

Evaluation of MPI-DING Glasses for Use as Electron Probe Standards.

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MPI-DING glasses were prepared as melts of relatively large volumes of natural rock specimens in a cooperative project between the Max Planck Institute and the USGS [1,2,3]. They are intended as isotope and trace element standards for micro-analytical techniques such as LA-ICP-MS. The glasses were analyzed for a very complete set of major, minor and trace elements, as well as isotope ratios. Here, I report on an investigation of the major and minor element compositions and homogeneity of these standard materials, and evaluate their use as reference materials for electron probe analysis. These glasses are potentially attractive as EPMA standards because of their wide range of compositions (~46-75.6 wt. % SiO₂, ~0.1-22.5 wt. % MgO, 10.8-17.6 % Al₂O₃, and ~3.3-11 % FeO). One approach to calibration for microprobe analysis supports the use of standards that are as close to matching the unknown compositions as possible, such that the impact of uncertainties in mass-absorption coefficients and matrix corrections is minimized. The availability of these glasses, with such a wide range of compositions, will enable researchers investigating natural and experimental glasses to more closely match their unknowns, and thus potentially improve their results. The data presented here confirm the major element homogeneity of these glasses. Proposed revised compositions of the glasses are also presented, as several of the MPI-DING standards have published compositions with somewhat low totals, which indicated the need for more investigation of their major element compositions.

The natural rock types used to produce these synthetic glasses include Gorgona Island komatiite, Iceland rhyolite, Kilauea and Mauna Loa Hawaiian basalts, a quartz diorite from the Italian Alps, and Mt. St. Helens andesitic ash. The proposed revised major element compositions for these standard materials have been determined by calibration with Smithsonian Institute natural glass standards: VG568 rhyolite, VG2 mid-ocean ridge basalt (MORB), and A99 Hawaiian basalt, as well as various mineral standards. VG568 rhyolite was used to calibrate Al, Na and K. VG2 basalt was used to calibrate Si. Hawaiian basalt A99 was used to calibrate Ca and Fe. San Carlos olivine was used to calibrate Mg. Chromium was calibrated on the Smithsonian standard, Tiebaghi chromite. Titanium was calibrated on rutile. Phosphorus was calibrated on a fluor-apatite. Manganese was calibrated with the mineral rhodonite. Ni was calibrated on Ni metal. The mineral standards used can be obtained commercially from Astimex, or SPI. Sodium, potassium and phosphorus were analyzed first on their respective spectrometers, both on primary standards and MPI glasses, in order to minimize potential effects from electron beam damage on the glasses.

In general, our data, presented in Table 1, matched the published compositions for the MPI glasses for most elements, within analytical uncertainties. However, somewhat better totals (all between 99.3 and 100.4) were achieved. The results presented here have somewhat higher Si, Ca and Fe for some standards, and these increases enabled the achievement of improved totals. The original published values for these standards showed totals of 98.4 to 99.8 wt. % oxides. In particular, the original published data for ML3B-G and KL2-G have low totals (~98.4 wt. % for both), and given that shortfalls were not explainable based on unanalyzed elements, they indicated that further analytical efforts were needed. Results presented here are improved compositions for major elements in these standards.

Among the seven MPI-DING glasses, one we received, GOR-128-G, a highly magnesian glass, had experienced abundant quench crystallization of olivine, and is not discussed here further. The other six glasses were found to be free of any crystals, and were found to be very homogenous in major element composition (see Table 2) [4].

Table 1: Proposed Revised Compositions of MPI-DING glasses

	GOR-132-G	ATHO-G	KL2-G	T1-G	StHs6-80-G	ML3B-G
SiO ₂	46.22	75.86	50.73	57.27	63.93	52.42
TiO ₂	0.30	0.24	2.56	0.77	0.69	2.08
P ₂ O ₅	0.03	0.02	0.27	0.22	0.18	0.24
Al ₂ O ₃	10.82	12.11	13.19	17.57	17.55	13.52
Cr ₂ O ₃	0.36	-	0.04	-	-	0.02
FeO ^t	10.30	3.23	10.87	6.82	4.39	11.08
MnO	0.15	0.10	0.16	0.14	0.07	0.17
MgO	22.51	0.10	7.43	4.02	2.03	6.67
NiO	0.15	-	0.02	-	0.01	0.02
CaO	8.66	1.73	11.27	7.60	5.42	10.68
Na ₂ O	0.82	3.55	2.28	3.04	4.44	2.31
K ₂ O	0.03	2.70	0.49	1.88	1.30	0.39
Total	100.35	99.64	99.31	99.33	100.01	99.60

Compositions were determined on a JEOL 8530F EPMA. Beam conditions were 15 kV and 15 nA. Glass standards and unknowns were analysed with a 10 µm diameter defocused beam. ZAF matrix-corrections were applied to convert x-ray yields into compositions. “-“ indicates below detection limits.

Table 2. Homogeneity of Major Elements in Glass Standards

	SiO ₂	Al ₂ O ₃	FeO	CaO	MgO	Na ₂ O	TiO ₂
GOR-132-G	0.40	1.19	1.05	1.32	0.63	6.8	8.4
ATHO-G	0.78	0.96	2.19	3.52	-	9.6	9.5
KL2-G	0.52	1.00	1.39	0.94	1.23	3.1	1.7
T1-G	0.57	0.89	1.40	1.38	1.33	3.3	3.7
StHs6-80-G	0.45	0.65	2.38	1.26	1.98	10.5	4.0
ML3B-G	0.52	0.83	1.00	1.27	1.32	3.8	2.1

2 standard deviations for 25-30 replicate analyses, expressed as a per cent relative to the abundance of each element present.

References:

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- [2] <http://www.mpic.de/en/research/111561/jochum-group/mpi-ding-reference-glasses.html>
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- [4] The author acknowledges support from NASA-JSC, and acknowledges the Max Planck Institute and USGS for the preparation, analysis and distribution of these standard glasses. I also gratefully acknowledge the receipt of standards from the Smithsonian.