

# PBS-72

## Iron Ore Reference Material Certificate

*Table 1: 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	%	62.38	0.194	0.133	62.32	62.44	0.31	31	113
SiO <sub>2</sub>	%	3.65	0.036	0.026	3.63	3.66	0.98	23	86
Al <sub>2</sub> O <sub>3</sub>	%	2.14	0.024	0.019	2.13	2.14	1.13	29	104
TiO <sub>2</sub>	%	0.105	0.0042	0.0024	0.104	0.106	4.00	32	115
Mn	%	0.404	0.0070	0.0026	0.402	0.406	1.73	30	110
P	%	0.064	0.0016	0.0012	0.063	0.064	2.48	30	109
K <sub>2</sub> O	%	0.041	0.0015	0.0009	0.040	0.041	3.56	25	90
CaO	%	0.053	0.0029	0.0015	0.052	0.054	5.47	25	90
Sr	%	0.059	0.0028	0.0019	0.057	0.060	4.66	12	43
V	%	0.020	0.0010	0.0005	0.019	0.020	5.05	18	66
LOI.371	%	2.90	0.046	0.029	2.86	2.95	1.59	6	20
LOI.425	%	3.04	0.060	0.037	3.01	3.07	1.97	16	56
LOI.650	%	3.61	0.040	0.027	3.60	3.63	1.11	15	56
LOI.1000	%	3.89	0.083	0.027	3.86	3.92	2.13	26	92
LOI.425 to LOI.650	%	0.57	0.044	0.027	0.55	0.59	7.70	16	56

*Table 2: 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			
MgO	%	0.081	0.0116	0.0064	0.077	0.085	14.32	28	101
S	%	0.020	0.0025	0.0017	0.019	0.021	12.50	26	95
Cr	%	0.005	0.0010	0.0005	0.005	0.006	18.30	16	58
LOI.371 to LOI.425	%	0.17	0.039	0.029	0.13	0.20	23.64	5	16
LOI.650 to LOI.1000	%	0.26	0.032	0.025	0.25	0.28	12.09	16	55

*Table 3: Informational Values.*

Analyte	unit	Value	Number of Labs	Number of Analysis
Cl	%	0.013	19	65
Na <sub>2</sub> O	%	0.023	22	74
As	%	0.002	13	46
Ba	%	0.011	15	51
Co	%	0.001	10	38
Cu	%	0.002	18	68
Ni	%	0.003	17	64
Pb	%	0.002	15	56
Sn	%	0.001	3	9
Zn	%	0.005	22	78
Zr	%	0.003	9	31

Note 1. SI units equivalent: 1 ppm, parts per million  $\equiv$  mg/kg  $\equiv$  ug/g  $\equiv$  0.0001 wt.%  $\equiv$  1000ppb, parts per billion

Note 2. The number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD.

Version Control					
Doc. Version	Analysis Version	Author	Certifying Officer	Date	Changes
PBS-72_R0	PBS-72_V1	Andrew Weatherstone	Bruce Armstrong	12/04/2021	Initial Document
PBS-72_R1				01/09/2021	Spelling correction

## Introduction

This document specifies preparation, analysis, and certification of reference material PBS-72.

## Method of Preparation

Approximately 150kg of iron ore material was used in this batch. The material was prepared as follows:

- Drying at 105°C for nominal 24 hours
- Multistage crushing and milling to target p95@53um
- Homogenisation in high shear blender
- Packaging into 10kg vessels awaiting final packaging at client request.

Samples were taken at intervals during the packaging stage to provide material for the Certification process. A total of 300 10g samples were subsampled from the stored material for the Certification process. The selection of laboratory for each sample was randomised, prior to the samples being boxed and sent to the respective laboratories. Two samples were sent to 32 laboratories at two months apart, each laboratory received four 10g samples in total.

## 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  $>12$ .
- Individual outliers with Z-score  $>3$  are then removed from the informing population.

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 25%, 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  $\alpha=0.05$  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  $\pm$  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 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 for Certified Values, Table 2 for Provisional Values, and Table 3 for Informational Values.

## Participating laboratories

A total of 32 laboratories participated in the interlaboratory proficiency testing. The number of analytes analysed varied between laboratories. Table 4 indicates the number of laboratories used for each analyte, the number of labs excluded, and total number of laboratories used for the certification process.

Table 4: Number of Labs used for each analyte.

Analyte	No of Labs used for Analysis	No of labs excluded in Analysis	Total No of Labs
Fe	32	1	31
SiO <sub>2</sub>	32	9	23
Al <sub>2</sub> O <sub>3</sub>	32	3	29
TiO <sub>2</sub>	32	0	32
Mn	32	2	30
CaO	31	6	25
P	32	2	30
S	27	1	26
MgO	32	4	28
K <sub>2</sub> O	30	5	25
Na <sub>2</sub> O	24	2	22
As	18	5	13
Ba	18	3	15
Cl	19	0	19
Co	14	4	10
Cr	17	1	16
Cu	21	3	18
Ni	17	0	17
Pb	16	1	15
Sn	8	5	3
Sr	13	1	12
V	21	3	18
Zn	24	2	22
Zr	9	0	9
LOI.371	6	0	6
LOI.425	16	0	16
LOI.650	16	1	15
LOI.1000	28	2	26
LOI.371.to.425	6	1	5
LOI.425.to.650	16	0	16
LOI.650.to.1000	16	0	16

## Preparer and supplier of reference material

The iron ore reference material PBS-72 has been prepared and certified, and is certified by:

Pilbara Standards Pty Ltd  
16 Durham Rd  
Bayswater, WA 6053  
Australia  
[www.pilbarastandards.com.au](http://www.pilbarastandards.com.au)

The material has been supplied in 10g heat sealed aluminised bags with unique labels which are randomised throughout the batches. These randomised numbers are supplied in a separate document.

## Intended use

This pulverised iron ore reference material is for use to monitor and test the accuracy of laboratory analysis for the Certified and Provisional analytes.

## 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

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

12/04/2021

## 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.