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CERTIFICATE OF ANALYSIS FOR CERTIFIED REFERENCE MATERIAL

AC18.10659

Description: Gabbronorite, Cambro-Ordovician, Adelaide Fold Belt, South Australia, Australia.

The material consists of rock sourced from an operating (Black Hill) quarry, located approx. 85 km east of the city of Adelaide (South Australia). This light grey, Cambro-Ordovician (~490 Ma) rock forms part of a large gabbroic intrusion. Mineralogy consists of mainly sodic labradorite plagioclase (49 %), K-feldspar (13 %), biotite (11 %), augite and hypersthene clinopyroxenes (10 %) with minor amounts of dolomite – ankerite (5 %), magnetite (4 %), quartz (4 %) and calcic amphibole (2 %).

AC18.10659 is available as 50 g units packed into glass, wide-mouth jars.

Intended use: For use in evaluating instrumental analytical methods for the chemical analysis of lithological samples.

Certified and informational values derived from analytical methods of analysis are provided in Tables 1 and 2, respectively.

Approving officer: Management of the interlaboratory certification program by Craig Hamlyn (Technical Manager, OREAS).

Minimum sample size: To relate analytical determinations to the values in this certificate, a minimum dry sample mass of 0.2 g should be used.

Storage and period of validity: The certification of AC18.10659 remains valid, within the specified measurement uncertainties, until June 2029, provided the CRM is stored in a clean and cool dry place away from direct sunlight. This certification is nullified if the CRM is any way changed or contaminated.

Maintenance of Certified Values: OREAS will monitor this CRM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, OREAS will notify the purchaser (using the contact's email address on the Sales Order).

Table 1. Certified Values and their associated 95% Expanded Uncertainty.

Constituent (wt.%)	Certified Value	95 % Expanded Uncertainty
Al_2O_3	16.65	0.21
CaO	9.32	0.18
Fe_2O_3	10.40	0.14
K_2O	2.07	0.03
MgO	5.10	0.07
MnO	0.166	0.005
Na_2O	2.58	0.04
P_2O_5	0.421	0.013
SiO_2	51.68	0.53
TiO_2	1.27	0.03

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95 % Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Constituent (ppm)	Certified Value	95 % Expanded Uncertainty
As	< 5	IND
B	< 50	IND
Ba	560	22
Be	1.90	0.29
Bi	< 0.1	IND
Cd	< 10	IND
Ce	51	4
Co	33.8	2.3
Cr	104	16
Cs	5.37	0.42
Cu	64	8
Dy	4.24	0.43
Er	2.30	0.22
Eu	1.68	0.17
Ga	20.5	1.8
Gd	5.26	0.56
Ge	1.66	0.33
Ho	0.80	0.10
In	< 0.2	IND
La	24.6	1.9
Li	15.9	2.3
Lu	0.309	0.037
Mo	< 5	IND
Nb	7.55	1.41
Nd	28.0	2.0
Ni	45.4	7.3
Pr	6.56	0.51
Rb	102	10
Sb	< 0.1	IND
Sc	25.0	3.0
Sm	5.90	1.03
Sn	2.23	0.55
Sr	578	29
Ta	< 5	IND
Tb	0.70	0.09
Te	< 1	IND
Th	8.22	0.58
Tl	< 0.5	IND
Tm	0.32	0.04
U	1.80	0.32
V	286	18
W	< 5	IND
Y	22.0	1.3
Yb	2.11	0.37
Zn	96	9
Zr	94	8

Table 2. Indicative Values for AC18.10659.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Ag	ppm	*< 5*	F	ppm	*262*	Pb	ppm	*11.1*
Au	ppm	*< 0.002*	Hf	ppm	*2.63*	Re	ppm	*< 0.1*
Br	ppm	*< 0.5*	Hg	ppm	*< 1*	S	wt.%	*0.014*
C	wt.%	*0.032*	Ir	ppm	*< 0.005*	Se	ppm	*< 3*
Cl	ppm	*248*	LOI^{1000}	wt.%	*0.184*	SG	Unity	*2.93*

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction). Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of OREAS' in-house CRM-specific LIMS.

Table 3. Physical properties of AC18.10659.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Colour‡
601	0.56	N7	Light Gray

‡The Munsell Rock Colour Chart helps geologists and archaeologists communicate with colour more effectively by cross-referencing ISCC-NBS colour names with unique Munsell alpha-numeric colour notations for rock colour samples.

Commutability: AC18.10659 is sourced from naturally occurring rock and will display similar behaviour to routine 'field' samples in the relevant measurement process. Commutability is not an issue for this CRM.

Instructions for handling, correct use and safety: Fine powders pose a risk to eyes and lungs. The use of safety glasses and dust masks are advised. Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [13]. After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. AC18.10659 contains a non-hygroscopic matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 3 in this certificate. The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration.

Certified values and their associated 95 % expanded uncertainties are reported according to ISO/IEC Guide 98-3:2008 [6,16] and are shown in Table 1. These values are metrologically traceable to the international measurement scale (SI) of mass with major elements expressed in % (mass ratio) and minor elements expressed in mg/kg. In line with popular use, data are expressed as the mass fraction in either weight percent (wt.%) or parts per million, 1×10^{-6} (ppm). They are the means of accepted laboratory means after outlier filtering and are the present best estimate of the true value.

Indicative values shown in Table 2 are metrologically traceable to the international measurement scale (SI) of mass and are expressed in % (mass ratio) or mg/kg. In line with popular use, data are expressed as the mass fraction in either weight percent (wt.%) or parts per million, 1×10^{-6} (ppm). Indicative values are present where interlaboratory consensus is insufficient to meet OREAS' criteria for certification. AC18.10659 was also tested by OREAS for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

Sample Preparation and Analysis: AC18.10659 was crushed to a nominal 14 mm particle size then dried to constant mass at 105 °C. The dry material then underwent multi-stage milling to achieve a particle size of 100 % passing 30 µm. Homogenisation was accomplished using OREAS' novel processing technologies and the final product was packaged into 50 g units in glass jars sealed with plastic lids.

Ten commercial analytical laboratories participated in the program to certify the elements reported in Table 1. Full ICP-OES and MS elemental suites were analysed using a lithium borate fusion. These same laboratories also undertook lithium borate fusion with X-ray fluorescence. Instrumental neutron activation analysis (INAA) was also performed at one laboratory. The results generated by these quantitative analytical methods were pooled for certification purposes.

The INAA data was also used for homogeneity verification whereby 20 x 1 g subsamples were analysed at Actlabs, Ancaster in Canada. These data comprised an Analysis of Variance (**ANOVA**) using paired samples taken systematically from 10 different sampling intervals (representative of the prepared batch) and were randomised prior to assigning sample numbers. The duplicate samples enabled an ANOVA by comparison of within- and between-unit variances across the 10 pairs to test:

- Null Hypothesis, H_0: Between-unit variance is no greater than within-unit variance (reject H_0 if p-value < 0.05);
- Alternative Hypothesis, H_1: Between-unit variance is greater than within-unit variance.

The INAA data was not filtered for outliers prior to the calculation of p-values and no significant p-values were observed across the 35 reported elements except for Barium (Ba). However, this p-value is not considered technically significant given the poor repeatability across the 20 analyses (16.7% RSD) compared to the tightly constrained certification data via the fusion with XRF and ICP methods (3.15% RSD). The Null Hypothesis is accepted accordingly.

Document history:

Revision No.	Date	Changes applied
0	22nd November, 2024	First publication.

References

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[2] Ingamells, C. O. and Switzer, P. (1973). A Proposed Sampling Constant for Use in Geochemical Analysis, Talanta 20, 547-568.

[3] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.

[4] ISO Guide 33401:2024-01. Reference materials – Contents of certificates, labels and accompanying documentation.

[5] ISO Guide 33405:2024-05. Reference materials – Approaches for characterization and assessment of homogeneity and stability.

[6] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[7] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.

[8] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.

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[10] ISO 17034:2016. General requirements for the competence of reference material producers.

[11] Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), Minnesota (USA).
[12] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.
[13] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.
[14] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.
[15] Thompson, A.; Taylor, B.N. (2008); Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed 22 November 2024).
[16] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.

Appendix

The semi-quantitative XRD results for AC18.10659 shown in Table 4 below, were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results are normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. Some amorphous material may be present including traces of rutile.

Table 4. Indicative mineralogy based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Chlorite	< 1
Annite - biotite - phlogopite	11
Calcic amphibole	2
Clinopyroxene	10
Plagioclase	49
K-feldspar	13
Quartz	4
Calcite	< 1
Dolomite - ankerite	5
Magnesite and/or ilmenite	1
Apatite Group	1
Magnetite	4

Participating laboratories

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. American Assay Laboratories, Sparks, Nevada, USA
4. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
5. Intertek, Cupang, Muntinlupa, Philippines
6. Intertek, Perth, WA, Australia
7. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
8. SGS Canada Inc., Vancouver, BC, Canada
9. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
10. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan