



# Reference Material Certificate

# IAG CRM 2 / CGL 001 MGL-GAS (Serpentinite)

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# Status of reference material certificate

This material was originally certified by the Central Geological Laboratory, Ulaanbaatar, Mongolia in December 1998, 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 10<sup>th</sup> 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

The MGL-GAS material was collected from the Naran Massif in the Khantaishir area of Mongolia. It was originally prepared, packaged and certified in December 1998 by the Central Geological Laboratory (CGL), Ulaanbaatar, Mongolia. The material consists of a homogeneous powder of which 98.5 wt. % passed a 74  $\mu$ m sieve. The mineralogy of the sample (in % m/m) has been determined to be as follows:

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Minerals	g/100 g	
Serpentine	95.1	
Magnetite	2.4	
Calcite	1.20	
Plagioclase	0.40	
Magnesite	0.30	
Chromite	0.30	
Goethite	0.25	
Sericite-muscovite	0.15	
Pyrite, pyrrhotite, olivine, chalcopyrite,		
and amphibole	minor	

Sample preparation, homogeneity and stability testing studies were performed by the CGL laboratories. This material has been produced in units of 100 g packaged in a polyethylene bottle for delivery to users.

The original preparation, homogeneity and stability testing were performed by the Central Geological Laboratory, Ulaanbaatar, Mongolia (Erdenetsetseg and Gantsetseg 1998). Two hundred and fifty 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 200 kg of powder, of which 98.5 wt % was  $<74 \mu m$ . Before bottling, homogeneity testing was done on the four components most likely to exhibit heterogeneity, namely, Ni, Co, CaO and  $Cr_2O_3$ ; all were found to be distributed homogeneously between 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 °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. Rehomogenisation 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**

Certified values listed in Table 1 are based on procedures that are summarised in the International Association of Geoanalysts' Certification Protocol (Kane et al., 2003). Laboratories pre-qualified to provide certification data through their participation in the IAG's GeoPT programme (Potts et al. 2003). Data from the original 1998 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.

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# 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-GAS (Serpentinite)

Certified Values (CVs) and their uncertainties (U)

Oxide/ element	Certified value	Expanded uncertainty	No. data sets	Element	Certified value	Expanded uncertainty	No. data sets
	CV	± U	p		$\mathbf{CV}$	± U	p
	g/100g	g/100g			mg/kg	mg/kg	
SiO <sub>2</sub>	38.54	0.23	43	Cr	2780	30	26
Fe <sub>2</sub> O <sub>3</sub> (total)	8.00	0.22	44	Ni	2300	120	26
MnO	0.082	0.009	36	Sr	7.3	0.4	12
MgO	38.22	0.34	42	U	0.80	0.04	12
LOI	13.33	0.14	26	V	33.4	2.0	10
	mg/kg	mg/kg	p	Zn	39	3	12
Co	106	3	27				

# **Table 2** MGL-GAS (Serpentinite)

# **Indicative Values (IVs) and their uncertainties (U)**

	Indicative value	Expanded uncertainty	No. data sets		Indicative value	Expanded uncertainty	No. data sets
Oxide	IV	± U	p	Oxide/ element	IV	± U	p
	g/100g	g/100g			g/100g	g/100g	
TiO <sub>2</sub>	0.022	0.007	32	$CO_2$	0.84	0.03	10
Al <sub>2</sub> O <sub>3</sub>	0.475	0.020	24	H <sub>2</sub> O <sup>-</sup>	0.58	0.24	12
FeO	0.27	0.20	9		mg/kg	mg/kg	
CaO	0.681	0.011	31	As	117	6	7
Na <sub>2</sub> O	0.038	0.021	9	Ba	8.4	0.6	7
K <sub>2</sub> O	0.018	0.009	24	Sm	0.037	0.004	8
P <sub>2</sub> O <sub>5</sub>	0.023	0.005	23				

#### Notes for Tables 1 and 2:

 $m{U}$  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  $m{k} > m{2}$  that varies as a function of  $m{p}$ .  $m{p}$  is the number of independent results used to determine the certified or information value. The between-laboratory standard deviation cannot be obtained simply by dividing  $m{U}$  by  $m{k}$ . See Appendix 3.

Fe<sub>2</sub>O<sub>3</sub>(total) is the total iron expressed as Fe<sub>2</sub>O<sub>3</sub>.

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

 $H_2O^-$  is the structural water bonded to minerals in the sample.

# **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.

# Metrological traceability

Traceability was demonstrated for this reference material by requesting participating laboratories to co-analyse the certified reference material ZGI-SW (serpentinite). This material was certified by the Zentrales Geologisches Institut, Berlin, Germany Democratic Republic. An assessment of the results for ZGI-SW was undertaken to confirm the absence of systematic bias, establishing a chain of comparisons between the present MGL-GAS certification and a previous geochemical certification project. A more complete assessment of the limitations of this approach is 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**

- 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.
- 2. In any event, and notwithstanding anything contained in these Terms and Conditions, IAG's liability in contract, tort (including negligence, defamation or breach of statutory duty) or otherwise arising by reason of or in connection with these Terms and Conditions (including as a result of proficiency testing) shall be limited to the price paid for the material giving rise to such liability
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# Revisions

This certificate is version 2.00 and is a revision of certificate 1.10 issued on 10<sup>th</sup> 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.

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# **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 Date: 12<sup>th</sup> December 2019

Certification Programme

Name Jacinta Enzweiler Position President of the International Date: 19th December 2019

Association of Geoanalysts

Name E. Tegshjargal Position Director of the Central Geological Date: 20th December 2019

Laboratory, Ulaanbaatar, Mongolia



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# **Revision schedule**

#### Revision 2.00 - December 2019

Reformatting of the re-certification certificate of analysis (dated 10<sup>th</sup> 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 Gantsetseg D. (1998)

Report on the Preparation and Certification of Certified Reference Material - Serpentinite GAS Central Geological Laboratory of Mongolia (in Mongolian), (2006 English translation, personal communication), 20pp.

#### ISO Guide 33 (2000)

Uses of Certified Reference Materials ( $2^{nd}$  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. (2003)

GeoPT12 - An International Proficiency Test for Analytical Geochemistry Laboratories - Report on Round 12/January 2003 (GAS Serpentinite). International Association of Geoanalysts report, 38pp.

# Appendix 1

# Institutions that provided data for the re-certification of MGL-GAS

# **IAG laboratories:**

- School of Science, University of Greenwich at Medway, Chatham Maritime, Kent, UK
- CERAM Testing and Environmental, Penkhull, Stoke-on-Trent, UK
- Instituto de Geociêcias, Universidade Estadual de Campinas, Brazil
- Savannah River National Laboratory, Aiken, SC, USA
- National Research Center for Geoanalysis, Beijing, PR China
- Laboratorio do INETI, S. Mamede de Infesta, Portugal
- VSEGEI All Russia Geological Research Institute Central Laboratory, St. Petersburg, Russia
- Southern and Eastern African Mineral Centre, Dar es Salaam, Tanzania
- Instituto de Geociêcias da USP, Cidade Universitária, São Paulo, Brazil
- GTK Geolaboratory, Geological Survey of Finland, Rovaniemi, Finland
- HuK Umweltlabor GmbH, Wenden, Germany
- Geoanalytical Laboratories, State Geological Institute of Dionyz Stur, Slovakia
- GeoForschungsZentrum Potsdam, Potsdam, Germany
- Central Geological Laboratory, Ulaanbaatar, Mongolia
- Mineral Resources Laboratory, US Geological Survey, Denver, CO, USA

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- Geoanalytical Laboratory, School of Earth and Environmental Sciences, Washington State University, Pullman, WA, USA
- ALS Chemex, North Vancouver, BC, Canada
- Laboratoire Pierre Süe, Gif-sur-Yvette, France
- Departamento de Geoquimica, Instituto de Geologia, UNAM, Mexico
- Central Laboratory, Czech Geological Survey, Prague, Czech Republic
- Instituto de Technologia Ceramica, Campus Universitario Riu Sec, Castellón, Spain
- Geoscience Laboratories, Ontario Geological Survey, Sudbury, ON, Canada

# Original certification laboratories whose data were retained for the recertification:

- Four laboratories within the Central Geological Laboratory, Mongolia
- National Center for Standardization and Metrology, Mongolia
- Central Chemical Laboratory of "Erdenet" Mongolian-Russian joint venture
- Physics and Technology Institute of the Academy of Science, Mongolia
- Amdel Laboratories, Ltd., Australia
- Shimadzu Corporation, Kyoto, Japan
- Geological Survey of Japan, Tsukuba, Japan
- Institute for Nuclear Physics, Korea

# Appendix 2

# Techniques that contributed to the re-certification of MGL-GAS

$SiO_2$	AAS, GRAV, ICP-AES, ICP-MS, PHOT, XRF
Fe <sub>2</sub> O <sub>3</sub> (total)	AAS, ICP-AES, ICP-MS, INAA, PHOT, VOL, XRF
MnO	AAS, ICP-AES, ICP-MS, INAA, VOL, XRF
MgO	AAS, GRAV, ICP-AES, ICP-MS, VOL, XRF
LOI	GRAV
Co	AAS, ICP-AES, ICP-MS, INAA, XRF
Cr	AAS, ICP-AES, ICP-MS, INAA, PHOT, XRF
Ni	AAS, ICP-AES, ICP-MS, INAA, XRF
Sr	ICP-AES, ICP-MS, XRF
U	ICP-MS, INAA
V	AAS, ICP-AES, ICP-MS, XRF
Zn	AAS, ICP-AES, ICP-MS, INAA, XRF

### Oxides/Elements which have uncertified (information) mass fraction values

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$TiO_2$	ICP-AES, ICP-MS, XRF
$Al_2O_3$	ICP-AES, ICP-MS, VOL, XRF
FeO	VOL
CaO	AAS, ICP-AES, ICP-MS, VOL, XRF
$Na_2O$	AAS, XRF
$K_2O$	AAS, ICP-AES, XRF
$P_2O_5$	ICP-AES, XRF
$CO_2$	VOL
$H_2O^-$	GRAV
As	AAS, ICP-MS, INAA, XRF

As AAS, ICP-MS, INAA, XRF Ba ICP-AES, ICP-MS, INAA, XRF

Sm INAA, ICP-MS

# $\label{lem:appendix 3} Pooled \ within-laboratory \ and \ between-laboratory \ standard \ deviations \ needed \ for \ evaluation \ of \ laboratory \ results \ for \ MGL-GAS$

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			
$SiO_2$	0.19	0.40		
$Fe_2O_3(total)$	0.07	0.20		
MnO	0.002	0.007		
MgO	0.26	0.52		
LOI	0.07	0.23		
	mg/kg			
Co	3	6		
Cr	40	70		
Ni	28	80		
Sr	0.7	0.4		
U	0.02	0.03		
V	3	2		
Zn	2	3		

Not certified, for information purposes only

Oxide/Element	sd(within)	sd(between)
	g/	′100g
$TiO_2$	0.003	0.019
$Al_2O_3$	0.015	0.030
FeO	0.08	0.03
CaO	0.009	0.026
$Na_2O$	0.006	0.011
$K_2O$	0.002	0.016
$P_2O_5$	0.003	0.011
$CO_2$	0.02	0.24
$H_2O^-$	0.06	0.17
	m	g/kg
As	5	4
Ва	2.6	0.5
Sm	0.005	0.004

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