Certificate of Analysis

IAG CRM 1
OU-6 (Penrhyn Slate)

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

Telephone: +44 (0)115 9375219

Status of certificate of analysis

This reference material was originally certified by the International Association of Geoanalysts with the certification report published in 2004/2005 (Kane et al., 2004, 2005) and a certificate of analysis published in 2003 (Potts and Kane, 2003). This present reference material certificate replaces the original certificate of analysis with detailed editorial and formatting changes and a revised expiry date. No further evaluation of certification data has been undertaken and certified values and uncertainties remain unaltered in relation to the original certificate of analysis, apart from a minor revision to the rounding of some uncertainties.

Description of the certified reference material

OU-6 Penrhyn Slate was obtained from the Penrhyn Slate Quarries, Bethesda, North Wales as powdered material prepared as a commercial product. The slate quarry is located in a north-south trending belt that cuts across part of Snowdonia. The slate is fine-grained, purplish grey, well-cleaved and of Cambrian age. The rock was originally deposited as a mud and was recrystallised during low grade metamorphism. Caledonian deformation aligned fine-grained mica in the rock to produce the slaty cleavage. The rock is mineralogically homogeneous on a fine scale, with the exception of occasional green reduction spots (Fe^{2+}) and crystals of pyrite.

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.

Sample preparation

Powdered material was blended and then split into ~ 35 g units for distribution initially as a GeoPT sample at The Open University, with all excess to become this certified reference material. Grain size distribution measurements indicated 98.33% of material < 63 µm and 0.03% > 125 µm.

Homogeneity testing

Homogeneity testing was carried out when the material was originally prepared as a GeoPT™ test sample and is reported in Potts et al. (2001). Further homogeneity assessment was carried out using the submitted certification data. Each of the participating laboratories analysed replicate samples from three units of the material and the data was subjected to analysis of variance. Material variance for the few elements that were found to be heterogeneously distributed is included in the uncertainty of the certified values.

Certification

The certification project was carried out under the direction of the IAG certification committee following the IAG protocol for the certification of reference materials (Kane et al. 2003). Analyses were provided by thirty participating IAG laboratories, qualified by their performance in the GeoPT proficiency testing programme round nine (Potts et al. 2001).

*Certification analyses were performed by a variety of analytical methods (**Appendix 1),** some of which were non-destructive and some of which provided for total decomposition. Laboratory means were averaged without weighting to provide certified values. Values were classified as indicative, rather than certified, if there were fewer than ten laboratories reporting results and/or (b) there was not statistical agreement between results from two or more methods of analysis. The uncertainties of these values include variance components for between-laboratory and between-method disagreements in the dataset, for non-reproducibility in drying, as well as a component for material inhomogeneity in the few cases where that was warranted. Participating laboratories are listed in **Appendix 2.***

Certified and indicative values

*The certified mass fractions are given in **Table 1** for ten major oxides and thirty-five trace elements; indicative values are given for nine other oxides/elements in **Table 2**. The values are based on measurements using two or more independent analytical techniques. In some cases, primary methods (e.g., gravimetry for major oxides, isotope dilution-mass spectrometry (ID-MS) for a select few elements) have been used, though generally, certified values were established using comparative analytical techniques.*

*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.*

*Test portion masses of 100 mg or larger were used for most certification analyses. Analysis of variance showed the material to be homogeneous for most of the certified constituents at this test portion mass. Material variance for the few elements heterogeneously distributed is included in the uncertainty of the certified values. **Traceability of values** was established to the fullest extent possible by concurrent analysis of JSl-1 produced by the Geological Survey of Japan (Terashima et al. 1990, Imai et al. 1996). A complete description of the certification project and its results can be found in Kane (2004, 2005).*

Expiry of the Certification

Geological materials are known to be stable for particularly long periods of time, provided the material is properly stored, and individual test portions are removed with care to avoid contamination of the remaining sample. The certification is expected to remain valid for period of not less than twenty years from the date of issue of this revised certificate, i.e., until April 2040. The IAG will monitor the material and will report any changes that are identified.

Table 1
IAG OU-6 Penrhyn Slate

Certified values

Certified values for elemental/oxide mass fractions and uncertainties from the 2003 certification report on a dried (105 °C) basis

Oxide / element	Certified value g 100 g^{-1}	Uncertainty g 100 g^{-1}		Element	Certified value mg kg^{-1}	Uncertainty mg kg^{-1}
SiO_2	57.35	0.30		**Hf**	4.7	0.4
TiO_2	0.99	0.01		**Ho**	1.04	0.05
Al_2O_3	20.45	0.31		**La**	33.2	1.8
Fe_2O_3 (T)	8.94	0.20		**Lu**	0.45	0.02
MnO	0.280	0.003		**Nb**	14.5	0.6
MgO	2.41	0.03		**Nd**	30.2	1.5
CaO	0.74	0.02		**Ni**	40	2
Na_2O	1.76	0.05		**Pb**	28.8	0.8
K_2O	3.03	0.03		**Pr**	7.91	0.26
P_2O_5	0.120	0.004		**Rb**	121	4
LOI	3.62	0.06		**Sc**	23.1	3.2
	mg kg^{-1}	mg kg^{-1}		**Sm**	6.01	0.42
As	13.2	0.7		**Sn**	2.7	0.2
Ba	480	13		**Sr**	132	3
Ce	77.1	2.7		**Ta**	1.02	0.12
Co	29.2	1.1		**Tb**	0.86	0.04
Cr	70.7	2.1		**Th**	11.3	1.0
Cs	8.10	0.28		**U**	1.9	0.1
Cu	40	5		**V**	130	6
Dy	5.06	0.14		**Y**	27.8	0.8
Er	2.93	0.22		**Yb**	2.98	0.10
Eu	1.36	0.05		**Zn**	111	4
Ga	24.2	0.8		**Zr**	174	6
Gd	5.30	0.38				

The uncertainty *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.
LOI *is the loss on ignition (nominally 1050 °C for 2 hours).*
Fe_2O_3 (T) *is the total iron expressed as Fe_2O_3.*

Table 2
IAG OU-6 Penrhyn Slate

Indicative values

Indicative values for elemental/oxide mass fractions and uncertainties from the 2003 certification report on a dried (105 °C) basis

Oxide / element	Indicative value g 100 g^{-1}	Uncertainty g 100 g^{-1}		Element	Indicative value mg kg^{-1}	Uncertainty mg kg^{-1}
Fe(II)O	1.65	0.08		**Be**	2.5	0.4
Fe_2O_3 (calc)	7.12	0.21		**Li**	95	5
CO_2	0.23	0.08		**Sb**	0.6	0.2
				Tl	0.54	0.06
				Tm	0.45	0.02

The uncertainty *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.***
Fe(II)O *is the ferrous iron estimated from direct measurements submitted by participating laboratories.*
Fe_2O_3(calc) *is the calculated ferric iron content (expressed as Fe_2O_3) calculated from the difference between Fe_2O_3(T) and Fe(II)O data.*

Minimum test portion mass for use

Because homogeneity testing was carried out with masses of 100 mg or more, analysts should use the same 100 mg mass for all measurements in order to avoid any potential degradation of the certified value uncertainty that might result from the use of a smaller analytical sample mass.

Instructions for drying

The material should be dried at 105 ± 5 °C for 2 hours before weighing samples to be analysed. The weight loss to be expected on drying, based on H_2O^- reported by certification laboratories, should be less than 0.2% m/m, unless the sample has been improperly stored and thus exposed to excess humidity.

Limitations in applicability to techniques

The certified values represent total elemental mass fractions. Therefore, analysts should not expect to achieve certified values for their analyses if they use any partial decomposition techniques. For volatile elements, care should be taken to avoid volatility losses during preparation of the sample for analysis.

Storage information

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

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 the certificate originally published in Geostandards and Geoanalytical Research (Potts and Kane, 2003). Any further revisions to this Certificate of Analysis will be made available on the IAGeo Limited web site (www.iageo.com).

Approvals

This Reference Material Certificate was approved on behalf of the International Association of Geoanalysts as follows:

Name *Phil Potts* **Position** *On behalf of the IAG Certification Programme* **Date: 5th May 2020**

Name *Jacinta Enzweiler* **Position** *President of the International Association of Geoanalysts* **Date: 5h May 2020**

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Kane J.S. (2005)
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Geostandards Newsletter, 14, 1-5.

Appendix 1

Analytical methods used for certification

Oxide/element (No. of labs)	Number of methods	Identification of methods (No. of labs for each)
SiO_2 (24)	3	XRF (19) ICP-AES (4) GRAV (1)
TiO_2 (24)	2	XRF (19) ICP-AES (5)
Al_2O_3 (24)	3	XRF (19) ICP-AES (4) GRAV (1)
Fe_2O_3(T) (28)	4	NAA (2) XRF (19) ICP-AES (6) GRAV (1)
MnO (27)	4	XRF (19) AAS (1) ICP-MS (1) ICP-AES (6)
MgO (23)	2	XRF (17) ICP-AES (6)
CaO (22)	2	XRF (18) ICP-AES (4)
Na_2O (25)	5	NAA (2) XRF (15) AAS (1) ICP-AES (6) Fl. Photom. (1)
K_2O (24)	5	XRF (16) AAS (1) ICP-AES (5) Fl. Photom (1) ID-TIMS (1)
P_2O_5 (24)	3	XRF (17) ICP-AES (6) COLOR (1)
As (12)	4	NAA (2) XRF (6) AAS-hydride (1) ICP-MS
Ba (24)	5	NAA (2) XRF (8) ICP-MS (9) ICP-AES (4) ID-TIMS (1)
Ce (24)	4	NAA (2) XRF (6) ICP-MS (15) ID-TIMS (1)
Co (20)	4	NAA (2) XRF (7) ICP-MS (8) ICP-AES (3)
Cr (21)	4	NAA (2) XRF (9) ICP-MS (4) ICP-AES (6)
Cs (17)	3	NAA (2) XRF (1) ICP-MS (14)
Cu (14)	3	XRF (7) ICP-MS (3) ICP-AES (4)
Dy (17)	3	ICP-MS (14) ICP-AES (2) ID-TIMS (1)
Er (17)	3	ICP-MS (14) ICP-AES (2) ID-TIMS (1)
Eu (19)	4	NAA (2) ICP-MS)14) ICP-AES (2) ID-TIMS (1)
Ga (16)	2	XRF (8) ICP-MS (8)
Gd (16)	3	ICP-MS (13) ICP-AES (2) ID-TIMS (1)
Hf (16)	3	NAA (2) XRF (1) ICP-MS (13)
Ho (17)	3	NAA (1) ICP-MS (14) ICP-AES (2)
La (21)	5	NAA (2) XRF (3) ICP-MS (14) ICP-AES (1) ID-TIMS (1)
Lu (16)	4	NAA (2) ICP-MS (12) ICP-AES (1) ID-TIMS (1)
Nb (22)	2	XRF (9) ICP-MS (13)
Nd (23)	5	NAA (2) XRF (4) ICP-MS (13) ICP-AES (3) ID-TIMS (1)
Ni (20)	3	XRF (9) ICP-MS (5) ICP-AES (6)
Pb (22)	3	XRF (10) ICP-MS (11) ICP-AES (1)
Pr (17)	3	XRF (1) ICP-MS (14) ICP-AES (2)
Rb (26)	4	NAA (2) XRF (10) ICP-MS (13) ID-TIMS (1)
Sc (16)	4	NAA (3) XRF (2) ICP-MS (5) ICP-AES (6)
Sm (18)	4	NAA (2) ICP-MS (13) ICP-AES (2) ID-TIMS (1)
Sn (18)	2	XRF (10) ICP-MS (8)
Sr (27)	4	XRF (12) ICP-MS (10) ICP-AES (4) ID-TIMS (1)
Ta (15)	2	NAA (2) ICP-MS (13)
Tb (18)	3	NAA (2) ICP-MS (14) ICP-AES (2)
Th (23)	4	NAA (2) XRF (6) ICP-MS (14) ICP-AES (1)
U (14)	2	NAA (1) ICP-MS (13)
V (19)	3	XRF (8) ICP-MS (4) ICP-AES (7)

Y (23)	3	XRF (8) ICP-MS (12) ICP-AES (3)
Zn (23)	4	NAA (2) XRF (11) ICP-MS (4) ICP-AES (6)
Zr (23)	4	NAA (1) XRF (11) ICP-MS (10) ICP-AES (1)

Appendix 2

Participating Laboratories

- Becquerel Laboratories Pty Ltd., Menai, NSW 2234, Australia
- Genalysis Laboratory Services Pty Ltd., Maddington, WA 6109, Australia
- Osterreichisches Forschungzentrum, Seibersdorf, Austria
- Geosciences Laboratory, Sudbury, Ontario P3E 6B5, Canada
- XRAL laboratories, Don Mills, Ontario M3B 3J4, Canada
- Geological Survey of Denmark and Greenland, DK-2400 Copenhagen NV, Denmark
- Department of Earth Sciences, Aarhus University, DK-8000 Aarhus C, Denmark
- Geological Survey of Estonia, 12618 Tallinn, Estonia
- GTK Geolaboratory, Geological Survey of Finland, Rovaniemi, Finland
- Centre de Geochemie de la Surface, CNRS, 67084 Strasbourg Cedex, France CRB
- Analyse Service GmbH, D-37181 Hardegsen, Germany
- Bayerisches Geologisches Landesamt, D-80797 München, Germany
- Lurgi Umwelt GmbH, 60388 Frankfurt am Main, Germany
- Geoforschungs Zentrum Potsdam, D-14473 Potsdam, Germany
- Polish Geological Institute, 00-975 Warsaw, Poland
- Geological Survey of Slovak Republic, 05240 Spisská Nová Ves, Slovakia
- Samsung Corning Ltd., Suwon City, Kyunggi-Do, South Korea
- Department of Earth Sciences, The Open University, Milton Keynes MK7 6AA, UK
- Department of Earth and Environmental Sciences, The University of Greenwich, Chatham, Maritime, Kent ME4 4TB, UK
- Department of Earth Sciences and Geography, Kingston University, Kingston-upon-Thames, KT1 2EE, UK
- Department of Geology, Washington State University, Pullman, WA 99164, USA
- Savannah River Site, Aiken, SC 29808, USA
- Minerals Technologies, Inc., Easton, PA 18042, USA
- Laboratoire de Tectonophysique, Université de Montpellier, 34095 Montpellier, France
- Huk Umweltlabor GmbH, D-57482 Wenden, Germany
- Geological Survey of Norway, N-7491 Trondheim, Norway
- Geologisches Institut der Universitaet zu Koeln, D-50674 Koeln, Germany
- CRPG, 15 rue Notre-Dame des Pauvres, 54501 Vandoeuvre-lès-Nancy Cedex, France
- U.S. Geological Survey, Mineral Resources Laboratory, Denver, CO 80225, USA
- Max Planck Institut für Chemie, D-55020 Mainz, Germany