Reference Material Certificate: Pulverised Bauxite

IMS-469

Table 1: IMS-469 Certified Values expressed as % w/w

Analyte	Certified Value (*y*)	Standard Deviation (*s*)		95% Confidence Interval (*CI*)		u_{CRM}^	$k^{\#}$	U_{CRM}~	No. of Labs	No. Samples
		1 SD	1 SD Within Lab	lower	upper					
Al_2O_3	47.19	0.247	0.103	46.99	47.39	0.11	2.31	0.26	8	39
SiO_2	2.26	0.041	0.025	2.23	2.29	0.021	2.31	0.049	8	39
Fe_2O_3	23.20	0.088	0.060	23.13	23.26	0.075	2.36	0.18	7	34
TiO_2	2.56	0.034	0.011	2.53	2.59	0.017	2.31	0.038	8	40
MnO	0.015	0.0044	0.0006	0.011	0.019	0.0019	2.31	0.0044	8	40
P_2O_5	0.091	0.0016	0.0008	0.090	0.093	0.0015	2.31	0.0035	8	38
SO_3	0.060	0.0074	0.0028	0.054	0.066	0.0038	2.31	0.0088	8	40
MgO	0.025	0.0088	0.0043	0.016	0.035	0.0049	2.45	0.012	6	27
K_2O	0.022	0.0039	0.0025	0.020	0.025	0.0039	2.31	0.0089	8	40
Na_2O	0.017	0.0084	0.0035	0.008	0.026	0.0055	2.45	0.013	6	26
Cr_2O_3	0.098	0.0038	0.0028	0.096	0.101	0.0018	2.31	0.0042	8	40
V_2O_5	0.129	0.0038	0.0011	0.125	0.132	0.0020	2.31	0.0047	8	40
ZrO_2	0.088	0.0101	0.0039	0.079	0.096	0.0062	2.31	0.014	8	40
LOI1000	24.33	0.171	0.045	24.18	24.48	0.089	2.31	0.20	8	40
Rx_SiO_2_LT	1.27	0.107	0.033	1.14	1.39	0.11	2.57	0.29	5	17
Tot_C	0.16	0.014	0.008	0.14	0.18	0.10	2.57	0.26	5	17
Org_C	0.11	0.036	0.024	0.07	0.15	0.10	2.57	0.27	5	17

Note 1. SI units equivalent: 1 ppm, parts per million ≡ grams per ton ≡ mg/kg ≡ ug/g ≡ 0.0001 wt.% ≡ 1000ppb, parts per billion
Note 2. The number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD.
^ Standard uncertainty.
# Coverage Factor.
~ Expanded Uncertainty.

Table 2: IMS-469 Informational Values expressed as % w/w

Analyte	Assigned Value (*y*)	Standard Deviation (s)		95% Confidence Interval (*CI*)		u_{CRM}^	$k^{\#}$	U_{CRM}~	No. of Labs	No. Samples
		1 SD	1 SD Within Lab	lower	upper					
CaO	0.013	0.0046		0.000	0.056	0.0035	4.3	0.015	2	8
BaO	0.007	0.0032		0.000	0.045	0.0032	4.3	0.014	2	8
Av_Al_2O_3_LT	41.39	1.084	0.658	39.76	43.02	0.84	2.78	2.3	4	12
Av_Al_2O_3_HT	42.80	0.654	0.401	41.14	44.46	0.57	3.18	1.8	3	11
Rx_SiO_2_HT	1.95	0.110	0.043	1.67	2.24	0.13	3.18	0.41	3	11

Material and Method of Preparation

IMS-469 is manufactured from a pulverised bauxite material. Following drying at 105°C, crushing and screening, this reference material was prepared by multi-stage pulverisation and homogenisation. During the discharge from the last blending stage, samples were drawn for homogeneity and characterisation studies. The samples taken were randomised before being submitted to independent ISO/IEC 17025 accredited laboratories for as part of homogeneity and inter-laboratory round-robin testing for material characterisation, with number of laboratories and samples summarised in Table 3.

Table 3: Batch Specific Parameters

Production Summary		
	Material Source	West African Bauxite Ores
	Material Description	High Grade Bauxite Ore
	Drying Temperature (°C)	105
	Production screen size (µm)	53
Characterisation Study – Un-normalised fusion XRF		
	No. of Laboratories	8
	No. samples per laboratory	5
	No. determinations per sample	1
	Mass per sample submitted (g)	10
Characterisation Study – Low Temp, High Temp Available Alumina, Reactive Silica		
	No. of Laboratories	5
	No. samples per laboratory	3
	No. determinations per sample	1
	Mass per sample submitted (g)	20
Homogeneity Study – Un-normalised XRF		
	No. of Laboratories	1
	No. samples per laboratory	18
	No. determinations per sample	2
	Mass per sample submitted (g)	10

Homogeneity Analysis

A homogeneity study was undertaken in accordance with ISO 33405:2024 and ISO 17034:2016 using systematically selected samples to be representative of the entire batch. The sample identifiers were randomised to ensure different production order and laboratory analytical order. These samples were submitted to a single laboratory for multiple analysis in a single batch under repeatable conditions. The homogeneity study results were reviewed to ensure all analytes were deemed homogeneous prior to the material progressing to the inter-laboratory round-robin stage for characterisation assessment. A summary of the study is presented in Table 3, with results summarised in Table 4.

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Table 4: Homogeneity Study Results

Analyte	Avg. Concentration Homogeneity Lab (% w/w)	SD between samples median value	Relative SD %
Al_2O_3	47.48	0.054	0.1
SiO_2	2.21	0.012	0.5
Fe_2O_3	23.10	0.055	0.2
TiO_2	2.54	0.007	0.3
MnO	0.020	0	0
P_2O_5	0.093	0.0006	0.6
SO_3	0.059	0.0023	3.9
MgO	0.019	0.0019	10
K_2O	0.021	0.0029	13.4
Na_2O	0.013	0.0040	31.6
Cr_2O_3	0.098	0.0008	0.8
V_2O_5	0.128	0.0008	0.6
ZrO_2	0.086	0.0040	4.7
LOI1000	24.11	0.060	0.3

Measurement Techniques used for Certification

Reactive Silica, Available Alumina

The sample (usually 1g) is digested in stainless steel vessels at either 148°C (Low Temperature) or 240°C (High Temperature) with a sodium hydroxide solution at a specified concentration. An aliquot is taken and analysed for available alumina by ICP-AES or MP-AES, expressed as Al_2O_3.

The remaining supernatant liquor is acidified with hydrochloric acid, then analysed for reactive silica by ICP-AES or MP-AES, expressed as SiO_2.

Leco for Total Carbon, Organic Carbon

Laboratories were requested to analysis total-carbon and organic-carbon via a LECO method. The method selected (acid treatment or temperature differential) was not specified in the instructions to laboratories.

Material Characterisation and Certification Methodology

The process of characterisation was undertaken in accordance with ISO 33405:2024 and ISO 17034:2016 following examination of grouped laboratory results for potential technical failures by way of comparison with the established CRM submitted for analysis with the candidate material. Where required, further investigation of outliers was conducted. Laboratory results deemed technical outliers were removed from the analysis pool prior to the determination of statistical parameters. The certifying officer, in some cases, may use their judgment in identifying or eliminating outliers outside of these statistical parameters.

- Certified value was determined by average of lab averages for analytes with no outlier laboratory results, or median of median for those with outlier laboratory results.
- Standard deviation (s) is the measure of spread of analyte determinations and includes inter-laboratory bias, method uncertainty, and material homogeneity uncertainty. Approximately 95% of determinations using the same analytical method are expected to be between two standard deviations either side of the certified value. The standard deviation is calculated from the validated laboratory group data less outlier laboratory and individual determinations.
- Within laboratory standard deviation (s_w) is the average spread of determination values across the reporting laboratories, less outlier laboratory and individual determinations. This is calculated by single factor ANOVA of the participating laboratory groups.
- Confidence Interval (*CI*) is an estimate of the true (unknowable) analyte concentration in the material at the 95% confidence interval. For example, a 95% *CI* could be interpreted as there is a 0.95 probability that the true value is between certified value ± *CI*. The narrower the interval, the more precise the certified value. The 95% *CI* should not be used for determination of quality control gates.
- Standard Uncertainty (u_{CRM}) is the sum of variance from characterisation, homogeneity and stability studies. The uncertainty of characterisation is derived from the standard deviation of average of laboratory averages divided by the square root of the number of laboratories. Uncertainty of material homogeneity (u_{hom}) is the sum of ANOVA within and between sample uncertainty derived from the homogeneity study. An allowance for stability has been included in accordance with ISO 33405:2024.
- Coverage Factor (k) is the students t-distribution value for two tailed test at 95%.
- Expanded Uncertainty (U_{CRM}) is the product of coverage factor and standard uncertainty, and represents the 95% confidence interval of the true unknowable analyte concentration of the batch combined with the bias from individual samples.

The certified value of any analyte may not be less than 0.00 % even though in some cases the uncertainty error bounds define a range outside of 0.00 %. These cases are due to low concentrations of some analytes relative to the analytical detection limits and increments of precision.

Analytes have been categorised as certified or informational based on:

- Confirmation of sufficient between-unit variance demonstrating material homogeneity.
- Minimum number of participating laboratories for that particular analyte.
- Sufficient agreement between participating laboratories.

Commutability

This pulverised reference material is not commutable to any other analytical methods than as stated by its intended use.

Metrological Traceability

Metrological traceability of the certified and informational values and their uncertainties listed in Table 1 and 2 have not been established, as values from laboratories and methods not accredited to ISO 17025 have been included in the characterisation studies.

Participating Laboratories

Laboratories used in the certification process are listed in Table 5 in alphabetical order, along with batch number.

Table 5: IMS-469 Participating Laboratories

Laboratory Name	Location	Job #
ALS Geochemistry	Stafford, Queensland	BR24178522
ALS Geochemistry	Loughrea, Ireland	LR24176473
ALS Minerals	Malaga, Western Australia	PH24168211
Bureau Veritas Minerals	Canning Vale, Western Australia	u360399
Bureau Veritas Minerals	Wingfield, South Australia	aa066781
Intertek Genalysis	Maddington, Western Australia	1771.0/2410019
Nagrom	Kelmscott, Western Australia	KM-2407-073513
SGS Geochemistry	Perth Airport, Western Australia	PER24-15866

Preparer and Supplier of Certified Reference material

The bauxite reference material IMS-469 was prepared and certified by:

Independent Mineral Standards Pty Ltd
16 Durham Rd
Bayswater, WA 6053
Australia
Ph: +61 8 6155 7616
imstandards.com.au

The material has been supplied in 10g heat sealed aluminised plastic bags and also 1kg press-seal plastic jars.

Intended Use

This pulverised reference material is intended for monitoring and testing the accuracy and precision of XRF, TGA, and low temperature and high temperature caustic bomb digest. Note that the caustic digest is partial, and digest parameters have significant impact on reported analyte values.

The estimate of material and measurement uncertainties reported in this certificate are the product of the participating laboratories, not any individual laboratory. Commercial laboratories typically have different measurement uncertainties to site-based laboratories. Application of the grouped uncertainties reported in this certificate to a specific laboratory for ongoing QC may lead to many false reports of out-of-control processes, or alternatively non reporting of out-of-control processes.

It is recommended that the centre line and control limits of a Shewhart chart used for ongoing monitoring of a particular laboratory are derived from averaged values and variation from replicate analysis of this CRM after removal of outliers.

Instructions for Correct Use

The recommended values for this CRM refer to the concentration levels after removal of hygroscopic moisture by drying in air to constant mass at 105°C. If the reference material is not dried prior to analysis, the recommended value should be corrected to the moisture bearing basis.

Minimum Sample Mass

This reference material has been certified using 0.2g to 0.5g aliquots for Li-B fusion XRF analysis. Uncertainty and homogeneity statements relating to this are only applicable if a minimum of 0.2g sample mass is used during Li-B fusion XRF analysis.

Period of Validity

This Certificate is valid 10 years from the date of original issue.

Stability and Storage Instructions

This CRM is an oxidised reference material and is stable in the sealed aluminised plastic bags, or press-sealed plastic jars under normal conditions of storage. It is recommended to keep jars sealed at all times when not in use.

Legal Notice

Independent Mineral Standards Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of ability. The purchaser by receipt hereof releases and indemnifies Independent Mineral Standards Pty Ltd from and against all liability and costs from the use of this material and information.

Certifying Officer

Bruce Armstrong

Certification Date

28th February 2025.

References

ISO 33405:2024, Reference materials — Approaches for characterization and assessment of homogeneity and stability.

ISO 17034:2016, General Requirements for the competence of reference material producers.

Version History

Batch	Author	Document Version	Date	Modifications
IMS-469	Harry Ooi	R0	28/02/2025	Initial Document