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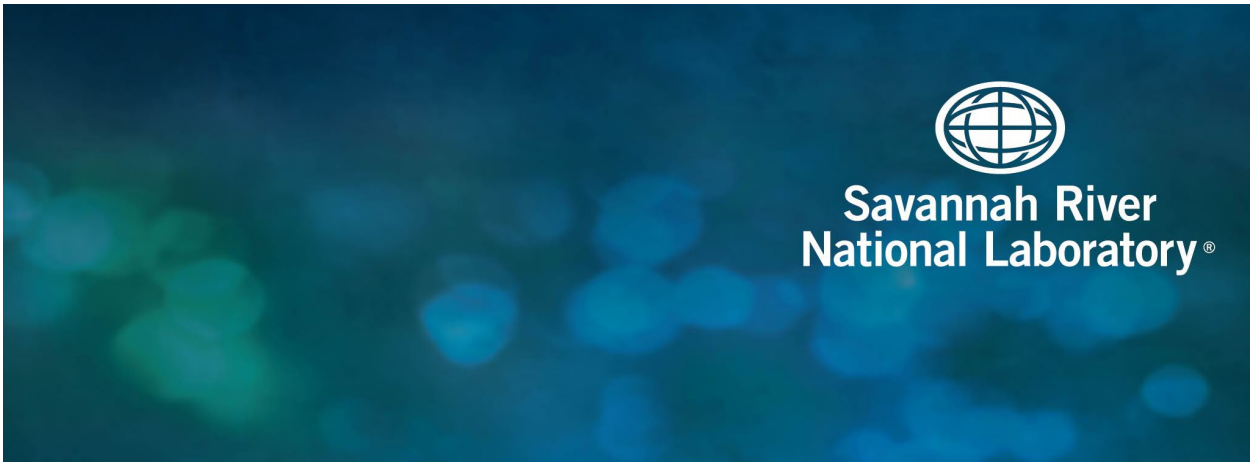

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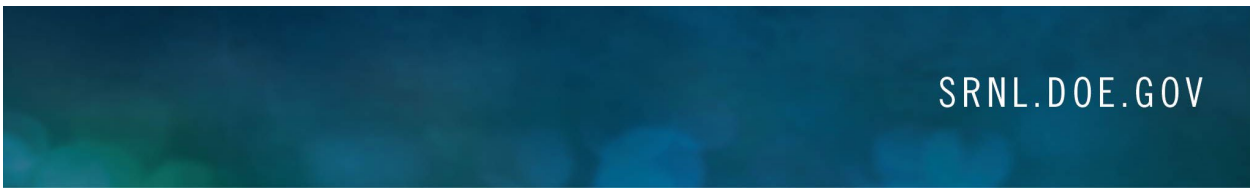
Characterization of the Sulfur-Saturated Melt Version of the EMHQ-LBE-04-B Glass

M. C. Hsieh

October 2022

SRNL-STI-2022-00366, Revision 0



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REVIEWS AND APPROVALS

AUTHORS:

M. C. Hsieh, Applied Materials Research Date

TECHNICAL REVIEW:

A. N. Stanfield, Applied Materials Research, Reviewed per E7 2.60 Date

APPROVAL:

J. Manna, Director, Materials Technology & Energy Sciences Division Date

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EXECUTIVE SUMMARY

This report provides the results from the chemical analyses of the sulfur-saturated melt version of the EMHQ-LBE-04-B glass. These data will be used in the development, validation, and implementation of enhanced property/composition models for waste glass vitrification at Hanford.

Chemical analyses were performed on a representative sample of the sulfur-saturated melt version of the glass to allow for comparisons with the targeted composition as well as the measured composition of the quenched glass. The relative differences between the targeted and measured concentration of Na_2O and SO_3 in the glass were greater than ±10%.

The wash solutions contained mainly sodium and sulfate ions.

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LIST OF ABBREVIATIONS

BDL	below detection limit
DOE	United States Department of Energy
hp std	High Purity Standards ICP
IC	ion chromatography
ICP-OES	inductively couple plasma – optical emission spectroscopy
ID	identifier
KH	potassium hydroxide fusion
LAW	low-activity waste
LM	lithium metaborate fusion
LRM	low-activity test reference material
ORP	Office of River Protection
PF	sodium peroxide fusion
PNNL	Pacific Northwest National Laboratory
seq	sequence
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SSM	sulfur-saturated melt
std	High Purity Standards ICP multi-element custom solution SM-744-013
TTQAP	Task Technical and Quality Assurance Plan
wt.%	weight percent
WTP	Waste Treatment and Immobilization Plant

1.0 Introduction

The U.S. Department of Energy (DOE) is responsible for building the Hanford Waste Treatment and Immobilization Plant (WTP) at the Hanford site in Washington to remediate 56 million gallons of radioactive waste that is temporarily stored in 177 underground tanks. The Office of River Protection (ORP) has requested that the Savannah River National Laboratory (SRNL) contribute in areas of recognized capabilities and expertise for glass waste form development to support successful startup of the WTP.

Successful efforts have allowed for demonstration of greatly enhanced treatment efficiencies of those projected from the minimum requirements set forth in the WTP Contract[a]. Additional flexibility and expansion of the qualified glass forming region are the current focus.[1] SRNL support of this work is defined in the Task Technical and Quality Assurance Plan (TTQAP).[2]

This report provides results from the chemical analyses of the sulfur-saturated melt (SSM) version of the EMHQ Low-Activity Waste (LAW) study glasses, a series of simulated nuclear waste glasses designed and fabricated at Pacific Northwest National Laboratory (PNNL). The glasses were selected as part of a broader study of the influence of glass composition on chemical durability, sulfur retention, and other properties.[3] The resulting data will be used in the development, validation, and implementation of enhanced property/composition models for nuclear waste glasses.[1]

2.0 Experimental Procedure

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Savannah River Site (SRS) Manual E7, Procedure 2.60.[4] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011.[5] Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiment L6390-00441-04. The glasses provided by PNNL were designed and fabricated following a Task Plan.[1]

2.2 Glass Selected for Study

The baseline (quenched) glass compositions in this study were designed and fabricated by PNNL. Characterization of the baseline glasses were reported earlier.[6] Samples of the SSM version of the study glass, along with samples of the wash solution resulting from the preparation of the SSM glass, were received at SRNL for chemical composition analysis. The PNNL identifiers (IDs) for the glass sample and wash solutions and associated SRNL sample identifiers are listed in Table 2-1.

Table 2-1. Identifiers for the LAW EMHQ-LBE-04-B SSM Samples

Sample Type	PNNL ID	Lab ID
SSM Glass	EMHQ-LBE-04-B-SSM-S	S-14282
SSM Wash Solution	EMHQ-LBE-04-B-SSM-W	S-14281
SSM Wash Solution	EMHQ-LBE-SSM-W-110221	S-14280

2.3 Glass Composition Analysis

Three dissolution techniques (potassium hydroxide fusion (KH), lithium metaborate fusion (LM), and sodium peroxide fusion (PF)) were used for preparing the EMHQ-LBE-04-B-SSM-S glass sample for analysis.[7-9]

[a] Contract DE-AC27-01RV14136, as amended, U.S. Department of Energy, Richland, WA (2000).

The EMHQ-LBE-04-B-SSM-S sample was analyzed twice for each element of interest by inductively coupled plasma – optical emission spectroscopy (ICP-OES)[10] or ion chromatography (IC),[11] for a total of two measurements per element per glass. A sample of the low-activity test reference material (LRM) was included with the sample glass measurements. The LRM composition reported as the "Consensus Average" is used as the reference composition of this glass.[12] The preparation and measurement methods used for each of the reported glass components are listed in Table 2-2.

Table 2-2. Preparation and Measurement Methods used in Reporting the Analyte Concentrations of the EMHQ-LBE-04-B-SSM-S Study Glass and LRM Standard Glass

Analyte	Measurement Method	Preparation Method
Al	ICP-OES	LM
B	ICP-OES	PF
Ca	ICP-OES	PF
Cl	IC	KH
Cr	ICP-OES	LM
F	IC	KH
Fe	ICP-OES	LM
K	ICP-OES	LM
Li	ICP-OES	PF
Mg	ICP-OES	LM
Mn	ICP-OES	LM
Na	ICP-OES	LM
P	ICP-OES	LM
S	ICP-OES	LM
Si	ICP-OES	PF
Ti	ICP-OES	LM
V	ICP-OES	LM
Zr	ICP-OES	PF

2.4 Wash Solution Analysis

The PNNL collaborators supplied a wash solution resulting from the preparation of the EMHQ-LBE-04-B-SSM-S glass along with a solution having a known composition. The samples were diluted at SRNL based on the expected concentrations of the species in solution in preparation for the analyses.

Each of the samples was analyzed in triplicate for each element of interest by ICP-OES[10] and IC[11]. Solution standards[b] and blanks were also intermittently measured to assess the performance of the ICP-OES and IC instruments over the course of these analyses. The measurement methods used for each of the reported wash solution components are listed in Table 2-3.

[b] ICP multi-element custom solutions, product numbers SM-744-013 and SM-744-063, High Purity Standards, North Charleston, SC.

Table 2-3. Measurement Methods Used in Reporting the Analyte Concentrations of the Wash Solution Samples

Analyte	Measurement Method
Al	ICP-OES
B	ICP-OES
Ca	ICP-OES
Cl^-	IC
Cr	ICP-OES
F^-	IC
Fe	ICP-OES
K	ICP-OES
Li	ICP-OES
Mg	ICP-OES
Mn	ICP-OES
Na	ICP-OES
P	ICP-OES
PO_4^{3-}	IC
S	ICP-OES
SO_4^{2-}	IC
Si	ICP-OES
Ti	ICP-OES
V	ICP-OES
Zr	ICP-OES

3.0 Results and Discussion

JMP® Version 16.0.0 (SAS Institute, Inc.)[13] was used to support these analyses.

3.1 Review and Evaluation of the SSM Glass Composition Measurements

Table A-1, Table A-2, and Table A-3 in Appendix A provide the elemental concentration measurements in weight percent (wt.%) from glasses prepared using KH, LM, and PF dissolution methods, respectively. Elemental measurements for the samples of the LRM standard glass are also included in these tables of Appendix A.

3.1.1 Treatment of Detection Limits

The elemental concentrations in Table A-1, Table A-2, and Table A-3 in Appendix A were converted to oxide concentrations by multiplying the values of each element by the gravimetric factor for the corresponding oxide. A concentration measurement that was reported to be below the detection limit was set to the detection limit for the purposes of data review and calculating a sum of oxides for the glasses. Concentration measurements that were below the detection limit (BDL) are denoted with a less than symbol (<).

3.1.2 Composition Measurements by Glass Identifier

Exhibit A-1 in Appendix A provides plots of the oxide concentration measurements by the PNNL glass ID (including the LRM glass samples) by Lab ID grouped by target concentration. A review of the plots presented in these exhibits indicates the repeatability of the four individual values for each oxide for each glass. There were no indications of errors in preparation or measurement that had to be addressed in the treatment of the data.

3.1.3 Results for the LRM Standard Glass

Exhibit A-2 in Appendix A provides a comparison of the LRM results to their acceptability limits utilized by SRNL.[10] The review is in the form of plots of the measurements arranged by element, framed by upper and lower acceptability limits for the concentration of each element of interest. The results show that all measurements of the LRM elements of interest were within the acceptability limits during the execution of these analyses.

3.1.4 Measured versus Target Composition

All measurements in Table A-1, Table A-2, and Table A-3 in Appendix A were used in calculating oxide values, which were then averaged to determine a representative chemical composition for the EMHQ-LBE-04-B-SSM glass sample. A sum of oxides was computed based upon the averaged oxide values.

Table A-4 in Appendix A provides a summary for the average compositions, targeted compositions, and some associated differences and relative differences. The measured sum of oxides for the EMHQ-LBE-04-B-SSM and LRM glasses are 100% and 103%, respectively, indicating acceptable recovery of the glass components.[14] Entries in Table A-4 show the relative differences between the measured and targeted values for the analytes with measured and targeted values above 1 wt.%. The relative differences are shaded if they are 10% or more and are summarized below.

- The Na_2O relative difference was 37%.
- The SO_3 relative difference was 48%.

3.2 Comparison of the Measured Compositions of the Baseline and SSM Glasses

Exhibit A-3 in Appendix A provides a comparison of the measured oxide concentrations among the baseline (quenched) and SSM version of EMHQ-LBE-04-B. A review of Exhibit A-3 led to the following observation:

- The measured SO_3 concentrations were higher in the SSM version of the study glass, as expected, due to the use of the sulfur saturation method in fabricating the glass.

The discussion of the analyses of the wash solutions, provided in Section 3.3, may provide further insight into the measured composition of the SSM glass.

3.3 Review and Evaluation of the Wash Solution Measurements

Table B-1 in Appendix B provides the elemental concentration measurements in mg/L for the wash solutions as measured by ICP-OES. Table B-2 in Appendix B provides the anion concentration measurements in mg/L for the wash solutions as measured by IC. Elemental measurements of the blanks and standard solutions are included in these tables.

3.3.1 Treatment of Detection Limits

The elemental and anion concentrations in Table B-1 and Table B-2 in Appendix B include measurements that were reported to be below the detection limit. These values were set to the detection limit for the purposes of data review and for calculating an average composition for each wash solution.

3.3.2 Composition Measurements by Wash Solution Identifier

Exhibit B-1 in Appendix B provides plots of the elemental and anion concentration measurements grouped by the wash solution identifier (including the blanks and standard solutions). Different symbols and colors are used to represent the different solutions. Plotting the data in this format provides an opportunity to review the values for each individual solution as a function of the triplicate measurements. A review of the plots presented in these exhibits reveals the repeatability of the three individual values for each analyte for each solution. These observations were not considered to indicate an error in preparation or measurement

that had to be addressed in the treatment of the data. Therefore, the entire set of measurement data was used in determining representative, measured compositions for the wash solutions.

3.3.3 Results for the Standard Solutions

Table B-3 in Appendix B provides comparisons of the standard solution results to their reference values. The results in the table indicate no issues with the performance of the analyses.

3.3.4 Measured Compositions of the Wash Solutions

From the discussion of Section 3.3.2, all the measurements for each analyte for each wash solution (Table B-1 and Table B-2 in Appendix B) were averaged to determine a representative chemical composition for each solution. Table B-4 in Appendix B provides a summary of the average measured compositions of the wash solutions. The results in Table B-4 are summarized below.

- The EMHQ-LBE-04-B-SSM-W wash solution contained mainly Na (182 mg/L), S (119 mg/L), and SO4 (297 mg/L).
- The EMHQ-LBE-SSM-W-110221 wash solution contained mainly Na (540 mg/L), S (379 mg/L), and SO4 (691 mg/L).
- The measured concentrations of B, Ca, Li, Si, and V were below 100 mg/L in the EMHQ-LBE-04-B-SSM-W wash solution.
- The measured concentrations of Cl-, F-, PO4, Al, Cr, Fe, K, Mg, Mn, P, Ti, and Zr were below detection limits for both solutions.

4.0 Summary

This report provides the results from the chemical analyses of the sulfur-saturated melt version of the EMHQ-LBE-04-B glass. These data will be used in the development, validation, and implementation of enhanced property/composition models for waste glass vitrification at Hanford.

Chemical analyses were performed on a representative sample of the sulfur-saturated melt version of the glass to allow for comparisons with the targeted composition, as well as the measured composition of the quenched glass. The relative differences between the targeted and measured concentration of Na_2O and SO_3 in the glass were greater than ±10%.

The wash solutions contained mainly sodium and sulfate ions.

5.0 References

1. R.L. Russell, "EMHQ LAW Glass Composition Boundary Expansion," Pacific Northwest National Laboratory, Richland, WA, EWG-TP-169, Revision 0.0, 2021.

2. K.M. Fox, "Task Technical and Quality Assurance Plan for Hanford Waste Glass Development and Characterization," Savannah River National Laboratory, Aiken, SC, SRNL-RP-2013-00692, Revision 1, 2016.

3. D.K. Peeler, D.S. Kim, J.D. Vienna, M.J. Schweiger, and G.F. Piepel, "Office of River Protection Advanced Low-Activity Waste Glass Research and Development Plan," Pacific Northwest National Laboratory, Richland, WA, PNNL-24883, EWG-RPT-008, Revision 0, 2015.

4. "Technical Reviews," Savannah River Site, Aiken, SC, Manual E7, Procedure 2.60, Rev. 20, 2021.

5. "Savannah River National Laboratory Technical Report Design Check Guidelines," Westinghouse Savannah River Company, Aiken, SC, WSRC-IM-2002-00011, Rev. 2, 2004.

6. M.C. Hsieh, "Composition Measurements and Product Consistency Test Results for the EMHQ-LBE-04-B Glass," Savannah River National Laboratory, Aiken, SC, SRNL-STI-2022-00118, Revision 0, 2022.

7. "Sample Dissolution Using Potassium Hydroxide Fusion," Savannah River National Laboratory, Aiken, SC, Manual L29, Procedure ITS-0035, Rev. 3, 2015.

8. "Lithium Metaborate Fusion Preparation," Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0071, Rev. 0, 2021.

9. "Dissolution of Glass, Sludge, and Slurry Samples Using Na_2O_2/NaOH/HCl," Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0040, Rev. 0, 2022.

10. "Calibration, Verification, and Operation of the Agilent 5110 ICP-OES Inductively Coupled Plasma-Optical Emission Spectrometer," Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0242, Rev. 1, 2021.

11. "Anion Analysis Using the Dionex ICS 6000 Ion Chromatograph," Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0244, Rev. 1, 2020.

12. W.L. Ebert and S.F. Wolf, "Round-Robin Testing of a Reference Glass for Low-Activity Waste Forms," Argonne National Laboratory, Argonne, IL, ANL-99/22, Revision 0, 1999.

13. JMP(R) Version 16.0.0, SAS Institute Inc., Cary, NC, 2021.

14. C.M. Jantzen, "Verification of Glass Composition and Strategy for SGM and DWPF Glass Composition Determination," E. I du Pont de Nemours & Co., Savannah River Laboratory, Aiken, SC, DPST-86-708, 1987.

Appendix A. Tables and Exhibits Supporting the Composition Measurements of EMHQ-LBE-04-B-SSM-S

Table A-1. KH Measurements (wt.%) of the LRM and EMHQ-LBE-04-B-SSM-S Glasses

PNNL ID	Block	Seq	Lab ID	Cl-	F-
1ppm ckstd	1	1	1ppm ckstd	1.03	0.989
LRM-SSM	1	2	LRMKH111	<0.0250	0.902
EMHQ-LBE-04-B-SSM-S	1	3	S-14282KH11	0.0428	0.0359
LRM-SSM	1	4	LRMKH112	<0.0250	0.902
EMHQ-LBE-04-B-SSM-S	1	5	S-14282KH21	0.0380	0.0376
LRM-SSM	1	6	LRMKH113	<0.0250	0.898
1ppm ckstd	1	7	1ppm ckstd	1.01	0.980
1ppm ckstd	2	1	1ppm ckstd	1.09	1.04
LRM-SSM	2	2	LRMKH121	<0.0250	0.898
EMHQ-LBE-04-B-SSM-S	2	3	S-14282KH12	0.0426	0.0434
LRM-SSM	2	4	LRMKH122	<0.0250	0.905
EMHQ-LBE-04-B-SSM-S	2	5	S-14282KH22	0.0442	0.0451
LRM-SSM	2	6	LRMKH123	<0.0250	0.906
1ppm ckstd	2	7	1ppm ckstd	0.994	1.02

Table A-2. LM Measurements (wt.%) for the study glass

PNNL ID	Block	Seq	Lab ID	Al	Cr	Fe	K	Mg	Mn	Na	P	S	Ti	V
LRM-SSM	1	1	LRMLM111	5.16	0.130	1.00	1.23	0.0659	0.0613	16.0	0.202	0.104	0.0622	<0.100
EMHQ-LBE-04-B-SSM-S	1	2	S-14282LM11	1.76	0.0261	0.0901	0.0505	0.117	<0.0100	10.2	<0.100	0.992	0.0455	2.11
LRM-SSM	1	3	LRMLM112	5.08	0.125	0.982	1.17	0.0694	0.0601	15.6	0.193	0.123	0.0614	<0.100
EMHQ-LBE-04-B-SSM-S	1	4	S-14282LM21	1.74	0.0258	0.0893	0.0470	0.116	<0.0100	9.70	<0.100	0.981	0.0451	2.07
LRM-SSM	1	5	LRMLM113	5.09	0.127	0.990	1.17	0.0781	0.0604	15.6	0.200	0.1481	0.0627	<0.100
LRM-SSM	2	1	LRMLM121	5.23	0.128	1.01	1.21	0.0737	0.0606	15.6	0.211	0.147	0.0623	<0.100
EMHQ-LBE-04-B-SSM-S	2	2	S-14282LM12	1.80	0.0309	0.0908	0.0509	0.122	<0.0100	10.2	<0.100	1.03	0.0477	2.14
LRM-SSM	2	3	LRMLM122	5.16	0.124	0.992	1.20	0.0733	0.0596	15.3	0.218	0.132	0.0613	<0.100
EMHQ-LBE-04-B-SSM-S	2	4	S-14282LM22	1.78	0.0298	0.0884	0.0484	0.118	<0.0100	9.70	<0.100	1.00	0.0465	2.08
LRM-SSM	2	5	LRMLM123	5.18	0.123	0.987	1.17	0.0746	0.0594	15.2	0.209	0.139	0.0615	<0.100

Table A-3. PF Measurements (wt.%) for the study glass

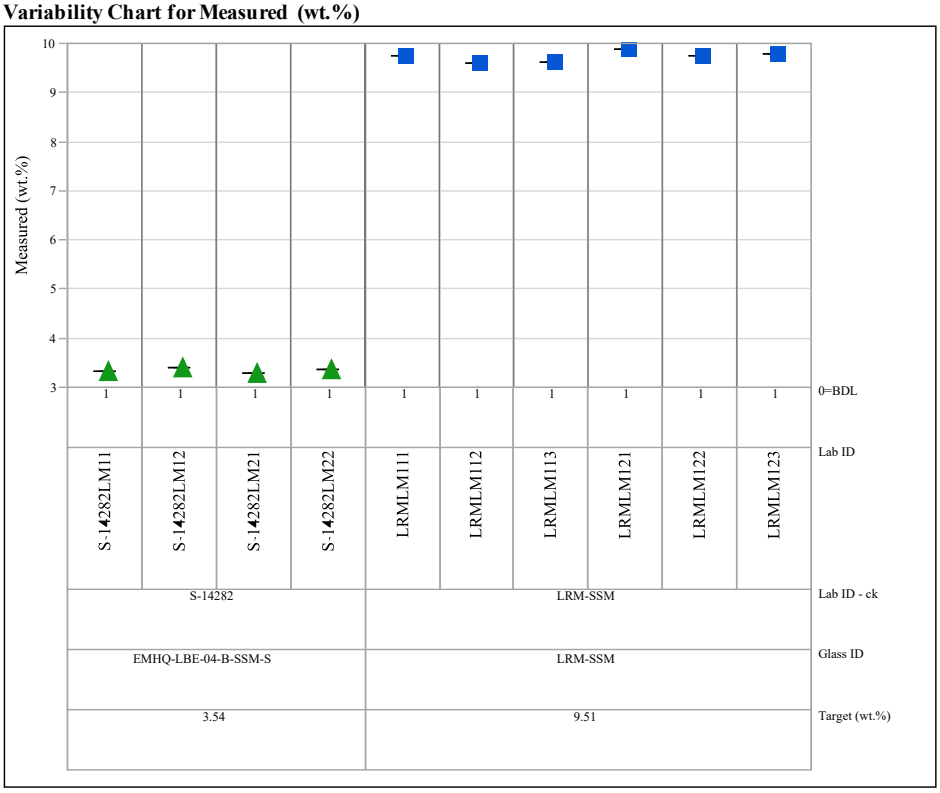
PNNL ID	Block	Seq	Lab ID	B	Ca	Li	Si	Zr
LRM-SSM	1	1	LRMPF111	2.46	0.452	<0.100	26.8	0.733
EMHQ-LBE-04-B-SSM-S	1	2	S-14282PF11	3.83	8.67	0.839	22.6	1.37
LRM-SSM	1	3	LRMPF112	2.44	0.409	<0.100	26.3	0.724
EMHQ-LBE-04-B-SSM-S	1	4	S-14282PF21	3.90	8.60	0.843	22.7	1.38
LRM-SSM	1	5	LRMPF113	2.43	0.456	<0.100	26.7	0.737
LRM-SSM	2	1	LRMPF121	2.43	0.431	<0.100	26.6	0.731
EMHQ-LBE-04-B-SSM-S	2	2	S-14282PF12	3.85	8.41	0.801	22.4	1.35
LRM-SSM	2	3	LRMPF122	2.42	0.436	<0.100	26.6	0.744
EMHQ-LBE-04-B-SSM-S	2	4	S-14282PF22	3.90	8.93	0.803	22.9	1.38
LRM-SSM	2	5	LRMPF123	2.44	0.428	<0.100	26.7	0.738

Table A-4. Comparison of Measured versus Target Compositions

Glass ID	Oxide	Measured (wt.%)	Target (wt.%)	Difference of Measured vs Target (wt.%)	% Difference Measured vs Target
EMHQ-LBE-04-B-SSM-S	Al_2O_3	3.34	3.54	-0.194	-5%
EMHQ-LBE-04-B-SSM-S	B_2O_3	12.5	13.5	-1.079	-8%
EMHQ-LBE-04-B-SSM-S	CaO	12.1	12.2	-0.133	-1%
EMHQ-LBE-04-B-SSM-S	Cl^-	0.0419	0.0760	-0.034	
EMHQ-LBE-04-B-SSM-S	Cr_2O_3	0.0411	0.0250	0.016	
EMHQ-LBE-04-B-SSM-S	F^-	0.0405	0.0950	-0.055	
EMHQ-LBE-04-B-SSM-S	Fe_2O_3	0.128	0.131	-0.003	
EMHQ-LBE-04-B-SSM-S	K_2O	0.0593	0.0870	-0.028	
EMHQ-LBE-04-B-SSM-S	Li_2O	1.77	1.67	0.104	6%
EMHQ-LBE-04-B-SSM-S	MgO	0.196	0.180	0.016	
EMHQ-LBE-04-B-SSM-S	MnO	<0.0129	0.0130	0	
EMHQ-LBE-04-B-SSM-S	Na_2O	13.4	9.81	3.607	37%
EMHQ-LBE-04-B-SSM-S	P_2O_5	<0.229	0.158	0.071	
EMHQ-LBE-04-B-SSM-S	SiO_2	48.5	49.6	-1.194	-2%
EMHQ-LBE-04-B-SSM-S	SO_3	2.50	1.69	0.814	48%
EMHQ-LBE-04-B-SSM-S	TiO_2	0.0771	0.0910	-0.014	
EMHQ-LBE-04-B-SSM-S	V_2O_5	3.75	4.03	-0.282	-7%
EMHQ-LBE-04-B-SSM-S	ZrO_2	1.85	2.00	-0.147	-7%
EMHQ-LBE-04-B-SSM-S	**Sum of Oxides**	**100**	**99.0**	**1.000**	**1%**
LRM-SSM	Al_2O_3	9.73	9.51	0.221	2%
LRM-SSM	B_2O_3	7.85	7.85	-0.004	0%
LRM-SSM	CaO	0.609	0.540	0.069	
LRM-SSM	Cl^-	<0.025	0	0.025	
LRM-SSM	Cr_2O_3	0.184	0.190	-0.006	
LRM-SSM	F^-	0.902	0.860	0.042	
LRM-SSM	Fe_2O_3	1.42	1.38	0.040	3%
LRM-SSM	K_2O	1.44	1.48	-0.045	-3%
LRM-SSM	Li_2O	<0.215	0.110	0.105	
LRM-SSM	MgO	0.120	0.100	0.020	
LRM-SSM	MnO	0.0778	0.0800	-0.002	
LRM-SSM	Na_2O	21.0	20.0	0.931	5%
LRM-SSM	P_2O_5	0.471	0.540	-0.069	
LRM-SSM	SiO_2	56.9	54.2	2.741	5%
LRM-SSM	SO_3	0.330	0.300	0.030	
LRM-SSM	TiO_2	0.103	0.100	0.003	
LRM-SSM	V_2O_5	<0.179	0	0.179	
LRM-SSM	ZrO_2	0.992	0.930	0.062	
LRM-SSM	**Sum of Oxides**	**103**	**98.2**	**4.800**	**5%**

Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration

Oxide=Al_2O_3, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=B_2O_3, Prep Method=PF
Variability Chart for Measured (wt.%)

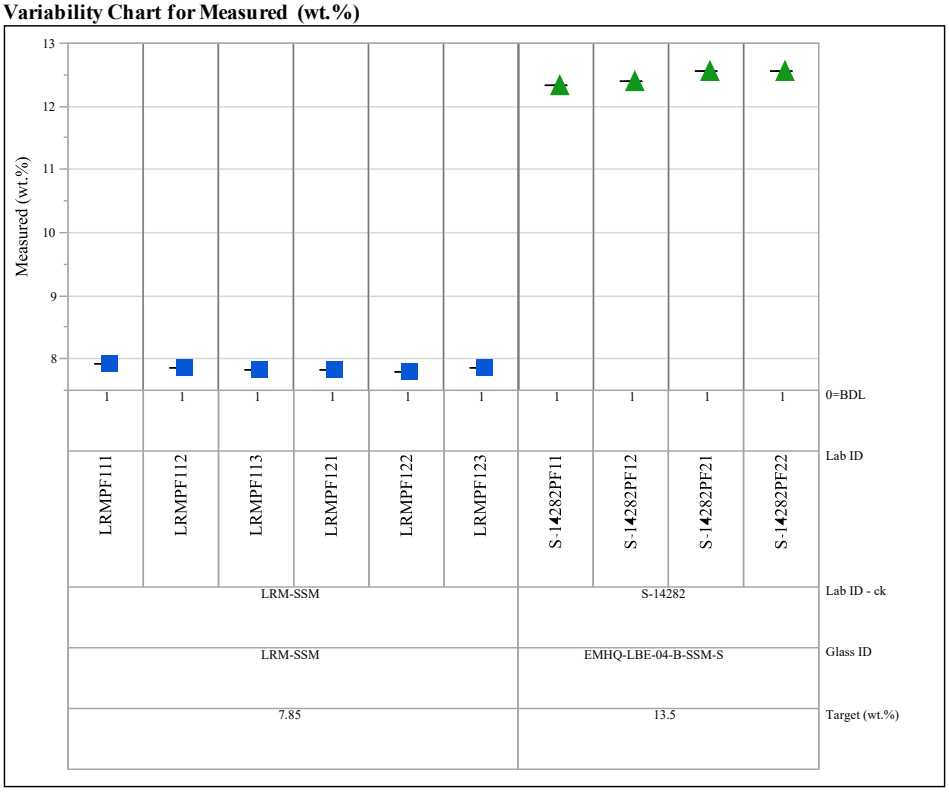
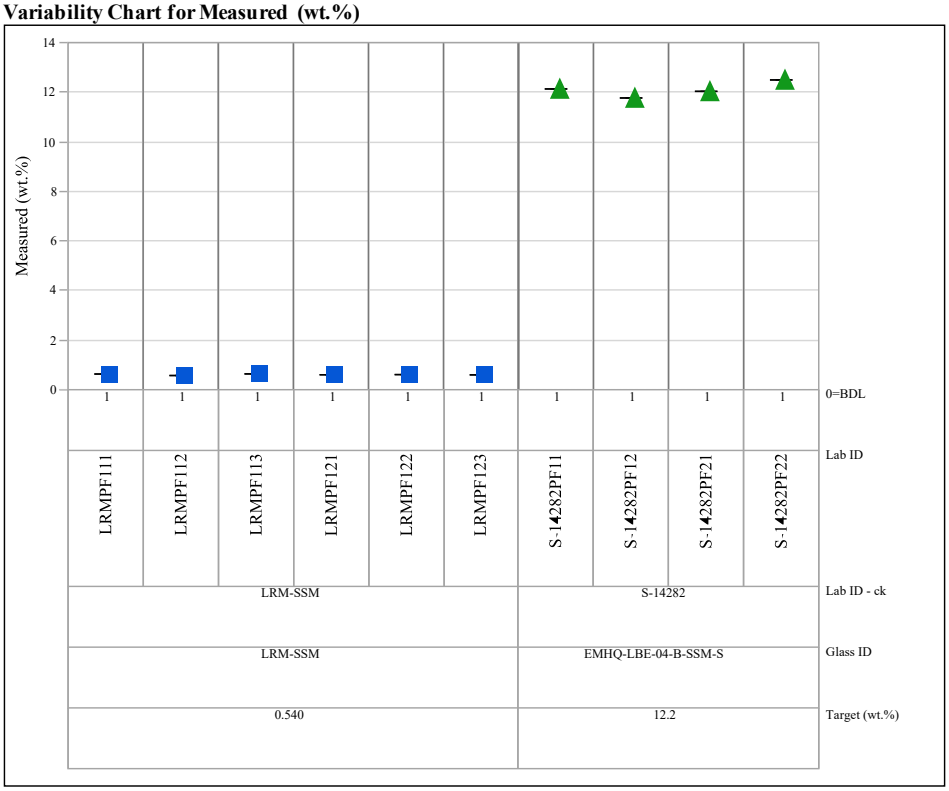


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=CaO, Prep Method=PF
Variability Chart for Measured (wt.%)



Oxide=Cl^-, Prep Method=KH
Variability Chart for Measured (wt.%)

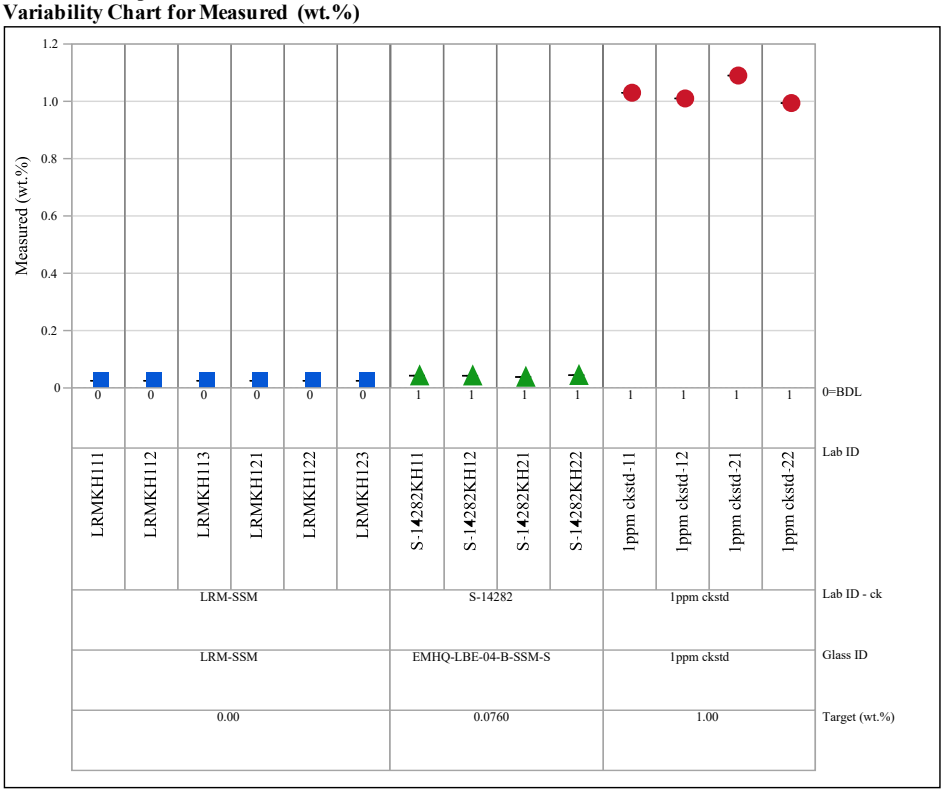
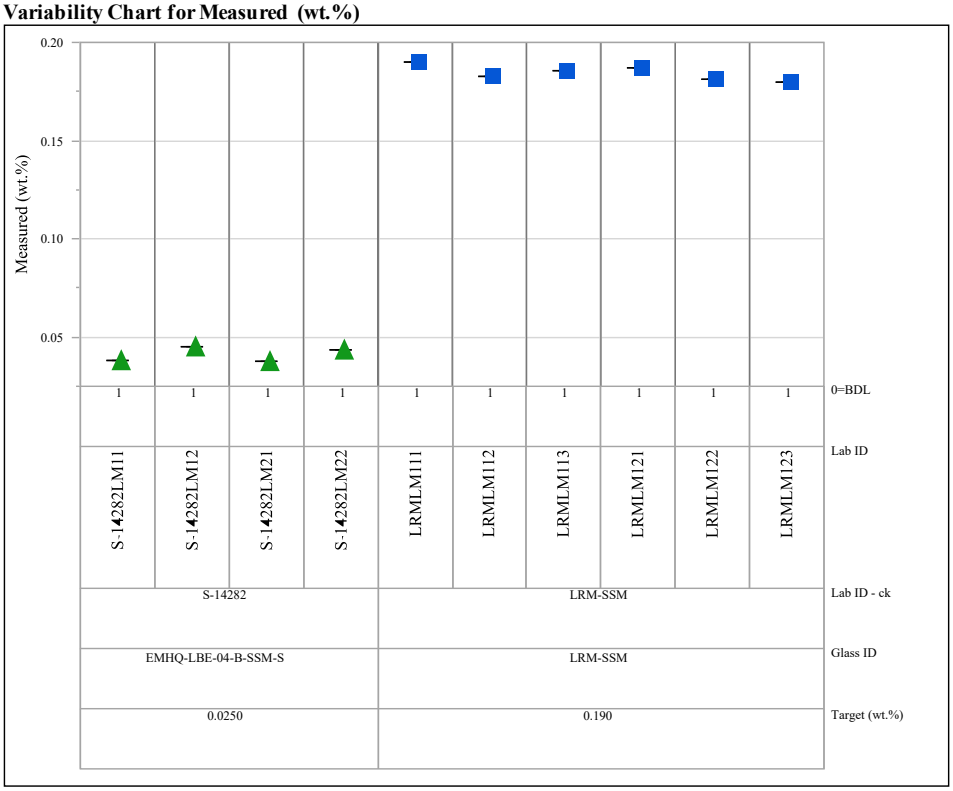


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=Cr_2O_3, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=F^-, Prep Method=KH
Variability Chart for Measured (wt.%)

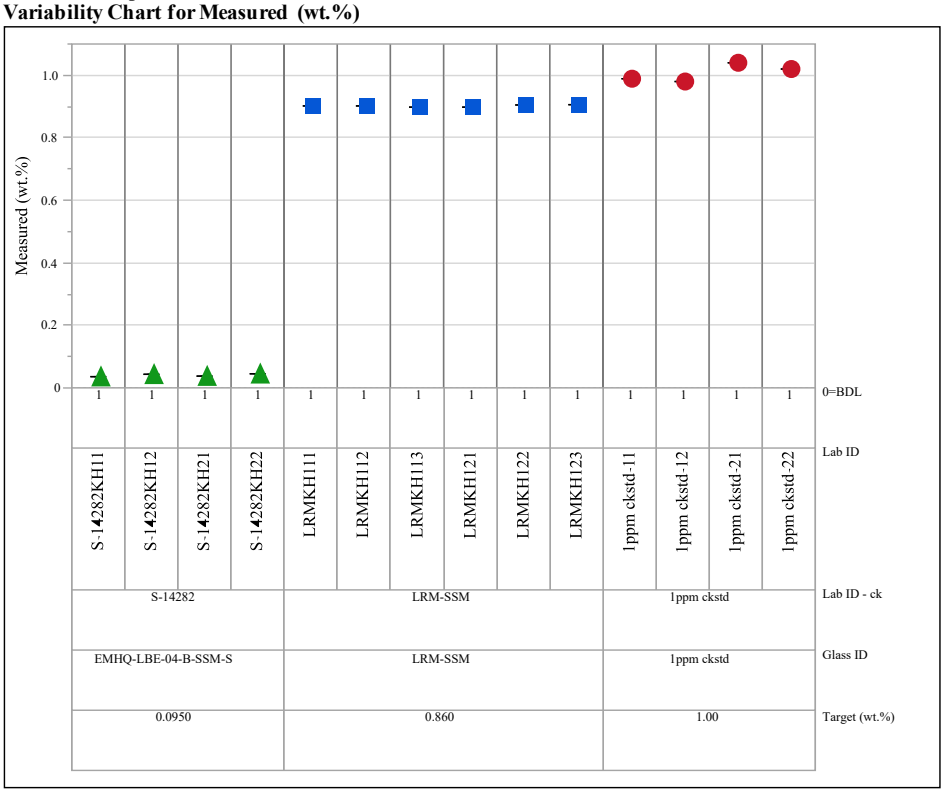
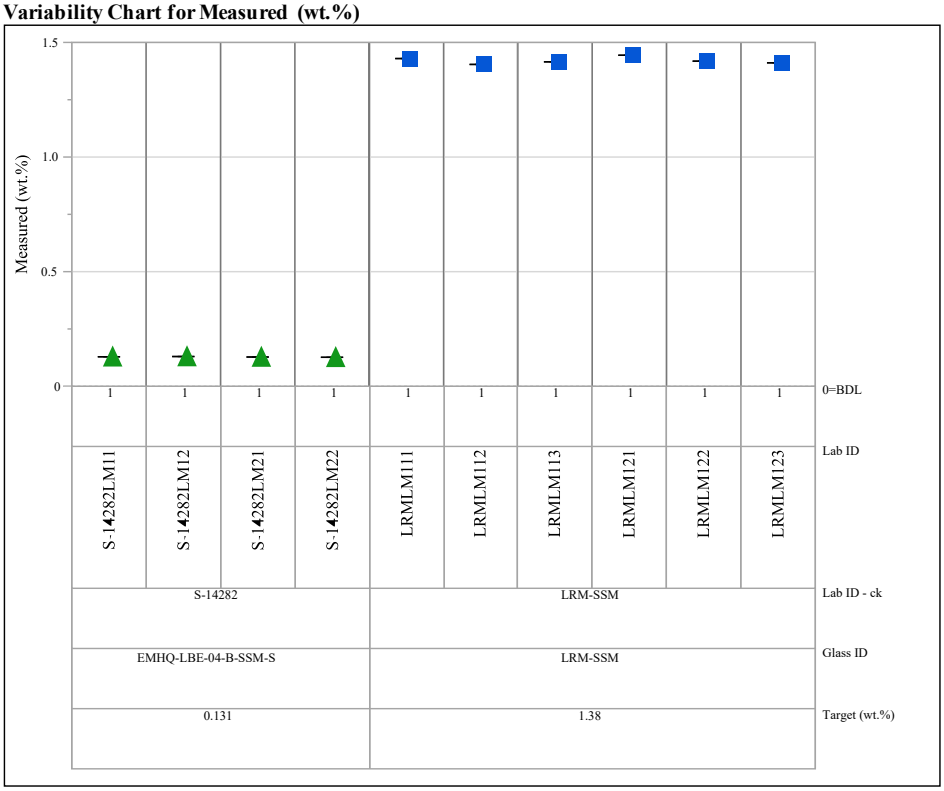


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=Fe_2O_3, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=K_2O, Prep Method=LM
Variability Chart for Measured (wt.%)

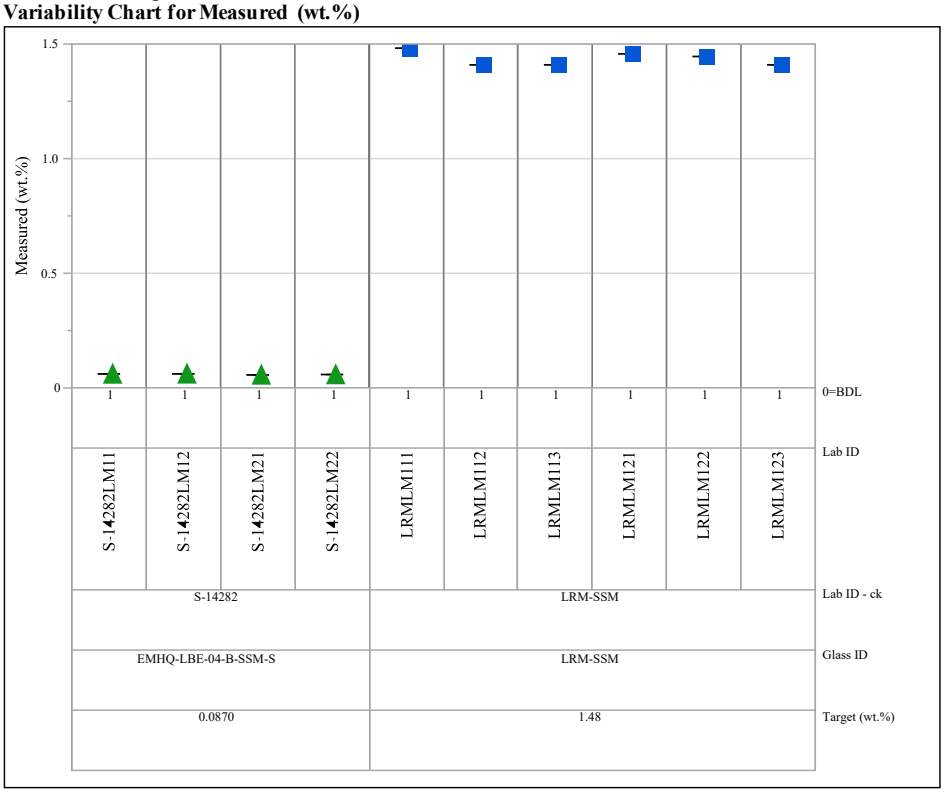
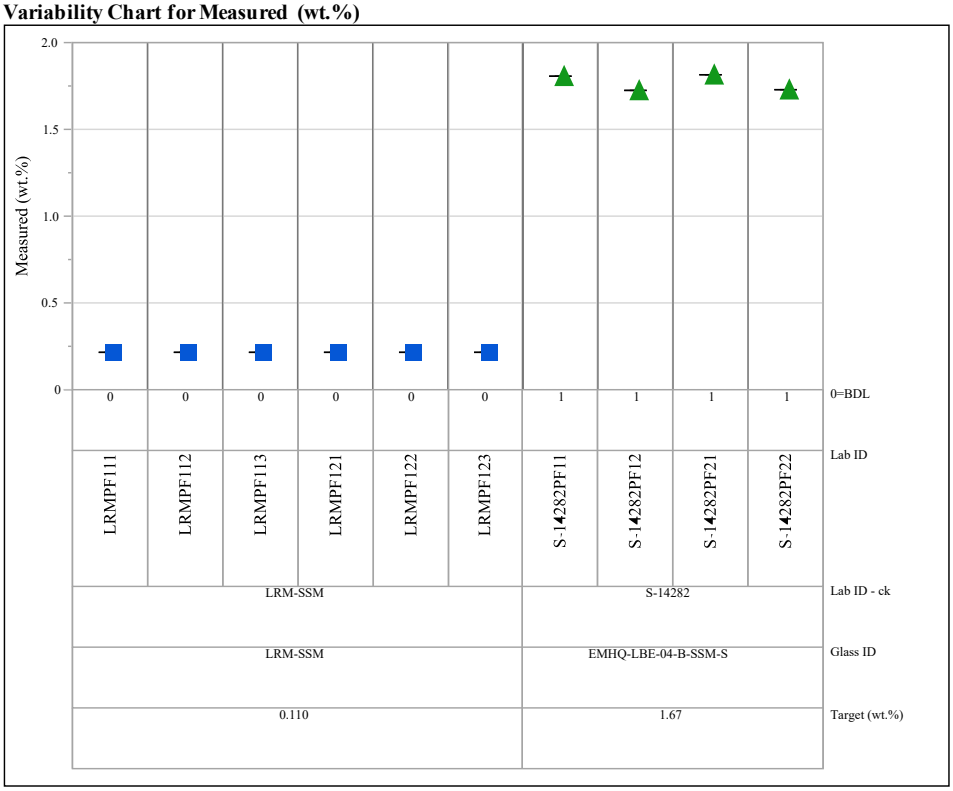


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=Li_2O, Prep Method=PF
Variability Chart for Measured (wt.%)



Oxide=MgO, Prep Method=LM
Variability Chart for Measured (wt.%)

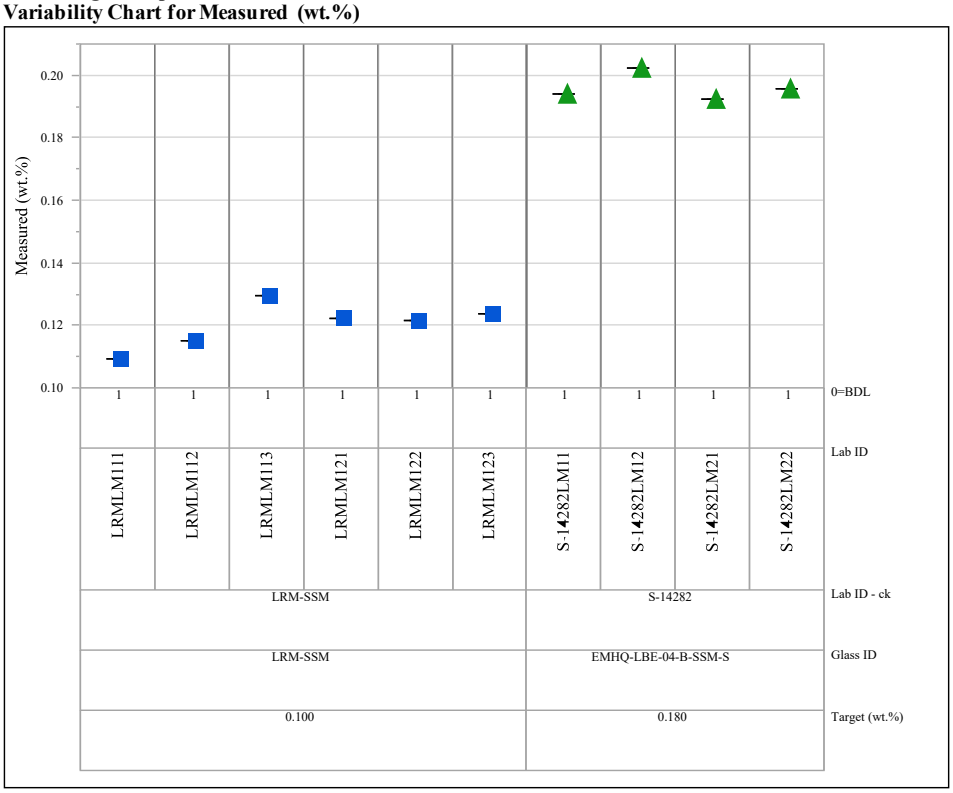
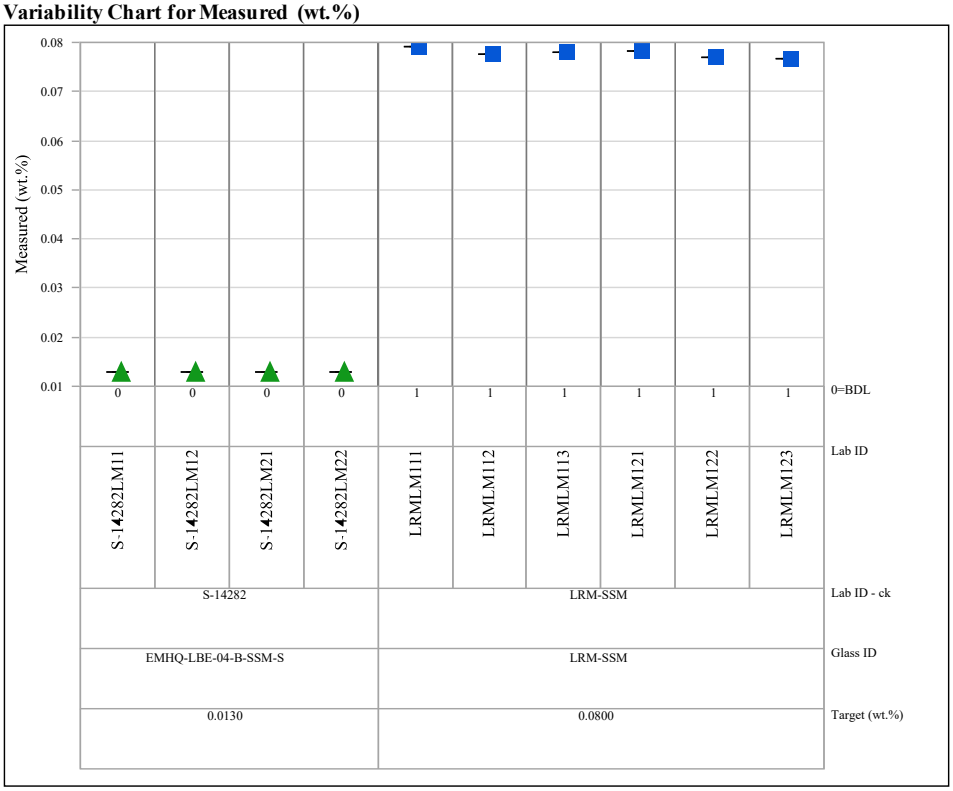


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=MnO, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=Na_2O, Prep Method=LM
Variability Chart for Measured (wt.%)

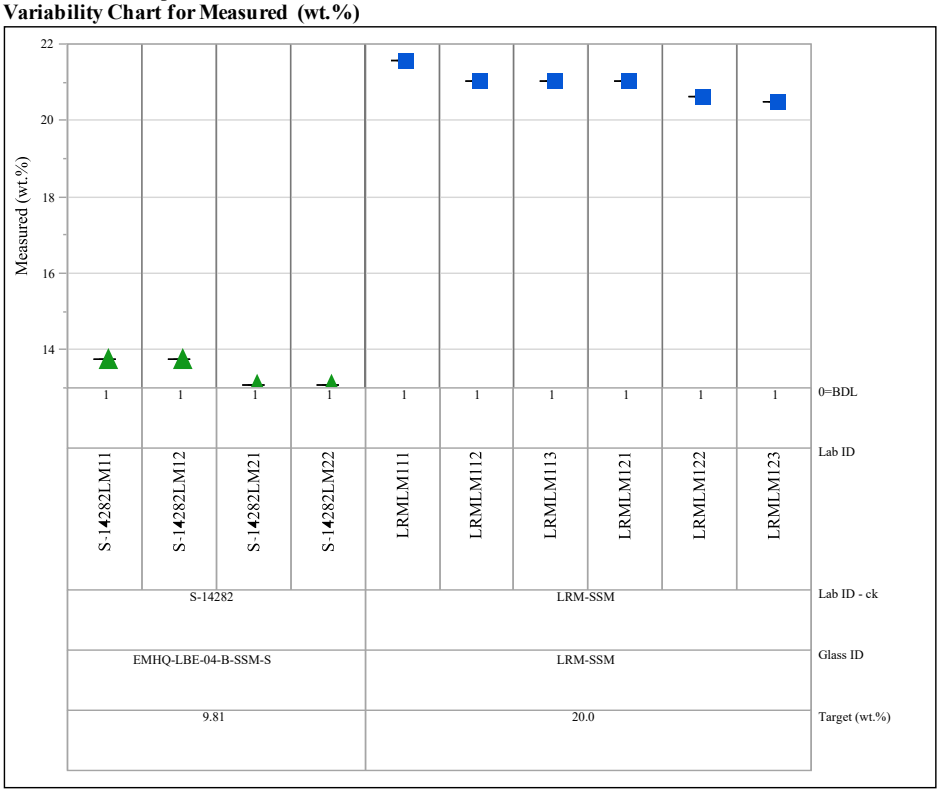
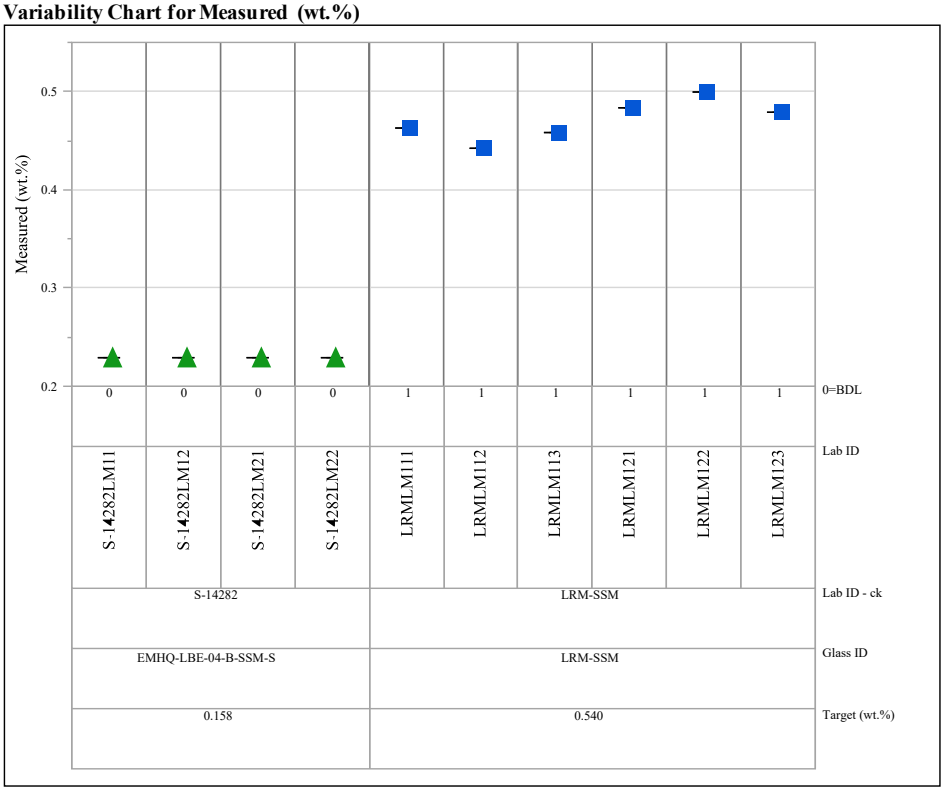


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=P_2O_5, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=SiO_2, Prep Method=PF
Variability Chart for Measured (wt.%)

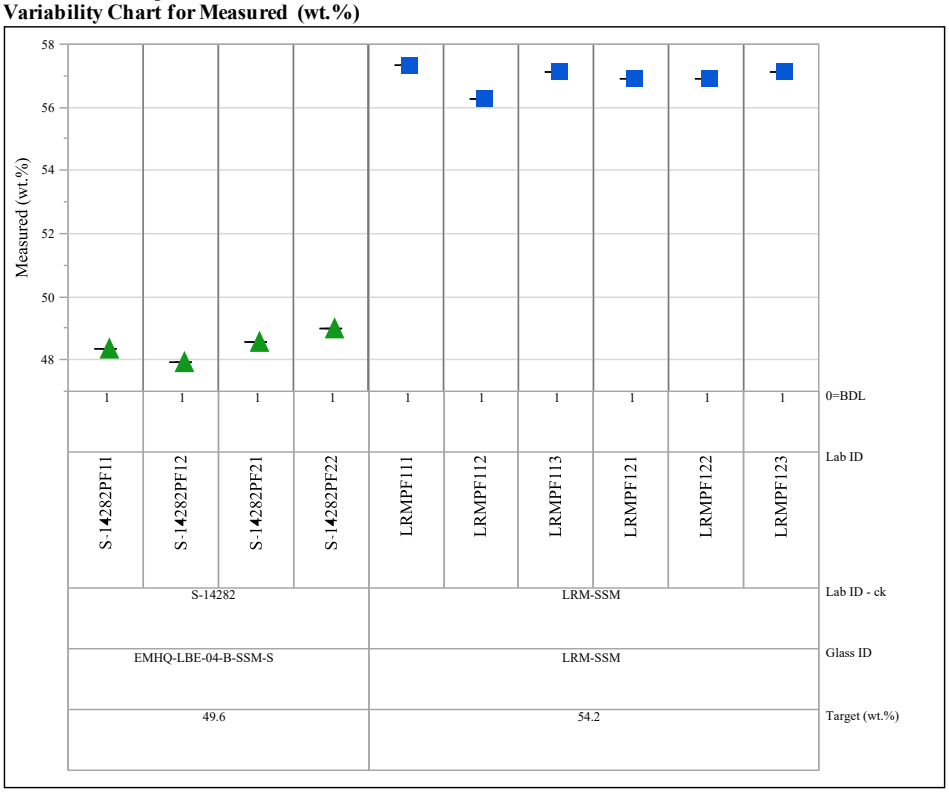
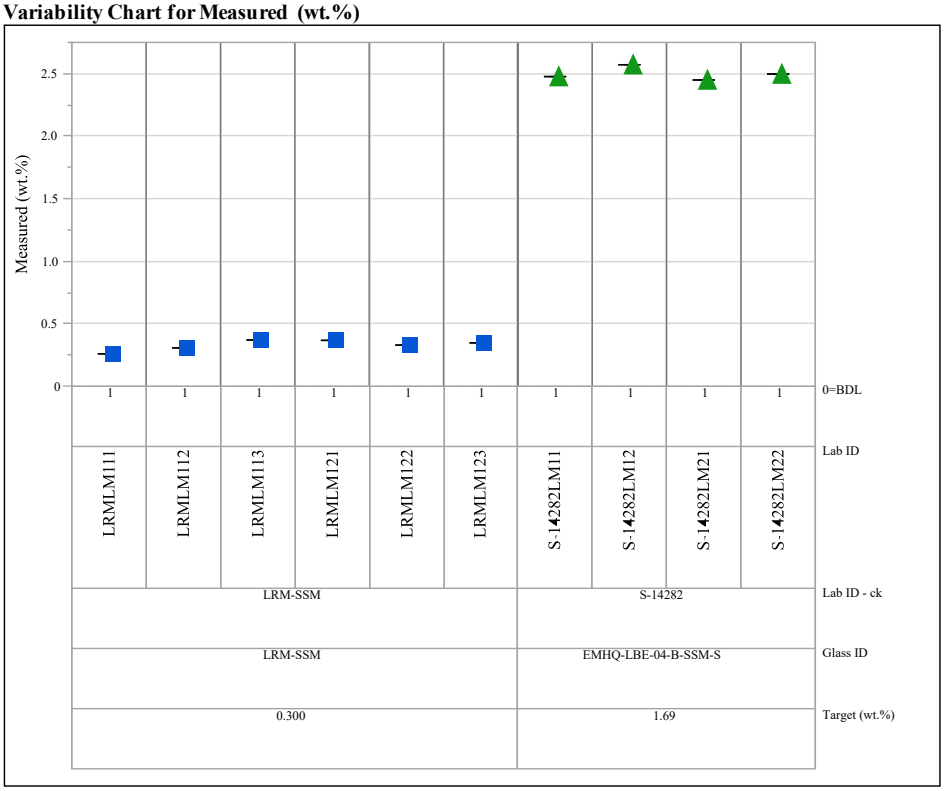


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=SO_3, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=TiO_2, Prep Method=LM
Variability Chart for Measured (wt.%)

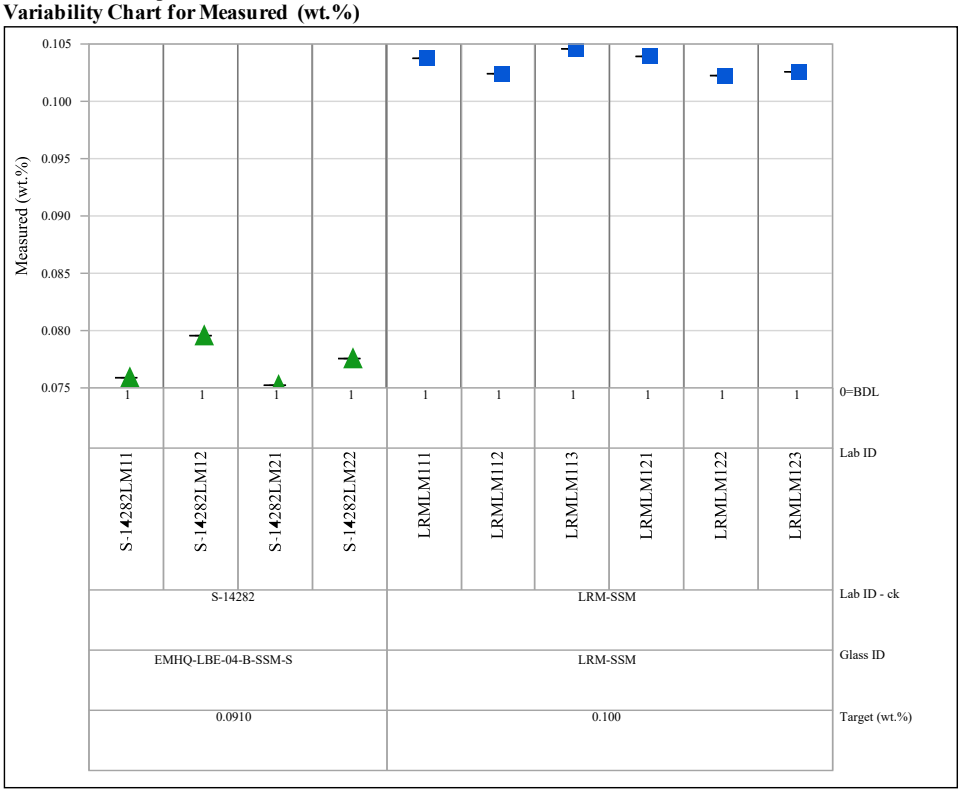
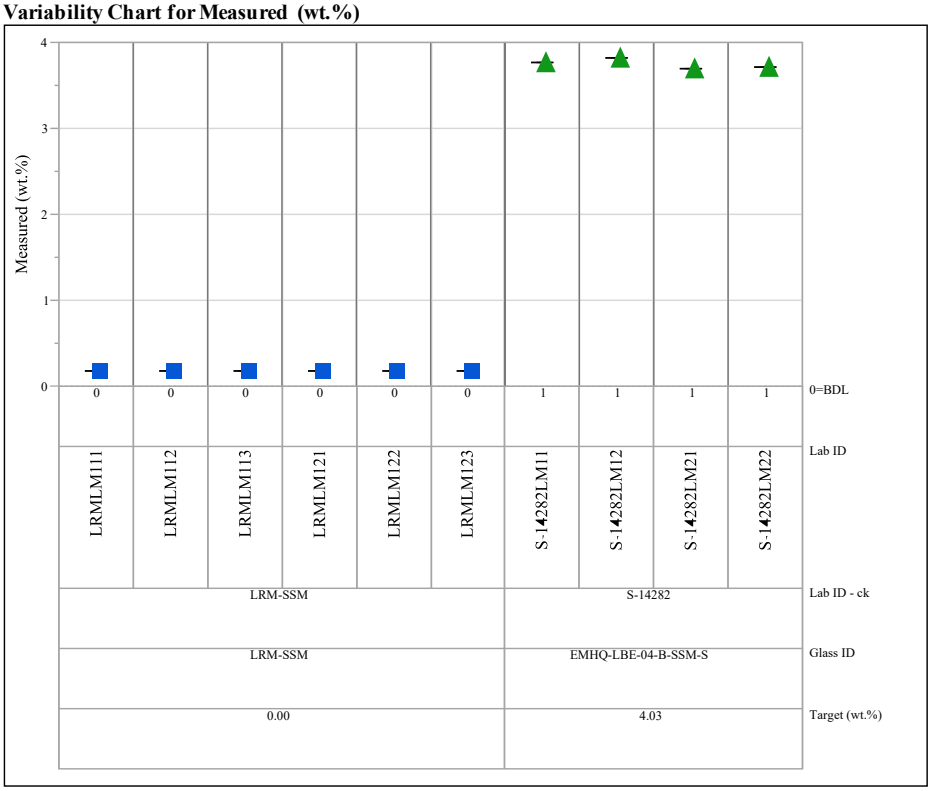


Exhibit A-1. Plots of Oxide Measurements by Glass Identifier by Target Concentration (continued)

Oxide=V_2O_5, Prep Method=LM
Variability Chart for Measured (wt.%)



Oxide=ZrO_2, Prep Method=PF
Variability Chart for Measured (wt.%)

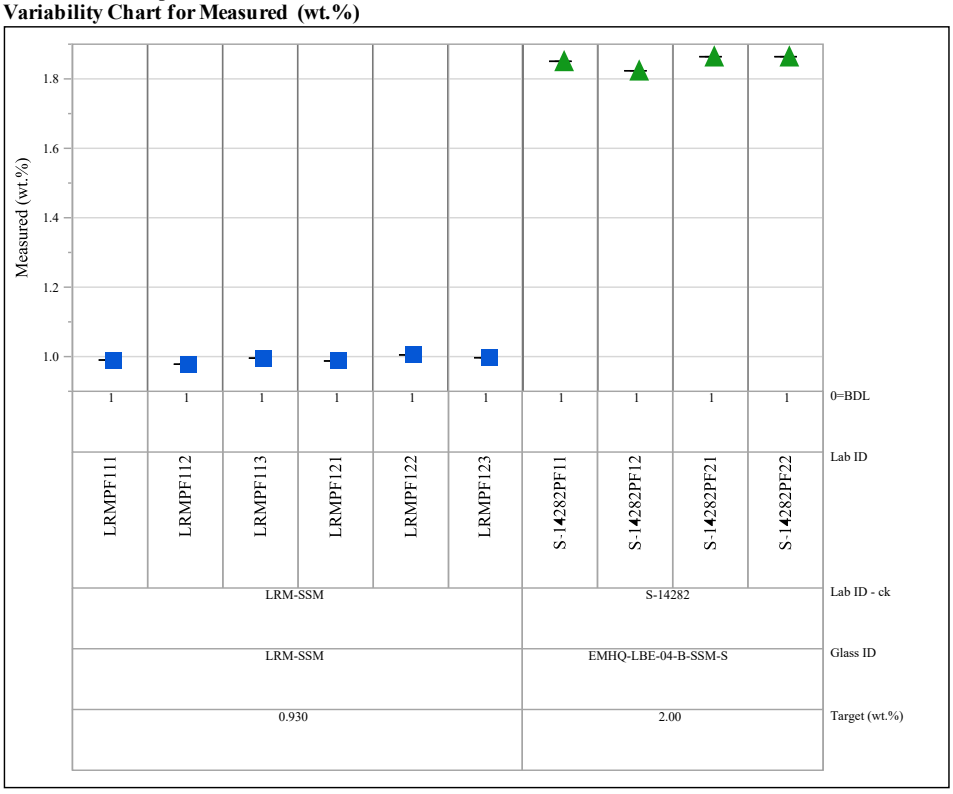
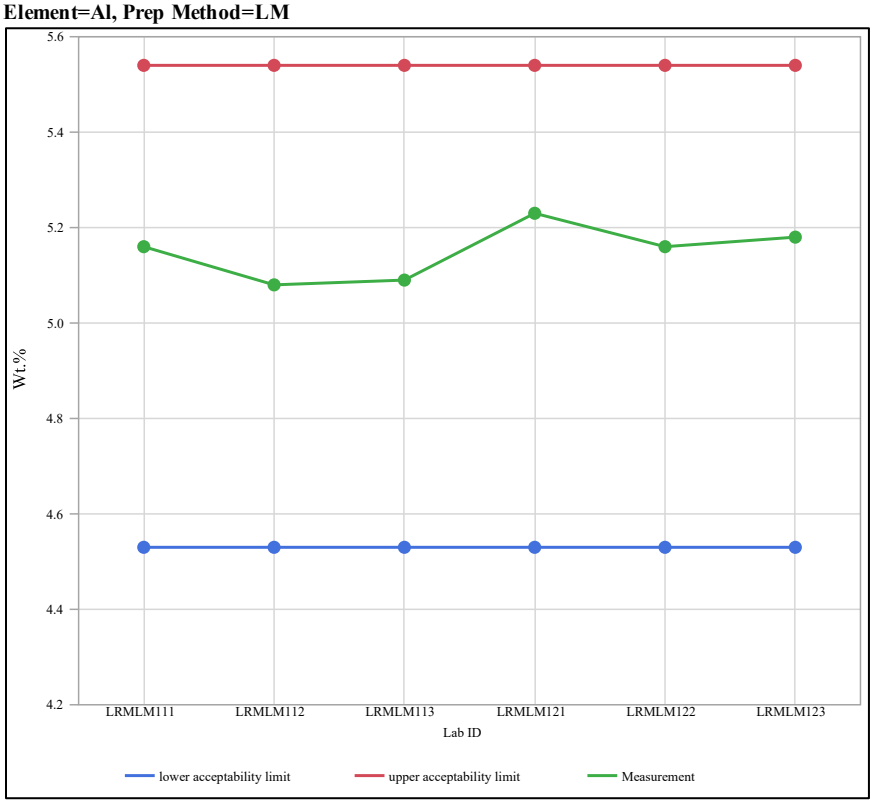


Exhibit A-2. Acceptability Evaluation for Measurements of the LRM Glass

Element=Al, Prep Method=LM



Element=B, Prep Method=PF

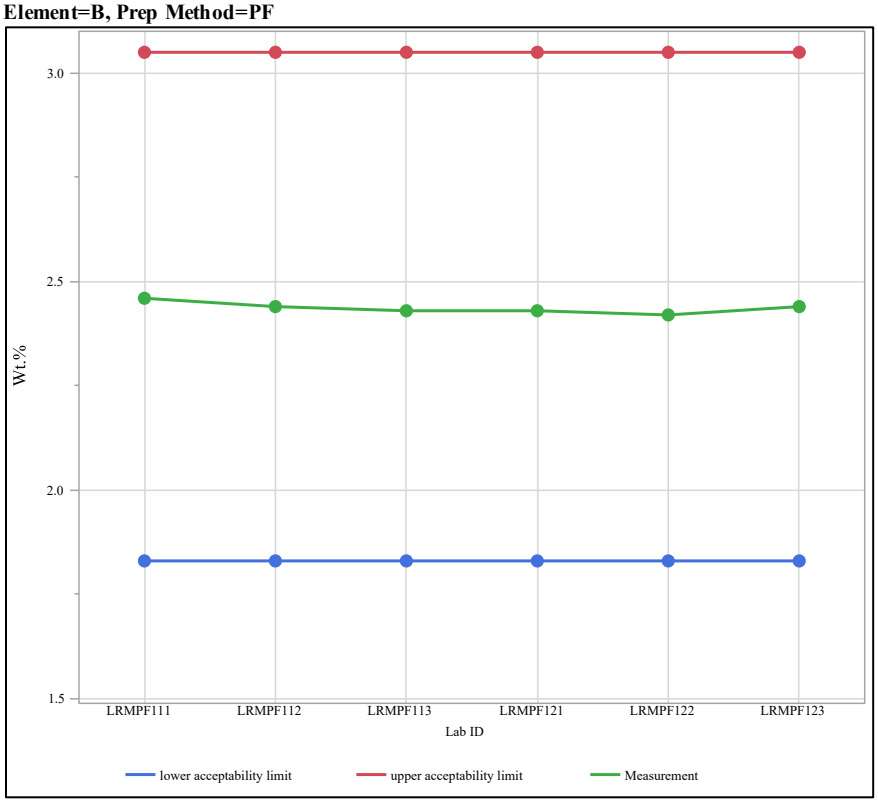
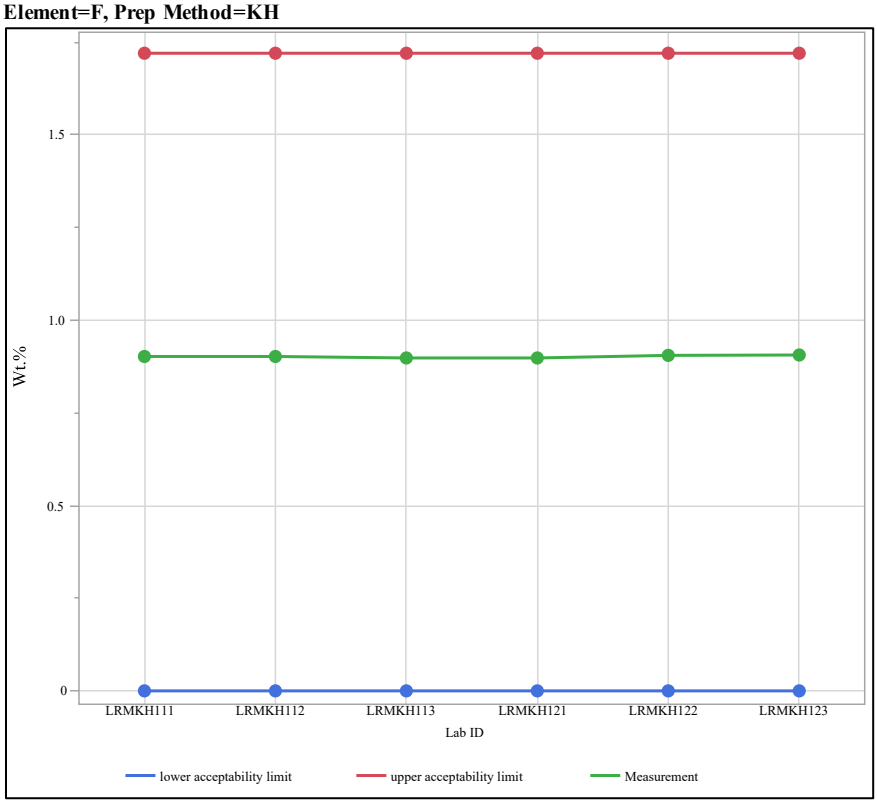


Exhibit A-2. Acceptability Evaluation for Measurements of the LRM Glass (continued)

Element=F, Prep Method=KH



Element=Fe, Prep Method=LM

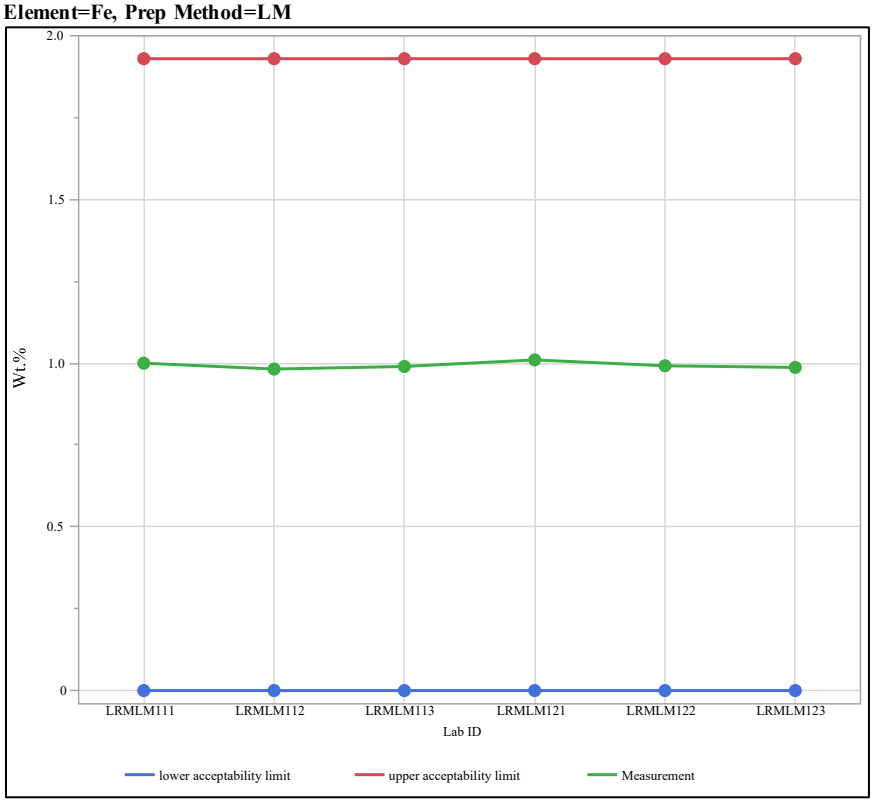
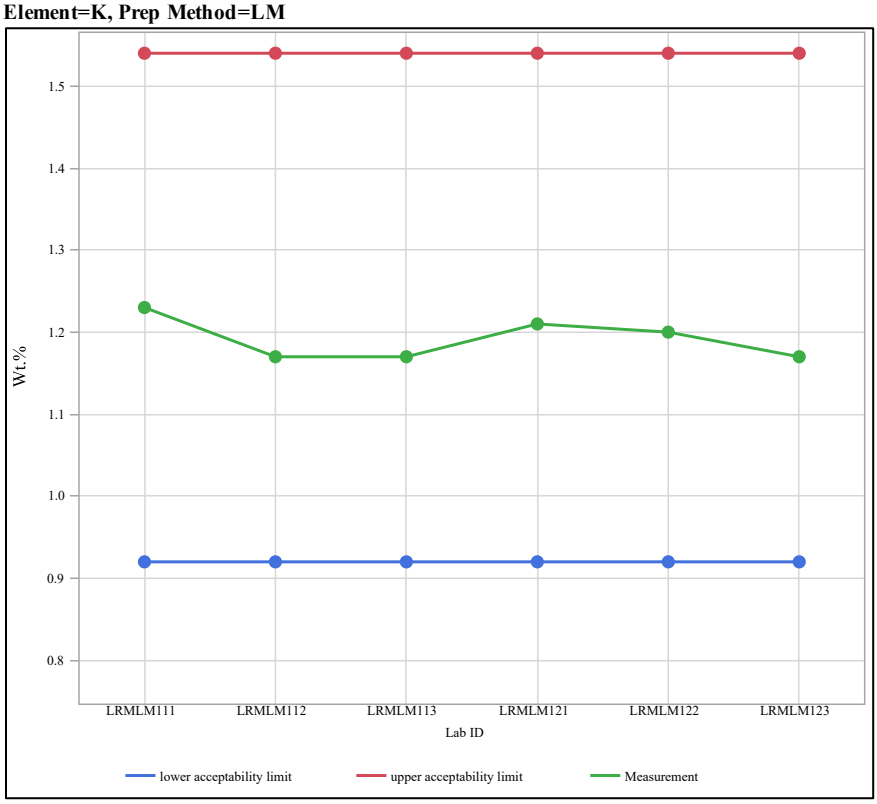


Exhibit A-2. Acceptability Evaluation for Measurements of the LRM Glass (continued)

Element=K, Prep Method=LM



Element=Na, Prep Method=LM

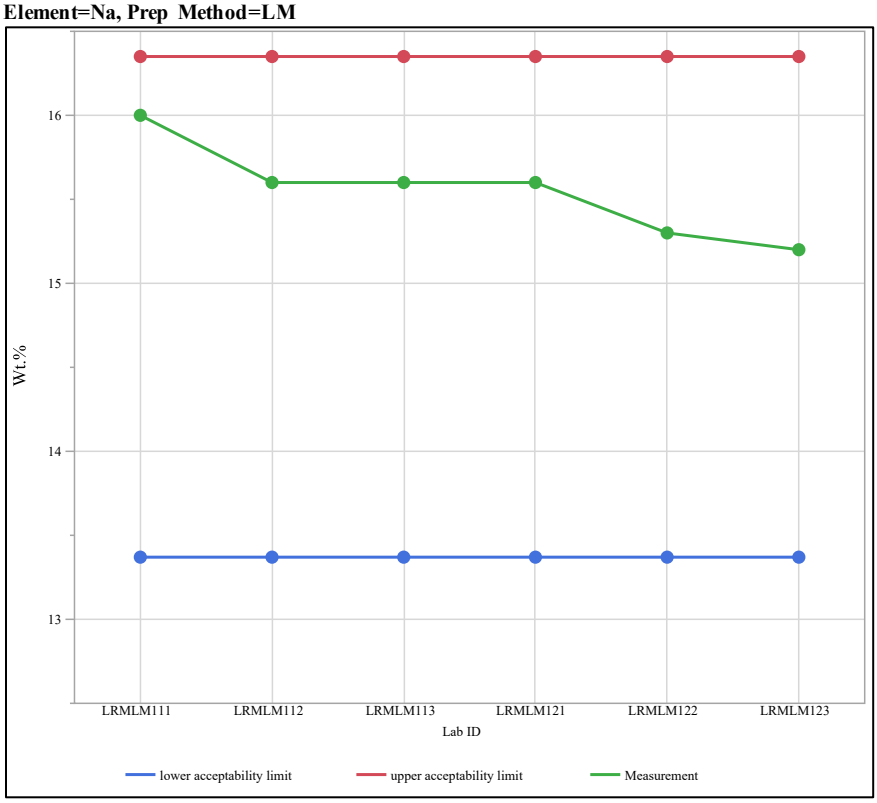
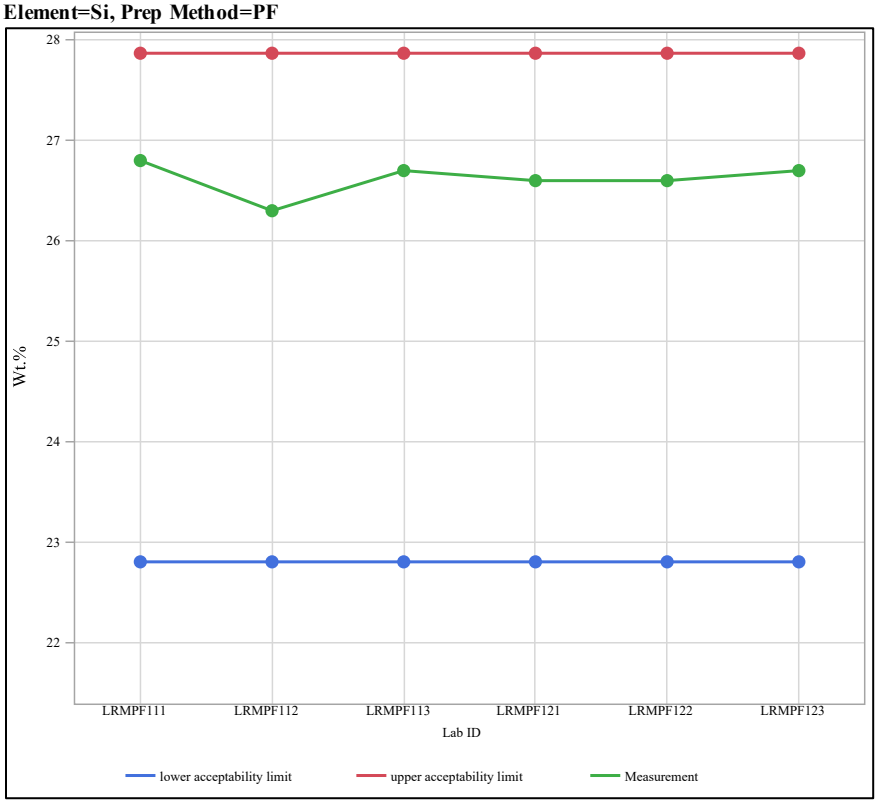


Exhibit A-2. Acceptability Evaluation for Measurements of the LRM Glass (continued)

Element=Si, Prep Method=PF



Element=Zr, Prep Method=PF

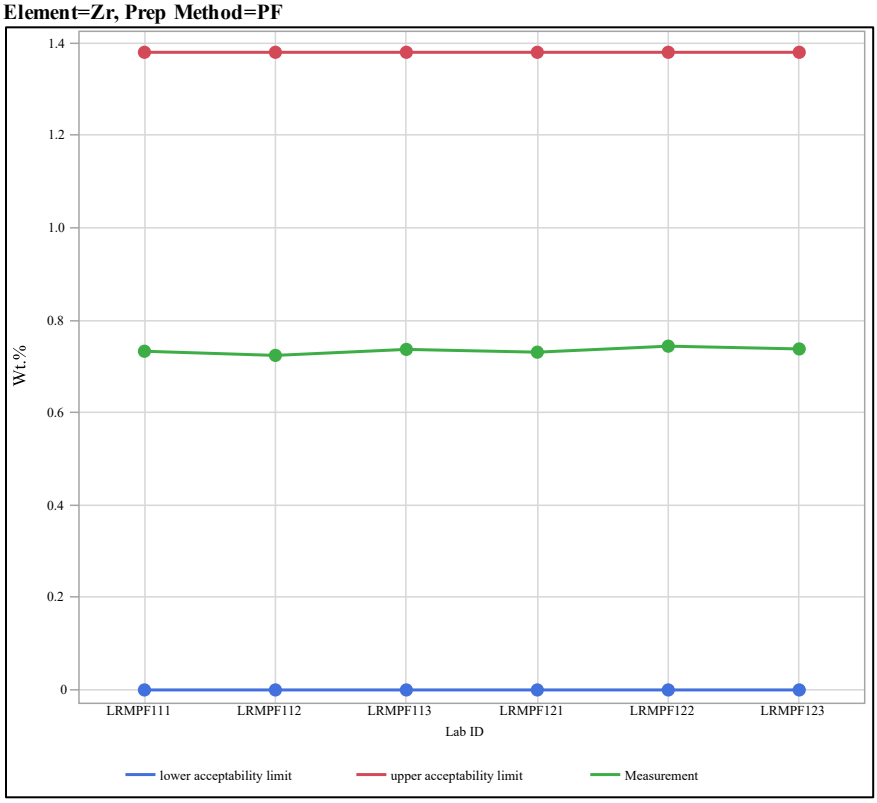
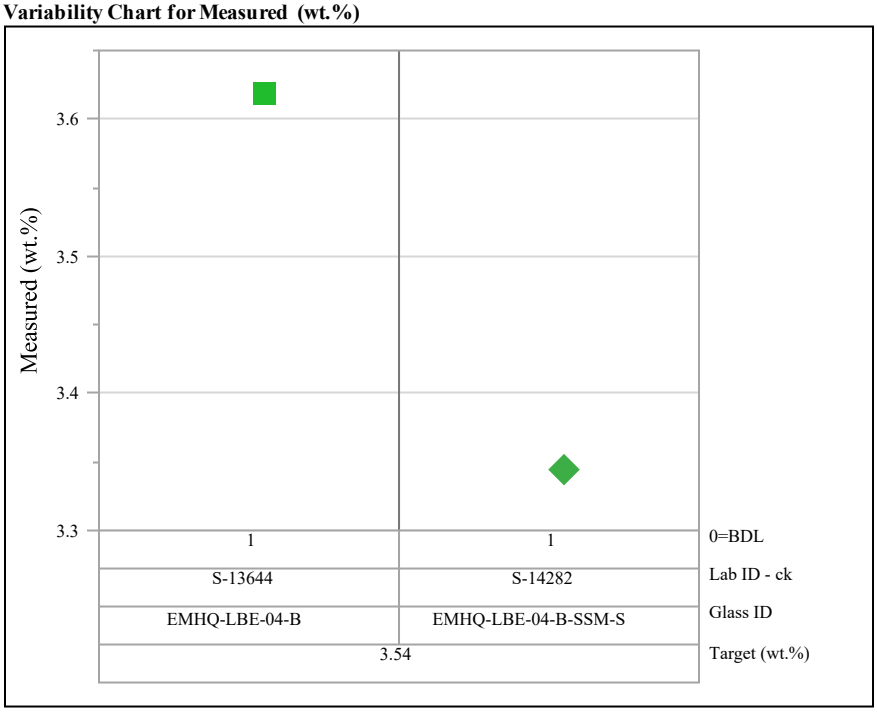


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass

Oxide=Al_2O_3
Variability Chart for Measured (wt.%)



Oxide=B_2O_3
Variability Chart for Measured (wt.%)

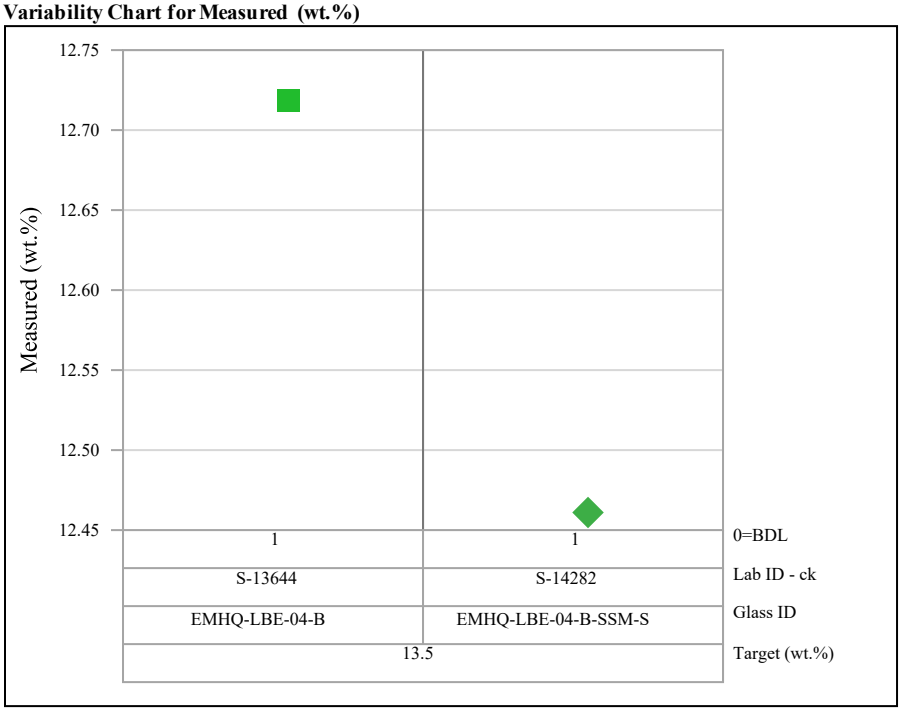
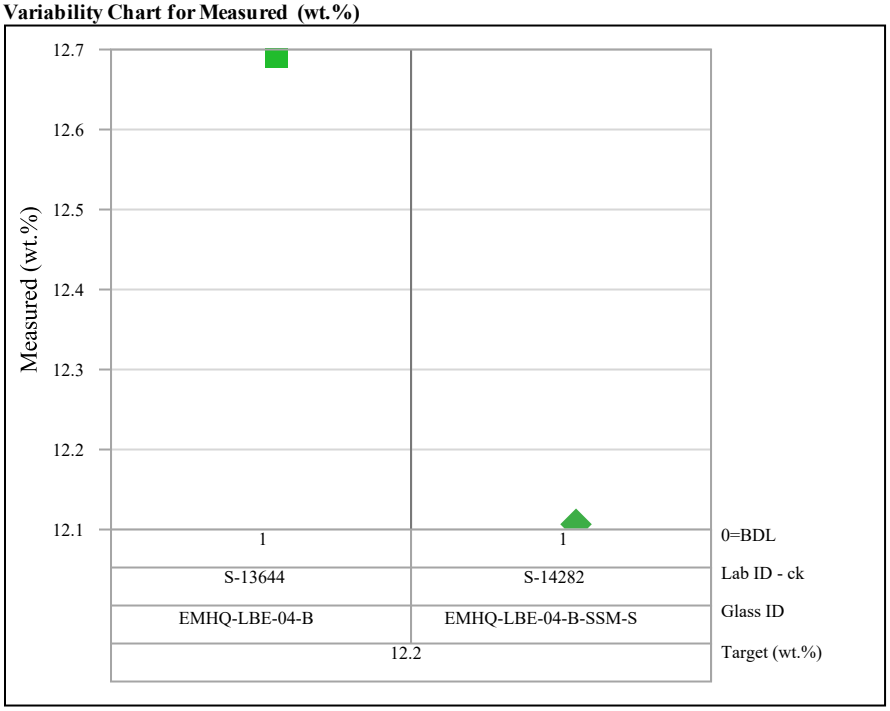


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=CaO
Variability Chart for Measured (wt.%)



Oxide=Cl⁻
Variability Chart for Measured (wt.%)

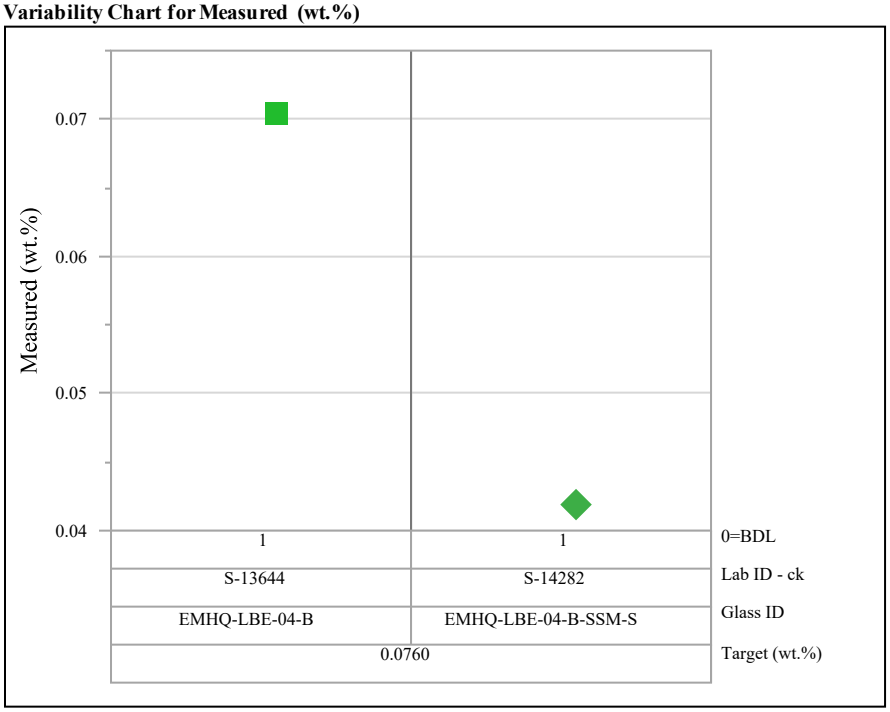
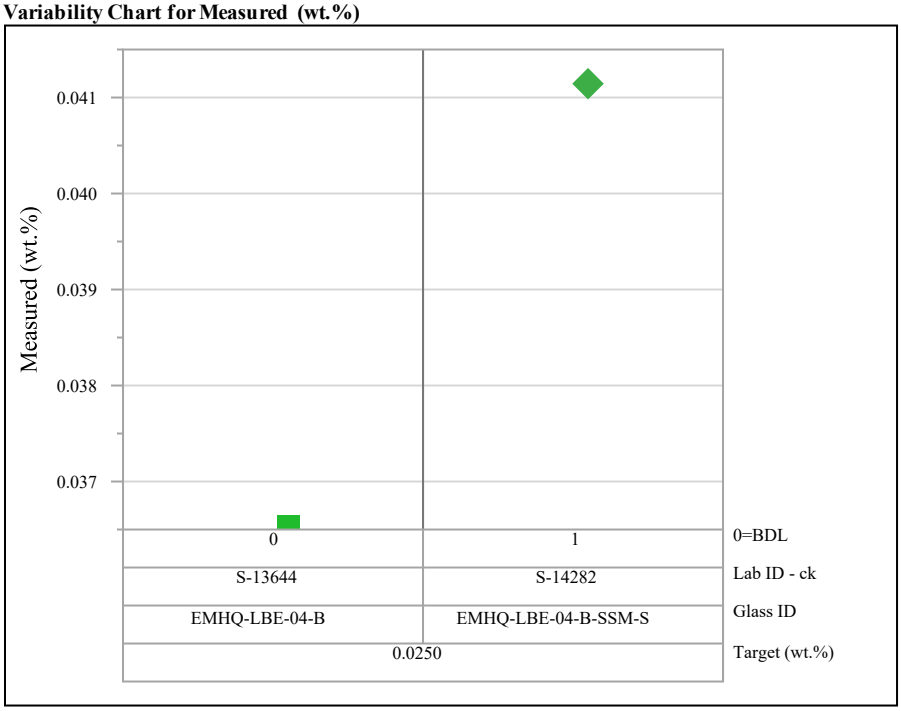


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=Cr_2O_3
Variability Chart for Measured (wt.%)



Oxide=F^-
Variability Chart for Measured (wt.%)

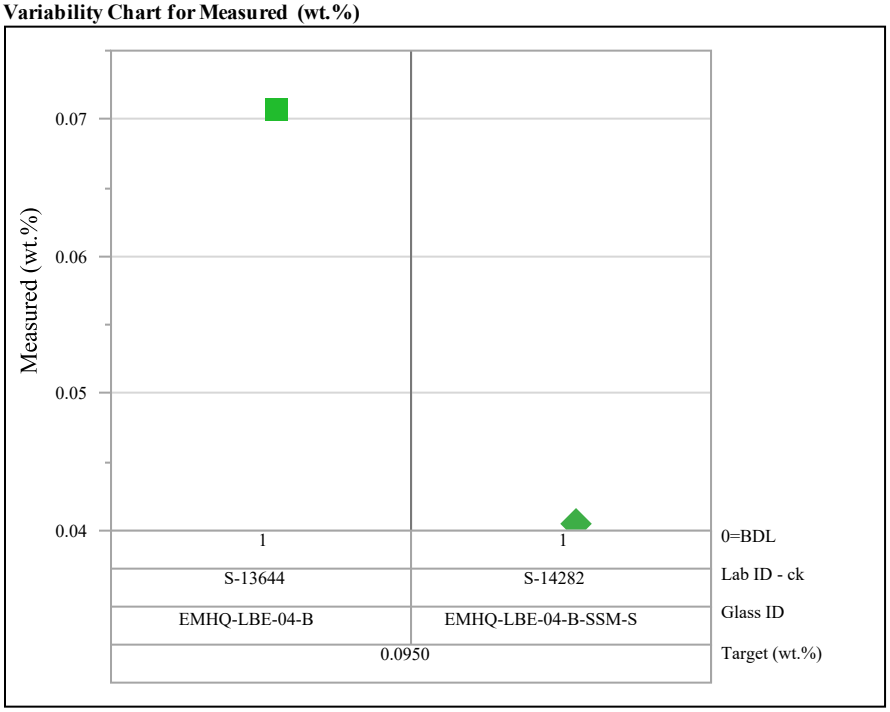
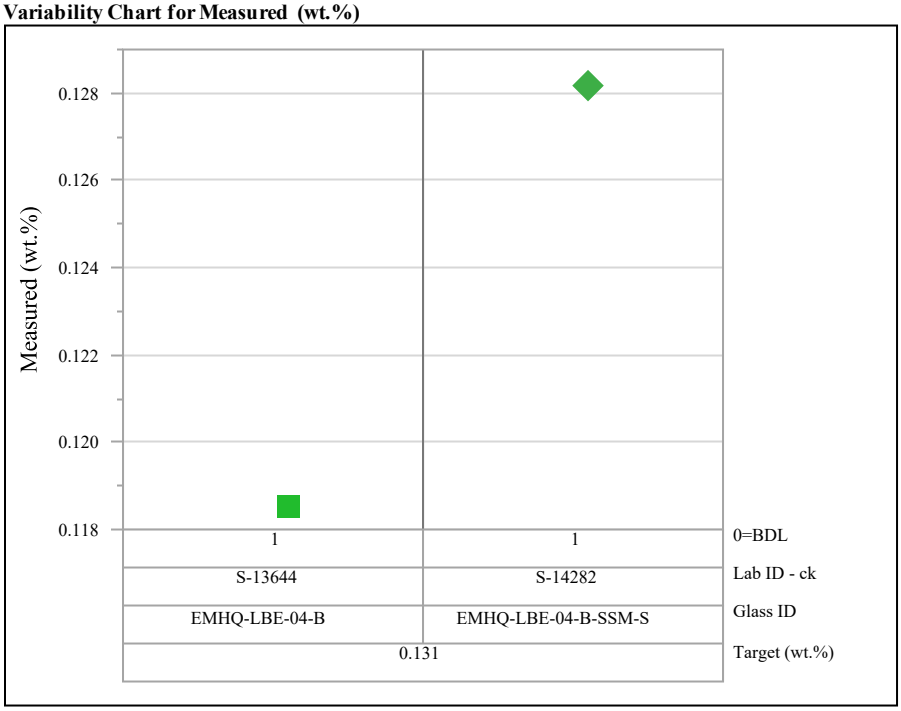


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=Fe_2O_3
Variability Chart for Measured (wt.%)



Oxide=K_2O
Variability Chart for Measured (wt.%)

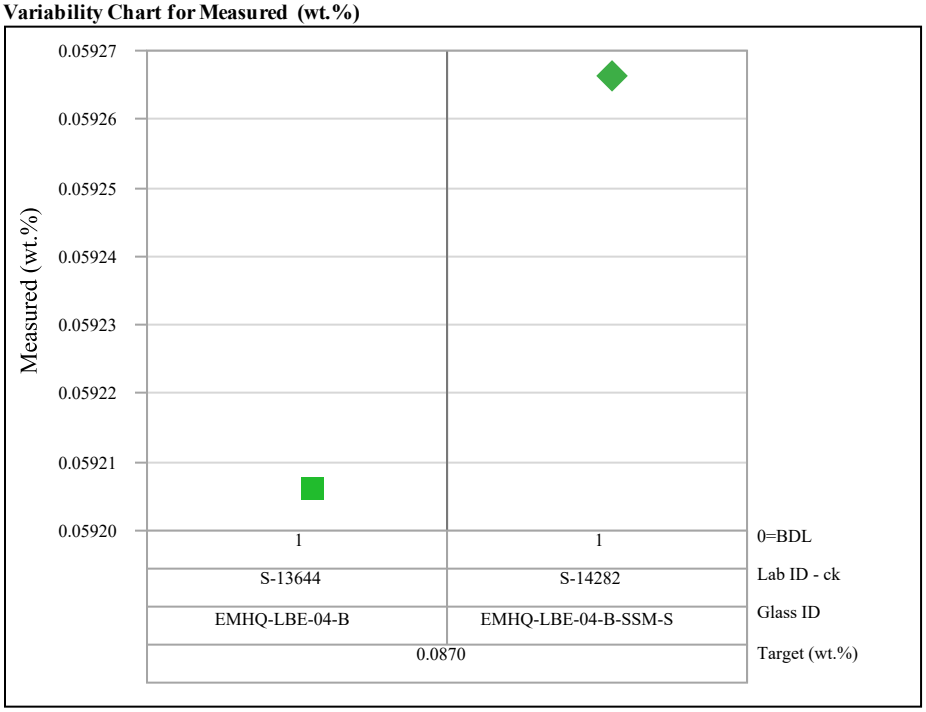
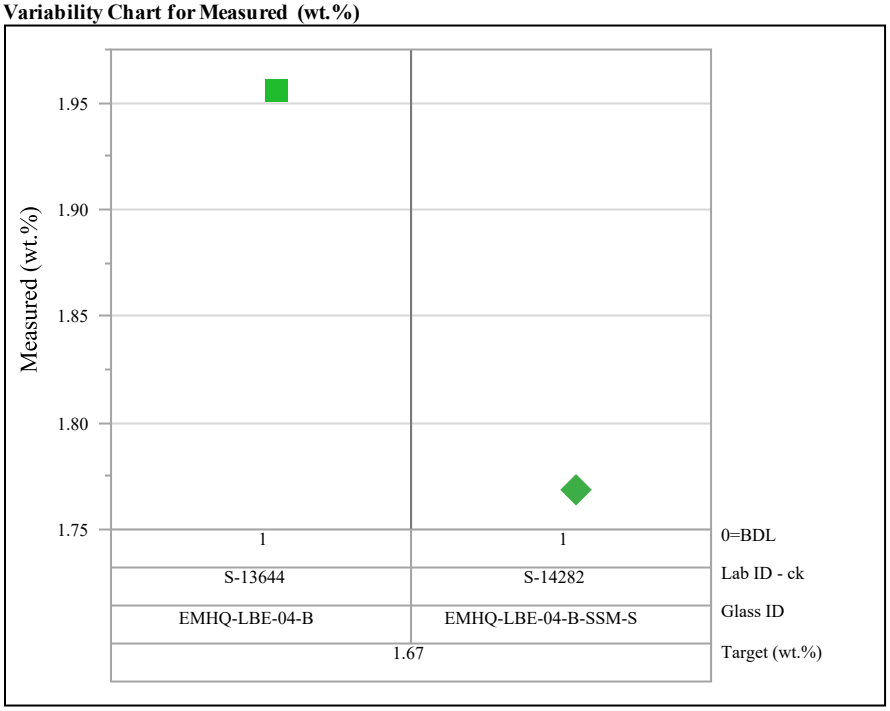


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=Li_2O
Variability Chart for Measured (wt.%)



Oxide=MgO
Variability Chart for Measured (wt.%)

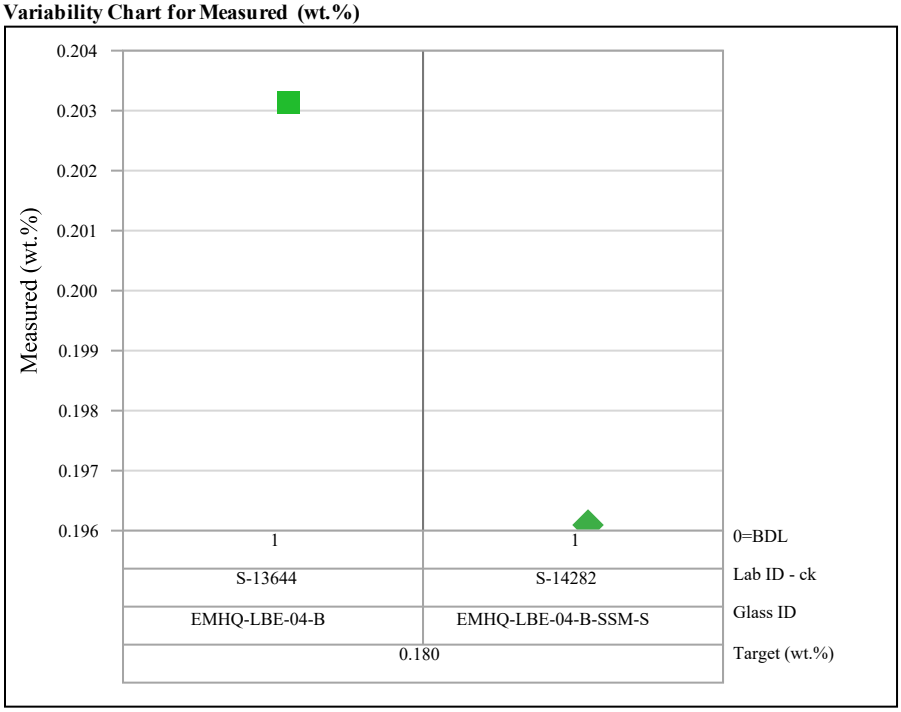
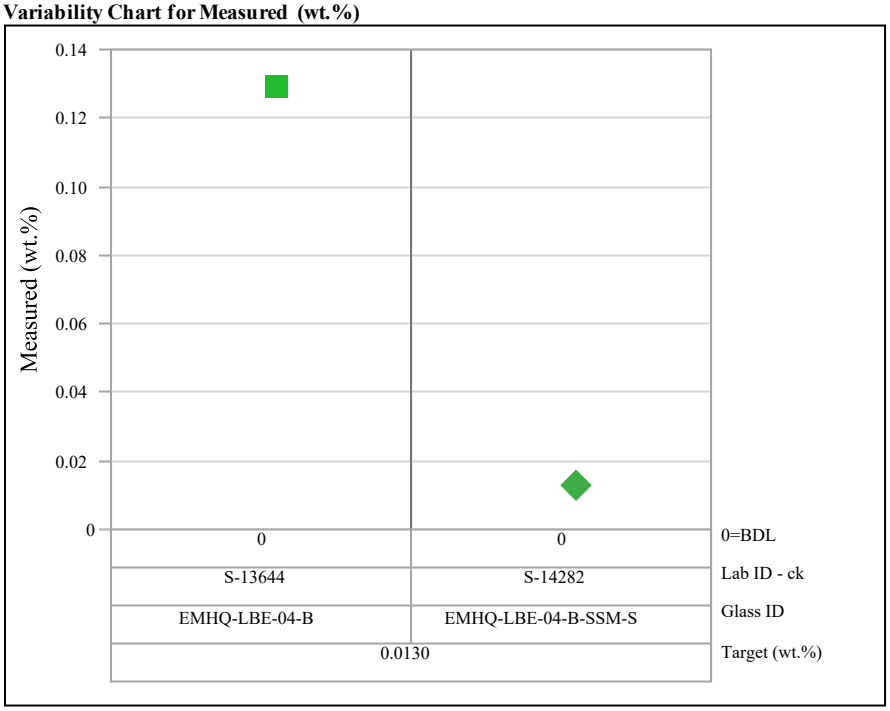


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=MnO
Variability Chart for Measured (wt.%)



Oxide=Na_2O
Variability Chart for Measured (wt.%)

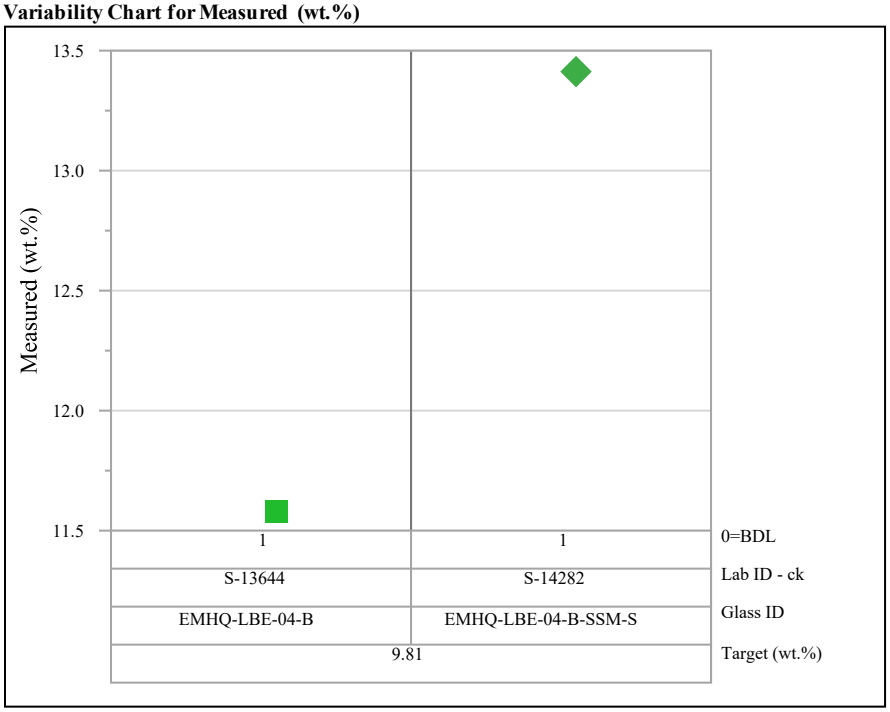
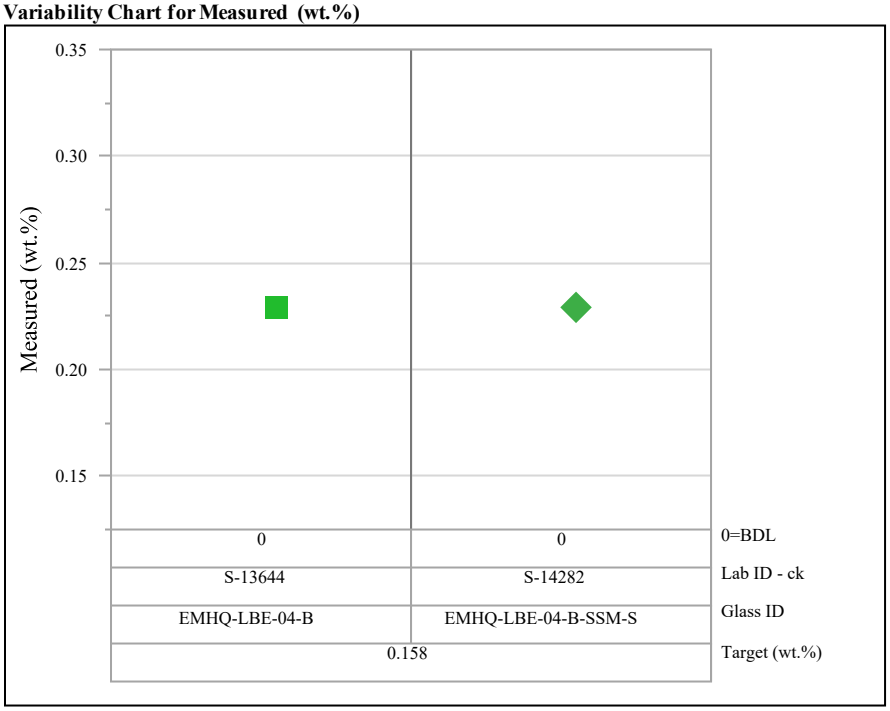


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=P_2O_5
Variability Chart for Measured (wt.%)



Oxide=SiO_2
Variability Chart for Measured (wt.%)

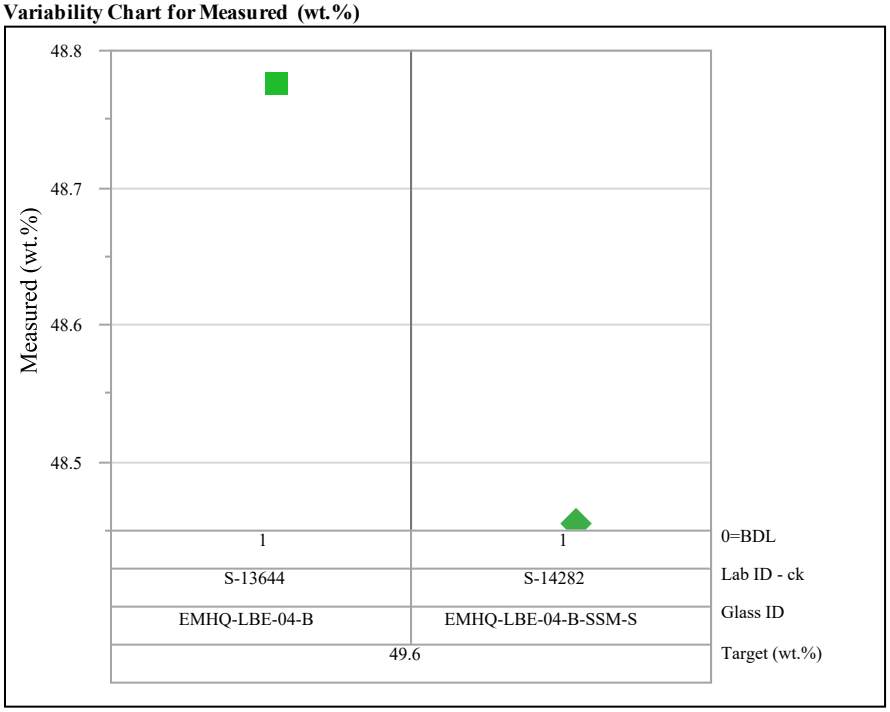
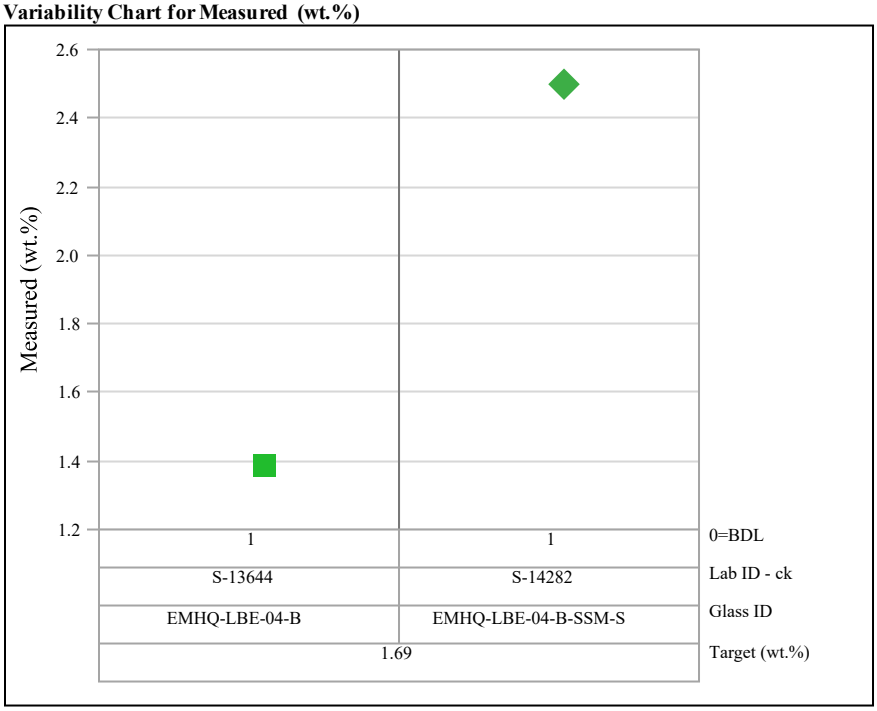


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=SO_3
Variability Chart for Measured (wt.%)



Oxide=TiO_2
Variability Chart for Measured (wt.%)

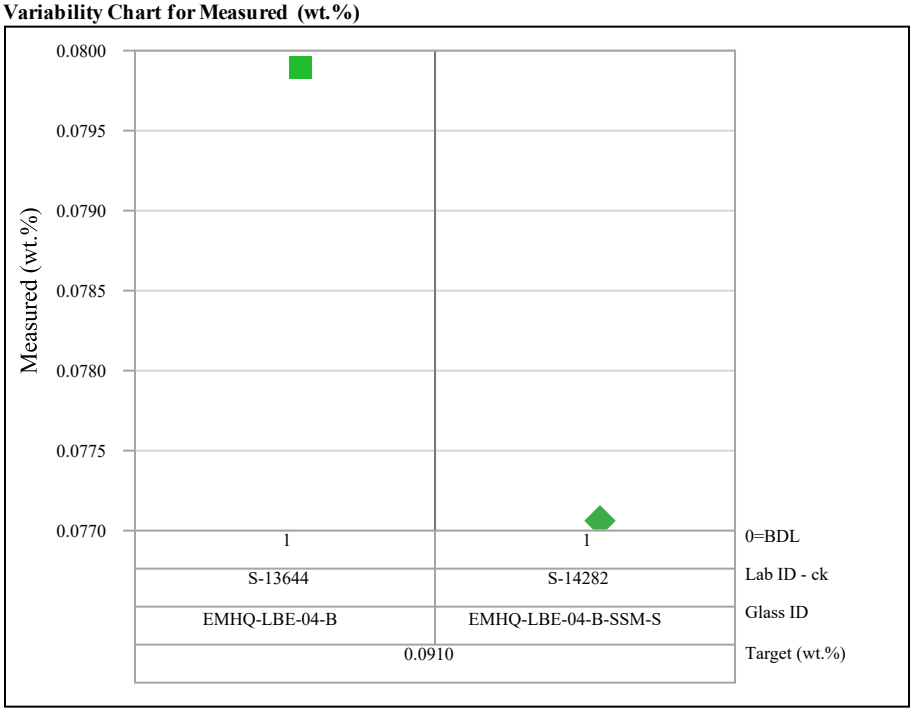
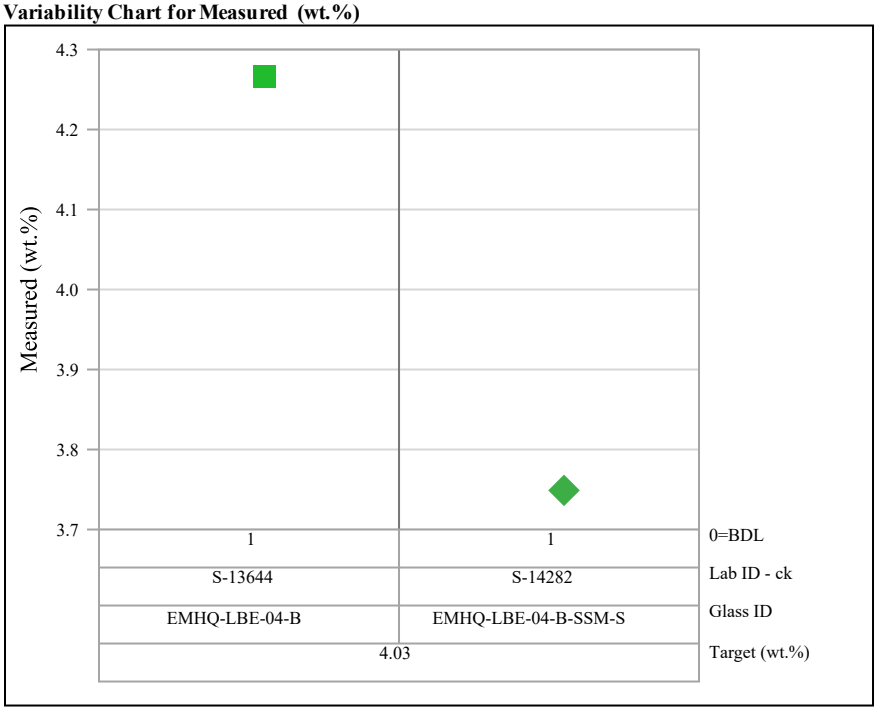


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=V_2O_5
Variability Chart for Measured (wt.%)



Oxide=ZrO_2
Variability Chart for Measured (wt.%)

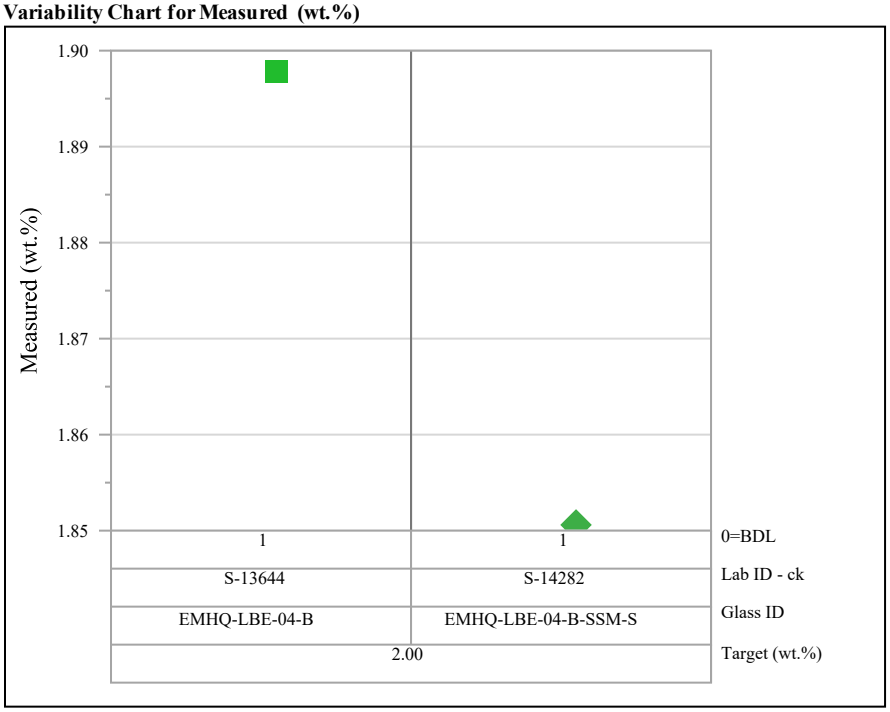
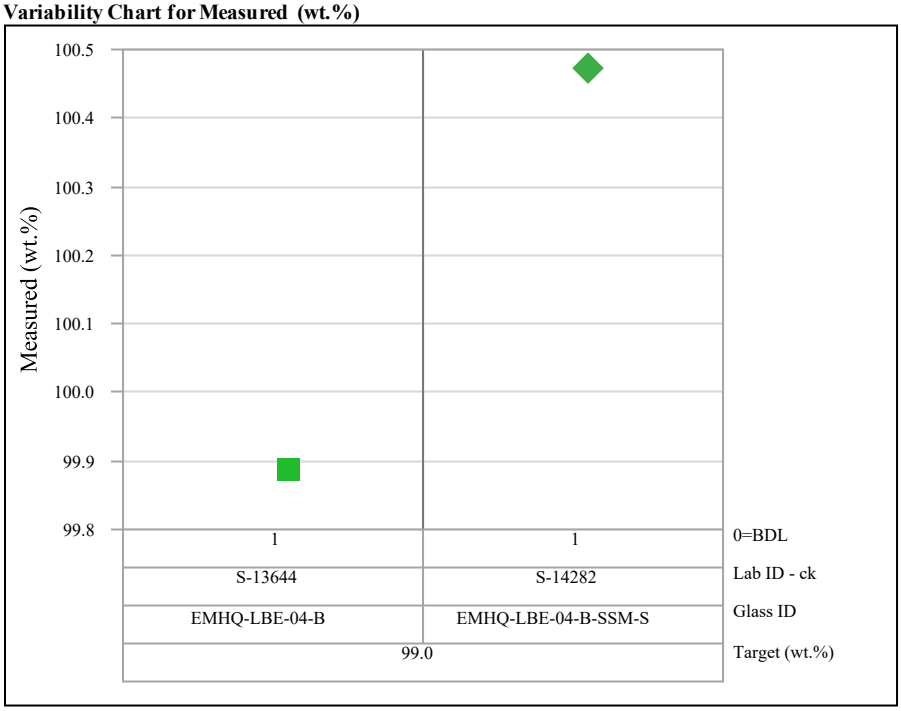


Exhibit A-3. Comparisons of the Measured Compositions of the Quenched and SSM Version of the EMHQ-LBE-04-B Glass (continued)

Oxide=Sum of Oxides
Variability Chart for Measured (wt.%)



Appendix B. Tables and Exhibits Supporting the Wash Solution Composition Measurements

Table B-1. Measurements (mg/L) of the SSM Wash Solutions Measured by ICP-OES

PNNL ID	Block	Seq	Lab ID	Al	B	Ca	Cr	Fe	K	Li	Mg	Mn	Na	P	S	Si	Ti	V	Zr
std	1	1	std-11	4.09	19.6	<1.00	<1.00	4.22	10.5	10.3	<1.00	<1.00	85.4	<1.00	<1.00	51.2	<1.00	<1.00	<1.00
hpstd	1	2	hpstd-11	49.6	<1.00	<1.00	<1.00	51.4	<1.00	<1.00	<1.00	20.9	150	<1.00	10.2	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-04-B-SSM-W	1	3	S-14281-1	<1.00	12.8	33.2	<1.00	<1.00	<1.00	6.32	<1.00	<1.00	179	<1.00	113	30.8	<1.00	10.5	<1.00
std	1	4	std-12	3.99	18.9	<1.00	<1.00	4.11	10.3	10.2	<1.00	<1.00	84.6	<1.00	<1.00	49.6	<1.00	<1.00	<1.00
hpstd	1	5	hpstd-12	49.2	<1.00	<1.00	<1.00	50.3	<1.00	<1.00	<1.00	20.4	150	<1.00	9.83	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-SSM-W-110221	1	6	S-14280-1	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	535	<1.00	373	<1.00	<1.00	<1.00	<1.00
std	1	7	std-13	3.96	19.0	<1.00	<1.00	4.16	10.3	10.3	<1.00	<1.00	84.9	<1.00	<1.00	50.1	<1.00	<1.00	<1.00
hpstd	1	8	hpstd-13	49.9	<1.00	<1.00	<1.00	51.5	<1.00	<1.00	<1.00	21.0	150	<1.00	10.1	<1.00	<1.00	<1.00	<1.00
std	2	1	std-21	3.93	19.6	<1.00	<1.00	4.10	9.83	9.49	<1.00	<1.00	80.3	<1.00	<1.00	50.2	<1.00	<1.00	<1.00
hpstd	2	2	hpstd-21	49.5	<1.00	<1.00	<1.00	50.8	<1.00	<1.00	<1.00	20.1	144	<1.00	10.4	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-SSM-W-110221	2	3	S-14280-2	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	574	<1.00	390	<1.00	<1.00	<1.00	<1.00
std	2	4	std-22	4.09	20.2	<1.00	<1.00	4.18	10.1	9.99	<1.00	<1.00	83.2	<1.00	<1.00	51.7	<1.00	<1.00	<1.00
hpstd	2	5	hpstd-22	49.6	<1.00	<1.00	<1.00	50.7	<1.00	<1.00	<1.00	20.2	145	<1.00	10.1	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-04-B-SSM-W	2	6	S-14281-2	<1.00	13.8	32.7	<1.00	<1.00	<1.00	6.15	<1.00	<1.00	184	<1.00	123	32.1	<1.00	10.7	<1.00
std	2	7	std-23	3.89	19.4	<1.00	<1.00	4.08	9.58	9.56	<1.00	<1.00	79.8	<1.00	<1.00	50.0	<1.00	<1.00	<1.00
hpstd	2	8	hpstd-23	52.3	<1.00	<1.00	<1.00	52.0	<1.00	<1.00	<1.00	20.7	148	<1.00	10.4	<1.00	<1.00	<1.00	<1.00
std	3	1	std-31	3.93	19.3	<1.00	<1.00	4.10	10.1	10.3	<1.00	<1.00	84	<1.00	<1.00	49.8	<1.00	<1.00	<1.00
hpstd	3	2	hpstd-31	51.3	<1.00	<1.00	<1.00	51.3	<1.00	<1.00	<1.00	20.3	146	<1.00	10.2	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-04-B-SSM-W	3	3	S-14281-3	<1.00	14.2	33.9	<1.00	<1.00	<1.00	6.77	<1.00	<1.00	183	<1.00	121	32.9	<1.00	10.9	<1.00
std	3	4	std-32	4.06	19.6	<1.00	<1.00	4.17	9.83	9.95	<1.00	<1.00	81.6	<1.00	<1.00	50.6	<1.00	<1.00	<1.00
hpstd	3	5	hpstd-32	50.9	<1.00	<1.00	<1.00	51.4	<1.00	<1.00	<1.00	20.3	148	<1.00	10.3	<1.00	<1.00	<1.00	<1.00
EMHQ-LBE-SSM-W-110221	3	6	S-14280-3	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	510	<1.00	373	<1.00	<1.00	<1.00	<1.00
std	3	7	std-33	4.18	20.0	<1.00	<1.00	4.21	9.83	9.90	<1.00	<1.00	81.2	<1.00	<1.00	51.6	<1.00	<1.00	<1.00
hpstd	3	8	hpstd-33	48.0	<1.00	<1.00	<1.00	49.6	<1.00	<1.00	<1.00	19.5	145	<1.00	9.9	<1.00	<1.00	<1.00	<1.00

Table B-2. Measurements (mg/L) of the SSM Wash Solutions Measured by IC

PNNL ID	Block	Seq	Lab ID	Cl-	F-	PO_4^{3-}	SO_4^{2-}
CHK	1	1	CHK	1.03	1.04	0.947	0.951
IC BLANK	1	2	IC BLANK-11	<5.00	<5.00	<10.0	<10.0
EMHQ-LBE-04-B-SSM-W	1	3	S-14281-1	<5.00	<5.00	<10.0	296
EMHQ-LBE-SSM-W-110221	1	4	S-14280-1	<5.00	<5.00	<10.0	688
IC BLANK	1	5	IC BLANK-12	<5.00	<5.00	<10.0	<10.0
CHK	1	6	CHK	1.00	1.03	0.923	1.04
CHK	2	1	CHK	1.06	1.03	1.08	0.951
IC BLANK	2	2	IC BLANK-21	<5.00	<5.00	<10.0	<10.0
EMHQ-LBE-04-B-SSM-W	2	3	S-14281-2	<5.00	<5.00	<10.0	297
EMHQ-LBE-SSM-W-110221	2	4	S-14280-2	<5.00	<5.00	<10.0	691
IC BLANK	2	5	IC BLANK-22	<5.00	<5.00	<10.0	<10.0
CHK	2	6	CHK	0.999	1.03	0.921	0.978
CHK	3	1	CHK	1.01	1.03	0.922	0.921
IC BLANK	3	2	IC BLANK-31	<5.00	<5.00	<10.0	<10.0
EMHQ-LBE-04-B-SSM-W	3	3	S-14281-3	<5.00	<5.00	<10.0	297
EMHQ-LBE-SSM-W-110221	3	4	S-14280-3	<5.00	<5.00	<10.0	693
IC BLANK	3	5	IC BLANK-32	<5.00	<5.00	<10.0	<10.0
CHK	3	6	CHK	1.00	1.03	0.942	0.996

Table B-3. Results for Standards and Blanks Utilized During the Measurements of the Wash Solutions

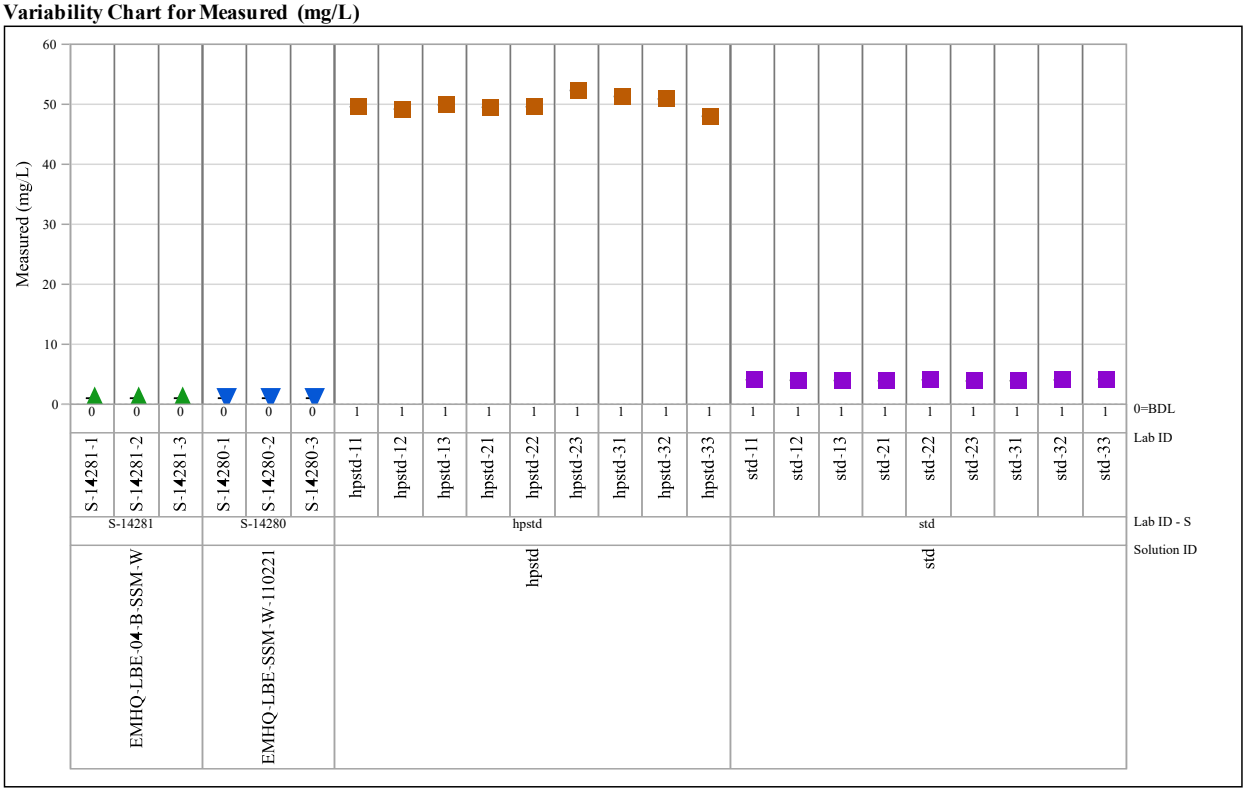
Solution ID	Analysis	Analyte	Reference (mg/L)	Measured (mg/L)
CHK	IC	Cl^-	1.00	1.02
CHK	IC	F^-	1.00	1.03
CHK	IC	PO_4	1.00	0.956
CHK	IC	SO_4	1.00	0.973
IC BLANK	IC	Cl^-	0	<5.00
IC BLANK	IC	F^-	0	<5.00
IC BLANK	IC	PO_4	0	<10.0
IC BLANK	IC	SO_4	0	<10.0
hpstd	ICP	Al	50	50.0
hpstd	ICP	B	0	<1.00
hpstd	ICP	Ca	0	<1.00
hpstd	ICP	Cr	0	<1.00
hpstd	ICP	Fe	50	51.0
hpstd	ICP	K	0	<1.00
hpstd	ICP	Li	0	<1.00
hpstd	ICP	Mg	0	<1.00
hpstd	ICP	Mn	20	20.4
hpstd	ICP	Na	150	147
hpstd	ICP	P	0	<1.00
hpstd	ICP	S	10	10.2
hpstd	ICP	Si	0	<1.00
hpstd	ICP	Ti	0	<1.00
hpstd	ICP	V	0	<1.00
hpstd	ICP	Zr	0	<1.00
std	ICP	Al	4	4.01
std	ICP	B	20	19.5
std	ICP	Ca	0	<1.00
std	ICP	Cr	0	<1.00
std	ICP	Fe	4	4.15
std	ICP	K	10	10.0
std	ICP	Li	10	10.0
std	ICP	Mg	0	<1.00
std	ICP	Mn	0	<1.00
std	ICP	Na	81	82.8
std	ICP	P	0	<1.00
std	ICP	S	0	<1.00
std	ICP	Si	50	50.5
std	ICP	Ti	0	<1.00
std	ICP	V	0	<1.00
std	ICP	Zr	0	<1.00

Table B-4. Average Measurements (mg/L) of the SSM Wash Solutions

		IC				ICP														
Solution ID	**Lab ID**	**Cl^- (mg/L)**	**F^- (mg/L)**	**PO_4 (mg/L)**	**SO_4 (mg/L)**	**Al (mg/L)**	**B (mg/L)**	**Ca (mg/L)**	**Cr (mg/L)**	**Fe (mg/L)**	**K (mg/L)**	**Li (mg/L)**	**Mg (mg/L)**	**Mn (mg/L)**	**Na (mg/L)**	**P (mg/L)**	**S (mg/L)**	**Si (mg/L)**	**Ti (mg/L)**	**V (mg/L)**	**Zr (mg/L)**
EMHQ-LBE-04-B-SSM-W	S-14281	<5.00	<5.00	<10.0	297	<1.00	13.6	33.3	<1.00	<1.00	<1.00	6.41	<1.00	<1.00	182	<1.00	119	31.9	<1.00	10.7	<1.00
EMHQ-LBE-SSM-W-110221	S-14280	<5.00	<5.00	<10.0	691	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	540	<1.00	379	<1.00	<1.00	<1.00	<1.00

Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte

Analyte=Al (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=B (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

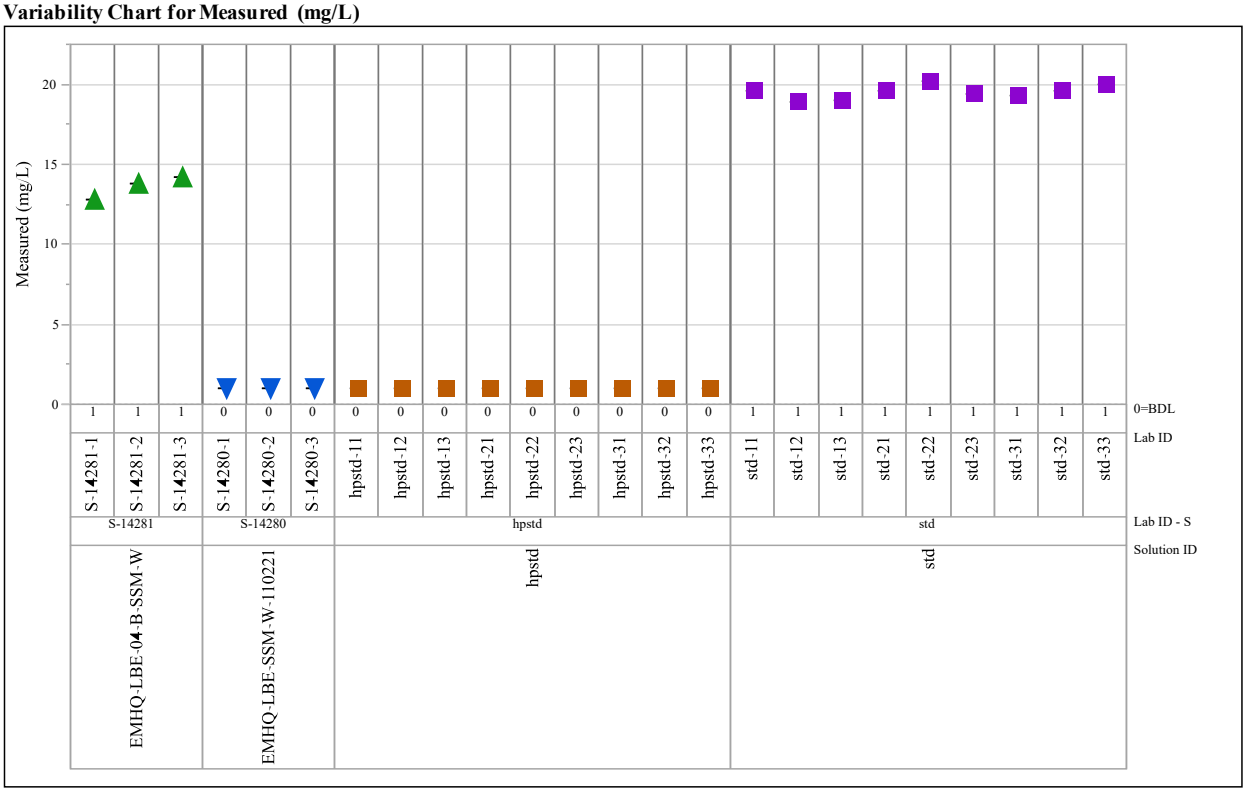
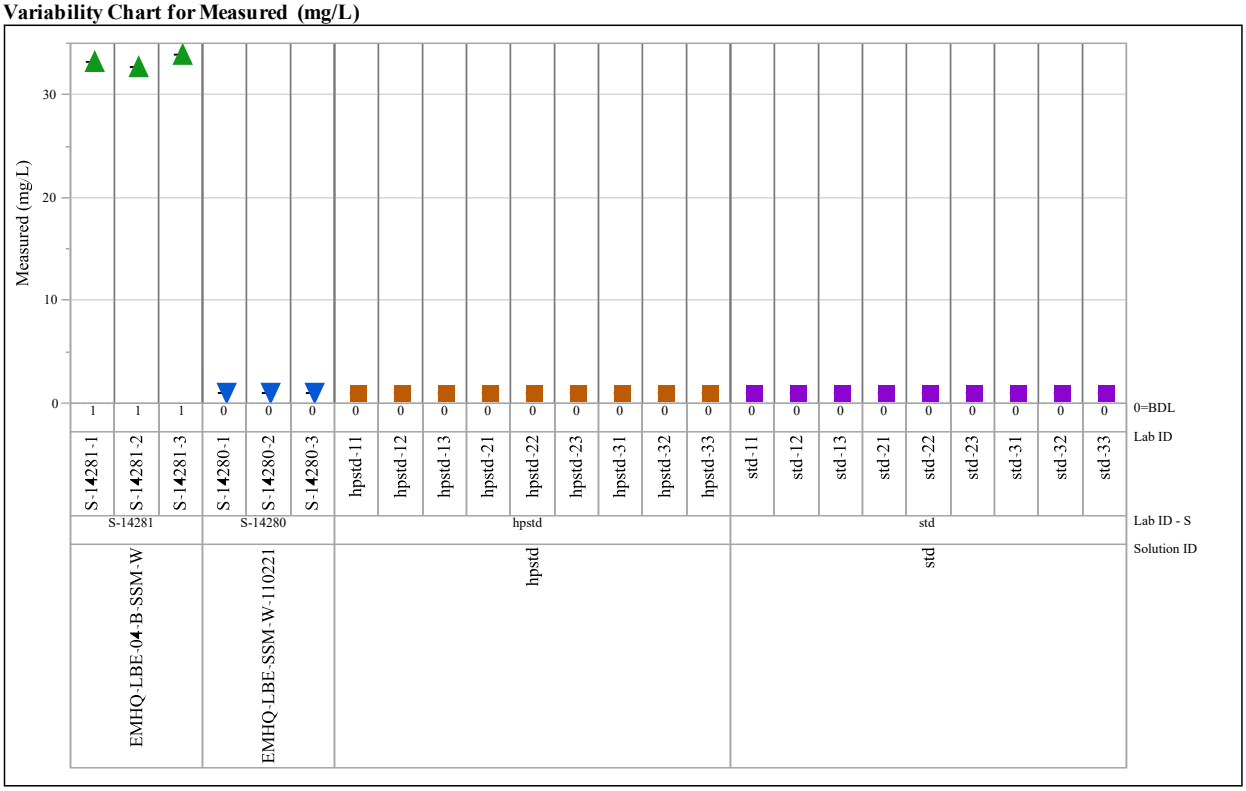


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=Ca (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=Cl (mg/L), Analysis=IC
Variability Chart for Measured (mg/L)

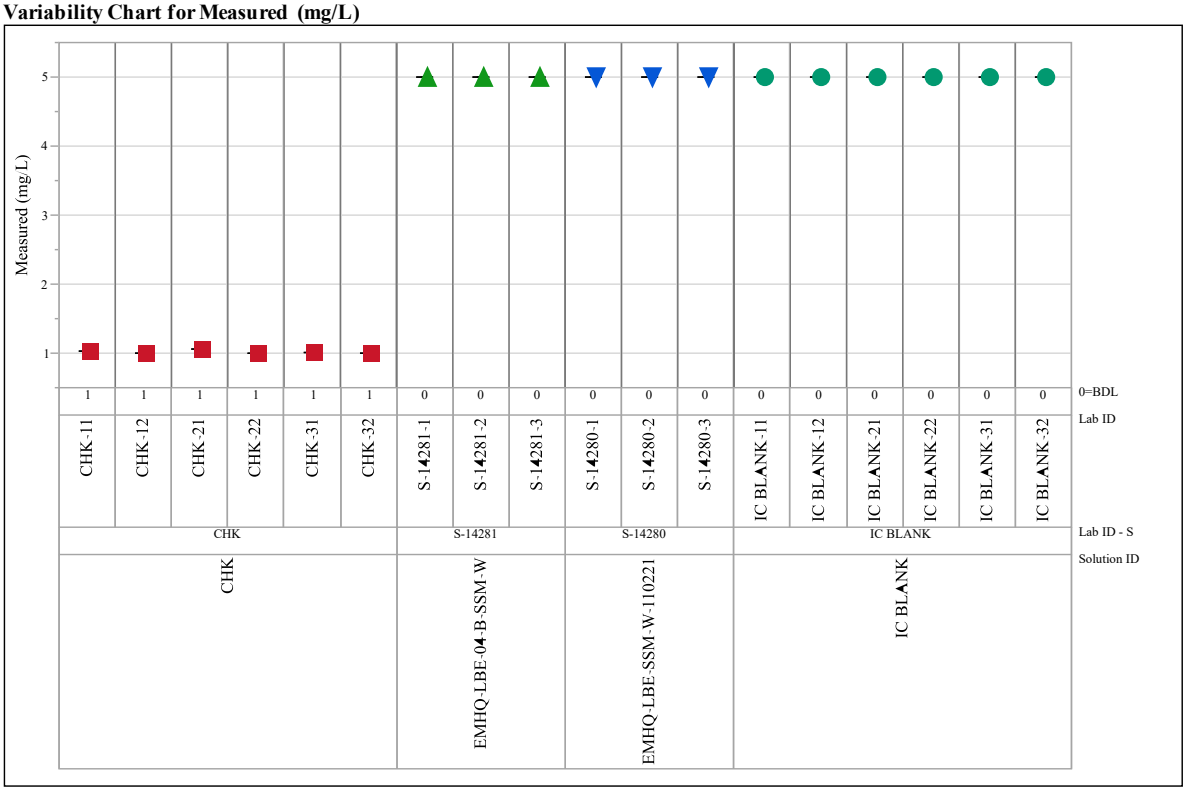
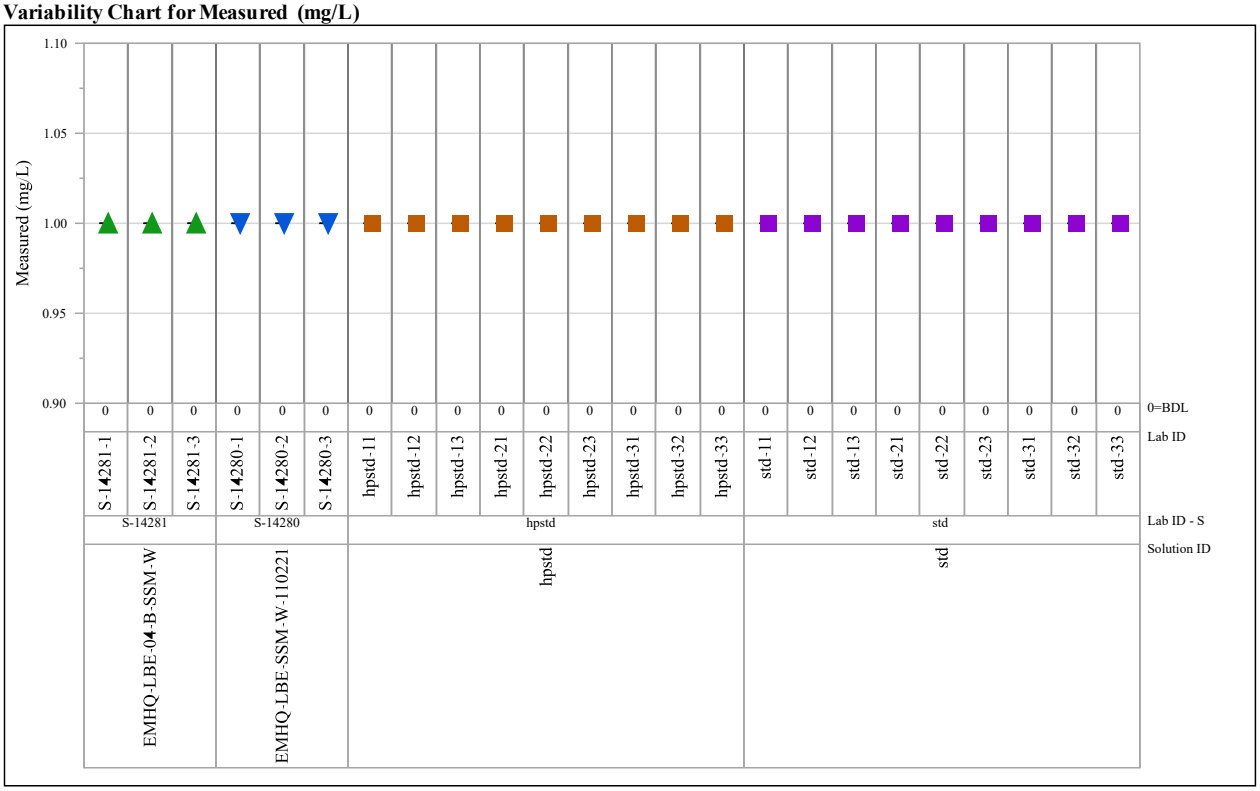


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=Cr (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=F (mg/L), Analysis=IC
Variability Chart for Measured (mg/L)

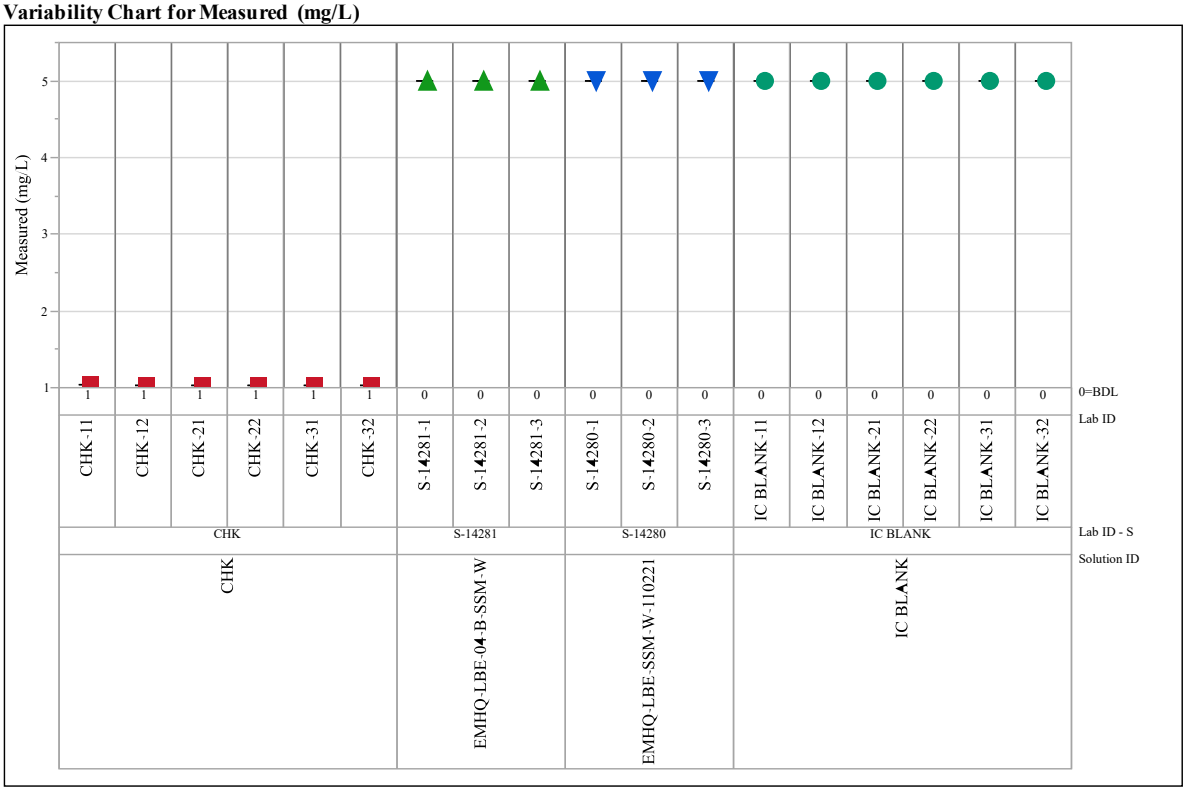
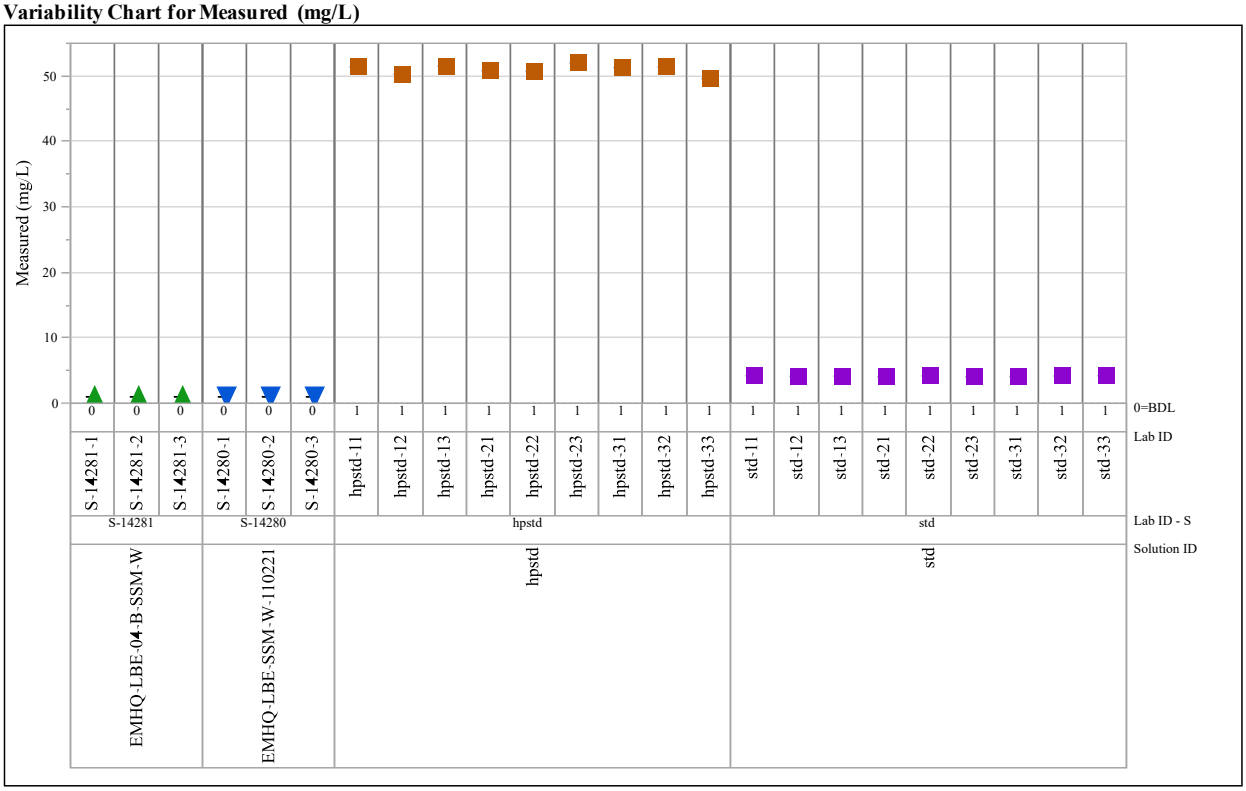


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=Fe (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=K (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

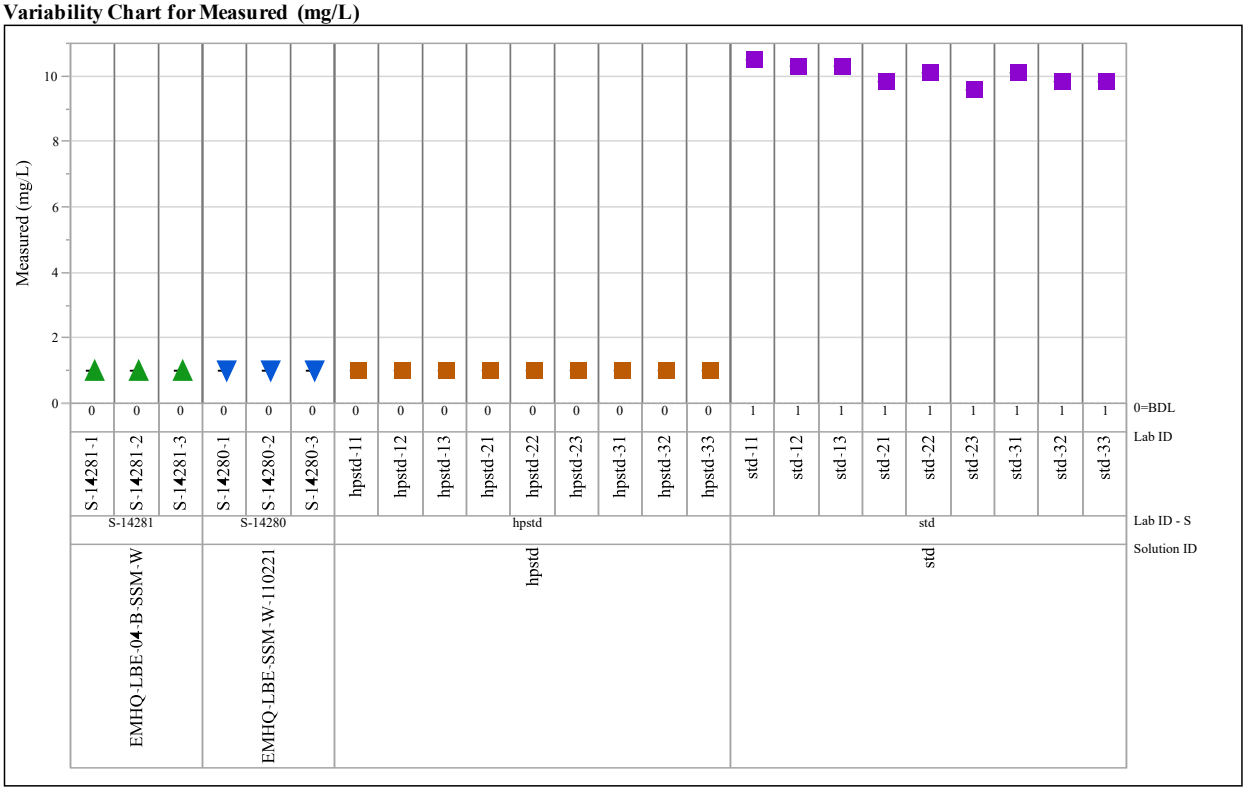
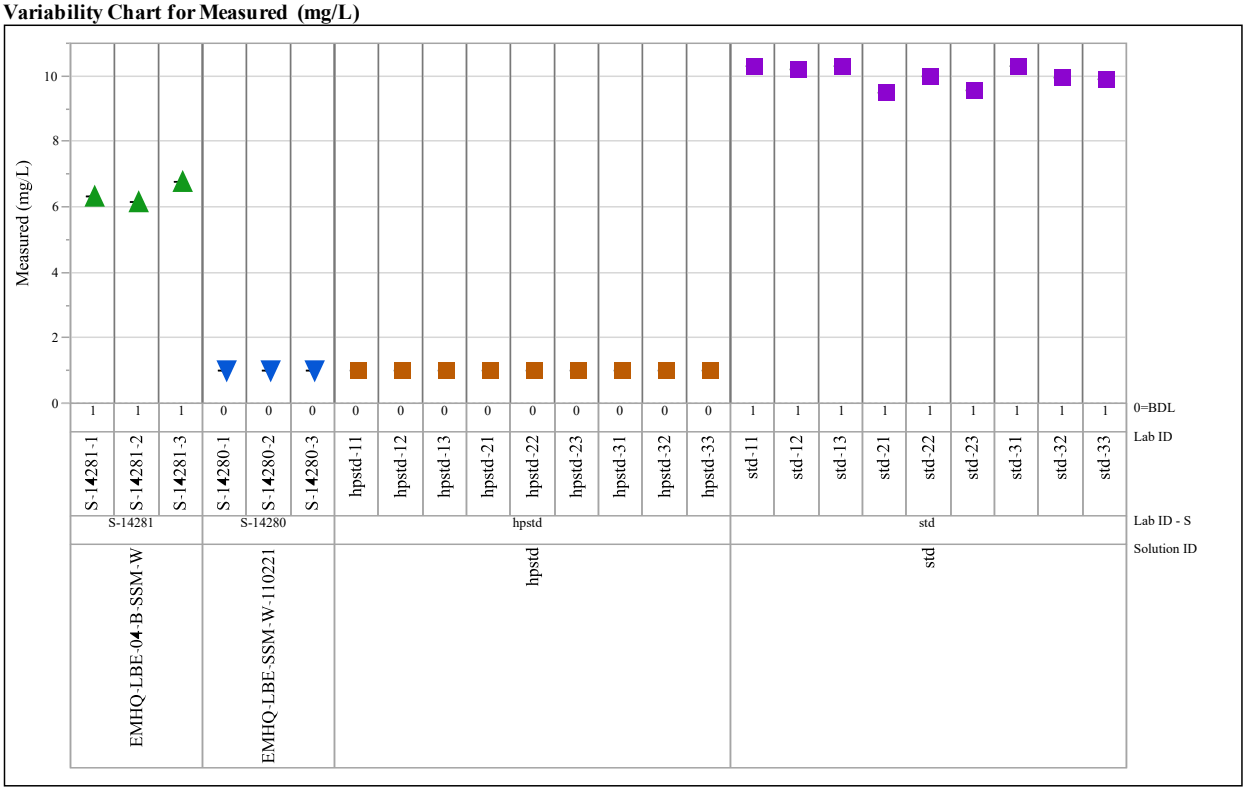


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=Li (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=Mg (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

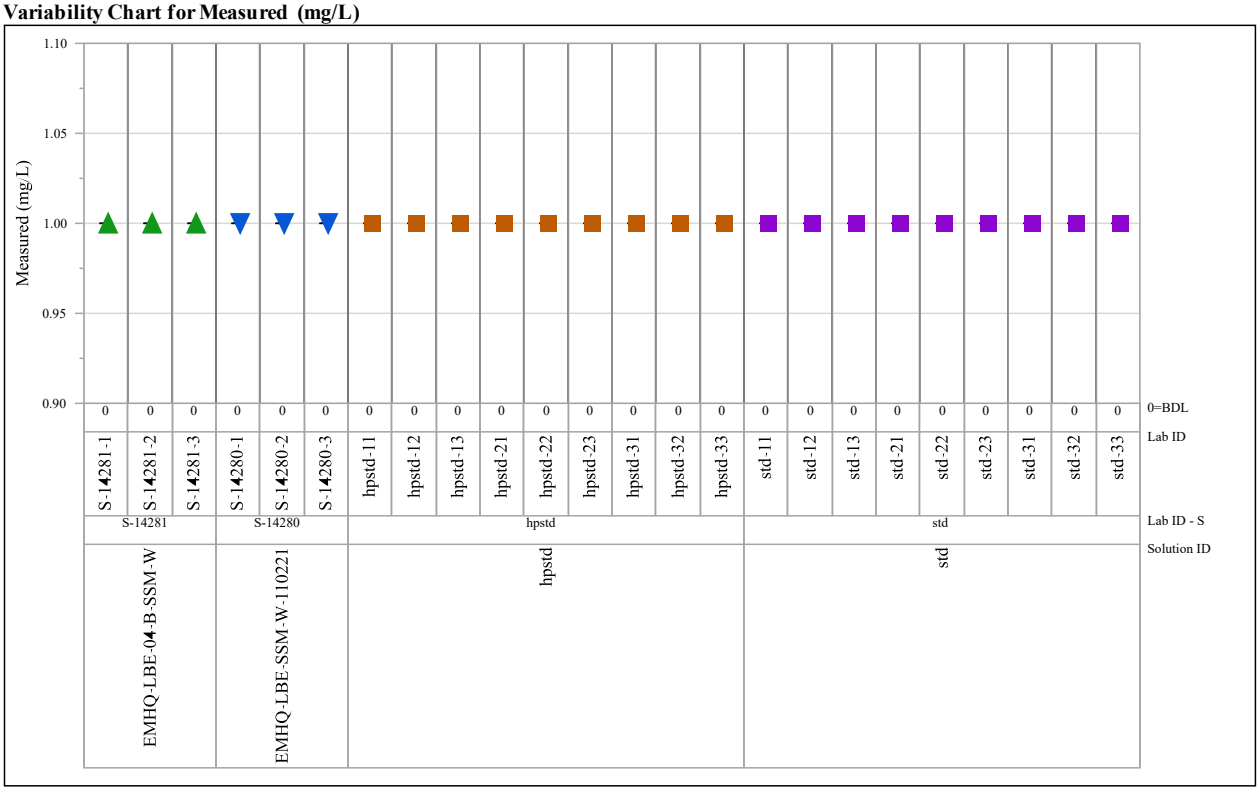
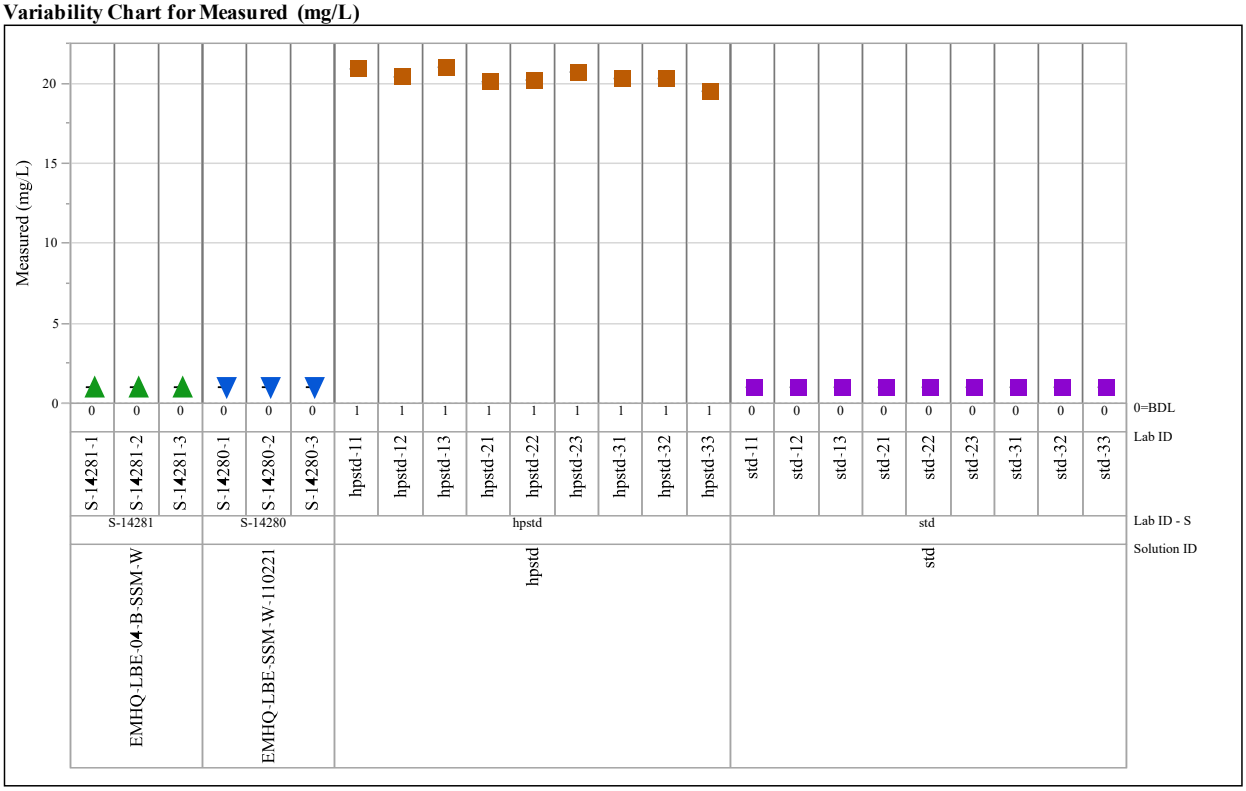


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=Mn (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=Na (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

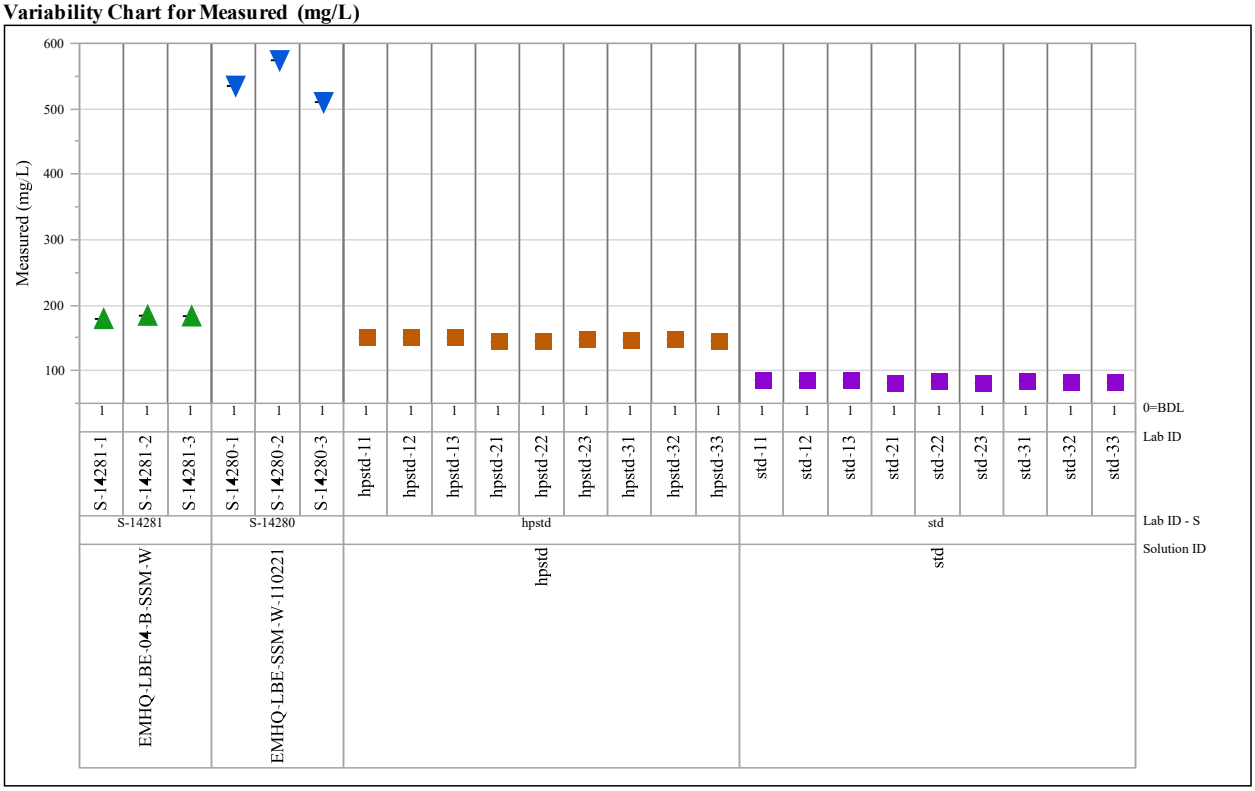
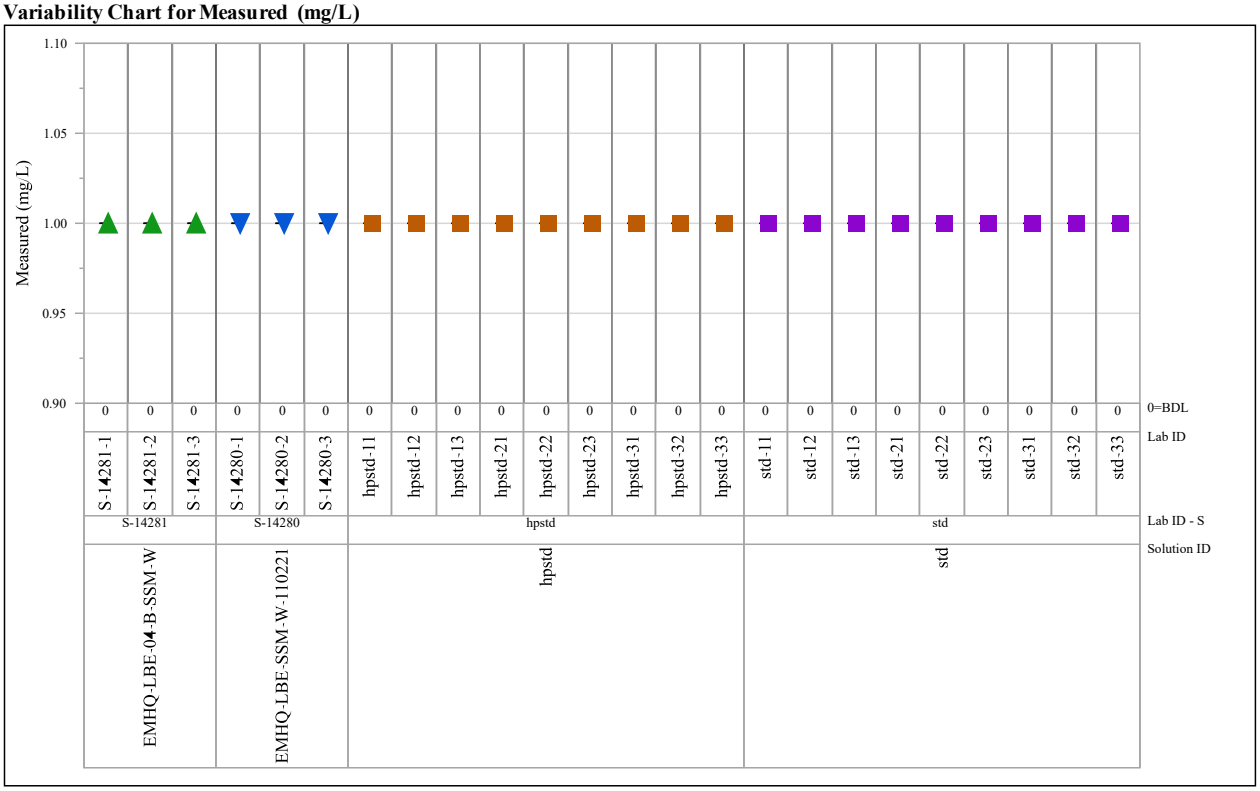


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=P (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=PO4 (mg/L), Analysis=IC
Variability Chart for Measured (mg/L)

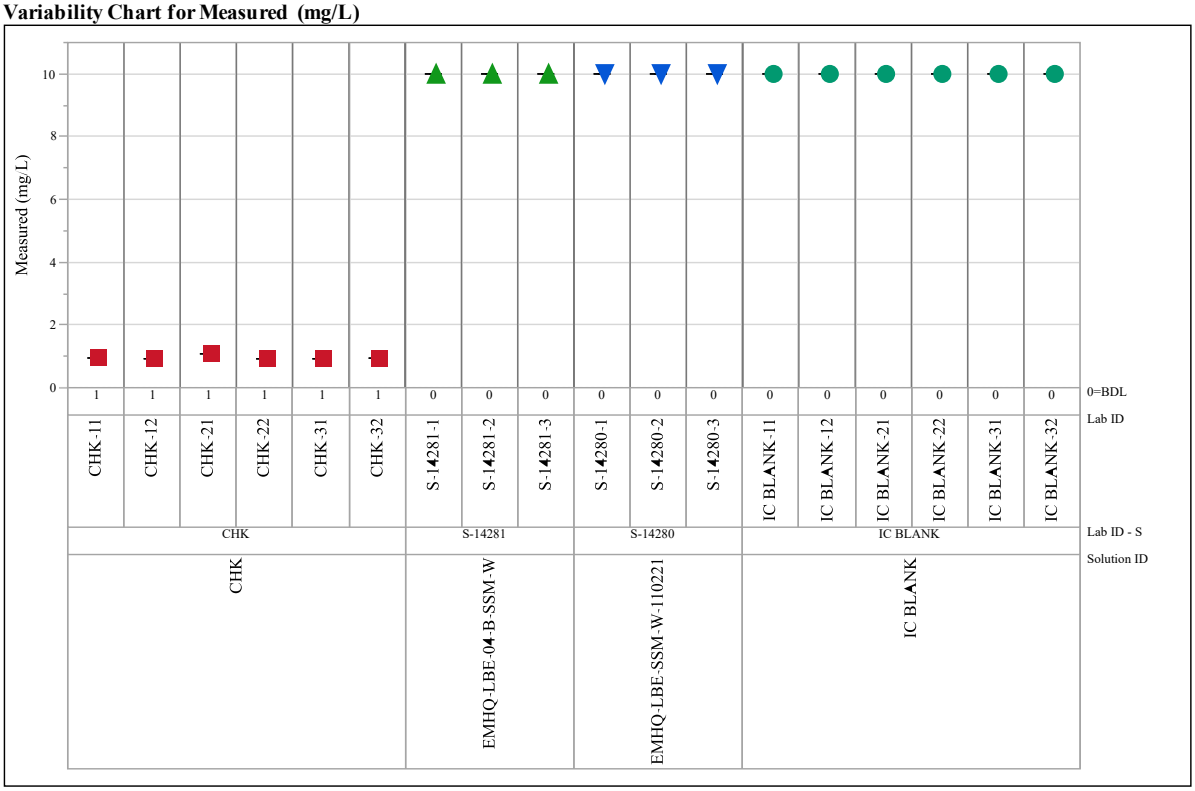
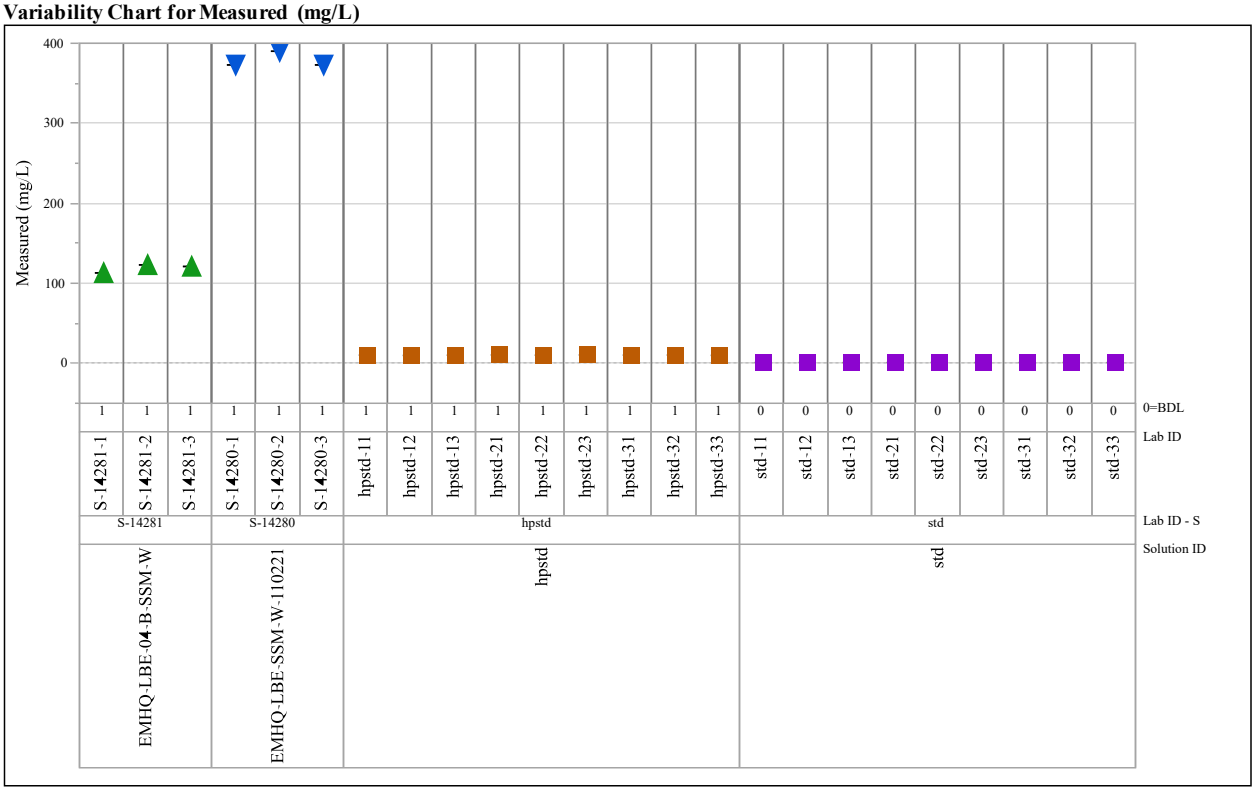


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=S (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=Si (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

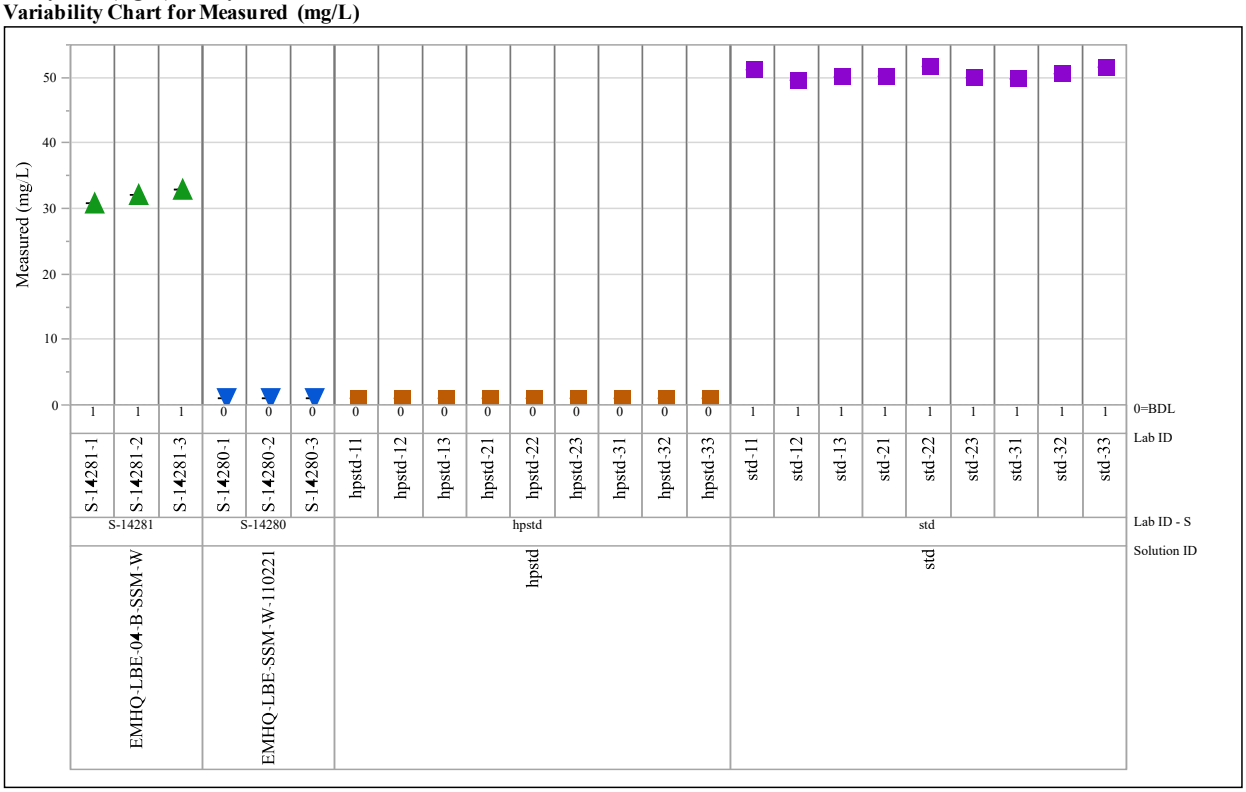
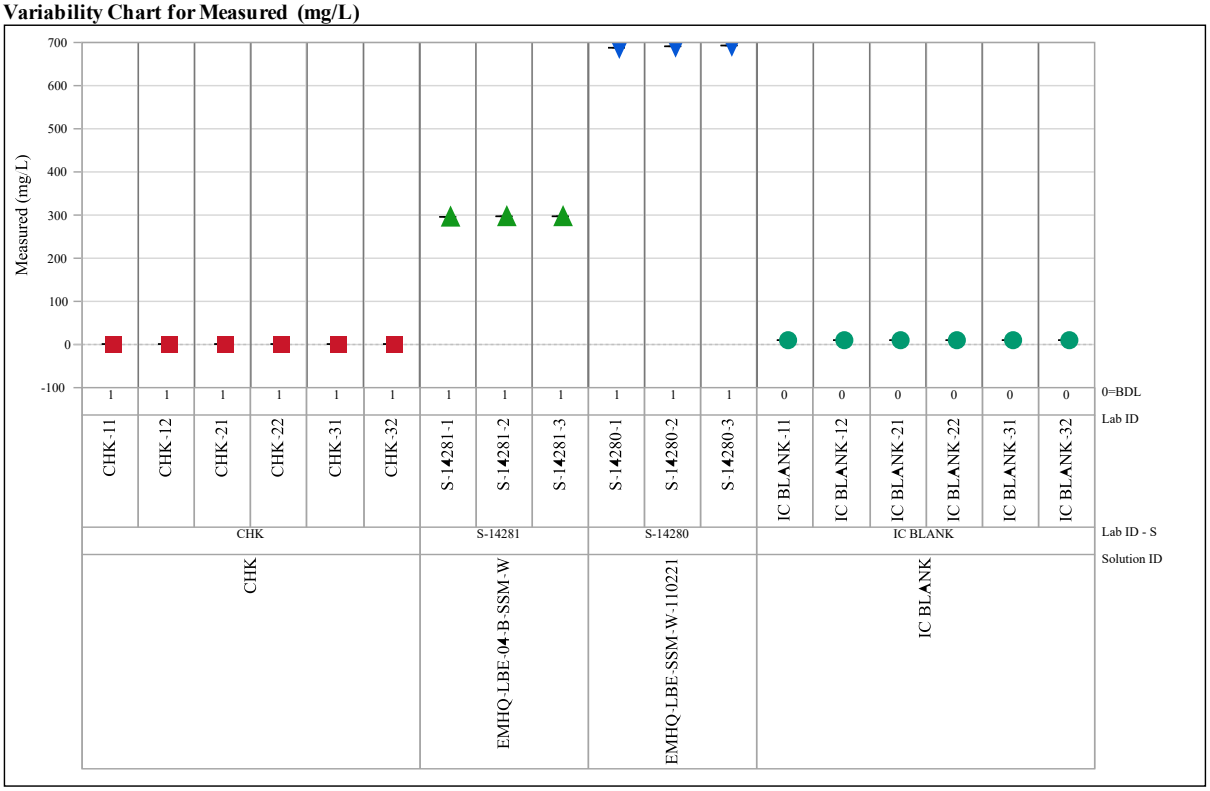


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=SO4 (mg/L), Analysis=IC
Variability Chart for Measured (mg/L)



Analyte=Ti (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)

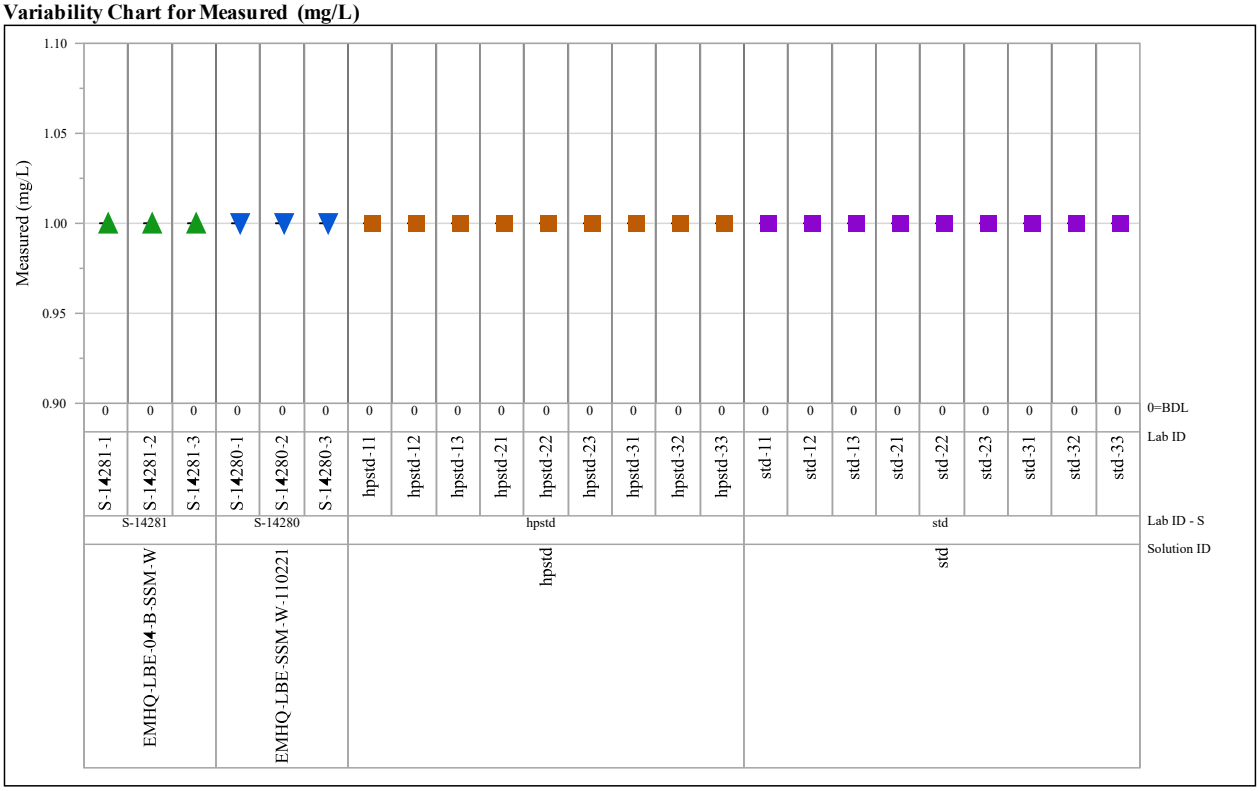
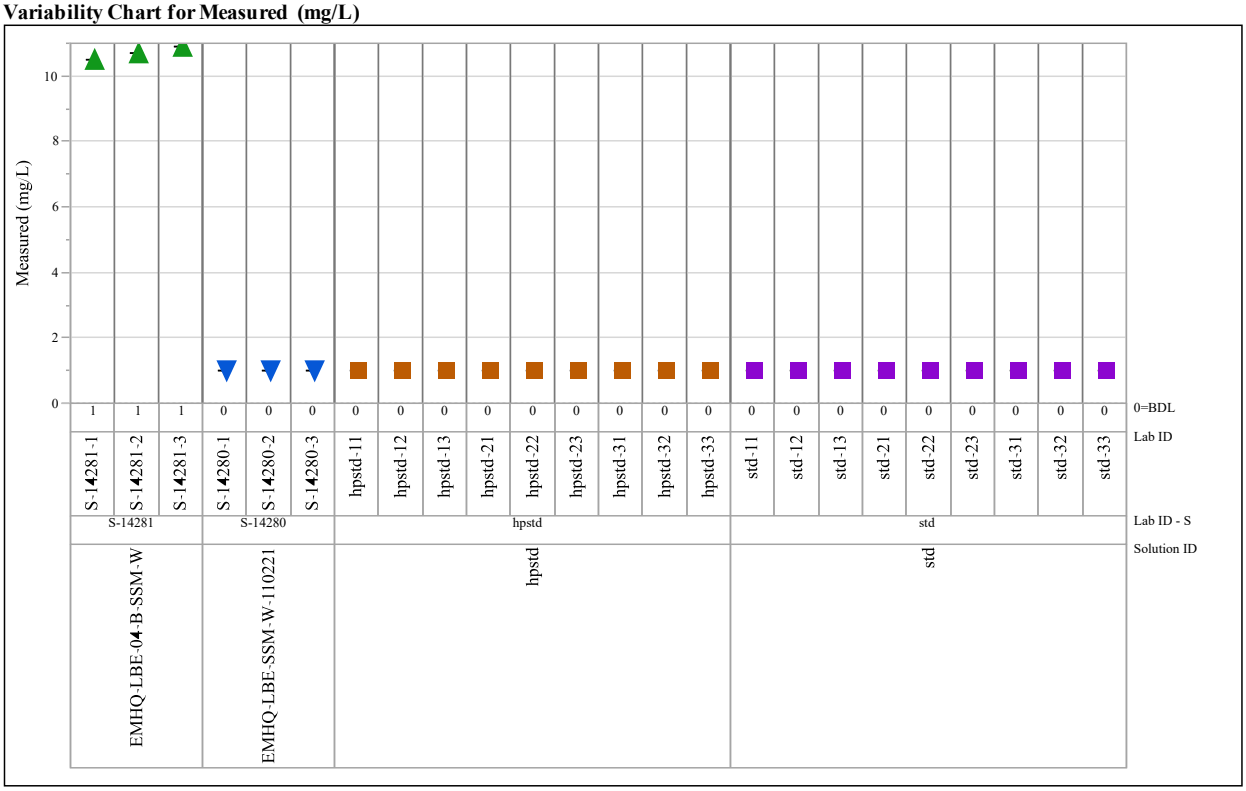
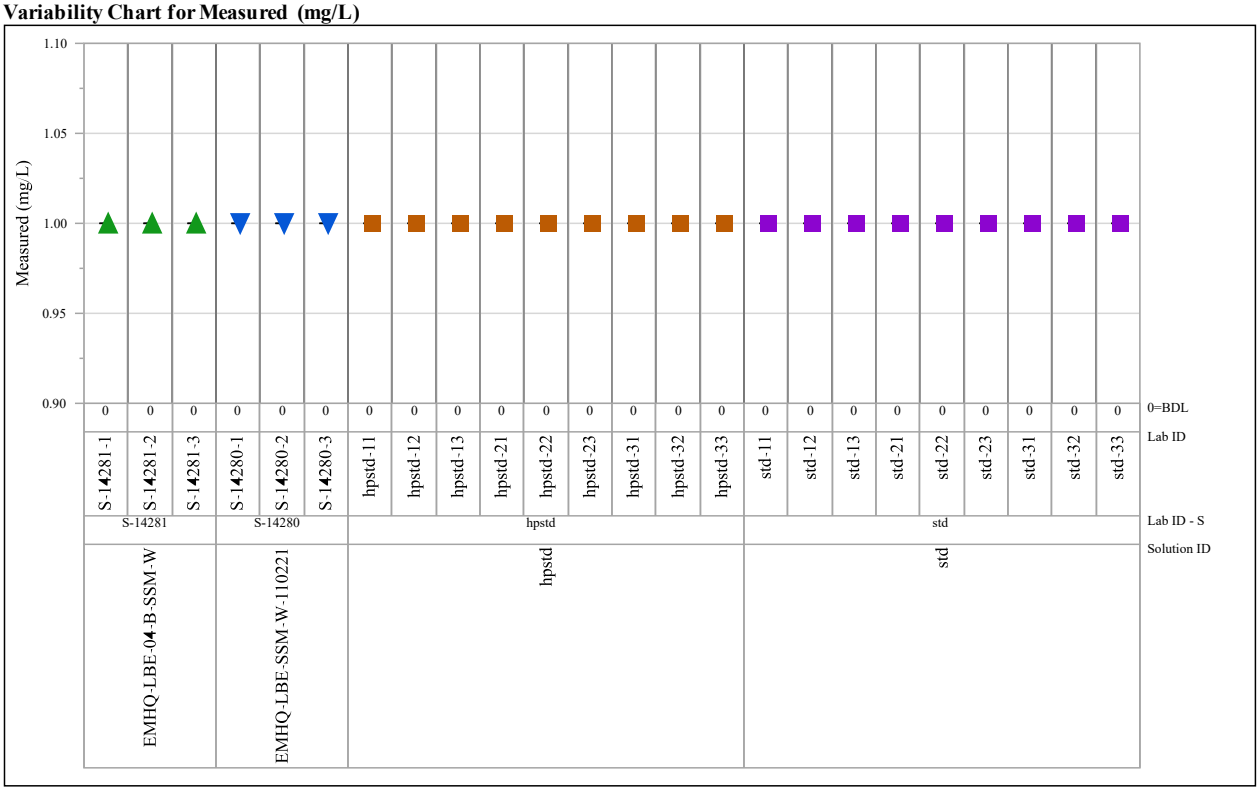


Exhibit B-1. Measurements of the SSM Wash Solutions by Solution ID by Analyte (continued)

Analyte=V (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



Analyte=Zr (mg/L), Analysis=ICP
Variability Chart for Measured (mg/L)



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