Iron Ore Certified Reference Material: Certificate of Analysis

PBS-209

Table 1: PBS-209 Certified Values

Analyte	unit	Value	Standard Deviation		95% Conf. Int.		Coeff. Of Var.	Number of Labs	Number of Analysis
			1 SD	1 SD Within Lab	lower	upper			
Fe	%	58.31	0.168	0.109	58.21	58.41	0.29	10	59
SiO2	%	4.83	0.062	0.039	4.79	4.86	1.28	10	59
Al2O3	%	2.57	0.042	0.018	2.54	2.6	1.65	10	60
TiO2	%	0.173	0.0085	0.0028	0.167	0.179	4.9	10	60
Mn	%	0.180	0.0048	0.0045	0.178	0.182	2.68	9	53
CaO	%	0.145	0.0091	0.0052	0.139	0.151	6.3	10	60
P	%	0.046	0.0010	0.0007	0.045	0.046	2.21	10	59
S	%	0.029	0.0015	0.0012	0.029	0.031	5.03	8	48
K2O	%	0.020	0.0008	0.0007	0.019	0.02	3.9	7	42
LOI371	%	7.23	0.088	0.032	7.16	7.3	1.22	8	48
LOI425	%	7.48	0.108	0.027	7.37	7.58	1.44	7	42
LOI650	%	8.02	0.069	0.036	7.97	8.08	0.86	8	48
LOI1000	%	8.29	0.051	0.036	8.26	8.32	0.62	9	54
LOI371 to 425	%	0.25	0.023	0.011	0.227	0.27	9.42	7	41

Table 2: PBS-209 Provisional Values

Analyte	unit	Value	Standard Deviation		95% Conf. Int.		Coeff. Of Var.	No. of Labs	No. of Analysis
			1 SD	1 SD Within Lab	lower	upper			
Cl	%	0.009	0.0010	0.0008	0.008	0.009	12.26	6	35
V	%	0.005	0.0010	0.0005	0.004	0.005	21.71	6	36
MgO	%	0.087	0.0134	0.0098	0.08	0.095	15.31	10	59
LOI425 to 650	%	0.54	0.057	0.024	0.485	0.59	10.64	7	42
LOI650 to 1000	%	0.26	0.057	0.016	0.215	0.313	21.57	8	48

Table 3: PBS-209 Informational Values

Analyte	unit	Value	Number of Labs	Number of Analysis
As	%	0.003	5	30
Ba	%	0.004	5	26
Co	%	0.004	4	24
Cr	%	0.004	4	22
Cu	%	0.003	5	28
Na2O	%	0.019	8	44
Ni	%	0.002	3	17
Pb	%	0.005	2	12
Sn	%	0.006	1	6
Sr	%	0.003	3	15
Zn	%	0.003	6	36
Zr	%	0.004	5	30

Table 4: Batch Specific Parameters

IMS Job #	PBS-209	Client CRM ID	N/A
Client Details			
Client Contact Name	IMS	IMS Quote # Client PO#	
Material Details			
Source Site Material Description Blending Instructions	Pilbara Region Composite of various iron ores. Medium Grade		
Production Details			
Sample mass (kg) Number Samples Total Mass (kg)	2 1728 3456	Screen size (mm) p95 (if spec'd)	5 N/A
Certification Details			
No Samples Samples / Lab Method Method	60 6 XRF Lithium-Borate fusion. Un-normalised TGA	No Labs Analytes Analytes	10 Fe, SiO2, Al2O3, TiO2, Mn, CaO, P, S, MgO, K2O, Na2O, Zn, As, Cl, Cu, Pb, LOI @ 371C, 425C, 650C, 1000C
Special Instructions			
Analysis Packaging Labelling Delivery Addresses Transport	12 x 2 Homogeneity samples Explorer bags, sealed drums, palletised heat printed TBA		

Version Control				
Doc. Version	Analysis Version	Author	Date	Changes
PBS-209_R1	PBS-209_R0	B. Armstrong	24/03/2022	Initial document
PBS-209_R2	As above. No change to values	B. Armstrong	18/08/2023	Updated certificate batch numbering format from PBS209 to PBS-209

Introduction

This document specifies preparation, analysis, and certification of custom coarse reference material.

Method of Preparation

The material preparation parameters specific to this batch are detailed in Table 4, with generic coarse reference material preparation outlined below.

All material was oven dried at 105C for about 24 hours, and multi-stage crushed and screened to pass determined sieve size.

To ensure complete blending and homogenisation, all material was passed through a multi-stage rotary sample division system to arrive at the final division with the determined number of samples and weights.

The final samples were bagged into heat sealed bags, each uniquely numbered with the batch number, order of division and sample number.

During the bagging process the determined number of samples were randomly selected for use in the certification process and marked prior to bagging. These samples were removed from the sample bagging stream during the bagging and sealing process. The selection of laboratory for each sample was again randomised, prior to the samples being boxed and sent to the respective laboratories.

The participating laboratories were each given samples for drying (105°C), sample preparation via pulverisation, and analysis via the techniques outlined in Table 4.

Method of Certification

Outlier laboratory and individual analytical results are removed from the informing sample population to remove erroneous values. The process used is:

- Remove below detection values which are imported as negative values. In addition, if laboratory groups of data contain 50% or more below detection values the entire laboratory group of results is discarded from subsequent analysis.
- Remove laboratory groups with median value evaluated by modified Z-scores of >3.0, using method of Iglewicz and Hoaglin (1993).
- Remove laboratory group data with excessive range which demonstrates out of control processes. This is calculated as laboratory group results with interquartile ranges with modified Z-scores of >6.
- Individual outliers with Z-score >3 are then removed from the informing population when confirmed using a α=0.01 on a two-tailed Grubbs test on the grouped data.

The above process is reviewed by the Certifying Officer, and in some cases will use their judgment in identifying or eliminating outliers outside of the above parameters.

Results have been grouped in Certified, Provisional, and Informational on the below general criteria:

- Certified values show good agreement with a low (<10%) coefficient of variation (CoV = Std. Deviation / Mean), a measure of the variability relative to the mean.
- Provisional are CoV 10% to 20%, or with significant disagreement between laboratories which cannot be resolved using statistical review techniques alone.

- Informational values are typically near the detection limit for the analysis. As such conventional standard deviation and confidence intervals are not appropriate controls. In these cases, it is likely that more appropriate analysis techniques are required for the analyte concentrations.

The results are displayed in Table 1 to Table 3

The Certified value is calculated from the mean of laboratory means; Standard Deviation is calculated as the standard deviation of all results. Within Laboratory Standard Deviation is calculated from ANOVA of the laboratory grouped results. Between Laboratory Standard Deviation when quoted is calculated according to ISO Guide 35, section B.6.

Confidence Interval is derived at the α=0.05 from the Students t-distribution for the number of participating laboratories, and the standard deviation of the laboratory means. The confidence interval is a measure of the reliability of the consensus value. In this case, it is a measure of the reliability of the certified value. For example, a 95% CI for Fe could be interpreted as there is a 0.95 probability that the certified value is between (mean ± CI). The narrower the interval, the more precise the certified value. A 95% CI is distinct from the lower limit and upper limit at 2SD which provides an estimate of the range of values for 95% of individual measurements for a given analyte. In the case of Fe, approximately 95% of replicates are expected to be between two SDs either side of the certified value.

The above calculations are in accordance with ISO 11459 and ISO Guide 35.

Consensus Values

Summary data of assigned values, standard deviations and confidence intervals, and number of laboratories and analysis used in calculating the values are shown in PBS-209

Table 1 for Certified Values, Table 2 for Provisional Values, and Table 3 for Informational Values.

Participating laboratories

Alphabetical order of laboratories used in the certification process are listed in Table 5, along with batch number.

Table 5: PBS-209 Participating Laboratories

Laboratory Name	Location	Job #
ALS Brisbane	Queensland, Australia	BR22027912
ALS Malaga	Western Australia	PH22018621
BV Canning Vale	Western Australia	u332078
BV Whyalla	South Australia	wh077025
BV Wingfield	South Australia	aa053592
Intertek Genalysis Maddington	Western Australia	1771_0_2201729
Nagrom Kelmscott	Western Australia	KM-2201-060285
PT Intertek Utama Services	Jakarta, Indonesia	220435
SGS Newburn	Western Australia	WM210791
Spectrolab	Western Australia	PBS209

Preparer and supplier of reference material

The iron ore reference material PBS-209 has been prepared and certified, and is certified by:

Independent Mineral Standards Pty Ltd
16 Durham Rd
Bayswater, WA 6053
Australia
www.imstandards.com.au

The material has been supplied in 2kg heat sealed plastic bags with unique labels showing batch number, order of division and sample number.

Intended use

The coarse iron ore reference material will be used to quantify a laboratory's ability to prepare, sub-sample and analyse coarse samples. Pulp iron ore reference materials are used to determine the laboratory's ability to analyse only (removing the preparation step). The combination of coarse and pulp iron ore reference materials permits the quantification of errors at the laboratory.

Stability and storage instructions

This CRM is an oxidised reference material and is stable in the sealed plastic bags under normal conditions of storage.

Instructions for the correct use of the reference material

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.

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

24th March 2022

References

ISO11459: 1997. Iron Ores- Certified reference materials – preparation and certification for use in chemical analysis.

ISO Guide 35 (2006), Reference materials – General and statistical principles for certification.

Boris Iglewicz and David Hoaglin (1993), "Volume 16: How to Detect and Handle Outliers", The ASQC Basic References in Quality Control: Statistical Techniques, Edward F. Mykytka, Ph.D., Editor.

Appendix 1

Tabulated and graphical presentation of certification data.

Appendix 2

Homogeneity report of key analytes.