Certificate of Analysis

IAG / CGL 020
ML-3 (Limestone)

International Association of Geoanalysts
13 Belvedere Close, Keyworth,
Nottingham, NG12 5JF, UK
e-mail: iageo.ltd@ntlworld.com

Telephone: +44 (0)115 9375219

Central Geological Laboratory
Trade Union Street, P.O. BOX 437,
Ulaanbaatar -18080, Mongolia
e-mail: info@cengeolab.com
cengeolab@magicnet.mn
Telephone: +976 70182904
Fax: +976 70184212

Description of the certified reference material

The starting material, a bulk limestone, was collected from the limestone occurrence in the Dund Baitlagiin gol area located in the Bayndelger Soum, Tuv Province,Mongolia. Sampling was performed in 2011 by the Central Geological Laboratory (CGL), Ulaanbaatar, Mongolia in accordance with relevant sampling procedures. In order to increase silica content, small amount of clay material was blended to the bulk material before homogenising.

The mineralogy of the sample has been determined as follows:

Minerals	*Percentage, % m/m*
Calcite	*92.6*
Magnesite	*2.9*
Quartz and Albite	*4.3*
Muscovite	*trace*
Amphiboles	*trace*
Magnetite	*trace*

Sample preparation, homogeneity and stability study tests were performed by the CGL laboratories between 2011 and 2014. After crushing and pulverization, the entire batch of selected bulk material passed a sieve with an opening of 75 μm using an ultrasonic sieving machine. The pulverized bulk material was homogenized using a high performance intensive mixer. After the primary homogeneity testing study, portions of 100 g were bottled by rotary splitting and then labelled.

After homogenisation and bottling, homogeneity testing study was performed under repeatability conditions. Fourteen measurands were determined in duplicate on 10 randomly selected units. Homogeneity test results were evaluated using the F-test, and against ISO 13528 and the "harmonized protocol" and confirmed that the material is sufficiently homogeneous.

Intended use

This certified reference material is designed for use by laboratories undertaking the major and trace element mass fraction analysis of limestone 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.

Minimum sample size

The minimum size of test portion is recommended to be 0.2 g, based on the results of a formal repeatability assessment procedure undertaken by the Central Geological Laboratory, Mongolia.

Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. On this basis, 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.

Storage information

Store in a sealed container in a cool dry environment.

Instructions for handling

When measurands are to be determined, test portions must be dried at 105 ± 5°C for at least 2 hours. Avoid contamination and cross-contamination of the test material during handling. The material should not be reground before use. The weight loss on drying has been found to be in the range of 0.06-0.24%.

Certified values

This material was certified by procedures based on the International Association of Geoanalysts Certification Protocol (Kane et al., 2003, Geostandards and Geoanalytical Research, 27, 227-244). Twenty-three laboratories (listed in ***Appendix 1****) participated in this certification round on the basis of their successful performance in round 30 of the IAG GeoPT proficiency testing programme (2012). These laboratories provided 25 technically valid data sets. A nested design was adopted for data acquisition as proposed in the IAG certification protocol. Participating labs received 2 packages of ML-3 and one package of CGL ML-2. The latter was supplied by the Central Geological Laboratory, Mongolia as a "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 packet of ML-3 and analyse the preparations on two different days. Participating laboratories were thus requested to submit 8 values (2x2x2 PacketxPrepxDay) for each measurand for which they had the analytical capability. For ML-2, the traceability sample, laboratories were requested to make two independent sample preparations (e.g., digestions) from the single supplied packet of ML-3 and analyse the preparations on two different days (i.e., 1x2x2 PacketxPrepxDay). After a careful assessment of the data distributions, certified values for Dy, Er, Gd, Ho, LOI, Nb, Nd, Pb, Pr, Sn, Tm and Y were derived from a single technique. Further details of the quality of data and data distributions may be found in the associated ML-3 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.

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.*

Information values

Information values are designed to provide guidance on the mass fraction concentrations of other selected elements and should not be used to validate analytical measurements. A minimum of 5 laboratory average results were used to calculate information values.

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Certified values and uncertainties

Measurand	Certified value	Uncertainty (expanded)	Unit	p
SiO_2	5.76	0.09	g/100 g	16
TiO_2	0.043	0.002	g/100 g	25
Al_2O_3	1.16	0.02	g/100 g	24
Fe_2O_3T	0.349	0.012	g/100 g	23
MnO	0.0231	0.0010	g/100 g	24
MgO	1.385	0.024	g/100 g	21
CaO	50.32	0.25	g/100 g	19
Na_2O	0.228	0.013	g/100 g	19
K_2O	0.229	0.009	g/100 g	20
P_2O_5	0.0659	0.0022	g/100 g	22
LOI	40.29	0.17	g/100 g	15
Ba	51	2	mg/kg	18
Be	0.58	0.06	mg/kg	13
Ce	6.08	0.14	mg/kg	17
Co	1.1	0.2	mg/kg	17
Cr	5.3	0.6	mg/kg	13
Cs	2.01	0.08	mg/kg	14
Cu	1.3	0.7	mg/kg	12
Dy	0.528	0.012	mg/kg	16
Er	0.298	0.007	mg/kg	17
Eu	0.143	0.007	mg/kg	18
Ga	1.48	0.11	mg/kg	13
Gd	0.611	0.013	mg/kg	16
Hf	0.395	0.029	mg/kg	10

Measurand	Certified value	Uncertainty (expanded)	Unit	p
Ho	0.103	0.004	mg/kg	16
La	3.71	0.10	mg/kg	20
Li	9.7	0.8	mg/kg	13
Lu	0.0412	0.0022	mg/kg	14
Nb	0.80	0.12	mg/kg	15
Nd	3.32	0.08	mg/kg	17
Ni	5	2	mg/kg	12
Pb	2.9	0.4	mg/kg	15
Pr	0.85	0.03	mg/kg	16
Rb	11.5	0.5	mg/kg	16
Sc	0.71	0.20	mg/kg	11
Sm	0.638	0.017	mg/kg	18
Sn	0.66	0.12	mg/kg	10
Sr	1018	30	mg/kg	22
Ta	0.093	0.013	mg/kg	11
Tb	0.092	0.004	mg/kg	15
Th	0.71	0.03	mg/kg	18
Tm	0.0437	0.0022	mg/kg	14
U	1.08	0.06	mg/kg	17
V	5.9	0.4	mg/kg	18
W	0.80	0.05	mg/kg	10
Y	3.43	0.22	mg/kg	17
Yb	0.276	0.007	mg/kg	19
Zn	8	2	mg/kg	17

***Certified value** and **uncertainty**- see text.*
***p:** number of technically valid data sets (after outlier rejection) that contributed to the certified value.*
***Fe_2O_3T** is the total iron expressed as Fe_2O_3*
***LOI** is the mass loss on ignition (typically 1050 °C for 2 hours)*

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Information values and uncertainties

Measurand	Information value	Uncertainty (expanded)	Unit	p
H_2OM	0.16	0.06	*g/100 g*	10
Cd	0.04	0.03	*mg/kg*	6
Mo	0.13	0.07	*mg/kg*	7
Sb	0.25	0.05	*mg/kg*	8
Tl	0.083	0.005	*mg/kg*	9
Zr	16	1	*mg/kg*	9

Information value *and* ***uncertainty*** *– see text.*
p: *number of technically valid data sets (after outlier rejection) that contributed to the certified value.*
H_2OM *is moisture (weight loss on drying at 105 °C for 2 hours).*

Metrological traceability

Traceability was demonstrated for this reference material by requesting participating laboratories to co-analyse the reference material CGL ML-2 (limestone), which had been distributed as the proficiency testing sample for the GeoPT 30 round. An assessment of the measurement results for CGL ML-2 obtained in the certification round was undertaken to confirm the absence of systematic bias compared with the assigned values obtained during the GeoPT 30 round. Thus, certification data are traceable to results obtained during the GeoPT 30 programme and through the calibration and validation procedures used by contributing laboratories.

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 IAG/CGL 020ML-3 characterisation report.

Safety information

Rock 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 made available on the IAGeo Ltd web site (www.iageo.com).

Acknowledgements

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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 29th February 2016**
Name*Thomas C. Meisel*	**Position** *President of the International Association of Geoanalysts***.**	**Date 1st March 2016**
Name*Tegshbayar Norov*	**Position** *Director of the Central Geological Laboratory, Ulaanbaatar, Mongolia*	**Date 2nd March 2016**

Appendix 1

Laboratories that participated in this ML-3 certification programme.

- *All-Russia Geological Research Institute, Central laboratory, Russia*
- *Analytical and Testing Center of Xi'an institute of Geology and Mineral Resources, Xi'an Center of China Geological Survey, China*
- *Bavarian Environment Agency, Analytical Geochemistry and Mineralogy BEA, Germany*
- *Central Chemical Laboratory, Poland*
- *Chemostrat Ltd, United Kingdom*
- *Czech Geological Survey, Czech Republic*
- *Delft University of Technology RID, Netherlands*
- *General and Analytical Chemistry Montanuniversität MUL, Austria*
- *Geological Survey of Denmark and Greenland (GEUS), Denmark*
- *Geology Department, Northwest University, State Key Laboratotry of Continental Dynamics, China*
- *Institute of Earth Sciences ¨Jaume Almera¨, labGEOTOP, Espana*
- *Institute of Geology and Geochemistry, Russia*
- *Intertek Genalysis, Australia*
- *Jochen Kuehnel XRAC, Germany*
- *LNEG –Laboratório, Portugal*
- *LSM Analytical Services, United Kingdom*
- *Lunar and Planetary Laboratory (LPL), University of Arizona, USA*
- *Ontario Geological Survey, Canada*
- *SGS Canada Inc., SGS Lakefield, Canada*
- *State Geological Institute of Dionyz Stur Geoanalytical Laboratories, Slovakia*
- *State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan, China*
- *SARM-CRPG-CNRS, France*
- *Seibersdorf Labor GmbH, Austria*