Reference Material Data Sheet

IAG OU-1 Bardon Volcanic Tuff

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Description of the reference material

Bardon Volcanic Tuff is a fine-grained grey-green volcanic tuff, partially recrystalised during low grade metamorphism. The mineral composition is largely plagioclase and feldspar with epidote, chlorite and some quartz. It was deposited at a volcanic centre active about 600 million years ago within the Charnian rocks of Leicestershire, UK.

Sample preparation

A 12-15 kg block of volcanic tuff, selected for its uniformity of colour and fine grain size, was collected during May 1996 from the north side of Number 7 Pit, Bardon Quarry, near Leicester, England. About 10 kg was split into 3-4 cm pieces and then passed twice through a Fritsch jaw crusher using first coarse, then fine, settings. The crushate was thoroughly homogenized in a plastic bin and divided into sixteen approximately equal portions using a two-way splitter. Portions of 650 g were ground in a Labtechnic swing mill using a chrome-free steel barrel. A trial particle size analysis by wet sieving showed that 80.9% of the powder passed a 63 µm sieve. A significant portion of the larger material comprised compacted flakes formed by impact between the heavy puck and the bowl. To disaggregate these flakes, the powder was re-milled in 80 g portions in a smaller Tema mill using an agate lined barrel. The re-milled powder was again homogenized and particle size determined as given below. The powder was divided into four 2.5 kg portions with a two-way divider. Each of these portions was then divided into eight portions of approximately 300 g each using a Retsch PT1000 eight-way sample divider, and each of these was divided into a further eight portions. The final product comprised 256 portions, each of 35 to 40 g powder, sealed into a flat plastic envelope.

Particle size distribution

Test portions (10 g) of the final sample powder were wet sieved and gave the following particle size distribution.

Particle size range	***proportion***
< 63 µm	*86.9%*
63-125 µm	*12.8%*
> 125 µm	*0.3%*

The proportion of material exceeding 63 µm (13%) was significantly larger than the desired specification (< 5%). However, most of the larger grains consisted of compacted flakes composed of much finer grains, and since the proportion of grains exceeding 125 µm in size was acceptably low, the powder was deemed to be acceptable for the trial.

Homogeneity testing

Homogeneity testing was based on an analysis of sixteen packets selected at random. These samples were analysed by WD-XRF at the Open University, UK. Duplicate glass discs and duplicate compressed powder pellets were prepared from each packet. Results for twenty-two major oxides and trace elements were obtained and analysed using standard analysis of variance (ANOVA) procedures. No significant differences between packets were identified at the 95% confidence level for any element in the study. Further details may be found in the GeoPT2 report (Geostandards Newsletter, 1998, 127-156).

Characterisation as a reference material

This material is characterised as a reference material using results from GeoPT2/1997 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme. The Proficiency Testing Steering Committee for this round was Prof M. Thompson (statistician), Prof P.J. Potts (results coordinator), Jean S. Kane, Dr P.C. Webb and J.S. Watson. The GeoPT2 report was published in Geostandards Newsletter, 1998, 127-156 and is available via the International Association of Geoanalysts web site (http://www.geoanalyst.org/index.php/proficiency-testing-proficiency-testing/geopt-programme/previous-rounds).

Intended use

This reference material is designed for use by laboratories measuring the major and trace element mass fractions in silicate rocks and similar materials 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

On the basis of the homogeneity results and an assessment of the methods used to contribute results to the GeoPT2 round, the minimum sample size recommended for use as a test portion is 0.2 g.

Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. On exposure to air, the material may absorb moisture, and instructions for handling must be followed.

Storage information

Store in a sealed container in a cool dry environment.

Assessment of reference values

The reference values were determined as 'consensus' values based on the statistical location of the participants' results in the GeoPT2 round. This location was determined as a robust mean if the distribution of results was unimodal and, outliers aside, close to symmetrical. If a slight asymmetry was apparent in a unimodal distribution, the median was chosen as an alternative. If a noteworthy skew was apparent and an objective explanation for the outcome was forthcoming, the mode of the results might be used. In other circumstances, usually when the number of valid results contributing to the location was less than 15 or their dispersion was unusually great, no reference value was assigned, although values may be reported as information values. These judgements were made by the IAG Proficiency Testing Steering Committee.

Instructions for handling

Before any measurements are made, every portion of the test sample must be dried at 105 ± 5 °C for at least 2 hours. Avoid contamination of the test material.

Reference material characterisation report

*Further details of the procedures used, the results, their original statistical analysis and assessment, on which the property values listed in this certificate are based, can be found in the GeoPT02 report (*http://www.geoanalyst.org/index.php/proficiency-testing-proficiency-testing/geopt-programme/previous-rounds*).*

IAG OU-1 Bardon Volcanic Tuff

Reference values

Measurand	Reference value	Uncertainty (expanded)	p		Measurand	Reference value	Uncertainty (expanded)	p
	g/100 g	*g /100 g*				*mg/kg*	*mg/kg*	
SiO_2	58.30	0.13	49		**Hf**	1.60	0.13	24
TiO_2	0.440	0.005	52		**Ho**	0.77	0.03	26
Al_2O_3	15.14	0.07	52		**La**	5.54	0.27	35
Fe_2O_3T	8.99	0.06	53		**Li**	20.0	0.9	13
MnO	0.129	0.002	52		**Lu**	0.39	0.04	29
MgO	4.73	0.04	52		**Nd**	7.00	0.12	29
CaO	6.50	0.03	52		**Pr**	1.60	0.03	22
Na_2O	2.48	0.03	53		**Rb**	2.0	0.2	35
K_2O	0.21	0.01	51		**Sc**	33.0	0.6	32
LOI	3.06	0.04	44		**Sm**	2.12	0.05	31
	mg/kg	*mg/kg*			**Sr**	105	2	51
Ba	130	5	51		**Ta**	0.14	0.02	12
Ce	12.3	0.6	36		**Tb**	0.48	0.04	25
Co	24.0	0.9	45		**Th**	1.62	0.06	31
Cs	0.137	0.011	14		**Tm**	0.36	0.02	19
Cu	61.6	2.7	44		**U**	0.38	0.03	26
Dy	3.38	0.08	25		**V**	221	4	42
Er	2.39	0.10	24		**Y**	21.9	0.5	48
Eu	0.51	0.02	31		**Yb**	2.49	0.06	31
Ga	13.7	0.4	30		**Zn**	76.0	1.4	46
Gd	2.74	0.10	24		**Zr**	54.5	2.6	45

Reference values *are the GeoPT assigned values obtained from a re-assessment using robust statistical analysis of results originally submitted to the GeoPT2 round. This reassessment took into account more recent experience of GeoPT data evaluation. Values are reported on a dried basis.*

Uncertainties *are the robust standard deviation of the mean or median or mode of the assigned value expanded by a coverage factor of two, and rounded up.*

Fe_2O_3T *is the total iron measured expressed as Fe_2O_3,* ***LOI*** *is the loss on ignition, nominally determined by heating a test portion to 1050 °C for 2 hours;* ***p*** *is the number of independent data sets that contributed to the reference value.*

Metrological traceability

Traceability was not formally demonstrated for this reference material. However, traceability could be demonstrated by the use of certified reference materials as calibrators or for performance assessment by the laboratories participating in this round and by the fact that some participating laboratories were formally accredited (although none of this information was recorded during the GeoPT02 round). However, traceability is implied by the overall consensus between datasets for individual elements/oxides submitted by laboratories that contributed to the GeoPT programme.

Safety information

Rock powders containing silicate minerals can cause harm especially if inhaled 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 are available on the relevant Material Safety Data Sheet.

IAG OU-1 Bardon Volcanic Tuff

Information values

Measurand	Information value	Uncertainty (expanded)	p		Measurand	Information value	Uncertainty (expanded)	p
	g/100 g	*g/100 g*				*mg/kg*	*mg/kg*	
Fe(II)O	5.06	0.24	12		**Mo**	0.9	0.2	19
P_2O_5	0.049	0.003	48		**Nb**	2.0	0.3	35
	mg/kg	*mg/kg*			**Ni**	12	3	43
As	8.4	0.6	22		**Pb**	3.9	0.8	32
Be	0.38	0.09	12		**Sb**	0.20	0.05	16
Cr	23	3	48					

Information values *are mainly 'provisional' values derived from the GeoPT02 dataset following a re-assessment of the source data originally submitted to the GeoPT02 round. This reassessment took into account more recent experience of GeoPT data evaluation, together with the opportunity to select median or mode values as information values, when justified by the data distribution. These data are provided for information purposes only and* ***not*** *for the calibration of methods or the assessment of data. Results are reported on a dried basis.*

Uncertainties *are the robust standard deviation of the mean, median or mode expanded by a coverage factor of two, and rounded up.*

Fe(II)O *is the mass fraction (g/100g) of ferrous iron expressed as its oxide;* ***p*** *is the number of independent data sets that contributed to the information value.*

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Revisions

Any revisions to this reference material data sheet will made available on the IAGeo Limited web site (www.iageo.com).

Acknowledgements

Peter Webb is acknowledged for undertaking a re-assessment of the GeoPT02 data set and for other contributions to this data sheet.

Approvals

This reference material information sheet was approved on behalf of the Certification and Reference Material Committee of the International Association of Geoanalysts.

Name *Philip J. Potts* **Position** *Chair of IAG Certification and Reference Material Committee* **Date** **4th August 2017**