Reference Material Certificate

IAG CRM 3 / CGL 002
MGL-OShBO (Alkaline Granite)

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Status of reference material certificate
This material was originally certified by the Central Geological Laboratory, Ulaanbaatar, Mongolia in 1999, but was re-certified in a collaborative project with the International Association of Geoanalysts using the IAG certification protocol described by Kane et al. (2007b). The results of this certification were published in a certificate of analysis issued on 10th March 2009. This present reference material certificate replaces the certificate of analysis issued in 2009 with detailed editorial and formatting changes and a revised expiry date and traceability statement. However, certified values and uncertainties are unaltered.

Description of the certified reference material
MGL-OShBO candidate CRM with a total mass of 400 kg was collected from "Tsagaan Horoot" of Buren somon in the Central Province of Mongolia following standard procedures and under the guidance of field geologists. It was originally prepared, packaged and certified in 1999 by the Central Geological Laboratory (CGL), Ulaanbaatar, Mongolia. The material consists of a homogeneous powder of which 93.3 wt. % passed a 63 µm sieve, while 0.44% was larger than 100 µm. The mineralogy of the material (in % m/m) has been determined to be:

Minerals	*g/100 g*
Albite	*32.2*
Potassium feldspar	*32.1*
Quartz	*31.5*
Muscovite, lepidolite	*3.7*
Topaz, apatite	*0.35*
Zircon, sphene, magnetite, ilmenite and pyrite	*minor*

The original preparation, homogeneity and stability testing were performed by the Central Geological Laboratory, Ulaanbaatar, Mongolia (Erdenetsetseg and Davaasuren 1999). Four hundred kg of material were collected. The raw material was first crushed using jaw and roll crushers and then further processed in a disc mill to produce powder, of which 97.8 wt % was <74 µm. A total of 288.5 kg of sample resulted. Homogenization had to be done in two batches because the total volume exceeded the blender capacity. Before bottling, homogeneity testing was done on each batch and on the three elements most likely to exhibit heterogeneity, namely, Li, Rb and Cs; all were found to be distributed homogeneously between batches and individual units of sample.

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 for the calibration of a measurement system, the assessment of a measurement procedure, 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

Certified values are for total mass fractions of oxides and elements, reported on a dry weight basis. Prior to analysis the test portion should be dried at 105 ^{o}C for 2 hours before weighing. Alternatively, moisture content may be determined on a second test portion, the results from which can be used to correct to a dry weight basis data acquired on material weighed on an "as received" basis. Also, analysts should not expect to achieve the certified values if they use any partial decomposition technique for their sample preparation. Re-homogenisation by manual shaking of the closed bottle is strongly recommended before a sub-sample is taken.

Minimum sample mass

The recommended minimum sample test portion mass is 100 mg. If an analytical technique requires a smaller test portion mass, it is recommended that more than 100 mg be weighed out and further pulverized in an agate mortar before weighing out the needed mass. No material that has been removed from the sample bottle should be returned to it, as this might cause contamination of all remaining material.

Storage information

The CRM should be stored at room temperature and tightly sealed to protect it from absorption of atmospheric moisture and laboratory chemicals.

Period of validity

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

Certified values

Table 1 (certified values) and Table 2 (indicative values) state the determined composition of MGL-OShBO with the associated expanded uncertainties. A full description of how these values and their uncertainties have been established can be found in Kane et al. (2003). Appendix 3 provides additional information that is essential for user laboratories to evaluate their own results for the CRM in the manner outlined in ISO Guide 33 (ISO 2000).

Data for the 2007-2008 certification were contributed by laboratories pre-qualified to provide certification data through their participation in the IAG's GeoPT programme (Potts et al. 2004). Data from the original 1999 certification analyses that fulfilled IAG quality criteria were also used in the re-certification. Those laboratories which provided data for the current Reference Material Certificate are listed in Appendix 1.

Number of values 'p'

*The number of technically valid data sets (**p**) that contributed to the estimation of the certified value after rejection of outliers is listed in the tables. Outliers were selected based in Youden plots, Mandel's k and detection limit criteria.*

Table 1

MGL-OShBO (Alkaline granite)

Certified Values (CVs) and their uncertainties (U)

Oxide/ element	Certified value CV *g/100g*	Expanded uncertainty ± U *g/100g*	No. data sets p		Element	Certified value CV *mg/kg*	Expanded uncertainty ± U *mg/kg*	No. data sets p
SiO_2	71.72	0.29	48		**La**	8.4	0.7	12
Al_2O_3	16.12	0.12	40		**Li**	1730	40	15
Fe_2O_3(Total)	0.500	0.029	23		**Lu**	0.326	0.021	10
FeO	0.299	0.004	11		**Nb**	64	4	19
MnO	0.149	0.017	40		**Nd**	15.5	0.5	10
CaO	0.388	0.011	31		**Ni**	10.7	1.6	17
Na_2O	5.34	0.26	34		**Pb**	63	6	18
K_2O	3.58	0.04	32		**Rb**	2360	110	29
P_2O_5	0.0293	0.0017	11		**Sc**	9.2	1.4	11
H_2O^-	0.074	0.020	15		**Sm**	6.0	0.4	10
F	1.13	0.16	10		**Sr**	12.3	1.1	17
LOI	1.10	0.04	23		**Ta**	46.7	2.4	12
					Th	13.3	0.8	10
	mg/kg	*mg/kg*			**Yb**	2.38	0.13	10
Ce	27.4	1.6	12		**Zn**	92	6	25
Cu	7.1	1.1	16		**Zr**	40.1	2.8	16

Notes for Tables 1 and 2:

***U** is the expanded uncertainty corresponding to a level of confidence of 95 %. It has been developed according to the Guide for the Expression of Uncertainty in Measurement (Ellison et al. 2000) with a coverage factor **k > 2** that varies as a function of **p**. **p** is the number of independent results used to determine the certified or indicative value. The between-laboratory standard deviation cannot be obtained simply by dividing U by k. See Appendix 3.*

***Fe_2O_3(total)** is the total iron expressed as Fe_2O_3.*

***LOI** is the mass loss on ignition (generally 1050 °C for 2 hours)*

Indicative values

Indicative values in Table 2 are designed to provide guidance on the mass fractions of other selected elements and should not be used to validate analytical measurements. A minimum of 5 laboratory average determinations were used to calculate indicative values.

Table 2

MGL-OShBO (Alkaline granite)

Indicative Values (IVs) and their uncertainties (U)

Oxide / element	Indicative value IV *g/100g*	Expanded uncertainty ± U *g/100g*	No. data sets p		Element	Indicative value IV *mg/kg*	Expanded uncertainty ± U *mg/kg*	No. data sets p
TiO_2	0.029	0.002	5		**Ga**	56.7	1.8	9
	mg/kg	*mg/kg*			**Gd**	4.11	0.19	9
Ba	12.3	1.0	8		**Hf**	7.89	0.28	8
Be	60	7	5		**Ho**	0.67	0.05	8
Cr	160	50	12		**Pr**	4.40	0.26	8
Cs	87	16	22		**Tb**	0.83	0.05	9
Dy	4.33	0.26	9		**Tm**	0.322	0.026	8
Er	1.78	0.11	9		**U**	3.84	0.25	9
Eu	0.027	0.009	5		**Y**	23.4	1.7	9

Metrological traceability

Traceability was demonstrated for this reference material by requesting participating laboratories to co-analyse the certified reference material ZGI-GM (granite). This material was certified by the Zentrales Geologisches Institut, Berlin, Germany Democratic Republic. An assessment of the results for ZGI-GM was undertaken to confirm the absence of systematic bias, establishing a chain of comparisons between the present MGL-OShBO certification and a previous geochemical certification project. A more complete assessment of the limitations of this approach are provided by Kane et al. (2007a) in the relevant certification report.

Certification characterisation report

Further details of the procedures used, the results, their statistical analysis and data assessment, on which the property values listed in this certificate are based, can be found in the GAS / OShBO certification report (Kane et al. 2007a).

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. Further details may be found on the relevant material safety data sheet.

Legal notice – terms and conditions

1. The IAG shall not be liable to the user of this material for loss (whether direct or indirect) of profits, business, anticipated savings or reputation or for any indirect or consequential loss or damage whatsoever even if previously advised thereof and whether arising from negligence, breach of these Terms and Conditions or howsoever occurring.
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Revisions

This certificate is version 2.00 and is a revision of certificate 1.10 issued on 10th March 2009. Any further revisions to this Reference Material Certificate will be made available on the IAGeo Ltd web site (www.iageo.com).

Acknowledgements

The original re-certification programme was directed by Jean Kane and the resultant certificate of analysis was approved by Jean Kane (certification project leader), Thomas Meisel (Certification Committee chair and Michael Wiedenbeck (IAG President) to whom grateful acknowledgement is given. Further contributions were made by members of the IAG Certification and Reference Material Committee, but especially the management and CRM teams of the Central Geological Laboratory, Ulaanbaatar for preparing and supplying this material. The IAG is very grateful to Mrs B. Davaasuren for coordinating the production of the original re-certification certificate and to the laboratories listed in Appendix 1 for providing data for this certification programme.

Approvals

This Certificate of Analysis was approved on behalf of the International Association of Geoanalysts and the Central Geological Laboratory, Mongolia, as follows:

Name *Phil Potts*	**Position** *On behalf of the IAG Certification Programme*	**Date; 12th December 2019**
Name *Jacinta Enzweiler*	**Position** *President of the International Association of Geoanalysts***.**	**Date: 19th December 2019**
Name *E. Tegshjargal*	**Position** *Director of the Central Geological Laboratory, Ulaanbaatar, Mongolia*	**Date: 20th December 2019**

Revision schedule

Revision 2.00 – December 2019
Reformatting of the re-certification certificate of analysis (dates 10th March 2009) with revised period of stability and traceability statements.

References:

Ellison S.L.R., Rosslein M. and Williams A. (2000)
Eurachem/CITAC Guide: Quantifying Uncertainty in Analytical Measurement (second edition). 126pp.

Erdenetsetseg D. and Davaasuren B. (1999)
Report on the Preparation and Certification of Certified Reference Material- Alkaline Granite, OShBO. Central Geological Laboratory of Mongolia (in Mongolian), (2006 English translation, personal communication), 35pp.

ISO Guide 33 (2000)
Uses of Certified Reference Materials (2nd edition). International Organization for Standardization (Geneva), 23pp.

Kane J.S., Batjargal B. and Erdenetsetseg D. (2007a)
Report on the 2007 recertification of the certified reference materials GAS (Serpentinite) and OShBO (alkaline granite). International Association of Geoanalysts Unpublished report, 17pp.

Kane J.S., Potts P.J., Meisel T. and Wiedenbeck M. (2007b)
International Association of Geoanalysts' Protocol for the Certification of Geological and Environmental Reference Materials: A Supplement. Geostandards and Geoanalytical Research, 31, 285-288.

Kane J.S., Potts P.J., Wiedenbeck M., Carignan J. and Wilson S.A. (2003)
International Association of Geoanalysts' Protocol for the Certification of Geological and Environmental Reference Materials. Geostandards Newsletter: The Journal of Geostandards and Geoanalysis, 27, 227-244.

Potts P.J., Thompson M., Chenery S.R.N., Webb P.C. and Batjargal B. (2004)
GeoPT14- An International Proficiency Test for Analytical Geochemistry Laboratories- Report on Round 14/January 2004 (OShBO Alkaline Granite). International Association of Geoanalysts report, 31pp.

Appendix 1

Institutions that provided data for the re-certification of MGL-OShBO

IAG laboratories:

- School of Science, University of Greenwich at Medway, Chatham Maritime, Kent, UK
- Instituto de Geociêcias, Universidade Estadual de Campinas, Brazil
- Savannah River National Laboratory, Aiken, SC, USA
- Laboratorio do INETI, S. Mamede de Infesta, Portugal
- Institute of Nuclear Physics, Tirana, Albania
- Instituto de Geociêcias da USP, Cidade Universitária, São Paulo, Brazil
- GTK Geolaboratory, Geological Survey of Finland, Rovaniemi, Finland
- HuK Umweltlabor GmbH, Wenden, Germany
- GeoForschungsZentrum, Potsdam, Germany
- Central Geological Laboratory, Ulaanbaatar, Mongolia
- Mineral Resources Laboratory, US Geological Survey, Denver, CO, USA
- Geoanalytical Laboratory, School of Earth and Environmental Sciences, Washington State University, Pullman, WA, USA
- ALS Chemex, North Vancouver, BC, Canada
- Central Laboratory, Czech Geological Survey, Prague, Czech Republic
- Geoscience Laboratories, Ontario Geological Survey, Sudbury, ON, Canada

Original certification laboratories whose data were retained for the recertification:

- Three laboratories within the Central Geological Laboratory, Mongolia
- Federal Institute for Geosciences and Natural Resources, Hannover, Germany
- Three laboratories of the Institute for Geochemistry, Irkutsk, Russia
- Chemical and Technology Center for New Materials, National University of Mongolia
- Physics and Technology Institute of the Academy of Science, Mongolia
- Central Chemical Laboratory of "Erdenet" Mongolian-Russian joint venture

Appendix 2

Techniques that contributed to the re-certification of MGL-OShBO

SiO_2	GRAV, ICP-AES, PHOT, XRF
Al_2O_3	AAS, ICP-AES, PHOT, VOL, XRF
Fe_2O_3	AAS, ICP-AES, PHOT, VOL, XRF
FeO	VOL
MnO	AAS, ICP-AES, ICP-MS, XRF
CaO	AAS, ICP-AES, ICP-MS, VOL, XRF
Na_2O	AAS, Fl PHOT, ICP-AES, XRF
K_2O	AAS, Fl PHOT, XRF
P_2O_5	ICP-AES, PHOT
H_2O^-	GRAV
LOI	GRAV

Ce	dc arc OES, ICP-AES, ICP-MS, INAA, XRF
Cu	AAS, dc arc OES, ICP-AES, ICP-MS, XRF
La	dc arc OES, ICP-AES, ICP-MS, INAA
Li	Fl PHOT, ICP-MS
Lu	ICP-AES, ICP-MS, INAA
Nb	dc arc OES, ICP-MS, XRF
Nd	ICP-AES, ICP-MS, INAA
Ni	AAS, dc arc OES, ICP-AES, ICP-MS, INAA, XRF
Pb	AAS, ICP-AES, ICP-MS, XRF
Rb	AAS, Fl PHOT, ICP-MS, INAA, XRF
Sc	dc arc OES, ICP-AES, ICP-MS, INAA, XRF
Sm	ICP-AES, ICP-MS, INAA
Sr	AAS, ICP-AES, ICP-MS, XRF
Ta	ICP-MS, XRF
Th	ICP-MS, INAA, XRF
Yb	ICP-AES, ICP-MS, INAA
Zn	AAS, dc arc OES, ICP-AES, ICP-MS, XRF
Zr	ICP-AES, ICP-MS, INAA, XRF

Oxides/Elements which have uncertified (information) concentration values

TiO_2	PHOT
Ba	ICP-AES, ICP-MS
Be	AAS, ICP-AES, ICP-MS
Cr	AAS, dc arc OES, ICP-MS, PHOT, XRF
Cs	Fl PHOT, ICP-MS, INAA, XRF
Dy	ICP-MS
Er	ICP-MS
Eu	ICP-MS
Ga	ICP-MS, XRF
Gd	ICP-MS
Hf	ICP-MS, INAA
Ho	ICP-MS
Pr	ICP-MS
Tb	ICP-MS, INAA
Tm	ICP-MS
U	ICP-MS, INAA
Y	ICP-AES, ICP-MS, XRF

Appendix 3

Pooled within-laboratory and between-laboratory standard deviations needed for evaluation of laboratory results for MGL-OShBO

This is additional information that is essential for user laboratories to evaluate their own results for the CRM in the manner outlined in ISO Guide 33 (ISO 2000).

	Mass fraction	
Oxide/Element	***sd(within)***	***sd(between)***
	g/100g	
SiO_2	*0.27*	*0.57*
Al_2O_3	*0.07*	*0.36*
Fe_2O_3(total)	*0.030*	*0.032*
FeO	*0.064*	*0.006*
MnO	*0.005*	*0.014*
CaO	*0.015*	*0.019*
Na_2O	*0.04*	*0.19*
K_2O	*0.11*	*0.11*
P2O5	*0.0034*	*0.0017*
H2O-	*0.028*	*0.036*
F	*nd*	*0.07*
LOI	*0.05*	*0.07*
	mg/kg	
Ce	*1.6*	*1.5*
Cu	*1.0*	*1.2*
La	*0.4*	*0.9*
Li	*50*	*90*
Lu	*0.014*	*0.018*
Nb	*4*	*7*
Nd	*0.5*	*0.6*
Ni	*1.7*	*3.0*
Pb	*4*	*5*
Rb	*100*	*130*
Sc	*0.4*	*0.9*
Sm	*0.2*	*0.4*
Sr	*0.7*	*2.0*
Ta	*2.7*	*3.0*
Th	*0.7*	*1.7*
Yb	*0.09*	*0.12*
Zn	*5*	*8*
Zr	*2.4*	*3.4*

Note: nd = not determined

Not certified, for information purposes only

Oxide/Element	***sd(within)***	***sd(between)***
	g/100g	
TiO_2	*0.001*	*0.001*
	mg/kg	
Ba	*0.8*	*0.8*
Be	*3*	*6*
Cr	*5*	*38*
Cs	*2*	*15*
Dy	*0.14*	*0.26*
Er	*0.07*	*0.11*
Eu	*0.042*	*0.073*

Ga	*3.1*	*3.2*
Gd	*0.15*	*0.23*
Hf	*0.21*	*0.32*
Ho	*0.03*	*0.05*
Pr	*1.44*	*0.23*
Tb	*0.03*	*0.05*
Tm	*0.015*	*0.025*
U	*0.19*	*0.32*
Y	*1.7*	*2.0*