Reference Material Certificate

Certified Reference Material

IAG GMN-1

Meissen Granite

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Geological origin

The Meissen Granite (IAG GMN-1) is a leucomonzogranite from the Meissen Massif. The source rock is known under various names in geological literature. The best known are 'Granit der Riesensteine' (Granite of the Giant Stones), 'Riesensteingranit' (Giant Stone Granite), 'Roter Granit (Meißen)' (Red Granite (of Meissen)), and 'Meißner Granit' (Meissen Granite). These names are essentially regional or coined from the local stone industry. The 'Meissen Granite' represents the youngest and probably smallest intrusive stage of the late Variscan Meissen Massif. The Meissen plutonic complex developed as a typical telescope intrusion with K-rich dioritic to predominantly monzonitic rocks at the margin and granodioritic to granitic rocks in the centre. The modal composition of the three main intrusive stages changed from dominantly monzonitic, to granodioritic to leuco-monzogranitic.

Intended use

This certified reference material is designed for use by laboratories undertaking the determination of major and trace element mass fractions in silicate rocks and equivalent matrices 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.

Certification programme

(i) *About 150 kg of rock material (Meissen Granite) was collected by Dr Axel Renno of the Helmholtz Institute Freiberg for Resource Technology from the Jansen Beton und Granitwerke GmbH granite quarry, Granitbruch Meissen-Cölln, in Meissen, Germany. The material came from the ongoing operation of the quarry. The material was processed initially at the Deutsches GeoForschungsZentrum GFZ, Telegrafenberg, Potsdam, Germany and subsequently at the British Geological Survey (BGS), Keyworth, UK, where it was milled (using hardened steel milling equipment), homogenised, divided and packeted.*

(ii) *Packets of the material were distributed for analysis to laboratories participating in Round 51 of the IAG GeoPT proficiency testing programme, together with packets of a supplementary test material comprising the established certified reference material CGL 008 (MGT-1 Granite). A full analysis of the results of this round are available as reports GeoPT 51 (Meissen Granite) and 51A (CGL 008) (Webb et al. 2022a,b).*

(iii) *Certification of IAG GMN-1 was undertaken using the IAG certification protocol based on the GeoPT proficiency testing programme (Potts et al. 2019). A full description of the application of this protocol to the certification of IAG GMN-1 is available (Potts et al. 2024).*

IAG MGN-1 material characteristics

(i) ***Particle size distribution*** *was undertaken by sieving: >98% by mass passed a 75 µm sieve and >93% passed a 53 µm sieve, as detailed in table 1 below:*

Table 1 IAG GMN-1 Meissen Granite Particle size distribution

Sieve size (µm)	Particle size retained (µm)	% by mass
125	>125	0.1
106	<125 - >106	0.2
75	<106 - >75	1.42
63	<75 - >63	2.35
53	<63 - >53	3.18
32	<53 - >32	14.08
	<32	78.68
	Total	100

(ii) ***Mineralogy*** *– modal analysis of IAG GMN-1 was undertaken following procedure by Rushton and Lakinska (2022).*

Table 2 Modal analysis of Meissen Granite

Mineral Group	*Area %	Mineral	*Area %
Quartz	33.56	Quartz	33.56
K-feldspar	26.45	K-feldspar	26.45
Plagioclase	36.29	Albite (AB)	20.50
		Oligoclase (ol)	15.01
		Andesine (an)	0.78
Mica	2.18	Muscovite (ms)	0.29
		Biotite (bt)	1.89
Chlorite	0.93	Chlorite (Fe rich)	0.01
		Chlorite (Mg rich)	0.37
		Chlorite (altered)	0.56
Phosphates	0.07	Apatite	0.07
Oxides	0.20	Fe-Ti oxides	0.02
		Fe oxide	0.12
		Ti oxide	0.06
Carbonates	0.28	Ca carbonate	0.24
		Ca carbonate (Mn rich)	0.01
		Siderite	0.04
Accessories	0.05	Fluorite	0.02
		Zircon	0.01
		Ca-REE phase	0.01

**Area normalised to 100%*

(iii) ***Homogeneity testing*** *was undertaken at the British Geological Survey (Keyworth, UK) on ten packets of Meissen Granite at random from the material prepared for certification. Duplicate test portions of 0.2 g were digested by sodium peroxide fusion, taken up in aqueous solution and measured in random order by ICP-MS. A standard F-test was undertaken on the test results which indicated that the material was sufficiently homogeneous for use as a geochemical certified reference material except for Cr. It was considered that Cr data was unsatisfactory because of contamination caused by slight abrasion of the hardened steel milling equipment used during sample preparation.*

(iv) ***Stability*** *– a formal test for stability was not undertaken but an evaluation of the characteristics of this material provided no indication that reported measurement values would be affected by significant changes over the period of availability of this material (Potts et al. 2024).*

(v) ***Derivation of certified values.*** *Estimates of the composition of this material were provided by assigned and provisional values derived from an analysis of the results submitted to the* Geo*PT 51 proficiency testing programme. Following the requirements of the* Geo*PT certification protocol (Potts et al. 2019),* Geo*PT assigned values are reported here as certified values and* Geo*PT provisional values as information/indicative values. In both cases, associated uncertainties are derived as standard deviations of the mean from the full* Geo*PT data set, expanded to give a confidence interval of 95%. Addition confidence in these data was obtained by assessing the results for CGL 008 (MGT-1) from round* Geo*PT51A and undertaking a comparison between* Geo*PT assigned values and independently assess certified values. Comparison of these data was considered to be satisfactory (Potts et al., 2024)*

Table 3 IAG GMN-1 Certified Values, Uncertainties and Quality Factors

	Certified Value	Uncertainty (95% confidence limit)	Quality factor (QF)		Certified Value	Uncertainty (95% confidence limit)	Quality factor (QF)
	g 100 g^{-1}	g 100 g^{-1}			mg kg^{-1}	mg kg^{-1}	
SiO_2	73.50	0.15	0.29	**Lu**	0.476	0.031	1.09
TiO_2	0.210	0.002	0.56	**Mo**	1.64	0.11	1.25
Al_2O_3	13.47	0.06	0.42	**Nd**	26.6	0.5	0.48
Fe_2O_3T	1.96	0.01	0.45	**Ni**	8.93	0.47	1.35
MnO	0.050	0.001	0.35	**Pb**	31.9	0.8	0.75
CaO	1.00	0.01	0.55	**Pr**	7.80	0.13	0.42
Na_2O	3.83	0.04	0.79	**Rb**	255	3	0.49
K_2O	4.80	0.03	0.59	**Sb**	0.095	0.006	0.83
P_2O_5	0.060	0.001	0.63	**Sc**	4.99	0.26	1.21
	mg kg^{-1}	mg kg^{-1}		**Sm**	5.24	0.12	0.53
Ba	344	5	0.65	**Sn**	5.62	0.34	1.45
Be	5.00	0.23	1.07	**Sr**	136	2	0.40
Bi	0.160	0.013	1.09	**Ta**	1.80	0.06	0.68
Ce	70.0	1.9	0.94	**Tb**	0.737	0.018	0.44
Co	2.11	0.08	0.78	**Th**	35.1	0.8	0.71
Cs	8.07	0.20	0.61	**Tl**	1.52	0.06	0.68
Dy	4.75	0.16	0.79	**Tm**	0.450	0.021	0.77
Er	2.98	0.08	0.56	**U**	12.4	0.3	0.66
Eu	0.60	0.02	0.52	**V**	13.2	0.5	1.05
Ga	17.6	0.6	0.90	**W**	3.90	0.30	1.77
Gd	4.51	0.12	0.58	**Y**	29.7	0.8	0.81
Hf	5.26	0.10	0.44	**Yb**	3.04	0.09	0.65
Ho	0.97	0.07	1.33	**Zn**	31.8	0.7	0.67
La	36.2	0.8	0.68	**Zr**	142	4	1.06
Li	56.4	1.3	0.77	**MEDIAN**			0.68

Table 4 IAG GMN-1 Information Values

	Information Value	Uncertainty (95% confidence limit)		Information Value	Uncertainty (95% confidence limit)
	g 100 g^{-1}	g 100 g^{-1}		mg kg^{-1}	mg kg^{-1}
MgO	0.37	0.01	**Cr**	545	24
	mg kg^{-1}	mg kg^{-1}	**Cu**	2.49	0.48
As	2.12	0.18	**Ge**	1.67	0.28
C(tot)	862	79	**In**	0.033	0.004
Cd	0.026	0.012	**Nb**	22.6	1.2

Certified values

Certified values listed in the Table 3 are based on procedures that are summarised in the International Association of Geoanalysts GeoPT certification protocol and derived from the GeoPT51 proficiency testing round. These values the best estimates of the true composition of this CRM, at the 95% confidence limit.

Indicative values

Indicative values in Table 4 are designed to provide guidance on the mass fractions of other selected elements and should not be used to validate analytical measurements without additional corroboration.

Quality factors

Quality factors listed with certified values are designed to give an indication of the reliability of certified values in assessing measurement results in routine geochemical analysis. Briefly, a quality factor of 1 or less indicates that the uncertainty in the certified value is at least three times smaller than the uncertainty expected of a laboratory operating to the higher data quality 1 performance standard in the GeoPT programme. In these circumstances, the certified value uncertainty will not have a significant effect in assessing data quality performance. The median QF value (0.68) places IAG GMN-1 (Meissen Granite) towards the lower, most beneficial, end of the range of median values for CRMs that were investigated by Potts and Webb (2022).

Instructions for handling

Certified values are for total mass fractions of oxides and elements, reported on a dry weight basis. Prior to use test portions should be dried at 105 ^{o}C for 2 hours before weighing. Alternatively, moisture content may be determined on a second test portion, the results from which can be used to correct to a dry weight. Re-homogenisation by manual shaking of the closed bottle is recommended before a sub-sample is taken.

Minimum sample mass

The recommended minimum sample test portion mass is 200 mg, as used in the homogeneity study. However, a significant number of the GeoPT 51 participating laboratories provided measurement results based on ≤ 100 mg test portions; an analysis of data distributions indicated no particular reason against taking 100 mg according to the above instructions to reproduce the reference values.

Storage information

The CRM should be stored at room temperature and tightly sealed to protect it from absorption of atmospheric moisture and laboratory chemicals.

Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. Consequently, the nominal period of validity of this certificate is selected as 20 years from the date of certification. On exposure to air, the material may absorb moisture, and instructions for handling to remove absorbed water before use of the material must be followed.

Metrological traceability

Certified values in IAG GMN-1 (Meissen Granite) are traceable according to the GeoPT certification protocol, which claims traceability based on: (i) Consensus values derived from a rigorous assessment of all measurement results submitted using robust statistical procedures, noting that a significant proportion of these laboratories are accredited to ISO 17025. (ii) The synchronous measurement by participating laboratories of an established CRM (CGL 008 – MGT-1) with a satisfactory evaluation of the equivalence between resultant GeoPT consensus values and corresponding certified values. (iii) Evidence from previous performance of the GeoPT programme when certified reference materials were used as test materials which has demonstrated the absence of statistically significant bias in GeoPT consensus values (see Potts et al., 2015).

Certification characterisation report

Further details of the procedures used, the results, their statistical analysis and data assessment, on which the property values listed in this certificate are based, can be found in the IAG GMN-1 certification report (Potts et al. 2024).

Safety information

Silicate powders can cause harm especially if ingested 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 may be found on the relevant material safety data sheet.

Legal notice – terms and conditions

1. The IAG shall not be liable to the user of this material for loss (whether direct or indirect) of profits, business, anticipated savings or reputation or for any indirect or consequential loss or damage whatsoever even if previously advised thereof and whether arising from negligence, breach of these Terms and Conditions or howsoever occurring.
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Revisions

This certificate is version 1.00. Any further revisions to this Reference Material Certificate will be made available on the IAGeo Ltd web site (www.iageo.com).

Acknowledgements

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References

Potts, P.J., Thompson, M. and Webb, P.C. (2015)
The reliability of assigned values from the GeoPT proficiency testing programme from an evaluation of data for six test materials that have been characterised as certified reference materials. Geostandards and Geoanalytical Research, 39, 407-417.

Potts, P.J. and Webb, P.C. (2022)
Simple Test for the Fitness for Purpose of Geological Reference Materials According to the Uncertainties Associated with Their Reference Values. Geostandards and Geoanalytical Research, 46, 477-492.

Potts, P.J., Webb, P.C., Gowing, C.J.B. and Renno, A. (2024)
Certification of the Meissen Granite IAG GMN-1 using the GeoPT proficiency testing certification protocol. Geostandards and Geoanalytical Research, (accepted for publication).

Potts, P.J., Webb, P.C. and Thompson, M. (2019)
The GeoPT Proficiency Testing Programme as a Scheme for the Certification of Geological Reference Materials. Geostandards and Geoanalytical Research, 43, 409-418.

Rushton, J.C. and Lakinska, A.M. (2022)
Modal and microanalysis of Karara and Meissen Granites and larvikite samples. British Geological Survey (Keyworth, UK) Commercial Report CR22/17, 17pp.

Webb, P.C., Potts, P.J., Thompson, M., Gowing, C.J.B. and Renno, A.D. (2022a)
GeoPT51 – An international proficiency test for analytical geochemistry laboratories – Report on Round 51 (Leucomonzogranite, GMN-1) / July 2022, International Association of Geoanalysts (Keyworth, UK), Unpublished Report, 49pp.

Webb, P.C., Potts, P.J., Thompson, M. and Gowing, C.J.B (2022b)
GeoPT51A – An international proficiency test for analytical geochemistry laboratories – Report on Round 51A (Granite, MEG-1) / July 2022, International Association of Geoanalysts (Keyworth, UK), Unpublished Report, 49pp.

Approvals

This Certificate of Analysis was approved on behalf of the International Association of Geoanalysts follows:

Name *Prof Jacinta Enzweiler*	**Position** *President of the International Association of Geoanalysts*	**Date:** *29th March 2024*
Name *Prof Phil Potts*	**Position** *Director of the GeoPT Certification Programme*	**Date:** *29th March 2024*
Name *Dr Peter Webb*	**Position** *Director of the GeoPT Proficiency Testing Programme*	**Date:** *29th March 2024*