



Certificate of Analysis

IAG MUH-1

(Ultramafic rock)

International Association of Geoanalysts

13 Belvedere Close, Keyworth
Nottingham, NG12 5JF, UK
e-mail iageo.ltd@ntlworld.com

Telephone +44 (0)115 9375219

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Description of the certified reference material

Material for this reference material was collected from the "Hartsteinwerke Preg" quarry near Kraubath, Styria, Austria. The sample consists of partially serpentinised harzburgite typical of the highly depleted serpentinites of the massive mantle section of the Spike Complex. Automated point counting gives an approximate volumetric mineral composition of 57 % serpentine, 35% olivine, 5% brucite, trace (0.2 - 1%) calcic amphibole, magnetite, talc, chromite, and enstatite, and ultra-trace (< 0.01%) Fe-Ni sulfide. Approximately 150 kg of crushed material were ground to $90\% \leq 63 \mu\text{m}$ (230-mesh) at the Ontario Geoscience Laboratories, using a continuous-feed Cr-steel ring mill, then blended for 24 hours in a large-capacity blender. Approximately 80 kg of the blended material was subsequently randomly split and transferred into 100 g jars for distribution.

Intended use

This certified reference material is designed for use by laboratories undertaking the determination of major and trace element mass fractions in silicate rocks and equivalent matrices. It is suitable for the calibration of a measurement system, for the assessment of a measurement procedure, for assigning values to other materials, and quality control. Note that the material may be used only for a single purpose in the same measurement process. For example, it must not be used for calibration and method validation at the same time.

Instructions for handling

When non-volatile analytes are to be determined, test portions of the test sample must be dried at $105 \pm 5^\circ\text{C}$ for at least 2 hours. Volatile analytes (e.g., As) should be determined on the material as received; separate portions should be dried as described above to obtain a correction factor for moisture. Avoid contamination and cross-contamination of the test material during handling. The material should not be reground before use. The mass loss on drying has been found to be in the range 0.06 to 0.75 g/100g.

Storage information

Store in a sealed container in a cool dry environment.

Minimum sample size

Based on an assessment of heterogeneity and a detailed evaluation of data submitted by laboratories contributing certification data by various techniques (e.g., ICP-MS, XRF, INAA), the minimum size of test portion is recommended to be 0.2 g.

Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. As a consequence, nominal period of validity of this certificate is set at 20 years. On exposure to air, the material may absorb moisture, and the instructions for removing absorbed water before use of the material must be followed.

Certified values

This material was certified based on procedures that are summarised in the International Association of Geoanalysts' Certification Protocol (Kane et al., 2003, Geostandards and Geoanalytical Research, 27, 227-244). Thirty-six laboratories were invited to participate in this certification round on the basis of their successful performance in round 20 of the IAG GeoPT proficiency testing programme. A nested design was adopted for data acquisition as proposed in the IAG certification protocol. Participating laboratories received 3 bottles of MUH-1 and one bottle of MGL-GAS (the "traceability" sample which was used here for quality control purposes). Participating laboratories were requested to make two independent sample preparations (e.g., digestions) of each bottle and analyse each preparation on two different days. Laboratories were thus requested to submit 12 values (3x2x2 bottles x Prep x Day) for each measurand for which they had the analytical capability.

Further details of the quality of data and data distributions may be found in the associated IAG MUH-1 certification report.

Uncertainties

U is the expanded uncertainty, corresponding to 95% confidence limits and incorporates the relevant student t factor (t) to account for the finite number of contributing laboratory average measurements. The standard uncertainty (u) may be calculated from $u = U/t$ and includes a random component, and a material variability (heterogeneity) component, as described in Kane et al. (2003). The stability component has not been included, as it is vanishingly small compared to the other components. An additional contribution has been added to uncertainty data for TiO₂, Cr, Cu, Lu, Rb, Sm, Sr and Yb to account for technique bias observed in the respective data sets.

Number of values, 'n'

The number of laboratory means, n, that contributed to the estimation of the certified value after rejection of outliers is listed in the tables. Outliers were selected based on Youden plots, Mandel's k and detection limit criteria.

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Certified values and uncertainties

Measurand	Certified value	Uncertainty (expanded)	Unit	n	Measurand	Certified value	Uncertainty (expanded)	Unit	n
SiO ₂	40.38	0.17	g/100g	22	Er	0.1082	0.0022	mg/kg	17
TiO ₂	0.0344	0.0026	g/100g	24	Eu	0.0262	0.0010	mg/kg	15
Al ₂ O ₃	1.334	0.019	g/100g	20	Ga	1.38	0.09	mg/kg	11
Fe ₂ O ₃ T	8.59	0.05	g/100g	23	Hf	0.044	0.013	mg/kg	13
MnO	0.1179	0.0014	g/100g	21	Ho	0.0357	0.0015	mg/kg	13
MgO	38.25	0.16	g/100g	23	Lu	0.0191	0.0013	mg/kg	16
CaO	1.213	0.010	g/100g	21	Nd	0.177	0.007	mg/kg	15
Na ₂ O	0.104	0.010	g/100g	18	Ni	2104	26	mg/kg	21
K ₂ O	0.012	0.003	g/100g	14	Pr	0.0351	0.0019	mg/kg	13
P ₂ O ₅	0.0075	0.0022	g/100g	13	Rb	0.27	0.04	mg/kg	14
LOI	9.38	0.09	g/100g	22	Sc	9.0	0.4	mg/kg	19
					Sm	0.0677	0.0026	mg/kg	17
Ba	4.98	0.29	mg/kg	17	Tb	0.0212	0.0007	mg/kg	15
Co	106.7	2.5	mg/kg	22	Th	0.018	0.004	mg/kg	11
Cs	0.099	0.003	mg/kg	13	Tm	0.0170	0.0007	mg/kg	14
Cu	19.1	1.2	mg/kg	17	Yb	0.118	0.005	mg/kg	18
Dy	0.153	0.007	mg/kg	17	Zn	44.5	1.8	mg/kg	23

Certified value and uncertainty - see text.

n: number of values (after outlier rejection) contributing to the certified value.

Fe₂O₃T = total iron expressed as Fe₂O₃.

LOI = loss on ignition.

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Information values and uncertainties

Measurand	Information value	Uncertainty (expanded)	Unit	n
As	3.6	0.7	mg/kg	8
Ce	0.20	0.02	mg/kg	14
Cr	2710	110	mg/kg	21
Gd	0.110	0.006	mg/kg	12
La	0.134	0.009	mg/kg	15
Li	1.8	0.5	mg/kg	7
Nb	0.062	0.022	mg/kg	8
Pb	0.42	0.07	mg/kg	8
Sb	0.134	0.019	mg/kg	5
Sr	8.5	0.4	mg/kg	24
U	0.014	0.004	mg/kg	12
V	41	4	mg/kg	19
Y	0.97	0.08	mg/kg	17

Information value and uncertainty – see text.

n: number of values contributing to the information value.

Information values

Information values are designed to provide guidance on the mass fractions of other selected elements; these values should not be used to validate analytical measurements.

Metrological traceability

Traceability was demonstrated for this reference material by requesting participating laboratories to co-analyse the certified reference material MGL GAS. This material is a serpentine, supplied by the Central Geological Laboratory, Mongolia, and had been previously certified using the International Association of Geoanalysts' Protocol. The certificate of analysis of this material may be found at http://www.iageo.com/Geoanalyst/IAG-CRM-2_10Mar2009.pdf. An assessment of the analytical results for MGL GAS was undertaken to confirm the absence of systematic bias, thus establishing an unbroken chain of comparisons between the present MUH-1 certification and a previous geochemical certification project.

Certification characterisation report

Further details of the procedures used, and of the results, their statistical analysis and data assessment on which the property values listed in this certificate are based, can be found in the IAG MUH-1 characterisation report.

Safety information

Silicate powders can cause harm especially if ingested or in contact with the skin. User organisations must undertake a health and safety risk assessment and ensure that the appropriate procedures are followed in the handling and use of this material.

Legal notice – terms and conditions

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Revisions

This certificate is version 1.00. Any revisions to this Certificate of Analysis will be made available on the IAGeo Limited web site (www.iageo.com).

Acknowledgements

Members of the IAG Certification Committee are gratefully acknowledged for their contributions to this certificate, but especially Marcus Burnham and Jean Kane.

Approvals

This Certificate of Analysis was approved on behalf of the International Association of Geoanalysts.

Name Philip J Potts **Position** Chair of the IAG Certification and Reference Material Committee **Date** 21st September 2015

Name Thomas Meisel **Position** President of the International Association of Geoanalysts. **Date** 3rd November 2015