



Reference Material Data Sheet

IAG OU-2 Belford Dolerite

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Description of the reference material

IAG OU-2 was collected from Craghill roadstone quarry, Belford, Northumberland, England (Grid reference NU 115 340) by John Holbrook, John Watson and Phil Potts during December 1991. The sample used to create this reference material was taken from surplus material remaining after preparation of the IWG-GIT reference material WS-E and a full field description can be found in Govindaraju et al. (Geostandards Newsletter, 18, 211-300). The sample was crushed, homogenised and hermetically sealed in packets at the Open University largely following procedures described in previous GeoPT reports.

Mineralogical description: *This dolerite comprises plagioclase, feldspar, clinopyroxene, orthopyroxene and opaque oxides, the plagioclase grains being up to 0.5 mm long with clinopyroxene as polycrystalline aggregates up to 1.2 mm across.*

Sample homogeneity: *Homogeneity testing was based on an analysis of sixteen packets, selected at random. These samples were analysed by WD-XRF at the Open University for the major elements (SiO₂, Al₂O₃, Fe₂O₃, MnO, MgO, CaO, Na₂O, K₂O, P₂O₅, TiO₂, LOI, Ba, Cr, Ni on glass disks and the trace elements (As, Ba, Co, Cr, Cu, Ga, Mo, Nb, Ni, Pb, Rb, S, Sc, Sr, Th, U, V, Y, Zn, Zr) on powder pellets. Duplicate glass disks and duplicate powder pellets were prepared from separate test portions taken from each packet. Results for twelve major/minor oxides and nineteen trace elements were analysed using standard analysis of variance (ANOVA) procedures, as described in the GeoPT4 report. No significant differences between packets were detected at the 95% confidence level for most of the elements in the present study. However, for SiO₂, Fe₂O₃, MgO, CaO and S measured on fused disks, small between-packet differences were observed and quantified. In every case, the conclusion resulted from atypically small “within” RSDs of the homogeneity testing data, which, for some packets, were derived from notionally zero differences. Had the within-differences been larger, as has been the case for previous Geo PT runs, and for the original WS-E homogeneity data (Govindaraju et al. 1994), the results would have satisfied the 95% confidence level criteria.*

Characterisation as a reference material

This material is characterised as a reference material using results from GeoPT04/1994 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 GeoPT04 report was published in Geostandards Newsletter (2000, v24, E1-E37). A re-assessment of datasets has been undertaken using more rigorous criteria than applied in the original report and closely aligned to those currently in use for GeoPT results evaluation.

Intended use

This reference material is designed for use by laboratories undertaking the determination of the major and trace element mass concentration fractions of 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 GeoPT04 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.

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 and cross-contamination of the test material.

Assessment of reference values

The reference values were determined as a 'consensus', based on the statistical location of the participants' results to the GeoPT04 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. In other circumstances, usually when the number of valid results contributing to the location was less than 12 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.

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 (although this information is not currently recorded by the GeoPT programme). Furthermore, traceability is implied by the overall agreement between datasets for individual elements/oxides submitted by laboratories that contributed to the GeoPT programme.

Reference to reference material characterisation report

Further details of the procedures used, the results, their statistical analysis and assessment, on which the property values listed in this certificate are based, can be found in the GeoPT04 report (<http://onlinelibrary.wiley.com/doi/10.1111/j.1751-908X.2000.tb00591.x/abstract>).

Safety information

Silicate powders containing heavy metals 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-2 Belford Dolerite Reference values								
Measurand	Reference value	Uncertainty (expanded)	n		Measurand	Reference value	Uncertainty (expanded)	n
	<i>g/100 g</i>	<i>g/100 g</i>				<i>mg/kg</i>	<i>mg/kg</i>	
SiO ₂	51.09	0.14	63		Ho	1.21	0.04	25
TiO ₂	2.425	0.016	65		La	27.3	0.6	48
Al ₂ O ₃	13.80	0.05	64		Lu	0.372	0.011	28
Fe ₂ O ₃ T	13.29	0.06	65		Mo	3.06	0.28	27
Fe(II)O	8.42	0.10	16		Nb	17.7	0.7	50
MnO	0.171	0.002	66		Nd	33.4	0.8	41
MgO	5.59	0.04	64		Ni	52.2	1.4	55
CaO	9.00	0.04	64		Pb	13.1	0.9	45
Na ₂ O	2.48	0.02	64		Pr	7.87	0.15	25
K ₂ O	0.99	0.01	63		Rb	25.4	0.7	55
P ₂ O ₅	0.300	0.005	60		Sc	28.2	0.7	35
	<i>mg kg⁻¹</i>	<i>mg kg⁻¹</i>			Sm	8.77	0.20	33
Ba	341	6	61		Sn	18.0	0.7	24
Be	1.1	0.1	14		Sr	407	4	63
Ce	60.2	1.5	50		Ta	1.20	0.05	24
Co	44.8	1.6	50		Tb	1.11	0.04	24
Cr	97	3	57		Th	3.02	0.15	42
Cs	0.50	0.04	20		Tm	0.427	0.017	21
Cu	63.0	0.9	49		U	0.635	0.038	26
Dy	6.10	0.18	27		V	340	5	54
Er	3.06	0.11	23		Y	30.9	0.8	55
Eu	2.22	0.06	30		Yb	2.56	0.08	31
Ga	23.1	0.5	35		Zn	114	2	56
Gd	7.14	0.33	23		Zr	201	4	54
Hf	5.20	0.13	30					

Reference values are the GeoPT assigned values obtained from a re-assessment using robust statistical analysis of results originally submitted to the GeoPT04 round. This reassessment took into account more recent experience of GeoPT data evaluation, together with the opportunity to select median values as the reference value, when justified by the data distribution. Values are reported on a dried basis.

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

Fe₂O₃T is the total iron expressed measured as Fe₂O₃.

IAG OU-2 Belford Dolerite Information values								
Measurand	Information value	Uncertainty (expanded)	n		Measurand	Information value	Uncertainty (expanded)	n
	<i>mg kg⁻¹</i>	<i>mg kg⁻¹</i>				<i>mg/kg</i>	<i>mg/kg</i>	
As	0.95	0.27	15		S	565	70	20
F	550	70	9		Sb	0.11	0.03	10
Ge	1.4	0.2	8		Tl	0.18	0.02	8
Li	12.9	0.9	14		W	0.75	0.21	12

Information values are 'provisional' values derived from the GeoPT04 dataset following a re-assessment of source data originally submitted to the GeoPT01 round. This reassessment took into account more recent experience of GeoPT data evaluation, together with the opportunity to select median 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 reports on a dried basis.

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

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Revisions

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

Acknowledgements

Peter Webb is gratefully acknowledged for undertaking a re-assessment of the GeoPT04 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

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