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CERTIFICATE OF ANALYSIS FOR CERTIFIED REFERENCE MATERIAL

AC18.10665

Description: Black Mass Li-NMC batteries (NMC Type 622).

The material consists of a black mass of mixed metal concentrate sourced from processed lithium-ion batteries. The nickel-manganese-cobalt (NMC) ratios appear to represent 622 NMC Type lithium-ion batteries.

AC18.10665 is available as 40 g units packed into glass, wide-mouth jars.

Intended use: For use in evaluating classical wet chemistry and instrumental analytical methods for the chemical analysis of black mass, mixed metal concentrate samples.

Certified and indicative 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 AC18.10665 remains valid, within the specified measurement uncertainties, until May 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	4.57	0.09
C	54.21	0.87
Ca	0.241	0.022
Co	2.97	0.01
Cu	0.564	0.015
F	1.43	0.16
Fe	0.874	0.027
K	0.040	0.005
Li	1.81	0.02
Mg	0.107	0.008
Mn	2.84	0.02
Na	0.290	0.023
Ni	9.19	0.06
P	0.145	0.024
S	0.552	0.030
Si	0.755	0.050
Ti	0.028	0.003
Zr	0.114	0.018

Constituent (ppm)	Certified Value	95 % Expanded Uncertainty
Ba	15.1	2.0
Cd	2.37	0.48
Ce	1.66	0.50
Cl	240	108
Cr	23.4	3.9
Dy	0.17	0.05
In	0.18	0.05
La	1.13	0.23
Mo	1.21	0.48
Pr	0.21	0.06
Sn	1.17	0.52
Sr	15.7	1.5
Tb	< 0.05	IND
Tm	< 0.05	IND
V	7.80	0.95
W	98	22
Y	3.01	0.57
Zn	756	41

SI unit equivalents: ppm (parts per million; 1 x 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).

Table 2. Indicative Values for AC18.10665.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Ag	ppm	*2.69*	Ge	ppm	*0.55*	Sb	ppm	*0.22*
As	ppm	*16.2*	H_2O-	wt.%	*1.14*	Sc	ppm	*1.71*
B	ppm	*148*	Hf	ppm	*19.4*	Se	ppm	*5.72*
Be	ppm	*0.14*	Hg	ppm	*0.23*	Sm	ppm	*0.20*
Bi	ppm	*0.92*	Ho	ppm	*0.035*	Ta	ppm	*0.073*
C-(Graphite)	wt.%	*35.36*	Lu	ppm	*0.039*	Te	ppm	*0.30*
Cs	ppm	*0.30*	Nb	ppm	*0.92*	Th	ppm	*0.23*
Er	ppm	*0.11*	Nd	ppm	*0.83*	Tl	ppm	*0.047*
Eu	ppm	*0.050*	Pb	ppm	*35.6*	U	ppm	*0.18*
Ga	ppm	*5.96*	Rb	ppm	*2.81*	Yb	ppm	*0.10*
Gd	ppm	*0.20*	Re	ppm	*0.007*			

SI unit equivalents: ppm (parts per million; 1 x 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 AC18.10665.

Bulk Density (kg/m³)	Munsell Notation‡	Munsell Colour‡
451	N1	Black

‡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: AC18.10665 is sourced from black mass, mixed metal concentrate samples and will display similar behaviour to routine 'process plant' 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. AC18.10665 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 2 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 on a dry samples basis and determined 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 x 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 x 10^{-6} (ppm). Indicative values are present where interlaboratory consensus is insufficient to meet OREAS' criteria for certification. AC18.10665 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: The metal concentrate consisting AC18.10664 originated from 622 NMC Type lithium-ion batteries that were discharged, shredded, granulated, dried and sieved at 1 mm to separate the black mass from most of the copper and plastics. The 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 40 g units in glass jars sealed with plastic lids.

Thirty-three commercial analytical laboratories participated in the program to certify the elements reported in Table 1. The results generated by these quantitative analytical methods were pooled for certification purposes.

The data underpinning the key commodities (Lithium, Nickel, Manganese and Cobalt) was acquired from trade/speciality laboratories whereby Nickel was predominantly assayed by DMG. For most analytes, various analytical methods were undertaken for the characterisation including acid digestions with ICP-OES, ICP-MS and AAS finishes, lithium borate fusion with XRF or ICP-OES finishes or sodium peroxide fusion with ICP-OES/MS. Fluorine analysis was undertaken by ion specific electrode, microwave digest with ICP-MS finish, aqueous leach with ion chromatography, sodium peroxide fusion with ion chromatography or pressed powder pellet with XRF finish. Chlorine analysis was undertaken by ion specific electrode, aqueous leach with ion chromatography, sodium cyanide leach with ICP-OES, pressed powder pellet with XRF finish, sodium carbonate dissolution with gravimetric finish and volumetric titration.

For the round robin program twelve 200 g test units were taken during packaging and are considered representative of the entire prepared batch. The homogeneity of AC18.10664 has been evaluated in a nested Analysis of Variance (ANOVA) within twenty of the participating laboratories. Each of these laboratories received six samples and these samples were made up of paired samples from three different, nonadjacent sampling intervals and were randomised prior to assigning sample numbers. The purpose of ANOVA is to test that no statistically significant difference exists in the variance between units to that of the variance within units. This allowed an assessment of homogeneity across the entire prepared batch of AC18.10664. The test was performed using the following parameters:

- 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.

No significant p-values were observed across the 35 reported elements. The Null Hypothesis is accepted with no evidence of heterogeneity.

Document history:

Revision No.	Date	Changes applied
1	13th January, 2025	Minor revisions to the values in Tables 1 and 2 were made after incorporating results from two additional laboratories into the pool of certification data.
0	12th December, 2024	First publication.

References

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[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).

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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.

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[16] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.

Participating laboratories

1. AFRILAB laboratory, Marrakesh, Marrakesh-Safi, Morocco
2. AGAT Laboratories, Calgary, Alberta, Canada
3. AH Knight, Spartanburg, SC, USA
4. AH Knight, St Helens, Merseyside, UK
5. AH Knight, Tianjin, China
6. Alex Stewart International, Liverpool, UK
7. ALS, Brisbane, QLD, Australia
8. ALS, Loughrea, Galway, Ireland
9. ALS, Vancouver, BC, Canada
10. ALS Inspection, Prescot, Merseyside, UK
11. American Assay Laboratories, Sparks, Nevada, USA
12. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
13. Bachelet, Angleur, Liege, Belgium
14. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
15. ESAN Istanbul, Istanbul, Turkey
16. FILAB, Dijon, Burgundy, France
17. Inspectorate (BV), Shanghai, Bao Shan District, China
18. Inspectorate (BV), Witham, Essex, UK
19. Inspectorate Griffith India Pvt. Ltd., Bhubaneswar, Odisha, India
20. Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
21. MSALABS, Vancouver, BC, Canada
22. Paragon Geochemical Laboratories, Sparks, Nevada, USA
23. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
24. RC Inspection SA (PTY) LTD, Johannesburg, Gauteng, South Africa
25. RCI Analytical Services BV, Oosterhout, Netherlands
26. Rigaku Corporation, Osaka, Kansai region, Japan
27. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
28. SGS, Randfontein, Gauteng, South Africa
29. SGS Australia Mineral Services, Perth, WA, Australia
30. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
31. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
32. SGS Nederland B.V., Spijkenisse, Zuid-Holland, Netherlands
33. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
34. SRL, Perth, WA, Australia
35. UIS Analytical Services, Centurion, South Africa