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CERTIFICATE OF ANALYSIS FOR CERTIFIED REFERENCE MATERIAL

AC18.10661

Description: Andesite (ferroan enstatite), Carboniferous, Blair Duguid Inlier, New South Wales, Australia.

The material consists of rock sourced from an operating (Allandale) quarry located approx. 40 km northwest of the city of Newcastle in New South Wales, Australia. This light grey, Carboniferous rock forms a small, hydrothermally altered, remnant volcanic (Blair Duguid) inlier within gentle to moderately folded Permian shallow marine sediments of the Sydney Basin. The mineralogy consists of andesine plagioclase (44 %), vermiculite, smectite (37 %), quartz amygdales (13 %) and hypersthene clinopyroxene (3 %).

AC18.10661 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 AC18.10661 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	16.70	0.29
CaO	5.62	0.11
Fe_2O_3	6.14	0.11
K_2O	0.763	0.024
LOI^{1000}	4.90	0.83
MgO	3.27	0.06
MnO	0.0885	0.0028
Na_2O	4.27	0.07
P_2O_5	0.233	0.010
SiO_2	57.67	0.92
TiO_2	0.713	0.017

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	292	10
Be	0.98	0.24
Bi	< 2	IND
Cd	< 10	IND
Ce	32.8	2.2
Co	18.6	1.8
Cr	66	16
Cs	0.83	0.21
Cu	47.9	8.7
Dy	3.47	0.37
Er	1.99	0.25
Eu	1.11	0.11
Ga	18.1	1.8
Gd	3.92	0.41
Ge	1.02	0.13
Ho	0.656	0.086
In	< 0.2	IND
La	15.3	1.0
Li	15.9	1.9
Lu	0.285	0.047
Mo	< 5	IND
Nb	3.71	0.98
Nd	18.9	2.1
Ni	31.3	7.1
Pr	4.27	0.37
Rb	17.43	0.89
Sb	< 2	IND
Sc	15.5	1.2
Sm	4.06	0.50
Sr	634	40
Ta	< 5	IND
Tb	0.56	0.12
Te	< 1	IND
Th	1.81	0.20
Tl	< 0.5	IND
Tm	0.272	0.035
U	0.53	0.13
V	130	12
W	< 5	IND
Y	18.3	1.3
Yb	1.85	0.36
Zn	79.3	7.6
Zr	127	11

Table 2. Indicative Values for AC18.10661.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Ag	ppm	*< 5*	Hf	ppm	*3.34*	S	wt.%	*0.011*
Au	ppm	*< 0.002*	Hg	ppm	*< 1*	Se	ppm	*< 3*
Br	ppm	*< 0.5*	Ir	ppm	*< 0.005*	SG	Unity	*2.64*
C	wt.%	*0.130*	Pb	ppm	*10.3*	Sn	ppm	*< 200*
F	ppm	*238*	Re	ppm	*< 0.1*			

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 AC18.10661.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Colour‡
559	1.73	10Y 8/2	Pale Greenish Yellow

‡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: AC18.10661 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. AC18.10661 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. AC18.10661 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: AC18.10661 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.

Only the results for elements present in concentrations well above detection levels (i.e. >20 x Lower Limit of Detection) have been considered for the objective of evaluating homogeneity. 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 Fe. However, this p-value are not considered technically significant given the tightly constrained certification data via the fusion with XRF and ICP methods. 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.

[9] ISO 17025:2017, General requirements for the competence of testing and calibration laboratories.
[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 AC18.10661 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. 'Clay mineral' appears to be mainly vermiculite, smectite, and palygorskite. 'Kandite group' appears to be mainly kaolinite. Some amorphous material may be present including traces of zeolite.

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

Mineral / Mineral Group	% (mass ratio)
Clay mineral	37
Chlorite	< 1
Kandite group	1
Serpentine	< 1
Annite - biotite - phlogopite	1
Muscovite	< 1
Clinopyroxene	3
Plagioclase	44
Quartz	13
Calcite	< 1
Dolomite - ankerite	< 1
Apatite Group	1

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