Pilbara Standards

Magnetite Ore Certified Reference Material: Certificate of Analysis

PBS70

Table 1: PBS70 Certified Values via fusion XRF (Un-Normalised)

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	%	30.46	0.100	0.071	30.37	30.54	0.3%	6	35
SiO2	%	47.54	0.122	0.111	47.46	47.61	0.3%	6	36
Al2O3	%	1.49	0.017	0.010	1.48	1.51	1.1%	6	36
TiO2	%	0.073	0.0038	0.0033	0.071	0.075	5.2%	8	48
Mn	%	0.117	0.0035	0.0023	0.115	0.120	3.0%	7	42
CaO	%	0.492	0.0071	0.0052	0.487	0.497	1.5%	7	41
P	%	0.102	0.0019	0.0009	0.101	0.104	1.9%	8	48
S	%	0.133	0.0017	0.0013	0.132	0.135	1.2%	7	41
MgO	%	2.26	0.024	0.011	2.24	2.28	1.1%	6	36
K2O	%	0.675	0.0115	0.0050	0.664	0.685	1.7%	7	42
Na2O	%	0.149	0.0140	0.0060	0.136	0.162	9.4%	7	42
LOI 1000	%	3.28	0.097	0.041	3.19	3.37	3.0%	7	42

Table 2: PBS70 Provisional Values via fusion XRF (Un-Normalised)

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	%	0.043	0.0032	0.0020	0.039	0.046	7.6%	5	30

Table 3: PBS70 Informational Values via fusion XRF (Un-Normalised)

Analyte	unit	Value	Number of Labs	Number of Analysis
As	%	0.001	1	4
Ba	%	0.006	5	30
Co	%	0.001	2	11
Cr	%	0.007	3	18
Cu	%	0.002	3	10
Ni	%	0.002	3	18
Pb	%	0.003	1	3
Sn	%	0.001	2	8
Sr	%	0.003	2	12
V	%	0.002	1	6
Zn	%	0.002	2	11
Zr	%	0.004	4	21

Table 4: PBS70 Certified Values of DTR Magnetite Conc. via fusion XRF (Un-Normalised)

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	%	62.01	1.030	0.364	61.15	62.87	1.7%	8	48
SiO2	%	11.77	1.053	0.323	10.88	12.66	8.9%	8	48
CaO	%	0.155	0.0153	0.0067	0.143	0.168	9.8%	8	48
P	%	0.039	0.0031	0.0012	0.037	0.042	7.9%	8	48
LOI 1000	%	1.70	0.168	0.095	1.57	1.82	9.9%	8	48
Distr Mags Pc*	%	32.90	0.971	0.256	31.97	33.83	3.0%	7	42

*Via Davis Tube Recovery at defined parameters

Table 5: PBS70 Provisional Values of DTR Magnetite Conc. via fusion XRF (Un-Normalised)

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			
Al2O3	%	0.31	0.041	0.016	0.28	0.35	13.2%	8	48
TiO2	%	0.030	0.0056	0.0030	0.026	0.035	18.4%	7	42
MgO	%	0.512	0.0547	0.0181	0.466	0.558	10.7%	8	48
K2O	%	0.132	0.0228	0.0076	0.113	0.151	17.3%	8	48
Cr	%	0.016	0.0009	0.0004	0.015	0.017	5.4%	4	24
V	%	0.004	0.0006	0.0005	0.003	0.005	15.4%	4	24

Table 6: PBS70 Informational Values of DTR Magnetite Conc. via fusion XRF (Un-Normalised)

Analyte	unit	Value	Number of Labs	Number of Analysis
Mn	%	0.028	7	42
S	%	0.018	7	42
Na2O	%	0.031	6	36
As	%	BDL	1	-
Ba	%	0.004	4	20
Cl	%	0.005	3	17
Co	%	0.001	2	10
Cu	%	0.004	4	23
Ni	%	0.003	3	18
Pb	%	0.003	3	17
Sn	%	0.002	2	12
Sr	%	0.001	2	11
Zn	%	0.002	3	18
Zr	%	0.004	3	18

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Version Control				
Doc. Version	Analysis Version	Author	Date	Changes
PBS70-05	PBS 70_v4_3_Head_Un-NORM, PBS 70_v4_3_MAG_Conc_Un-NORM,	B. Armstrong	10/08/2020	Un-normalised XRF data

Introduction

This document specifies preparation, analysis, and certification of reference material PBS70.

Origin of Material

The source material is from a magnetite mine located in Western Australia.

Method of preparation

The material was prepared as follows:

- Drying at 105°C to constant mass.
- Multistage crushing and milling to target size representative of milling process.
- Homogenisation via high shear blenders. Blended from two similar materials in a relative proportion of 45% / 55% by mass.
- 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. The material was tested for:

- XRF / LOI on head (feed).
- Davis Tube Wash.
- XRF / LOI on Davis Tube magnetic concentrate.

Measurement techniques used for certification

XRF Analysis

Eight laboratories were each given 6 x 50g randomly selected samples for analysis of both head sample and magnetite concentrate 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 1,000°C only.

Davis Tube Wash

Davis Tube Wash (DTW) was undertaken at eight laboratories with six samples each laboratory. Parameters for DTW were specified as following:

1. No sample preparation required. Use as supplied.
2. Stroke Frequency   60/minute
3. Stroke Length   38 mm
4. Magnetic Field Strength   3000 gauss
5. Tube Angle   45 degrees
6. Tube Diameter   25 mm
7. Washing Time   15 minutes or until water is clear

The concentrate sample is collected in a small container after washing is complete. The concentrate is then vacuum filtered, washed, dried and weighed.

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.

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 Table 1 Table 4 for Certified Values , Table 2 and Table 5 for Provisional Values, and Table 3 and Table 6 for Informational Values.

Participating laboratories

Table 7: PBS70 Participating Laboratories

Laboratory	Location	XRF Analysis	DTR
ALS Brisbane	Queensland, Australia	y	
ALS Burnie	Tasmania		y
ALS Malaga	Western Australia	y	y
BV Wingfield	South Australia	y	y
Intertek Genalysis Maddington	Western Australia	y	y
Nagrom, Kelmscott	Western Australia	y	y
BV Whyalla	South Australia	y	y
SGS Metallurgy	Western Australia	y	y
BV Canning Vale Metallurgy	Western Australia	y	y
Count		8	8

Preparer and supplier of reference material

The reference material PBS70 has been prepared and certified, and is certified by:

Pilbara Standards Pty Ltd
16 Durham Rd,
Bayswater,
WA6053,
Australia

www.pilbarastandards.com.au

The material is supplied in 50g sealed mylar bags and 1kg sealed plastic jars.

Intended use

PBS70 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 and Table 6.

Stability and storage instructions

PBS70 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 PBS70 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

10th August 2020

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

Fusion XRF Analysis. Tabulated and graphical presentation of certification data.

Appendix 2

Fusion XRF Analysis of Davis Tube Recovery Magnetic Concentrate. Tabulated and graphical presentation of certification data