Reference Material Certificate
Quantitative XRD
Pulverised Iron Ore
IMS-103

Table 1: IMS-103 QXRD analysis including amorphous content, Certified Values expressed as % w/w dry basis

Analyte	Certified Value (y)	Standard Deviation		95% Confidence Interval (CI)		u_{CRM}^	k#	U_{CRM}~	No. of Labs	No. Samples
		1 SD (s)	1 SD Within Lab (s_w)	lower	upper					
Amorphous	13.1	3.26	0.77	11.2	14.9	1.1	2	2.3	14	32
Goethite	40.7	1.91	1.03	39.6	41.8	1.1	2	2.3	13	30
Hematite	40.2	2.30	0.91	38.9	41.5	1.1	2	2.2	14	32
Quartz	2.6	0.29	0.10	2.4	2.8	0.13	2	0.26	12	28
Kaolinite	2.0	0.85	0.36	1.5	2.5	0.43	2	0.86	13	26
Cryptomelane	0.8	0.19	0.02	0.7	1.0	0.073	2.31	0.17	8	17

Table 2: IMS-103 QXRD analysis normalised to no amorphous content; Certified Values expressed as %w/w dry basis

Analyte	Certified Value (y)	Standard Deviation		95% Confidence Interval (CI)		u_{CRM}^	k#	U_{CRM}~	No. of Labs	No. Samples
		1 SD (s)	1 SD Within Lab (s_w)	lower	upper					
Goethite	47.2	1.73	1.09	46.4	48.1	1.2	2	2.3	17	38
Hematite	45.8	1.42	0.86	45.1	46.5	0.93	2	1.9	15	34
Quartz	3.0	0.28	0.13	2.8	3.2	0.15	2	0.30	12	28
Kaolinite	2.4	1.00	0.41	1.8	3.0	0.49	2	0.99	14	27
Cryptomelane	0.9	0.21	0.03	0.7	1.1	0.083	2.31	0.19	8	18

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

Version History

Batch	Author	Document Version	Date	Modifications
IMS-103	Harry Ooi	R0	13/10/2023	Initial Document
IMS-103	Harry Ooi	R1	3/11/2023	Review classification Magnetite. Text edits

Table 3: IMS-103 QXRD analysis including amorphous content, Informational Values expressed as % w/w dry basis

Analyte	Assigned Value (y)	Standard Deviation		95% Confidence Interval (CI)		u_{CRM}^	k#	U_{CRM}~	No. of Labs	No. Samples
		1 SD (s)	1 SD Within Lab (s_w)	lower	upper					
Magnetite	0.3	0.18	0.12	0.1	0.6	0.15	2.78	0.41	4	10
Anatase	0.26	0.129	0.012	0	0.5	0.071	2.78	0.2	4	8
Gibbsite	0.2	0.10		0	1.1	0.075	4.3	0.32	2	4

Table 4: IMS-103 QXRD analysis normalised to no amorphous content, Informational Values expressed as %w/w dry basis

Analyte	Assigned Value (y)	Standard Deviation		95% Confidence Interval (CI)		u_{CRM}^	k#	U_{CRM}~	No. of Labs	No. Samples
		1 SD (s)	1 SD Within Lab (s_w)	lower	upper					
Magnetite	0.4	0.21	0.14	0.1	0.6	0.16	2.57	0.41	5	12
Anatase	0.3	0.15	0.015	0	0.6	0.082	2.78	0.23	4	8
Gibbsite	0.2	0.11		0	1.3	0.084	4.3	0.36	2	4

Table 5: IMS-103 XRF analysis, Indicative oxide concentrations expressed as % w/w dry basis

Analyte	Units	Indicative Value
Fe_2O_3	% w/w	80.2
SiO_2	% w/w	7.1
LOI	% w/w	6.9
Al_2O_3	% w/w	3.1
MnO	% w/w	1.7
TiO_2	% w/w	0.2
P_2O_5	% w/w	0.14
MgO	% w/w	0.09
SO_3	% w/w	0.08
K_2O	% w/w	0.07

Analyte	Units	Indicative Value
CaO	% w/w	0.06
Na_2O	% w/w	0.05
BaO	% w/w	0.02
Cl	% w/w	0.02
SrO	% w/w	0.03
V_2O_5	% w/w	0.02
As_2O_3	% w/w	0.01
ZnO	% w/w	0.01
ZrO_2	% w/w	0.01
Sum	% w/w	99.8

Table 6: Indicative analyte chemical formulae

Analyte	Formulae
Goethite	FeOOH
Hematite	Fe_2O_3
Quartz	SiO_2
Kaolinite	$Al_2Si_2O_5(OH)_4$
Amorphous	-

Analyte	Formulae
Cryptomelane	KMn_8O_{16}
Anatase	TiO_2
Magnetite	Fe_3O_4
Gibbsite	$Al(OH)_3$

Introduction

This document specifies preparation, analysis, and certification of a pulverised mineral reference material certified for mineralogy via quantitative X-ray Diffraction (XRD) analysis via a multiple laboratory round-robin.

Material and Method of Preparation

The material is a blend of hematite-goethite iron ores sourced from the Pilbara region, Western Australia.

Following drying at 105 °C, crushing and screening, this reference material was prepared by multi-stage pulverisation and multi-stage air classified prior to homogenisation. During the discharge from the homogenisation stage, samples were drawn for the characterisation study. The samples taken were randomised before being submitted to independent laboratories for the inter-laboratory round-robin testing for material characterisation. Material was packaged into 5 g heat-sealed aluminised sachets.

Table 7: Batch Specific Parameters

Production Summary		
	Material Source	Various sources, Pilbara Region, Western Australia
	Material Description	Single material composited from multiple iron ores
	Packaged Sample Mass (g)	5
	Drying Temperature (°C)	105
	Screening size (µm)	p50 @ 4 µm, p80 @ 6 µm, p90 @ 8 µm
Characterisation Study		
	No. of Laboratories	18
	No. samples per laboratory	2
	No. determinations per sample	1

Material Characterisation

Participating laboratories were instructed sample preparation was not required, as the material has been pulverised to a suitable particle size distribution.

After homogenisation, three 20 g samples were provided to a single laboratory for single analysis via both fusion XRF and Quantitative XRD analysis to provide elemental and mineralogy concentration guidance to participating laboratories. The XRF analysis results are presented in Table 5.

Laboratories were invited to participate in the round robin based on prior experience in mineral analysis. Laboratories were provided with instructions specifying approximate mineral and elemental concentrations to aid in analysis and interpretation.

Laboratories were requested to perform quantitative XRD including amorphous content after drying at 45°C. Analysis results including amorphous content were reported by 15 laboratories, with three laboratories reporting results on crystalline content only basis. Those results including amorphous content have been normalised to crystalline only content basis, providing reference for both analysis scenarios.

Analysis parameters were at the discretion of each participant and are summarised in Table 9. Some laboratories have made multiple analysis per sample using differing analytical parameters or equipment in which case some have been treated as separate laboratories during data processing.

The participating laboratories analysis parameters are summarised in Table 8 and Table 9.

Table 8: Summary of x-ray tubes and Standard methods

x-ray tube type	Count		Internal / External Standard Method	Count
Cobalt	15		External	8
Copper	1		Internal	5
Not Recorded	2		Not Recorded	5

Table 9: Summary of analysis parameters

	Data start angle (°2θ)	Data end angle (°2θ)	step size (°2θ)	total collection time (minutes)	time step (s)
Count Recorded	17	17	15	12	6
Min.	2	55.15	0.01	29.25	0.3
Max.	12	145	0.05	1000	7
Median	5	90	0.02	40.00	0.63

NR = Not Reported

Certification Methodology

The process of characterisation was undertaken in accordance with ISO Guide 35:2017 and ISO17034:2016 following examination of grouped laboratory results for potential technical failures. 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 laboratory averages for analytes with no outlier laboratory results, or median of laboratory medians 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

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 Guide 35.

- 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 concentration may not be negative, even though in some cases the uncertainty error bounds define a range less than 0%. 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.

Intended Use

This pulverised reference material is intended for monitoring and testing the accuracy and precision of quantitative XRD analysis of iron ores.

The estimate of material and measurement uncertainties reported in this certificate are the product of the participating laboratories, not any individual laboratory. Each laboratory has unique measurement uncertainties. 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.

Participating Laboratories

Laboratories used in the certification process are listed in Table 10 in alphabetical order.

Table 10: Participating Laboratories

Laboratory Name	Location
ALS Global	Malaga, Western Australia
Brisbane Met Lab	Coopers Plains, Queensland
Bruker AXS GmbH	Karlsruhe, Germany
Bruker Singapore	Singapore
Bureau Veritas	Wingfield, South Australia
CSIRO Mineral Resources	Urrbrae, South Australia
Institute of Geology	Czech Republic
International Centre for Diffraction Data, ICDD	Newton Square, PA, USA
Intertek Genalysis	Maddington, Western Australia
Malvern Panalytical	Almelo, The Netherlands
MicroAnalysis Australia	Mount Lawley, Western Australia
Queensland University of Technology (QUT)	Brisbane, Queensland
Rigaku	Shibuya-ku, Tokyo, Japan
SGS Burnaby	Burnaby, British Colombia, Canada
SGS Chile	Santiago, Chile
SGS Lakefield	Lakefield, Ontario, Canada
SGS South Africa	Bryanston, South Africa

Preparer and supplier of reference material

This reference material has been prepared and is certified by:

Independent Mineral Standards Pty Ltd
16-18 Durham Rd
Bayswater, WA 6053
Australia

www.imstandards.com.au

Period of Validity

This Certificate is valid 10 years from the date of original issue. Monitoring of stability will be undertaken to during the term of validity.

Commutability

This 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 to Table 4 has not been established as values from laboratories and methods not accredited to ISO 17025 have been included in the characterisation study.

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 recommended values for this CRM refer to the concentration levels after removal of hygroscopic moisture by drying in air to constant mass at 45°C. If the reference material is not dried prior to analysis, the recommended value should be corrected to the moisture bearing basis. Sample preparation is not required, as the material has been pulverised to a suitable particle size distribution.

Legal notice

Independent Mineral Standards Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of our 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

3rd November 2023.

References

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

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

Appendices

Appendix:1: Graphical Analysis of Results Including Amorphous Content

Appendix 2: Graphical Analysis of Results Normalised to No Amorphous Content