Certificate of Analysis

IAG OKUM

(Ultramafic rock)

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Description of the certified reference material

Material for this reference material was collected from the spinifex-textured top of a komatiite flow at Serpentine Mt., McArthur Township, Ontario, Canada by the Ontario Geological Survey. The sample consists of black massive rocks containing randomly oriented spinifex blades. It is typical of a Mg-poor komatiite or komatiitic basalt.

Approximately 500 kg of material was jaw crushed and ground to 90% < 63 μm (230-mesh) at the Ontario Geoscience Laboratory, using a continuous feed Cr-steel ring mill, then blended, and bottled into 500 mL containers. A fraction of this original material was rebottled into 100 g splits for distribution through the IAG.

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 ± 5°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 0.11 to 0.36 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-three laboratories (listed in **Appendix 1**) 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 OKUM and one bottle of MGL GAS (the "traceablility" 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. 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.*

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

Uncertainties

U is the expanded uncertainty, corresponding to 95% confidence limits and incorporates the relevant Student's 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 Co, Sc, Sr and Zr to account for technique bias observed in the respective data sets.

Number of values, 'n'

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 on Youden plots, Mandel's k and detection limit criteria.

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Certified values and uncertainties

Measurand	Certified value	Uncertainty (expanded)	Unit	p		Measurand	Certified value	Uncertainty (expanded)	Unit	p
SiO_2	44.14	0.14	*g/100g*	22		**Hf**	0.551	0.023	*mg/kg*	15
TiO_2	0.380	0.004	*g/100g*	23		**Ho**	0.355	0.009	*mg/kg*	15
Al_2O_3	7.97	0.04	*g/100g*	22		**La**	0.412	0.017	*mg/kg*	18
Fe_2O_3T	11.81	0.05	*g/100g*	22		**Lu**	0.148	0.005	*mg/kg*	19
MnO	0.1813	0.0027	*g/100g*	23		**Nb**	0.37	0.06	*mg/kg*	11
MgO	21.29	0.10	*g/100g*	21		**Nd**	1.494	0.020	*mg/kg*	16
CaO	7.85	0.06	*g/100g*	20		**Ni**	886	10	*mg/kg*	21
Na_2O	1.136	0.022	*g/100g*	22		**Pr**	0.235	0.008	*mg/kg*	17
K_2O	0.044	0.002	*g/100g*	16		**Rb**	0.96	0.06	*mg/kg*	14
P_2O_5	0.0266	0.0023	*g/100g*	17		**Sc**	27.9	1.5	*mg/kg*	21
						Sm	0.715	0.011	*mg/kg*	18
Ce	1.27	0.03	*mg/kg*	18		**Sr**	16.1	1.0	*mg/kg*	23
Co	88.9	1.5	*mg/kg*	23		**Ta**	0.0264	0.0038	*mg/kg*	12
Cr	2460	31	*mg/kg*	22		**Th**	0.031	0.004	*mg/kg*	11
Cs	0.184	0.003	*mg/kg*	13		**V**	167.8	3.1	*mg/kg*	19
Cu	43.5	1.2	*mg/kg*	20		**Y**	9.08	0.29	*mg/kg*	19
Dy	1.61	0.04	*mg/kg*	17		**Yb**	1.009	0.023	*mg/kg*	20
Er	1.041	0.014	*mg/kg*	18		**Zn**	61.2	1.9	*mg/kg*	22
Eu	0.300	0.007	*mg/kg*	19		**Zr**	17.0	1.4	*mg/kg*	16
Ga	8.79	0.16	*mg/kg*	17						

Certified value *and* ***uncertainty*** *- see text.*
p *= The number of technically valid data sets that contributed to the estimation of the certified value after rejection of outliers.*
Fe_2O_3T *= total iron expressed as Fe_2O_3*

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Information values and uncertainties

Measurand	Information value	Uncertainty (expanded)	Unit	p
LOI	4.49	0.14	*g/100g*	22
Ba	6.2	0.5	*mg/kg*	16
Be	0.065	0.004	*mg/kg*	6
Gd	1.17	0.07	*mg/kg*	16
Li	4.4	0.3	*mg/kg*	8
Pb	0.26	0.02	*mg/kg*	7
Sb	0.079	0.012	*mg/kg*	5
Sn	0.25	0.02	*mg/kg*	5
Tb	0.229	0.010	*mg/kg*	18
Tm	0.155	0.006	*mg/kg*	16
U	0.012	0.005	*mg/kg*	12

Information values *and* ***uncertainty*** *– see text.*
p *= The number of technically valid data sets that contributed to the estimation of the information value after rejection of outliers.*
LOI *= loss on ignition.*

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 serpentinite, 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/. An assessment of the measurement results for MGL GAS was undertaken to confirm the absence of systematic bias, thus establishing an unbroken chain of comparisons between the present OKUM 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 OKUM 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

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.
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.
3. The IAG does not grant any warranties in relation to Geo*PT* products or the supply of analytical services or distribution of the proficiency test. All other conditions, warranties, stipulations or other statements whatsoever, whether express or implied, by statute, at common law or otherwise howsoever, relating to the Geo*PT* products, analytical services or proficiency tests are hereby excluded. In particular, (but without limitation to the foregoing) no warranties are granted regarding the fitness for purpose, performance, use, quality or merchantability of the Geo*PT* products, whether express or implied, by statute, at common law or otherwise howsoever.

Revisions

This certificate is version 1.10. 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 Jean Kane and Marcus Burnham. We are very grateful 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.

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**

Appendix 1

Laboratories that contributed to this OKUM certification programme.

- *Allgemeine und Analytische Chemie, Montanuniversität Leoben, Leoben, Austria*
- *Department für Lithospährenforschung, University of Vienna, Vienna, Austria*
- *Institut für Erdwissenschaften, University of Graz, Graz, Austria*
- *Seibersdorf Labor GmbH, Seibersdorf, Austria*
- *Instituto de Geociências , Universidade Estadual de Campinas, UNICAMP, Campinas, Brazil*
- *IPEN, Cidade Universitária, São Paulo, Brazil*
- *ALS, Vancouver, Canada*
- *Dept. Earth Sciences, Laurentian University, Sudbury, Canada*
- *Dept Geologie et Genie Geologique, Universite Laval, Laval, Canada*
- *Geoscience Laboratories, Ontario Geologial Survey, Sudbury, Canada*
- *SGS Mineral Services – Lakefield, Lakefield, Canada*
- *National Key Laboratory of Continental Dynamics, Northwest University, 710069, China*
- *State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan, China*
- *Central Laboratory, Czech Geological Survey, Prague, Czech Republic*
- *Géosciences Montpellier, Université de Montpellier II, Montpellier, France*
- *Laboratoire des Mécanismes et Transferts en Géologie, Toulouse, France*
- *Federal Institute for Geosciences and Natural Resources, Hanover, Germany*
- *Helmholtz-Zentrum Potsdam, Deutsches GeoForschungsZentrum GFZ, Potsdam, Germany*
- *HUK-Umweltlabor, Wenden, Germany*
- *Lapp Insulators GmbH, Wunsiedel, Germany*
- *Mineralogie-Geochemie, University of Freiburg, Freiburg, Germany*
- *Dipartimento di Scienze della Terra, Università di Pisa, Pisa, Italy*
- *Central Geological Laboratory, Ulaanbaatar, Mongolia*
- *LNEG, S.Mamede de Infesta, Portugal*
- *Central Laboratory, VSEGEI, St. Petersburg, Russia*
- *Institute of Geology and Geochemistry, Ekaterinburg, Russia*
- *KOREA BASIC SCIENCE INSTITUTE, Daejon, South Korea*
- *British Geological Survey, Nottingham, UK*
- *CERAM, Stoke-on-Trent, UK*
- *Dept of Earth & Environmental Sciences, The Open University, Milton Keynes, UK*
- *GeoAnalytical Lab, Washington State University, Pullman, USA*
- *Savannah River Nuclear Solutions (SRNS), Aiken, USA*
- *U. S. Geological Survey, Denver, USA*

Revision Schedule

1.00 (9th November 2015) – Original certificate approved.

1.10 (4th November 2016) – Addition of Appendix 1 (participating laboratories) and associated revisions to the text.