Pilbara Standards

Iron Ore Certified Reference Material: Certificate of Analysis

PBS-32

Table 1: PBS-32 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	%	61.71	0.226	0.179	61.61	61.81	0.4%	12	72
SiO2	%	4.24	0.040	0.020	4.21	4.26	0.9%	11	65
Al2O3	%	1.52	0.022	0.013	1.51	1.54	1.4%	12	70
TiO2	%	0.063	0.0040	0.0026	0.061	0.065	6.3%	12	71
Mn	%	0.298	0.0068	0.0032	0.294	0.302	2.3%	12	72
P	%	0.035	0.0013	0.0010	0.034	0.035	3.7%	11	65
S	%	0.031	0.0021	0.0008	0.030	0.033	6.7%	9	53
LOI 371'C	%	4.63	0.019	0.013	4.62	4.65	0.4%	8	48
LOI 425'C	%	4.77	0.020	0.013	4.75	4.78	0.4%	6	36
LOI 650'C	%	5.13	0.031	0.013	5.11	5.15	0.6%	10	59
LOI 1000'C	%	5.34	0.031	0.012	5.31	5.37	0.6%	7	42

Table 2: PBS-32 Provisional 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			
CaO	%	0.025	0.0046	0.0026	0.023	0.028	18.2%	11	66
MgO	%	0.039	0.0070	0.0048	0.035	0.043	18.0%	11	63
Cl	%	0.006	0.0011	0.0010	0.005	0.007	17.9%	7	38
Zn	%	0.012	0.0017	0.0008	0.011	0.013	14.7%	11	66
Loss 371 to 425'C	%	0.14	0.011	0.0068	0.13	0.15	7.8%	6	36
Loss 371 to 650'C	%	0.51	0.020	0.0082	0.50	0.52	3.9%	10	60
Loss 425 to 650'C	%	0.36	0.013	0.0083	0.35	0.37	3.5%	8	48
Loss 650 to 1000'C	%	0.20	0.009	0.0052	0.19	0.21	4.7%	7	41

Table 3: PBS-32 Informational Values

Analyte	unit	Value	Number of Labs	Number of Analysis
K2O	%	0.005	7	41
Na2O	%	0.008	4	18
As	%	0.003	7	39
Ba	%	0.004	6	24
Co	%	0.002	1	6
Cr2O3	%	0.002	3	18
Cu	%	0.002	3	16
Ni	%	0.003	2	11
Pb	%	0.003	3	15
Sn	%	0.006	1	6
Sr	%	0.002	2	10
V	%	0.002	5	25
Zr	%	0.002	3	17

Introduction

This document specifies preparation, analysis, and certification of reference material PBS-32. This document replaces earlier version of 19th June 2019, with revision of category for LOI intervals.

Origin of Material

The source material is from an active iron ore 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-32 XRD analysis of mineralogy

Phase	Formula	Units	Value
Hematite	Fe2O3	wt%	50
Goethite	FeO(OH)	wt%	39
Amorphous Content		wt%	7
Magnetite	Fe3O4	wt%	3
Kaolin	Al2Si2O5(OH)4	wt%	1
Ilmenite	FeTiO3	wt%	<1
Quartz	SiO2	wt%	<1

Method of preparation

The material was prepared as follows:

- Drying at 105°C to constant mass
- Multistage crushing and milling
- Homogenisation
- Packaging into sealed 10kg 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, SiO_2, $Al2O_3$, TiO_2, Mn, CaO, P, S, MgO, K_2O, Na2O, Ba, As, Cu, Pb, Zn, Cl, Co, Cr_2O_3, 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

Outlier laboratory and individual analytical results are removed from the informing sample population to remove erroneous values. The system 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 modified Z-scores >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 standard deviations with Z-score >3.
- 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.

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. 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.

Participating laboratories

Table 5: PBS-32 Participating Laboratories

ALS Brisbane	Queensland, Australia
ALS Malaga	Western Australia
BV Canning Vale	Western Australia
BV Cardiff	New South Wales, Australia
BV Whyalla	South Australia
BV Wingfield	South Australia
Intertek Genalysis Maddington	Western Australia
IRL Cloudbreak	Western Australia
Nagrom Kelscott	Western Australia
PT Intertek Utama Services	Jakarta, Indonesia
SGS Lakefield	Ontario, Canada
SGS Newburn	Western Australia

Version History

Batch	Author	Document Version	Date	Modifications
PBS-32	Harry Ooi	R1	8th August 2023	Updated certificate batch numbering format from PBS 32 to PBS-32.

Preparer and supplier of reference material

The iron ore reference material PBS-32 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-32 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-32 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-32 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, Managing Director, Pilbara Standards Pty Ltd

Certification date

3rd July 2019

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.