



Reference Material Data Sheet

IAG OU-5 Leaton Dolerite

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Description of the reference material

OU-5 is an olivine dolerite which was collected during May 1999 from Leaton Quarry, near Wellington, Telford, Shropshire, TF6 5HA, England, by courtesy of Johnstone Roadstone Ltd., the quarry operator. Several broken blocks were collected from the secondary crusher and prepared at the Open University (OU) for the GeoPT11-2001 proficiency testing round. Procedures for crushing, homogenisation and hermetically sealing in packets largely following procedures described in previous GeoPT reports. The sample was tested for homogeneity by selecting at random twelve packets of the sample prepared for distribution. Duplicate test portions from each packet were analysed by WD-XRF at the OU. For the elements that were given assigned values, homogeneity was considered to be satisfactory for use in the GeoPT11 round. Further details may be found in Appendix 1.

Characterisation as a reference material

This material is characterised as a reference material using results from GeoPT11/2001 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) Dr S.R.N. Chenery, Dr P.C. Webb and J.S. Watson. The GeoPT11 report was published on 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 GeoPT11 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.

IAG OU-5 Leaton Dolerite								
Reference values								
Measurand	Reference value	Uncertainty (expanded)	n		Measurand	Reference value	Uncertainty (expanded)	n
	g/100 g	g/100 g				mg/kg	mg/kg	
SiO ₂	49.09	0.15	66		Ho	1.93	0.07	35
TiO ₂	2.719	0.018	70		La	18.2	0.4	48
Al ₂ O ₃	13.62	0.06	71		Li	22.0	1.1	21
Fe ₂ O ₃ T	14.60	0.07	74		Lu	0.77	0.03	37
Fe(II)O	8.74	0.13	21		Nb	9.4	0.5	52
MnO	0.310	0.003	73		Nd	29.0	0.8	45
MgO	5.17	0.04	69		Pb	4.7	0.3	46
CaO	6.64	0.04	73		Pr	6.30	0.14	35
Na ₂ O	4.30	0.04	72		Rb	19.1	0.4	62
K ₂ O	0.83	0.01	73		Sb	0.42	0.04	17
P ₂ O ₅	0.440	0.005	62		Sc	42.7	0.8	39
	mg/kg	mg/kg			Sm	7.64	0.20	38
Ba	311	6	69		Sn	2.0	0.1	21
Be	1.35	0.15	18		Sr	227	3	67
Ce	44.2	1.1	53		Ta	0.55	0.04	32
Co	38.6	1.4	52		Tb	1.50	0.04	36
Cs	0.55	0.03	27		Th	2.26	0.09	42
Dy	9.2	0.3	35		Tm	0.81	0.04	30
Er	5.53	0.15	34		U	0.46	0.03	34
Eu	2.39	0.06	38		V	450	11	52
Ga	21.3	0.3	45		Y	52.2	1.2	62
Gd	8.70	0.21	34		Yb	5.17	0.18	39
Ge	2.03	0.16	15		Zn	133	3	58
Hf	5.65	0.17	37		Zr	220	4	63

Reference values are the GeoPT assigned values obtained from a re-assessment using robust statistical analysis of results originally submitted to the GeoPT11 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 of the assigned value expanded by a coverage factor of two, and rounded up.

Fe₂O₃T is the total iron expressed measured as Fe₂O₃, *Fe(II)O* is the ferrous iron composition.

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 'consensus' values based on the statistical location of the participants' results in the GeoPT11 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.

IAG OU-5 Leaton Dolerite								
Information values								
Measurand	Information value	Uncertainty (expanded)	n		Measurand	Information value	Uncertainty (expanded)	n
	<i>g/100 g</i>	<i>g/100 g</i>				<i>mg/kg</i>	<i>mg/kg</i>	
H₂O⁺	2.6	0.2	11		Cr	38.4	2.3	59
LOI	2.1	0.1	56		Cu	27.6	1.5	52
	<i>mg/kg</i>	<i>mg/kg</i>			F	670	140	13
As	2.5	0.3	23		Ni	15.0	1.2	56
Cd	0.20	0.04	15		Tl	0.13	0.03	12
Cl	160	25	7		W	0.8	0.2	17

Information values are mainly 'provisional' values derived from the GeoPT11 dataset following a re-assessment of source data originally submitted to the GeoPT11 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 reports on a dried basis.

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

H₂O⁺ is the structural water, **LOI** is the loss on ignition, usually determined by heating a test portion to 1050 °C for 2 hours.

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 consensus 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 GeoPT11 report (<http://www.geoanalyst.org/index.php/proficiency-testing-proficiency-testing/geopt-programme/previous-rounds>).

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.

Legal notice – terms and conditions

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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 GeoPT11 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** 3rd May 2016

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Appendix 1 - GeoPT11 Homogeneity Report

Homogeneity testing was based on analysis of duplicate test portions taken from each of 12 packets, which had been selected at random. These samples were analysed in duplicate by WD-XRF at the Open University for the major and minor elements (SiO₂, TiO₂, Al₂O₃, Fe₂O₃, MnO, MgO, CaO, Na₂O, K₂O, P₂O₅, LOI) on glass discs 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, following the procedures described in the GeoPT1 report.

Statistical analysis of homogeneity data was carried out using a new sequence of tests developed by Fearn and Thompson (Analyst, 2001, 126, 1414-1417) as follows: DATATEST is the outcome of a range of tests designed to identify analytical problems and discrepancies that could mask differences in analytical results related to inhomogeneity effects, particularly the identification of outliers. All elements listed in the Tables below, passed these tests. CONC is the average concentration derived from the XRF results. SIGMA is the target value for the standard deviation derived using the same modified form of the Horwitz function that was used to calculate the target precision for pure geochemistry laboratories (data quality = 1), described above. F is the well-known F-statistic for one-way analysis of variance. When compared to the relevant critical value of 4.94 (majors) or 2.78 (traces) all elements passed the F-test. VARSAM is the estimated between-sample variance, which is used to calculate: SAMRATIO, which is the ratio of the square root (VARSAM) / SIGMAP. Where this ratio has a value of less than 0.3, the element data is considered to have passed the harmonised protocol (HP-TEST). Results for LOI, As, Co, Ga, Mo, Ni, S, Th and U were, however, significant (SAMRATIO > 0.3). Careful re-evaluation of the source data for these elements indicated that inadequate analytical precision had been achieved in the determination of most of these elements and that interpretation of homogeneity may not be reliable.

In the typical interpretation of homogeneity data, the principal criterion is normally that elemental results 'pass' the classical F-test. However, this is not the ultimate arbiter of homogeneity, since the Harmonised Protocol requires that homogeneity should have an insignificant effect on the interpretation of proficiency testing results. If the within-packet variance is particularly small, the F-test may detect a significant level of between-packet variance (indicating inhomogeneity effects), that is, in fact, unimportant in relation to the target precision against which the results from participating laboratories are evaluated. In order to test the significance of data that 'fails' the F-test, the ratio of sampling precision to target precision is calculated. If this ratio is less than 0.3, elemental results are considered to be compatible with those of a homogeneous sample, in the context of this proficiency testing programme. Although data for LOI, As, Co, Ga, Mo, Ni, S, Th and U all passed the F-test, these elements gave a SAMRATIO of > 0.3 indicating results for these elements failed the harmonised protocol test. It is considered that the most likely explanation for this is that the precision of the XRF data for these elements was not adequate for homogeneity testing, noting that several approach the detection limit range of the technique. Participating laboratories are, therefore, advised to take these considerations into account when interpreting the z-score values for these elements.

Table H1 showing the results of homogeneity testing on the WD-XRF major element data and LOI.

ANALYTE	DATA TEST	CONC	SIGMAP	VAR-SAM	F	F-TEST	SAMRATIO	HP-TEST
SiO2	OK	44.4382	0.502075	0.0063983	1.35683	OK	0.159318	OK
TiO2	OK	0.5867	0.012714	0.0000042	3.3242	SIG	0.161994	OK
Al2O3	OK	11.9951	0.16505	0.00053	1.47088	OK	0.13948	OK
Fe2O3	OK	4.623	0.073	0.000	2.219	OK	0.093	OK
MnO	OK	0.287	0.007	0.000	1.084	OK	0.027	OK
MgO	OK	2.211	0.039	0.000	1.344	OK	0.146	OK
CaO	OK	12.697	0.173	0.000	0.985	OK	0.000	OK
Na2O	OK	2.781	0.048	0.000	1.244	OK	0.120	OK
K2O	OK	2.013	0.036	0.000	0.584	OK	0.000	OK
P2O5	OK	0.118	0.003	0.000	1.351	OK	0.209	OK
LOI	OK	18.221	0.235	0.000	0.437	OK	0.000	OK

Table H2 showing the results of homogeneity testing on the WD-XRF trace element data.

ANALYTE	DATA TEST	CONC	SIGMA	VAR-SAM	F	F-TEST	SAMRATIO	HP-TEST
Rb	OK	113.8	4.464	0	0.29095	OK	0	OK
Sr	OK	506.1	15.857	0.34	1.23549	OK	0.036756	OK
Y	OK	24.9	1.229	0	0.71457	OK	0	OK
Zr	OK	137.3	5.235	0	0.86637	OK	0	OK
Nb	OK	13.6	0.737	0	0.9398	OK	0	OK
Ba	OK	428.7	13.773	2.473	1.33997	OK	0.114171	OK
Pb	OK	19.6	1.004	0.186	1.17584	OK	0.429977	SIG
Th	OK	13.2	0.715	0.062	1.21999	OK	0.347474	SIG
U	OK	3.3	0.22	0	0.67849	OK	0	OK
Sc	OK	15.6	0.825	0.272	1.37964	OK	0.631635	SIG
V	OK	92.4	3.739	0.373	1.27503	OK	0.163268	OK
Cr	OK	67.4	2.861	0	0.37642	OK	0	OK
Co	OK	9.6	0.546	0	0.60509	OK	0	OK
Ni	OK	46.6	2.091	0	0.42918	OK	0	OK
Cu	OK	23.3	1.159	0	0.23776	OK	0	OK
Zn	OK	91.8	3.718	0	0.50769	OK	0	OK
Ga	OK	14.7	0.785	0	0.81497	OK	0	OK
Mo	OK	-0.3	*	0.045	2.84955	OK	*	SIG
As	OUT	*	*	*	*		*	
S	OK	1034.1	29.098	22.763	1.71501	OK	0.163967	OK
TiO2	OK	5999	129.562	406.806	1.48072	OK	0.155674	OK
Fe2O3	OK	44048.5	704.725	0	0.88687	OK	0	OK