



Reference Material Data Sheet

IAG G94 Threlkeld Microgranite

International Association of Geoanalysts

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Description of the reference material

Collection

The sample was collected from the southern part of the upper level of 'Threlkeld Quarry, a disused roadstone quarry situated about 5 km East of Keswick, Cumbria within the English Lake District. The field party that collected the sample comprised delegates attending the International Conference 'Geoanalysis 94' in Ambleside during September 1994 and who were participating in a post-conference workshop on geological reference materials. The sample was designated G94 - Threlkeld microgranite. Threlkeld Quarry had been worked for roadstone until 1982, and had been reopened as the 'Threlkeld Quarry and Mining Museum', containing displays and exhibits that demonstrated quarrying operations, particularly those aspects associated with the Lake District.

Geological setting

Threlkeld quarry is situated in an outcrop of felsic intrusive rock of Upper Ordovician / Lower Silurian age which has been dated at 468 ± 6 Ma. Several outcrops of this rock, a quartz-feldspar-phyric microgranite, apparently form part of a single, irregularly shaped body intruded into the Ordovician Skiddaw Group, very close to the overlying contact with the Borrowdale Volcanic Group. Xenoliths of the Skiddaw Group sediments are common in the Threlkeld intrusion.

Mineralogical description

Examination of thin sections showed a strongly altered rock in which the extensive alteration (50 – 60%) made it difficult to recognise primary minerals - especially the feldspars. The black specks, which had been observed in the field, but could not then be identified positively, were absent in the thin section examined as was garnet, which had also been observed occasionally in the field. The volume percentages of minerals were determined with the aid of a point counter. The severe alteration, particularly of feldspar minerals, hindered quantification. Plagioclase and alkali feldspar were counted together. The results of this mineralogical assessment were approximately as follows:

Feldspar (66%); quartz (25%); alteration products, i.e. chlorite (9%). sericite, calcite; accessories (titanite, epidote, zircon, rutile, ilmenite).

Sample homogeneity

Twelve packets of the sample, selected at random from the batch prepared for distribution were analysed for both major and trace elements using routine multi-element programmes by:

(a) X-ray fluorescence analysis (P.C. Webb and J.S. Watson, The Open University). The major elements were determined from glass discs and the trace elements from compressed powder pellets using routine analytical

conditions. Two glass discs and two powder pellets were prepared from each packet and each was analysed in duplicate.

(b) ICP-AES and ICP-MS (K. Govindaraju, CRPG, Nancy). Duplicate test portions from each packet were fused with lithium metaborate, taken up into solution and analysed by ICP-AES and ICP-MS using routine operating conditions. Results indicated that homogeneity was satisfactory for the proficiency testing trial.

Particle size distribution

Test portions (10 g) of the final sample powder were wet sieved and gave the following particle size distribution, which was deemed to be acceptable for the Trial:

G94 Threlkeld microgranite - particle size distribution

<63 μm	98.33%
63 - 125 μm	1.64%
>125 μm	0.03%

Results courtesy of J.S. Watson

Characterisation as a reference material

This material is characterised as a reference material using results from GeoPT01/1996, the first 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) and Dr P.C. Webb. The GeoPT01 report was published in Geostandards Newsletter (1996, v20, 295-325). 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 GeoPT01 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 GeoPT01 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.

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Reference Values							
Measurand	Reference value	Uncertainty	Number of reported results	Measurand	Reference value	Uncertainty	Number of reported results
	g / 100 g				mg / kg		
SiO ₂	69.94	0.14	43	Hf	3.38	0.20	18
TiO ₂	0.314	0.004	43	Ho	0.55	0.04	13
Al ₂ O ₃	14.66	0.07	43	La	22.0	0.6	26
Fe ₂ O ₃ T	3.05	0.03	44	Lu	0.227	0.013	16
MnO	0.075	0.002	42	Nb	12.1	0.6	35
MgO	1.04	0.02	43	Nd	16.7	0.7	25
CaO	1.34	0.01	43	Ni	16.9	1.1	33
Na ₂ O	4.52	0.04	44	Pr	4.64	0.30	12
K ₂ O	2.97	0.02	43	Rb	148	3	40
P ₂ O ₅	0.168	0.004	41	Sb	0.62	0.06	14
LOI	1.97	0.04	34	Sc	6.1	0.4	17
	mg / kg	mg / kg		Sm	3.59	0.15	19
Ba	357	8	44	Sr	89.0	1.8	43
Ce	42.0	1.3	28	Ta	1.33	0.10	14
Co	6.4	0.4	34	Tb	0.50	0.02	14
Cs	3.11	0.19	12	Th	7.1	0.3	31
Cu	16.0	1.0	35	U	3.08	0.22	28
Dy	2.79	0.15	16	V	37.8	1.6	38
Er	1.46	0.10	12	Y	16.1	1.0	37
Eu	0.74	0.03	18	Yb	1.45	0.05	20
Ga	17.0	0.5	28	Zn	39.4	1.7	37
Gd	3.27	0.17	12	Zr	133	4	40

Reference values are the GeoPT assigned values obtained from a re-assessment using robust statistical analysis of results 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 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.

LOI is the loss on ignition (normally at 1050 °C).

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 GeoPT01 report (<http://onlinelibrary.wiley.com/doi/10.1111/j.1751-908X.1996.tb00191.x/abstract>).

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Information Values			
Measurand	Information Values	Uncertainty	Number of reported results
	g / 100 g	g / 100 g	
Fe(II)O	2.57	0.09	10
	mg / kg	mg / kg	
As	3.0	0.4	22
Be	1.7	0.3	10
Cr	33	3	38
Li	52	2	8
Pb	8	2	31
Tm	0.21	0.02	9

Information values are 'provisional' values derived from the GeoPT01 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.

Fe(II)O is the ferrous iron content of the sample.

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.

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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 GeoPT01 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 **24th November 2015**

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