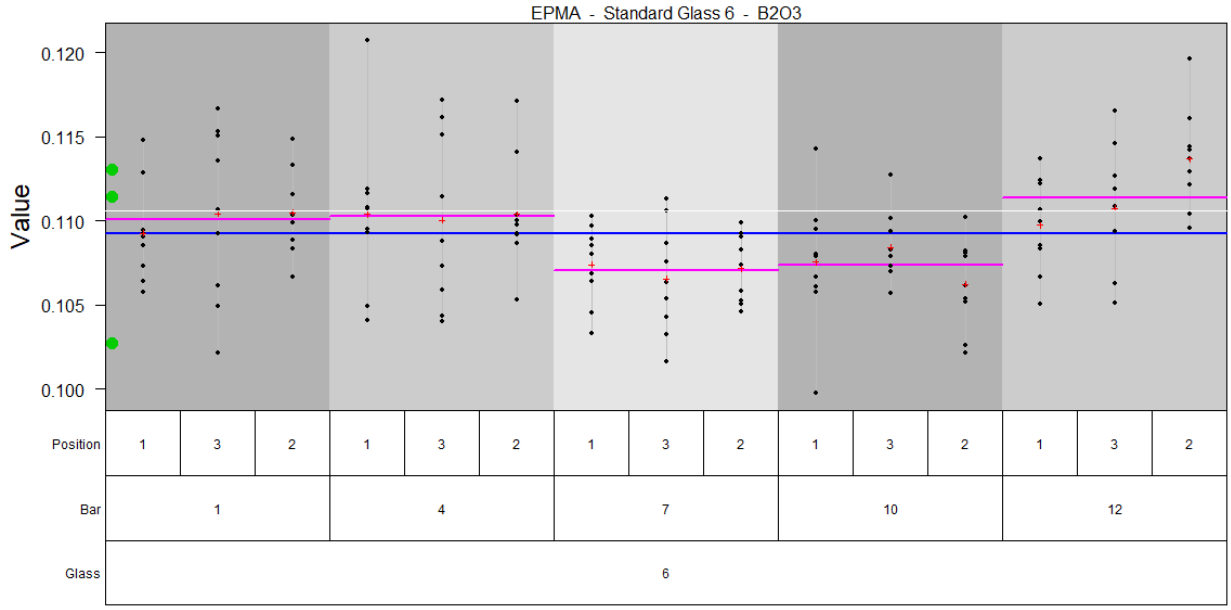

Figure A.49. Measured B_2O_3 concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

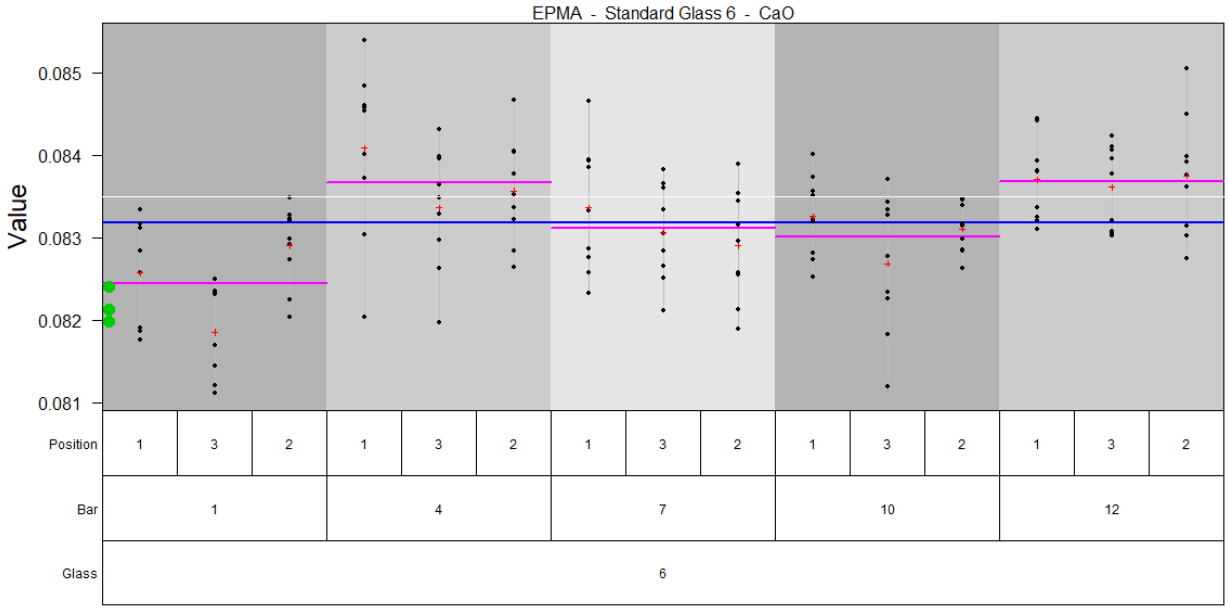

Figure A.50. Measured CaO concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

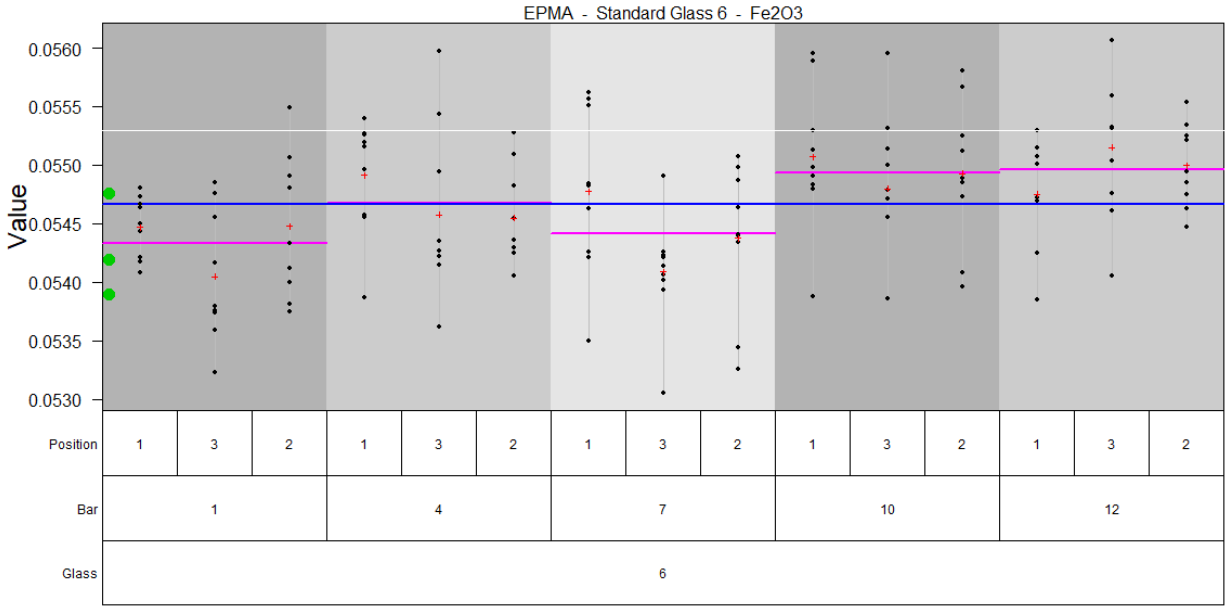

Figure A.51. Measured Fe_2O_3 concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

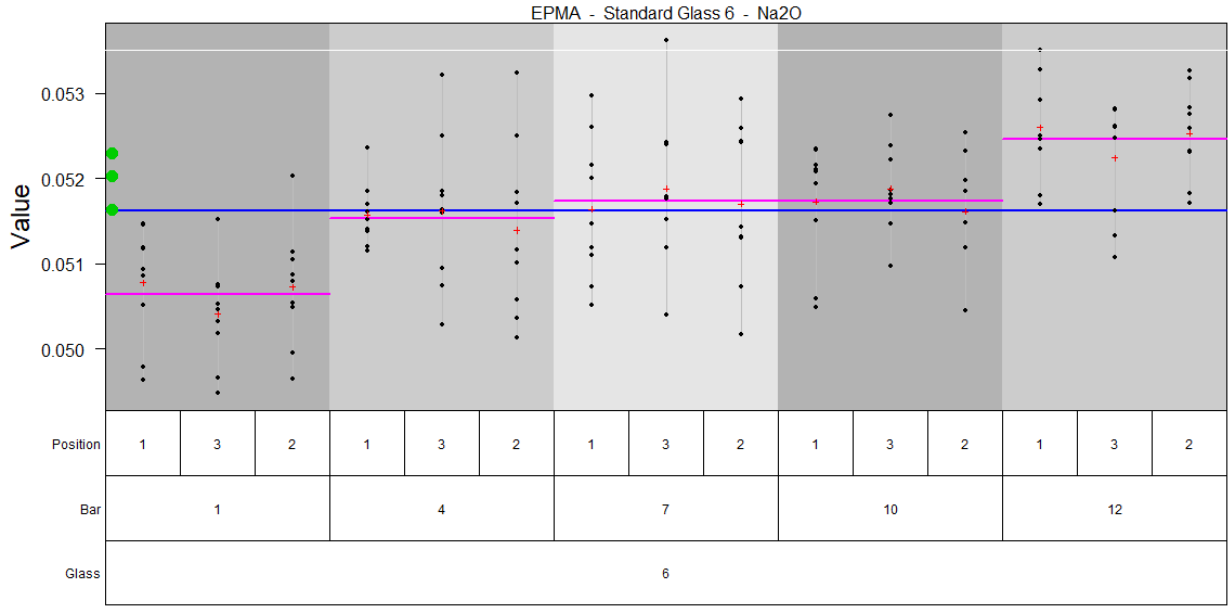

Figure A.52. Measured Na_2O concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

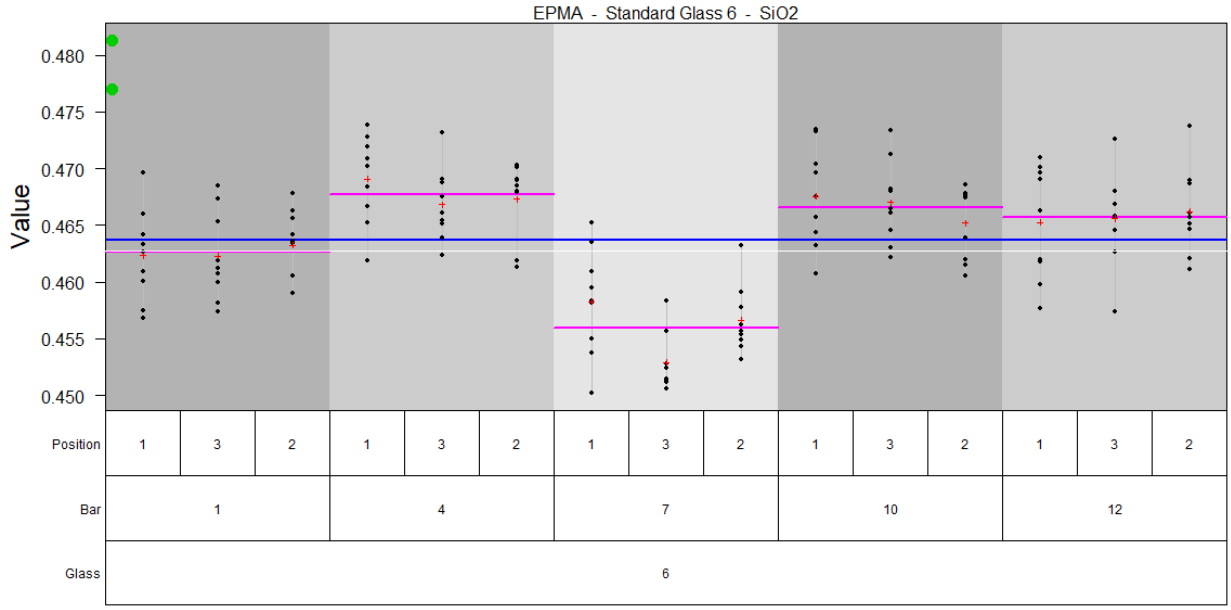

Figure A.53. Measured SiO_2 concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

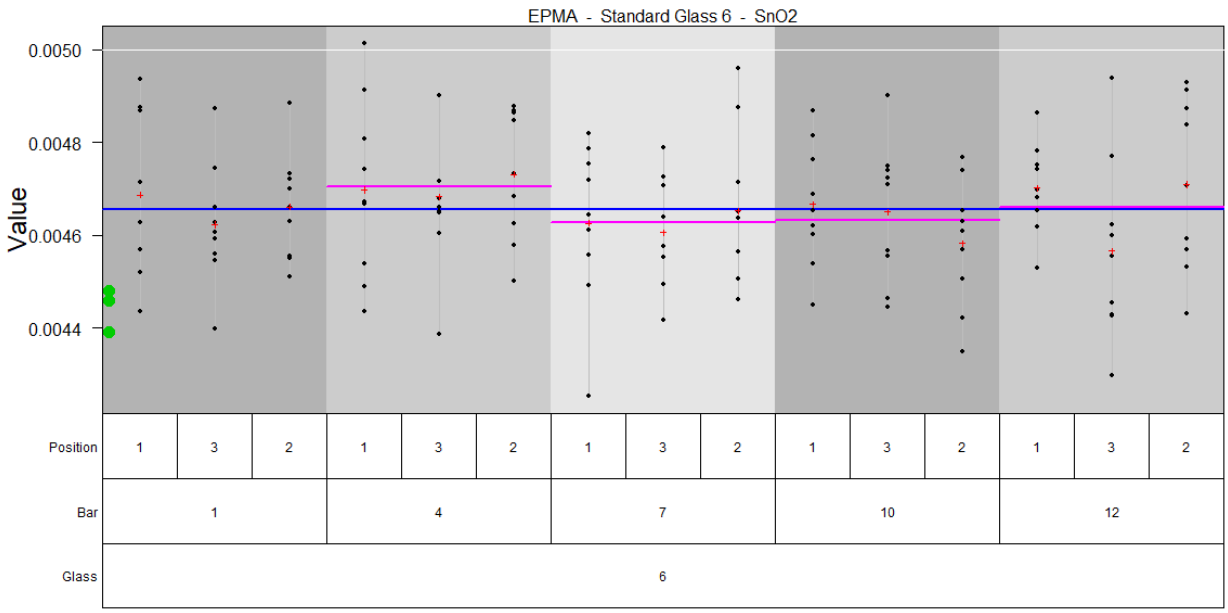

Figure A.54. Measured SnO_2 concentrations of LGS-19-06 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

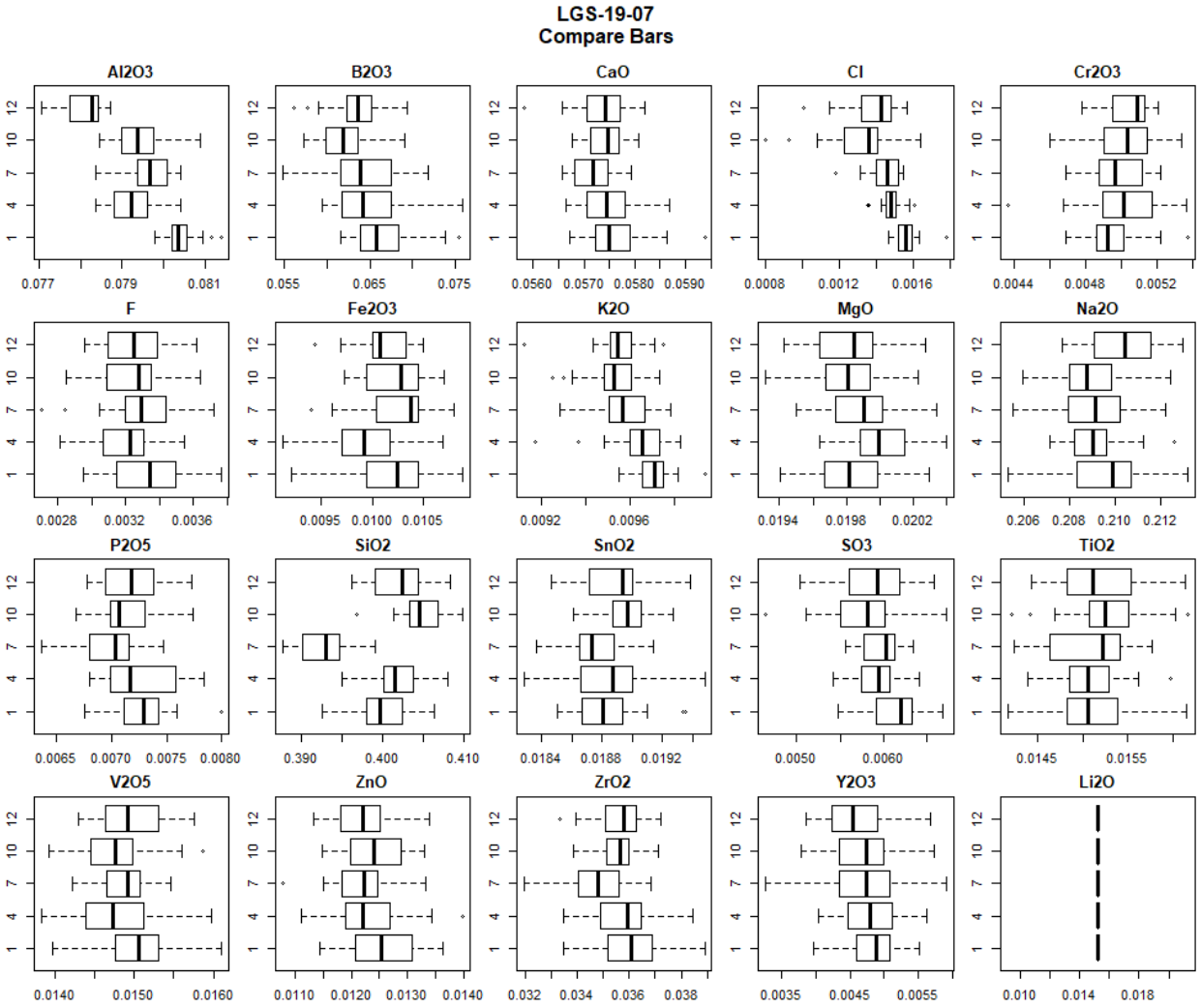

Figure A.55. Component distribution plots for LGS19-07 glass measured by EPMA, plotted by bar number on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

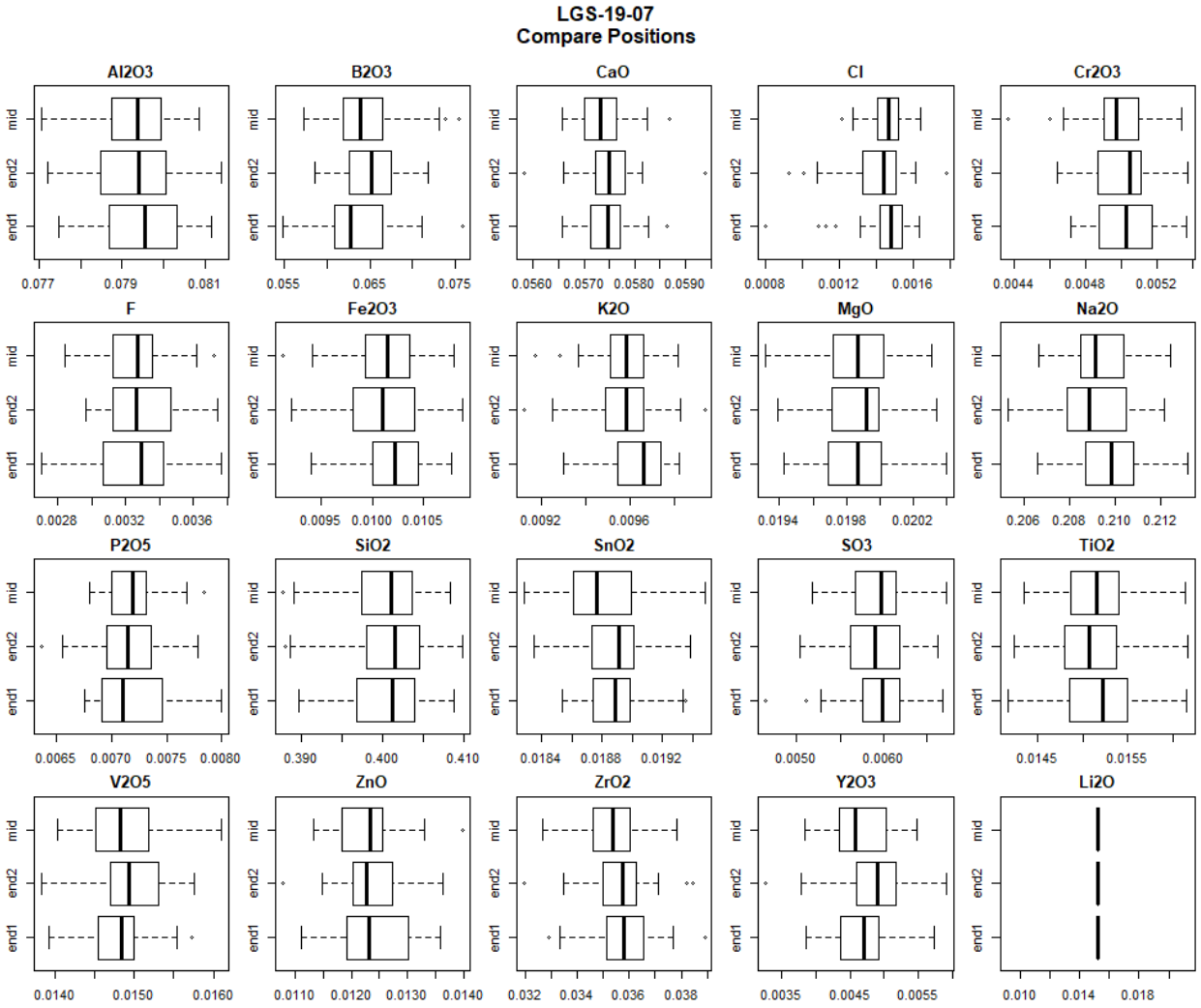

Figure A.56. Component distribution plots for LGS19-07 glass measured by EPMA, plotted by coupon position within bars (end 1, middle and end 2) on y-axis. Component concentration is on the x-axis, in mass fraction. The thick line inside the box is the median. The box shows 25% (left) and 75% (right) percentiles. The outer whiskers extend to the min and max of the dataset or the most extreme non-outlier values. Outliers are marked with a dot.

[LGS-19-07] "Al2O3"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	7.2507	0	100	0.001	1.2344		
2	Bar	4	0.0001	0	0	72.7725	0.0008	1.053
3	Bar:Position	10	0	0	0	1.1929	0.0001	0.1348
4	error	120	0	0	0	26.0346	0.0005	0.6299

Mean: 0.0794 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-07] "B2O3"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	92.9955	0	100	0.004	6.2819		
2	Bar	4	0.0003	0.0001	0	9.6976	0.0013	1.9562
3	Bar:Position	10	0.0002	0	0	3.8425	0.0008	1.2314
4	error	120	0.0017	0	0	86.4599	0.0038	5.8411

Mean: 0.0644 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-07] "CaO"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	110.6491	0	100	0.0005	0.8943		
2	Bar	4	0	0	0	7.372	0.0001	0.2428
3	Bar:Position	10	0	0	0*	0*	0*	0*
4	error	120	0	0	0	92.628	0.0005	0.8607

Mean: 0.0574 (N = 135)
Experimental Design: balanced | Method: ANOVA | * VC set to 0 |
adapted MS used for total DF

[LGS-19-07] "Fe2O3"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	109.1925	0	100	0.0003	3.3694		
2 Bar	4	0	0	0	7.1523	0.0001	0.9011
3 Bar:Position	10	0	0	0	1.6186	0	0.4287
4 error	120	0	0	0	91.2291	0.0003	3.2183

Mean: 0.0101 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-07] "Na2O"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	98.7327	0	100	0.0016	0.7818		
2 Bar	4	0	0	0	3.6282	0.0003	0.1489
3 Bar:Position	10	0.0001	0	0	14.2471	0.0006	0.2951
4 error	120	0.0003	0	0	82.1247	0.0015	0.7084

Mean: 0.2094 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-07] "SiO2"

Result Variance Component Analysis:

Name	DF	SS	MS	VC	%Total	SD	CV[%]
1 total	8.3379	0	100	0.0056	1.3889		
2 Bar	4	0.0023	0.0006	0	66.2652	0.0045	1.1306
3 Bar:Position	10	0.0002	0	0	5.0769	0.0013	0.313
4 error	120	0.0011	0	0	28.6579	0.003	0.7435

Mean: 0.4003 (N = 135)
Experimental Design: balanced | Method: ANOVA

[LGS-19-07] "SnO2"

Result Variance Component Analysis:

	Name	DF	SS	MS	VC	%Total	SD	CV[%]
1	total	107.7785	0	100	0.0002	1.2147		
2	Bar	4	0	0	0	4.7273	0	0.2641
3	Bar:Position	10	0	0	0	8.2631	0.0001	0.3492
4	error	120	0	0	0	87.0096	0.0002	1.133

Mean: 0.0189 (N = 135)
Experimental Design: balanced | Method: ANOVA

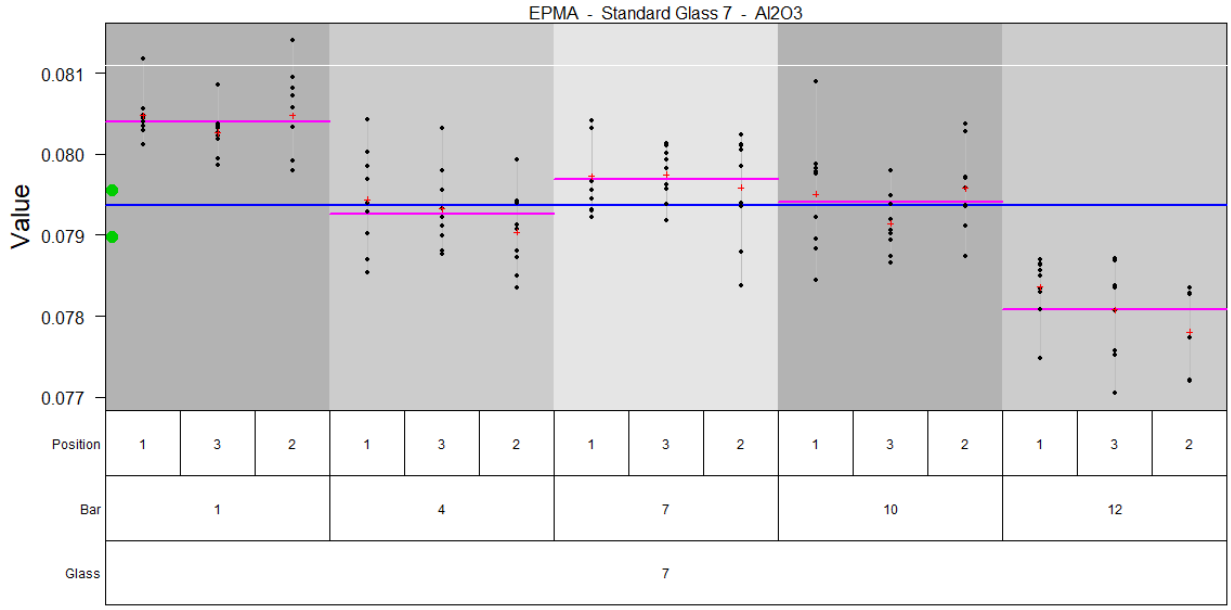

Figure A.57. Measured Al_2O_3 concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

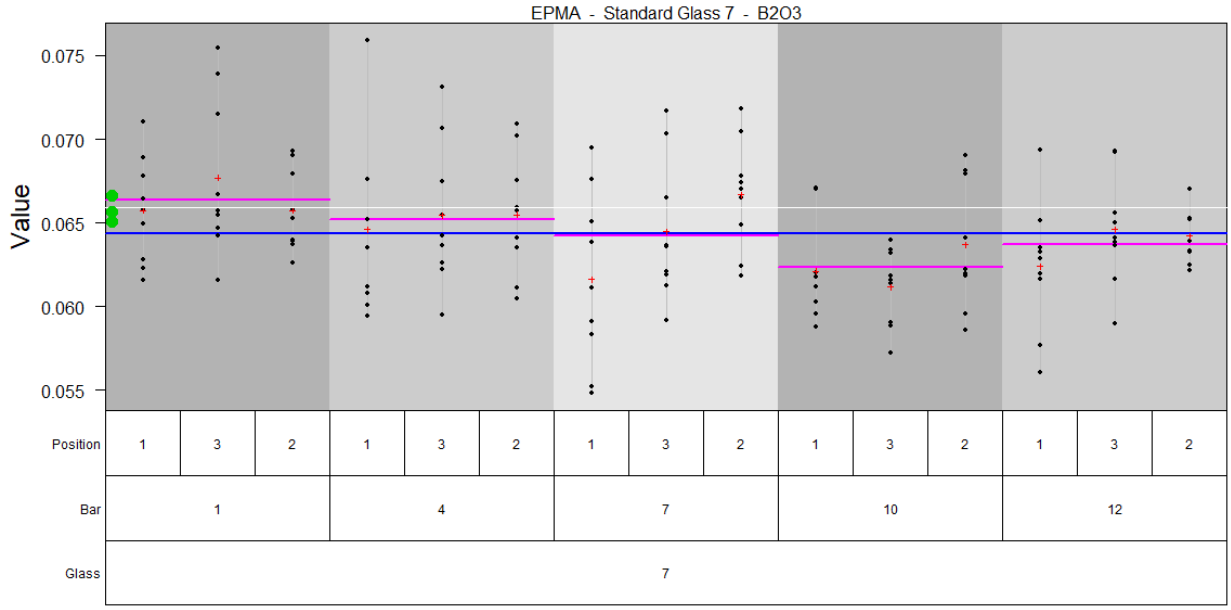

Figure A.58. Measured B_2O_3 concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

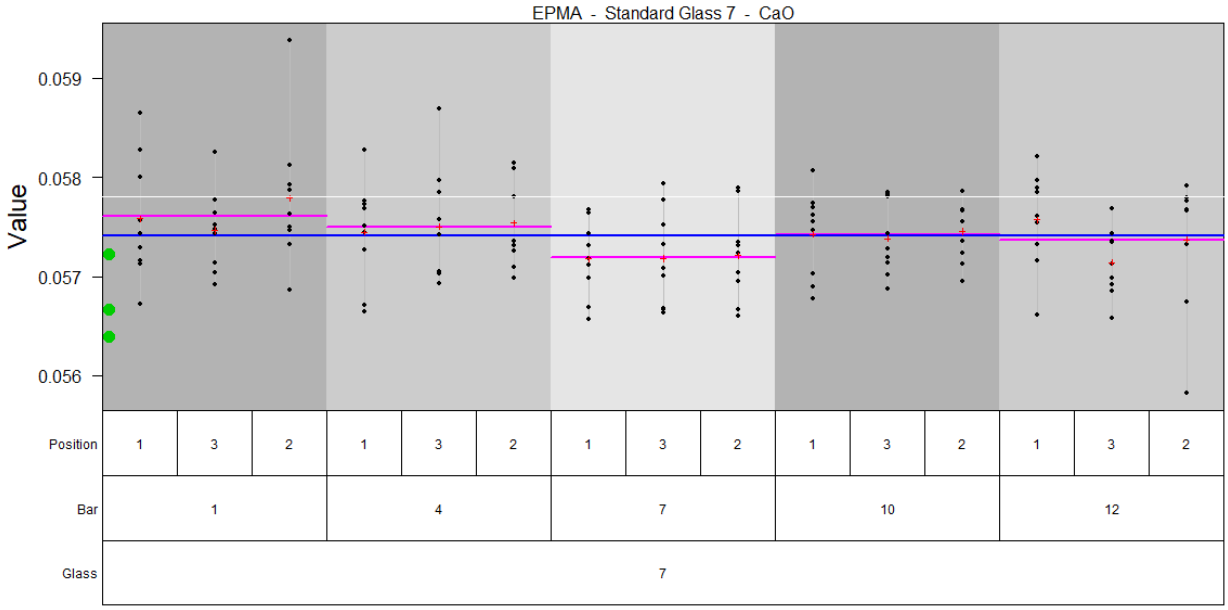

Figure A.59. Measured CaO concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

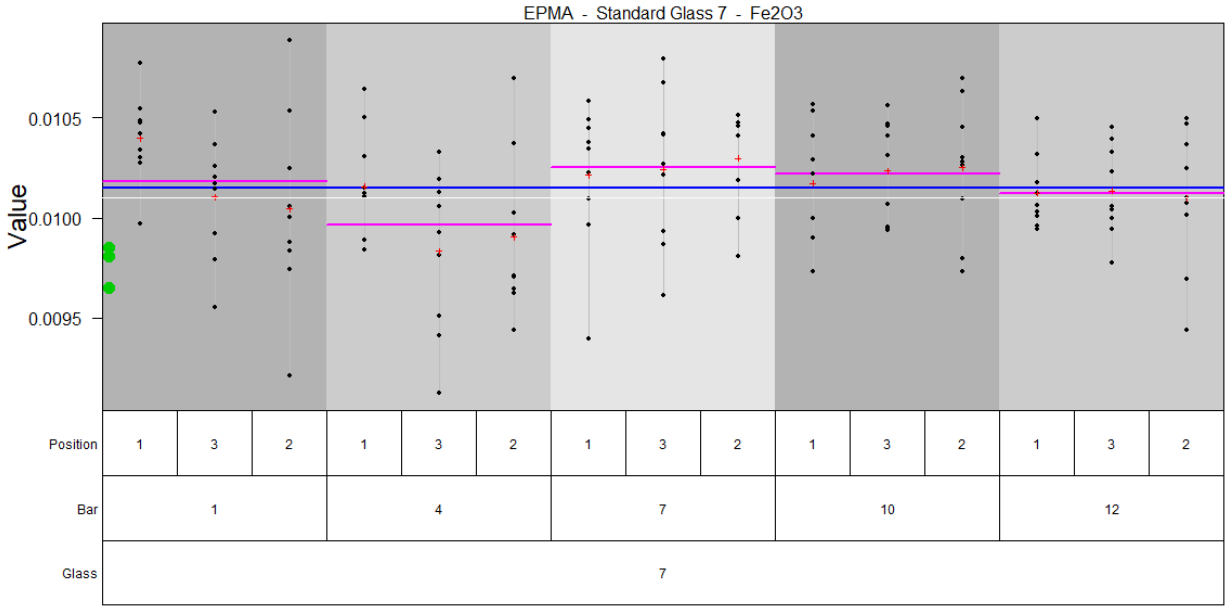

Figure A.60. Measured Fe_2O_3 concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

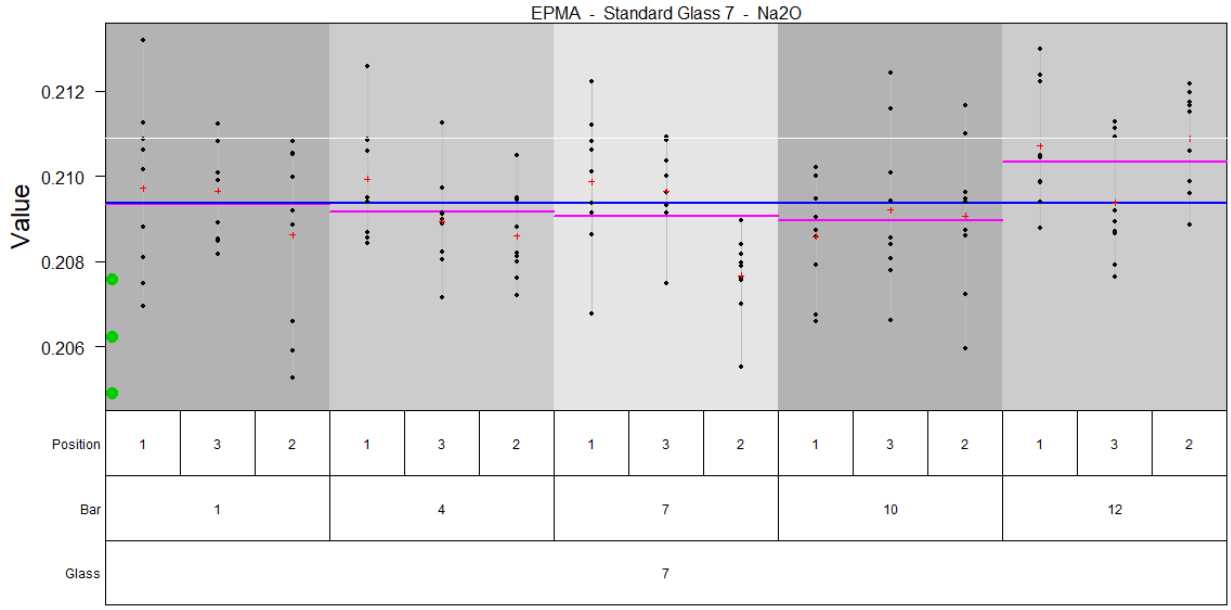

Figure A.61. Measured Na_2O concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

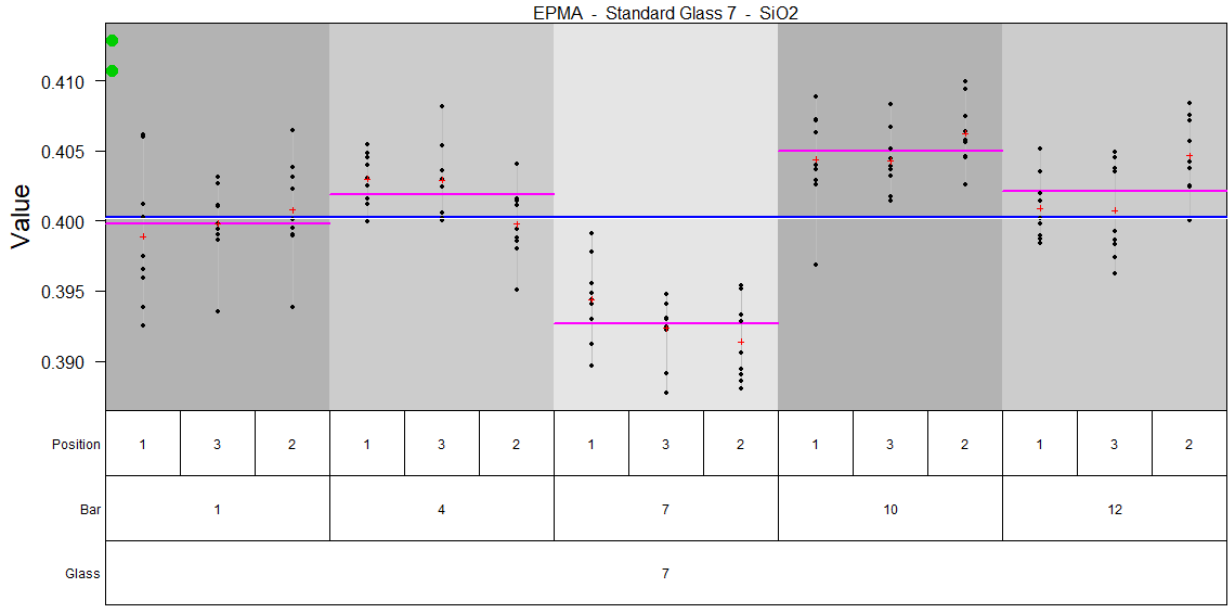

Figure A.62. Measured SiO_2 concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

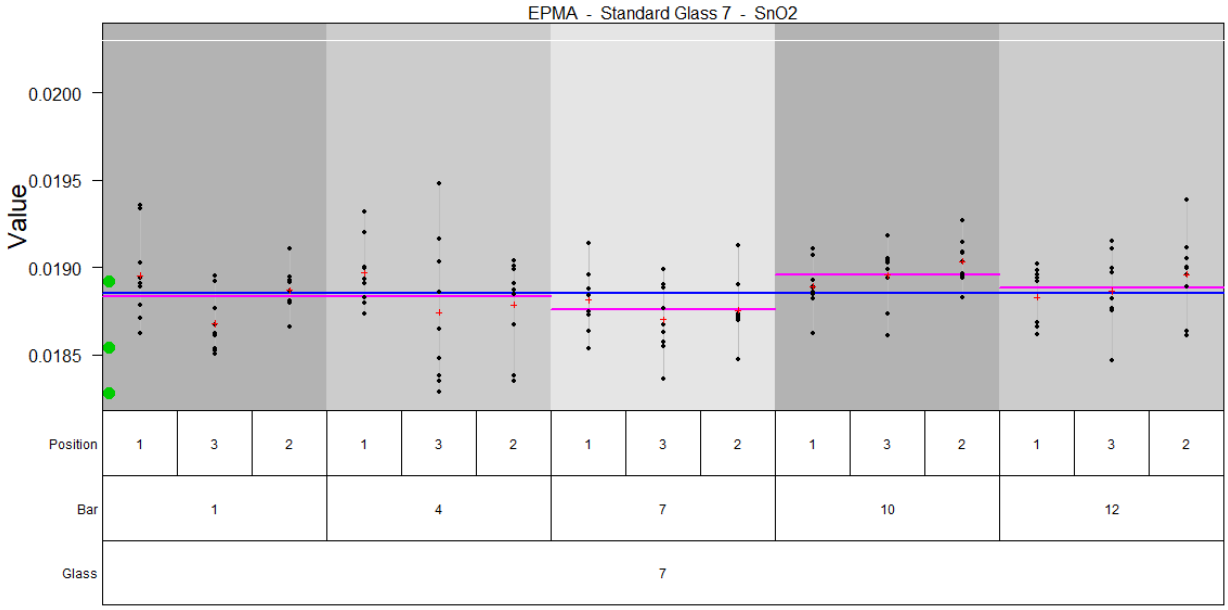

Figure A.63. Measured SnO_2 concentrations of LGS-19-07 glass plotted (y-axis) versus coupon location and bar number, where positions 1 and 2 are end coupons and 3 is the middle coupon. Black data points are individual measurements by EPMA and green points are ICP. Pink lines are the mean value of each bar analyzed, blue line is the mean of all bars analyzed, and the white bar is the batched concentration.

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