Pilbara Standards
Certified Reference Materials

Pilbara Standards Pty Ltd
16 Durham Rd, Bayswater,
WA 6053, Australia
ABN: 37 158 615 395

Gold Certified Reference Material: Certificate of Analysis

PBS-86

Table 1: PBS-86 Certified Values

Analyte	unit	Value	Standard Deviation		95% Conf. Int.		SD Coeff. Of Var.	Number of Labs (ISO/IEC 17025)	Number of Analysis Determinations
			1 SD	1 SD Within Lab	lower	upper			
Au	ppm	0.976	0.021	0.013	0.964	0.987	2.1%	13	50

Note 1. SI units equivalent: 1 ppm, parts per million ≡ mg/kg ≡ ug/g ≡ 0.0001 wt.% ≡ 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.

Table 2: PBS-86 Indicative Values

Analyte	XRF Value (wt.%)	ICP Value (ppm)
Ag		<1
Al	-	74850
Al2O3	14.185	-
As	0.063	601
Ba	0.014	140
Be	-	1
Bi	-	0.4
Ca	-	65360
CaO	9.065	-
Cd	-	1
Ce	-	24
Cl	0.036	-
Co	0.005	35
Cr	0.017	200
Cs	-	<0.5
Cu	0.007	80
Fe	9.82	98050
Ga	-	23
Ge	-	<5
Hf	-	9
In	-	0.3
K	-	3200
K2O	0.392	-
La	-	10.5
Li	-	8
Mg	-	32510
MgO	5.489	-
Mn	0.146	1285
Mo	-	3
Na	-	22350
Na2O	2.971	

Analyte	XRF Value (wt.%)	ICP Value (ppm)
Nb	-	24
Ni	0.004	35
P	0.091	910
Pb	0.02	210
Rb	-	8.5
Re	-	<0.05
S	1.527	15500
Sb	-	2.5
Sc	-	33
Se	-	<5
SiO2	50.889	-
Sn	0.003	3.5
Sr	0.026	-
Ta	-	2.5
Te	-	<1
Th	-	1.7
Ti	-	11570
TiO2	1.991	-
Tl	-	0.95
U	-	0.3
V	0.029	255
W	-	5
Y	-	35.7
Zn	0.026	280
Zr	0.014	120
LOI371	0.39	-
LOI425	0.51	-
LOI650	0.83	-
LOI1000	0.98	-

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Introduction

This document specifies preparation, analysis, and certification of gold reference material PBS-86.

Method of Preparation

PBS-86 is manufactured from a pulverised Bunbury basalt base blended with minor pulverised pyrite and spiked with gold (Au).

The base input materials are oven dried at 105°C for 24 hours prior to pulverisation. The blended material underwent a multi-stage homogenisation process and was discharged into storage drums. During the discharge the material was sub-sampled at regular intervals.

The samples taken were randomised before being submitted to independent ISO/IEC 17025 accredited laboratories for Au analysis by fire assay followed by atomic absorption spectrometry (AAS) or inductively coupled plasma (ICP) determinations as part of homogeneity and inter-laboratory round-robin testing.

Multi-element results provide valuable analytical information to assist laboratories in selecting the optimal procedure when performing a digest and analysis of the reference material. A single sample was analysed by both lithium-borate fusion with x-ray fluorescence spectrometry (XRF) determination, and 4-acid digest with ICP determination. The multi-elemental analysis results presented in Table 2 are for indicative purpose only.

Homogeneity Analysis

A homogeneity study was undertaken using 30 randomly selected 50g samples. These samples were submitted to a single laboratory for 25g fire-assay with ICP-OES analysis in a single batch under repeatable conditions. The homogeneity study results were reviewed, and the material was deemed suitable for progressing to the inter-laboratory round-robin stage. A summary of the study results is presented in Table 3.

Table 3: PBS-86 Homogeneity Study Results

	Au
Number of Samples Submitted	30
Number of Samples tested	29
Total Samples in Analysis	28
Duplicates performed	No
Number of outlying results removed	1
Mean concentration (ppm)	0.999
Standard Deviation (ppm)	0.0241
Relative Standard Deviation	2.41%

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Method of Certification

A total of 56 x 50g samples were selected for inter-laboratory round-robin analysis, 4 samples were provided to 14 laboratories. Laboratories analysed samples via fire-assay digestion followed by either AAS or ICP. All laboratories returned results in this round.

Outlier laboratory and individual analytical results were removed from the informing sample population to eliminate erroneous values. The process used was:

- Removal of laboratory group data with a median evaluated by modified Z-scores of >3.0, using a method of Iglewicz and Hoaglin (1993).
- Removal of laboratory group data with excessive range indicating unstable control processes occurring within the laboratory. This is calculated as laboratory group results with interquartile ranges with modified Z-scores of >6.
- Individual outliers with Z-score >3 were then removed from the informing population.

The above process was reviewed by the certifying officer. In some cases the certifying officer may use their judgment in identifying or eliminating outliers outside of the above parameters.

The results were grouped into certified and indicative categories on the below general criteria:

- Certified values show high agreement between replicates, indicated with a low, (<25%) coefficient of variation (CoV = Std. Deviation / mean), a measure of the variability relative to the mean
- Indicative values are typically for analytes below the detection limit for the analysis technique chosen, or in cases with insufficient number of laboratories returning accepted analytical determinations. Statistical measures such as standard deviation and confidence intervals are not statistically robust measures in scenarios where analytes are at or below instrument detection limits, or when an insufficient number of analytical determinations are available.

The **Certified value** is calculated from the mean of laboratory mean (grand mean).

Standard deviation is calculated for pooled laboratory results.

Single factor ANOVA is used to calculate **within laboratory standard deviation**.

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 could be interpreted as there is a 0.95 probability that the true 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. Approximately 95% of measurements are expected to be between two SDs either side of the certified value.

The above calculations are in accordance with ISO Guide 35.

Assigned Values

Summary of certified values, standard deviations, confidence intervals, the number of laboratories and analysis used in calculating the values are shown in Table 1 for Certified Values. Indicative values are displayed in Table 2.

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Participating laboratories

Samples were sent to 14 participating laboratories which are listed in Table 4, together with the laboratory job number, sample mass, and analysis method.

Table 4: Participating Laboratories

Laboratory Name	Location	Job #	Mass (g)	Analysis method
Activation Lab - Ancaster	Hamilton, Canada	A20-15485	30	AAS
Activation Lab - Kamloops	Kamloops, Canada	A20-16807	30	AAS
ALS - Malaga	Perth, Australia	PH20275528	50	AAS
Bureau Veritas Gauge Circuit	Perth, Australia	u318827	40	ICP
Bureau Veritas Wingfield	Adelaide, Australia	aa047973.b	40	AAS
Corem - Analytical Services Laboratory	Quebec, Canada	127471		ICP
INTERTEK GENALYSIS (WA)	Perth, Australia	1771.0/2022881	50	AAS
Intertek JK	Jakarta, Indonesia	202892	50	AAS
Intertek Wingfield	Adelaide, Australia	1771.0/2023316	25	ICP
Intertek-Townsville	Townsville, Australia	1771_02_2100503	50	ICP
SGS - Burnaby, B.C	British Columbia, Canada	BBM21-06481	30	AAS
SGS - Lakefield	Lakefield, Canada	CA02030-Jan21	35	AAS
SGS - Macreas Flat, NZ	Macreas Flat, New Zealand	Pilbara RR Jan 2021	30	AAS
SGS - Perth Airport	Perth, Australia	WM204555 & WM204612	30	AES

Preparer and supplier of reference material

The gold reference material PBS-86 has been prepared and certified, and is certified by:

Pilbara Standards Pty Ltd
16 Durham Rd
Bayswater, WA 6053
Australia
www.pilbarastandards.com.au

The material is available in sealed 1 kg and 2kg PET jars, with unique labels showing the batch number.

Minimum sample mass

This reference material has been certified using 25g to 50g aliquots for fire assay. Uncertainty and homogeneity statements relating to this are only applicable if a minimum of 25g sample mass is used.

Intended use

The pulp gold reference material is intended for monitoring and testing the accuracy of fire-assay analysis of gold ores.

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Stability and storage instructions

Jars should be stored in a cool dry location, and mixed before opening for first use. This product contains a low level of sulphide material. Once opened it is recommended to re-seal opened jars when not in use.

All jars have been labelled with a recommended use by date. The long-term storage of this product is monitored, and purchasers will be notified if changes are observed during the period of validity of the product.

Instructions for correct use

The certified values derived from fire-assay digestion and analysis is based on the concentration level in the packaged state, and no further drying is required before weighing and analysis.

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

9th February 2021

References

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.

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Appendix 1

Tabulated and graphical presentation of certification data.

	Laboratory Number														
Determination No.	**1**	**3**	**4**	**5**	**6**	**7**	**9**	**10**	**19**	**39**	**40**	**41**	**42**	**43**	**Overall**
1	0.954	NA	0.958	0.950	0.999	0.970	0.990	0.970	0.896	0.850	0.980	0.972	0.998	0.969	
2	0.969	1.000	0.963	0.960	0.994	0.990	0.960	0.960	0.952	0.860	1.010	0.981	0.989	0.963	
3	0.962	1.010	0.955	0.950	0.963	0.970	0.970	0.970	0.920	0.940	1.000	0.992	0.982	0.991	
4	0.971	1.020	0.984	0.970	1.018	0.970	0.960	0.970	0.931	0.880	0.990	0.971	0.994	1.001	
Count	4	3	4	4	4	4	4	4	3	0	4	4	4	4	50
Min	0.954	1.000	0.955	0.950	0.963	0.970	0.960	0.960	0.920	NA	0.980	0.971	0.982	0.963	0.920
Max	0.971	1.020	0.984	0.970	1.018	0.990	0.990	0.970	0.952	NA	1.010	0.992	0.998	1.001	1.020
Median	0.966	1.010	0.961	0.955	0.997	0.970	0.965	0.970	0.931	NA	0.995	0.977	0.992	0.980	0.970
Mean	0.964	1.010	0.965	0.958	0.994	0.975	0.970	0.968	0.934	NA	0.995	0.979	0.991	0.981	0.976
Std Dev	0.008	0.010	0.013	0.010	0.023	0.010	0.014	0.005	0.016	NA	0.013	0.010	0.007	0.018	0.0209
Coeff. Variation	0.799	0.990	1.356	1.000	2.296	1.026	1.458	0.517	1.740	NA	1.298	0.997	0.696	1.831	2.1
Dev. From Cert Mean	-1.23	3.48	-1.13	-1.90	1.79	-0.10	-0.61	-0.87	-4.27	NA	1.95	0.31	1.52	0.51	
95% Confidence Interval															0.0112
SD Within Labs															0.0129
SD Between Labs															0.0356
M-Score	0.24	2.16	0.51	0.81	1.43	0.00	0.27	0.00	2.40	5.40	1.35	0.35	1.16	0.54	3
M-IQR	0.51	0.42	0.23	0.05	0.98	1.35	0.51	1.81	1.86	4.70	0.51	0.05	0.84	2.56	6

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