

International Association of Geoanalysts

Certificate of Analysis:

Certified Reference Material OU-6 (Penrhyn Slate)

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This certified reference material is intended to be used in the analysis of geological materials - rocks, soils and sediments - or other materials that have a silicate matrix. A unit of OU-6 includes approximately 35 g of pulverised material (98.33% of material < 63 μm and 0.03% > 125 μm) and is packaged in a plastic envelope.

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).

The certified concentrations are given in Table 1 for ten major oxides and thirty-five trace elements; information values are given for nine other oxides/elements in the same table. The values are based on measurements using two or more independent analytical techniques. In some cases, definitive 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.

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 that test portion mass. Material variance for the few heterogeneously distributed elements is included in the uncertainty of the certified values. Traceability of values was established to the fullest extent possible by concurrent analysis of JSI-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).

Table 1.
Certified and information
values and their uncertainties

Constituent	Certified value	Uncertainty
SiO ₂	57.35	0.30
TiO ₂	0.99	0.01
Al ₂ O ₃	20.45	0.31
Fe ₂ O ₃ (T)	8.94	0.20
MnO	0.28	0.003
MgO	2.41	0.025
CaO	0.74	0.011
Na ₂ O	1.76	0.05
K ₂ O	3.03	0.03
P ₂ O ₅	0.12	0.004
LOI	3.62	0.06
TOTAL	99.52	
As	13.23	0.66
Ba	480	13
Ce	77.1	2.7
Co	29.2	1.1
Cr	70.7	2.1
Cs	8.10	0.28
Cu	40.4	4.9
Dy	5.06	0.14
Er	2.93	0.22
Eu	1.36	0.05
Ga	24.17	0.73
Gd	5.30	0.38
Hf	4.70	0.32
Ho	1.04	0.05
La	33.2	1.8
Lu	0.45	0.02
Nb	14.49	0.58
Nd	30.2	1.5
Ni	40.2	1.3
Pb	28.80	0.79
Pr	7.91	0.26
Rb	121.3	3.9
Sc	23.1	3.2
Sm	6.01	0.42
Sn	2.67	0.19
Sr	131.7	2.6
Ta	1.02	0.12
Tb	0.86	0.04
Th	11.3	1.0

**Table 1 (continued).
Certified and information
values and their uncertainties**

Constituent	Certified value	Uncertainty
U	1.92	0.09
V	129.8	5.1
Y	27.75	0.74
Yb	2.98	0.10
Zn	111.4	3.4
Zr	174.2	5.7
Constituent	Information value	Uncertainty
Fe(II)O	1.65	0.08
Fe ₂ O ₃ (calc)	7.12	0.21
H ₂ O ⁻	0.14	0.13
CO ₂	0.23	0.08
Be	2.53	0.31
Li	95.3	4.5
Sb	0.56	0.13
Tl	0.54	0.06
Tm	0.45	0.02

Major oxide values are expressed in % m/m, whereas trace elements are expressed in mg kg⁻¹. Uncertainties for all certified values for IAG CRM OU-6 are expressed as 95% confidence intervals.

Source and preparation of the 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 (Fe²⁺) and crystals of pyrite. This powdered material was blended and then split into ~ 350 gram units for distribution initially as a GeoPT[™] sample at The Open University, with all excess to become this certified reference material.

Homogeneity testing

Homogeneity testing was carried out for the GeoPT[™] sample and is reported in Potts *et al.* (2001). Further homogeneity testing was done based on 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 heterogeneously distributed elements is included in the uncertainty of the certified values.

Certification

Certification analyses were performed by a variety of analytical methods (Table 2), 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 are given for information, rather than being certified, if (a) 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 data-set, 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 Table 3.

Conditions of appropriate use

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 ten years from the date of certification, i.e., until July 2013 or later. The IAG will monitor the material and will report any changes that are identified.

Minimum test portion mass for use

Because homogeneity testing was done with masses of 100 mg or more, analysts should use the same 100 mg mass for all analyses 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 hr before weighing samples to be analysed. The weight loss to be expected on drying, based on H₂O⁻ reported by certification laboratories, should be less

Table 2.
Analytical methods used for the 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 ₂ O ₃ (24)	3	XRF (19) ICP-AES (4) GRAV (1)
Fe ₂ O ₃ (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 ₂ O (25)	5	NAA (2) XRF (15) AAS (1) ICP-AES (6) Fl. Photom. (1)
K ₂ O (24)	5	XRF (16) AAS (1) ICP-AES (5) Fl. Photom (1) ID-TIMS (1)
P ₂ O 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)

**Table 3.
Participating laboratories**

Becquerel Laboratories Pty Ltd., Menai, NSW 2234, Australia
 Genalysis Laboratory Services Pty Ltd., Maddington, WA 6109, Australia
 Österreichisches Forschungszentrum, 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

than 0.2% m/m, unless the sample has been improperly stored and thus exposed to excess humidity.

Preparation of sample for analysis

The certified values represent total elemental concentrations. 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.

References

Imai N., Sakuramachi H., Terashima S., Itoh S. and Ando A. (1996)

1996 Compilation of analytical data on nine GSJ geochemical reference materials, "sedimentary rock series". *Geostandards Newsletter*, 20, 165-216.

Kane J.S. (2004)

Report of the IAG on the certification of Penrhyn Slate, OU-6. *Geostandards and Geoanalytical Research*, 28, 53-80.

Kane J.S. (2005)

Correction: Report of the International Association of Geoanalysts on the certification of Penrhyn Slate, OU-6 (*Journal*, 2004, vol. 28, 53-80). *Geostandards and Geoanalytical Research*, 29, 237-239.

Kane J.S., Potts P.J., Wiedenbeck M., Carignan J. and Wilson S. (2003)

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., Webb P.C. and Watson J.S. (2001)

GeoPT9. An international proficiency test for analytical geochemistry laboratories- Report on round 9/July 2001 (OU-6 Penrhyn slate). IAG unpublished report.

Terashima S., Ando A., Okai T., Kanai Y., Taniguchi M., Takizawa F. and Itoh S. (1990)

Elemental concentrations in nine new GSJ rock reference samples "sedimentary rock series". *Geostandards Newsletter*, 14, 1-5.