

Reference Material Certificate: Alpha-Alumina Powder IMS-135

Table 1: IMS-135 Certified Values

Analyte	unit	Certified Value (y)	Standard Deviation (s)	U_{CRM}^{\wedge}	$k^{\#}$	U_{CRM}^{\sim}	No. of Labs	No. Samples
Alpha-Alumina	% w/w	99.04	0.79	0.58	2	1.16	1	10

Note 1. SI units equivalent: 1 ppm, parts per million \equiv grams per ton \equiv mg/kg \equiv ug/g \equiv 0.0001 %w/w \equiv 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.

\wedge Standard uncertainty.

$\#$ Coverage Factor.

\sim Expanded Uncertainty.

Table 2: IMS-135 Indicative Values

Analyte	XRF Value (% w/w)	Analyte	LA-MS Value (mg/kg)	Analyte	LA-MS Value (mg/kg)	Analyte	LA-MS Value (mg/kg)
SiO ₂	0.06	Ag	<0.1	Hf	0.02	Sm	0.01
Al ₂ O ₃	99.863	As	<0.2	Ho	<0.01	Sn	<0.2
Fe ₂ O ₃	<0.01	Ba	<0.5	In	<0.05	Sr	<0.1
CaO	<0.01	Be	<0.2	La	<0.01	Ta	0.02
MgO	<0.01	Bi	<0.02	Lu	<0.01	Tb	<0.01
MnO	<0.001	Cd	<0.1	Mn	<1	Te	<0.2
TiO ₂	0.005	Ce	0.02	Mo	0.2	Th	<0.01
SO ₃	0.002	Co	0.2	Nb	<0.01	Ti	1
P ₂ O ₅	0.001	Cr	1	Nd	0.02	Tl	<0.2
K ₂ O	<0.001	Cs	0.01	Ni	<2	Tm	<0.01
Na ₂ O	<0.01	Cu	<2	Pb	<1	U	<0.01
LOI1000	0.15	Dy	0.01	Pr	<0.01	V	0.2
		Er	<0.01	Rb	<0.05	W	<0.5
		Eu	<0.01	Re	<0.01	Y	<0.02
		Ga	10.3	Sb	<0.1	Yb	<0.01
		Gd	<0.01	Sc	<0.1	Zn	<5
		Ge	<0.05	Se	<5	Zr	<0.5

Material and Method of Preparation

IMS-135 is a manufactured high purity alumina powder (corundum structure), calcined and milled to a d95 of 2.5µm.

Material Characterisation and Certification Methodology

Ten sub-samples were sent for Quantitative X-ray Diffraction (QXRD) analysis at a single laboratory for value transfer from NIST SRM676a. Each sample was analysed in duplicate. These results are reported in Table 1 as certified values.

The X-ray Diffraction pattern was measured using a PANalytical Cubix³ diffractometer with copper radiation operating at 45kV and 40mA and graphite monochromator inserted in the diffracted beam. The collection parameters are listed in Table 3.

Table 3: Instrument parameters

Parameter	Setting
Start angle (deg 2θ)	4
End angle (deg 2θ)	80
Step size (deg 2θ)	0.02
Time/ active length (s)	150
Active length (deg 2θ)	4.01

The crystalline alumina phase content was determined using Rietveld Refinement (TOPAS V5.0 and ICSD 2021 Database), with NIST SRM676a as an external standard.

The certification methodology is as described below.

- Certified value (y) was determined by the average difference in crystalline alumina phase content between all refinements of the established CRM (NIST SRM676a) and the candidate material (IMS-135).
- Standard deviation (s) is the measure of spread of analyte determinations and 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 total of duplicate determinations of 10 samples, a total of 20 determinations.
- Standard Uncertainty (u_{CRM}) is the square root of the sum of the established CRM uncertainty and the standard error of the mean of the difference between established and candidate CRM. This is in accordance with ISO Guide 35.
- Coverage Factor (k) is the students t-distribution value for two tailed test at 95%. This is approximated as 2 for this study.
- 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.

An additional four sub-samples were analysed at a single laboratory via lithium-borate fusion with x-ray fluorescence spectrometry (XRF) for whole rock analysis, with subsequent Laser-

Ablation ICP-MS for trace analyte analysis on the same fusion beads. These results are reported in Table 2 for indicative purposes only.

Participating laboratories

Samples were sent to 2 participating laboratories which are listed in Table , along with analysis method.

Table 4: Participating Laboratories

Laboratory Name	Location	Analysis method
Intertek	Maddington, Australia	QXRD
Bureau Veritas	Canning Vale, Australia	XRF, LA-ICP-MS

Preparer and Supplier of Certified Reference Material

This certified reference material, IMS-135, 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 is available in sealed 250g PET jars, with unique labels showing the batch number.

Minimum Sample Mass

This reference material has been certified using 3-5g aliquots for QXRD. Uncertainty and homogeneity statements relating to this are only applicable if a minimum of 3g sample mass is used.

Intended Use

The pulverised reference material is intended for inclusion as a standard addition, or an external standard during QXRD analysis.

Period of Validity

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

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 assigned values and their uncertainties has been established by transfer from NIST SRM676a.

Stability and Storage Instructions

Jars should be stored in a cool dry location, and mixed by shaking the sealed container before opening for first use. Once opened it is recommended to re-seal opened jars when not in use. All jars have been labelled with a recommended use by date. The long-term storage of this product is monitored, and purchasers will be notified if changes are observed during the period of validity of the product.

Instructions for Correct Use

The certified values derived from QXRD and analysis is based on the concentration level in the packaged state, and no further drying is required before weighing and analysis.

Legal Notice

Independent Mineral Standards Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its 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, Operations Manager

Certification Date

20th July 2022

References

ISO Guide 35:2017, Reference materials – General and statistical principles for certification.

ISO17034:2016, General Requirements for the competence of reference material producers.

Version History

Batch #	Document Version	Date	Modification
IMS-135	IMS135_Certificate_R0	20/07/2022	Initial Document
IMS-135	IMS135_Certificate_R1	27/07/2022	Text edits