

Iron Ore Certified Reference Material: Certificate of Analysis

PBS 11

Table 1: PBS 11 Certified Values

Analyte	unit	Assigned Value	Standard Deviation			95% Confidence Interval			Coeff of Var.	Number of Laboratories	Number of Analysis
			Absolute SD	SD within lab	SD between lab	Interval	lower	upper			
Fe	%	44.56	0.21	0.12	0.45	0.14	44.42	44.70	0.5%	11	66
SiO ₂	%	21.51	0.08	0.06	0.15	0.06	21.45	21.57	0.4%	11	65
Al ₂ O ₃	%	6.12	0.04	0.03	0.07	0.03	6.09	6.15	0.6%	11	66
TiO ₂	%	0.528	0.011	0.006	0.023	0.007	0.521	0.535	2.0%	12	72
Mn	%	0.814	0.010	0.004	0.023	0.008	0.806	0.822	1.2%	9	54
CaO	%	0.146	0.006	0.003	0.013	0.004	0.142	0.150	3.9%	12	72
P	%	0.047	0.002	0.001	0.004	0.001	0.046	0.048	3.8%	12	71
S	%	0.033	0.002	0.001	0.005	0.002	0.031	0.035	6.6%	8	48
K ₂ O	%	0.122	0.004	0.002	0.007	0.002	0.120	0.124	2.9%	12	69
V	%	0.011	0.001	0.001	0.003	0.001	0.010	0.012	9.7%	11	66
LOI ₃₇₁	%	4.65	0.06	0.02	0.15	0.05	4.60	4.70	1.3%	9	54
LOI ₄₂₅	%	4.99	0.11	0.03	0.28	0.09	4.90	5.08	2.2%	8	48
LOI ₆₅₀	%	6.04	0.06	0.03	0.13	0.04	6.00	6.08	0.9%	9	54
LOI ₁₀₀₀	%	6.28	0.06	0.04	0.12	0.04	6.24	6.32	1.0%	11	66

Table 2: PBS 11 Provisional Values

Analyte	unit	Assigned Value	Standard Deviation			95% Confidence Interval			Coeff of Var.	Number of Laboratories	Number of Analysis
			Absolute SD	SD within lab	SD between lab	Interval	lower	upper			
MgO	%	0.118	0.013	0.005	0.031	0.009	0.109	0.127	11.4%	12	70
Ba	%	0.013	0.003	0.002	0.006	0.002	0.011	0.015	21.8%	9	53
Cr ₂ O ₃	%	0.021	0.004	0.003	0.008	0.003	0.018	0.024	19.6%	9	53
Zr	%	0.014	0.003	0.002	0.003	0.002	0.012	0.016	18.6%	8	48

Table 3: PBS 11 Informational Values

Analyte	unit	Assigned Value	Number of Laboratories	Number of Analysis
Na ₂ O	%	0.028	10	60
Zn	%	0.006	9	54
Pb	%	0.004	5	27
Cu	%	0.003	4	23
Cl	%	0.009	5	30
As	%	0.003	5	30
Co	%	0.001	1	6
Ni	%	0.004	6	28
Sn	%	0.002	2	8
Sr	%	0.004	6	29

Introduction

This document specifies preparation, analysis, and certification of reference material PBS 11.

Origin of Material

The source material is from an active mine located in the Pilbara region of Western Australia.

Constituent Mineralogy

Indicative mineralogy concentrations are provided as a guidance only from a single XRD analysis detailed in Table 4.

Table 4: PBS 11 XRD analysis of mineralogy

Phase	Formula	Units	
Hematite	Fe ₂ O ₃	wt%	33
Goethite	FeO(OH)	wt%	29
Amorphous Content		wt%	18
Quartz	SiO ₂	wt%	15
Magnetite	Fe ₃ O ₄	wt%	2
Gibbsite	Al(OH) ₃	wt%	1
Kaolin	Al ₂ Si ₂ O ₅ (OH) ₄	wt%	1
Anatase	TiO ₂	wt%	<1
Total		wt%	99

Method of preparation

The material was prepared as follows:

- Drying at 105°C to constant mass
- Multistage crushing and milling
- Homogenisation
- Packaging into sealed 20kg vessels awaiting final packaging at client request.

Samples were taken at intervals during the packaging stage to provide material for the Certification process.

Measurement techniques used for certification

Twelve laboratories were each given 6 x 10g randomly selected samples for analysis via lithium borate fusion XRF for the following:

Fe, SiO₂, Al₂O₃, TiO₂, Mn, CaO, P, S, MgO, K₂O, Na₂O, Ba, As, Cu, Pb, Zn, Cl, Co, Cr₂O₃, Ni, Sn, Sr, V, Zr.

Results are quoted as **un-normalised**.

In addition, loss on Ignition (LOI) was requested via Thermal gravimetric analysis (TGA) at 371°C, 425°C, 650°C and 1,000°C.

Method of Certification

Individual laboratory results identified as outliers using a $\alpha=0.05$ on a two-tailed Grubbs test by laboratory group are reviewed via normal probability plots for verification, and if deemed true outliers of the entire sample population are removed from the analysis. Laboratory performance was reviewed using box and whisker plots to identify extreme laboratory values. Laboratories with analyte means greater than 3 standard deviations from the remaining results were removed from the certification process. Results flagged as possible outliers were only removed from the analysis if it could be confidently deemed to be an excessive analytical variation rather than variation arising from inhomogeneity of the sample material.

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 Certified value is calculated from the mean of laboratory means, Standard deviation is calculated as the standard deviation of all results, and confidence interval derived at the $\alpha=0.05$ from the Students t-distribution for the number of participating laboratories. 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 \pm 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.

Participating laboratories

Table 5: PBS 11 Participating Laboratories

1	Activation Laboratories, Ancaster, Canada
2	ALS Brisbane, QLD
3	ALS Minerals, Wangara, WA
4	Bureau Veritas, Cardiff, NSW
5	Bureau Veritas- Ultratrace, Canning Vale, WA
6	Bureau Veritas, Whyalla, SA
7	Bureau Veritas, Wingfield SA
8	Intertek Jakarta, Indonesia
9	Intertek Genalysis, Maddington, WA
10	Nagrom, Kelmscot, WA
11	SGS Lakefield, Canada
12	SGS Newburn, WA

Preparer and supplier of reference material

The iron ore reference material PBS 11 has been prepared and certified, and is certified by:

Pilbara Standards Pty Ltd
Unit 6, 190 Star St,
Carlisle, WA 6101
Australia

www.pilbarastandards.com.au

The material is supplied in 250g and 1kg sealed plastic jars, or to client specification.

Intended use

PBS 11 is intended for the monitoring of laboratory performance in the analysis of analytes in geological samples; the verification of analytical methods; and the calibration of instruments used in the determination of the concentration of analytes reported in Table 1.

Stability and storage instructions

PBS 11 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 PBS 11 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

Pilbara 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 Pilbara Standards Pty Ltd from and against all liability and costs from the use of this material and information.

Certifying officer

Bruce Armstrong

Certification date

27th February 2019

References

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