

Reference Material Certificate: Pulverised Sediments and low grade Banded Iron Formation PBS-310

Table 1: PBS-310 Certified Values expressed as % w/w

Analyte	Certified Value (y)	Standard Deviation		95% Confidence Interval (CI)		UCRM [^]	k [#]	UCRM [~]	No. of Labs (ISO 17025)	No. Samples
		1 SD (s)	1 SD Within Lab (s _w)	lower	upper					
Fe	22.98	0.161	0.078	22.85	23.11	0.095	2.31	0.22	8	48
SiO ₂	30.24	0.139	0.064	30.14	30.34	0.11	2.26	0.26	9	54
Al ₂ O ₃	14.20	0.091	0.031	14.13	14.27	0.056	2.26	0.13	9	54
TiO ₂	0.830	0.0080	0.0056	0.825	0.835	0.0086	2.26	0.019	9	54
Mn	1.73	0.038	0.011	1.70	1.76	0.018	2.31	0.042	8	48
CaO	2.84	0.026	0.011	2.82	2.86	0.021	2.31	0.048	8	48
P	0.040	0.0008	0.0007	0.040	0.040	0.0013	2.26	0.0030	9	54
S	0.027	0.0017	0.0006	0.026	0.028	0.0014	2.26	0.0032	9	54
MgO	2.64	0.033	0.011	2.62	2.66	0.020	2.26	0.046	9	54
K ₂ O	0.981	0.0081	0.0034	0.975	0.987	0.011	2.31	0.025	8	47
Na ₂ O	1.70	0.040	0.016	1.67	1.73	0.033	2.31	0.076	8	48
Ba	0.021	0.0044	0.0020	0.017	0.025	0.0031	2.45	0.0076	6	36
Cl	1.73	0.187	0.074	1.58	1.88	0.17	2.57	0.43	5	30
Cr	0.012	0.0022	0.0009	0.010	0.014	0.0014	2.36	0.0033	7	42
Cu	0.007	0.0030	0.0006	0.003	0.011	0.0020	2.45	0.0050	6	35
Ni	0.009	0.0010	0.0007	0.008	0.010	0.0013	2.36	0.0032	7	42
Zn	0.007	0.0017	0.0005	0.006	0.008	0.0012	2.36	0.0029	7	42
LOI425	3.44	0.467	0.068	2.81	4.07	0.29	2.45	0.71	6	36
LOI650	10.04	0.155	0.052	9.91	10.17	0.073	2.36	0.17	7	42
LOI1000	10.93	0.097	0.052	10.85	11.01	0.056	2.36	0.13	7	42
LOI425 to 650	6.57	0.446	0.054	5.97	7.17	0.25	2.45	0.62	6	36
LOI650 to 1000	0.93	0.187	0.055	0.77	1.09	0.089	2.36	0.21	7	42

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

[^] Standard uncertainty.

[#] Coverage Factor.

[~] Expanded Uncertainty.

Table 2: PBS-310 Informational Values expressed as % w/w

Analyte	Assigned Value (y)	Standard Deviation		95% Confidence Interval (CI)		U_{CRM}^{\wedge}	k#	U_{CRM}^{\sim}	No. of Labs	No. Samples
		1 SD (s)	1 SD Within Lab (s_w)	lower	upper					
As	0.003	0.0015	0.0006	0.002	0.005	0.0012	2.36	0.0027	7	42
Co	0.004	0.0026	0.0006	0.002	0.007	0.0014	2.36	0.0034	7	41
Pb	0.004	0.0020	0.0010	0.002	0.006	0.0013	2.45	0.0031	6	31
Sr	0.006	0.0014	0.0005	0.004	0.008	0.0014	2.57	0.0037	5	30
V	0.013	0.0008	0.0005	0.013	0.014	0.0011	2.31	0.0026	8	47
Zr	0.020	0.0029	0.0009	0.017	0.023	0.0017	2.36	0.0040	7	42

Version History

Batch	Author	Document Version	Date	Modifications
PBS-310	Harry Ooi	R0	4/8/2023	Initial Document

Introduction

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

Material and Method of Preparation

Following drying at 105 °C, crushing and screening, this reference material was prepared by multi-stage pulverisation and homogenisation. During the discharge from the last blending stage, samples were drawn for homogeneity and characterisation studies. The samples taken were randomised before being submitted to independent ISO/IEC 17025 accredited laboratories for as part of homogeneity and inter-laboratory round-robin testing for material characterisation. Material was packaged into 1kg sealed PET containers.

Table 3: Batch Specific Parameters

Production Summary	
Material Source	Blend of sedimentary waste rocks and low-grade banded iron formation from Pilbara Region, Western Australia.
Material Description	Spiked for elevated NaCl, Ca concentrations.
Packaged Sample Mass (g)	10
Drying Temperature (°C)	105
Screening size (µm)	54
Homogeneity Study	
Sample mass /unit (g)	10
No. samples submitted	20
No. determinations per sample	2
Characterisation Study	
No. of Laboratories	9
No. samples per laboratory	6
No. determinations per sample	1

Homogeneity Analysis

A homogeneity study was undertaken in accordance with ISO Guide 35:2017 and ISO17034:2016 using systematically selected samples to be representative of the entire batch. The sample identifiers were randomised to ensure different production order and laboratory analytical order. These samples were submitted to a single laboratory for multiple analysis in a single batch under repeatable conditions. The homogeneity study results were reviewed to ensure all analytes were deemed homogeneous prior to the material progressing to the inter-laboratory round-robin stage for characterisation assessment. A summary of the study is presented in Table 3 with results summarised in Table 4.

Table 4: Homogeneity Study Results

Analyte	Mean Concentration Homogeneity Lab (% w/w)	SD between samples median value	Relative SD %
Fe	23.09	0.045	0.2
SiO ₂	30.22	0.054	0.2
Al ₂ O ₃	14.14	0.034	0.2
TiO ₂	0.831	0.0048	0.6
Mn	1.730	0.0085	0.5
CaO	2.844	0.0058	0.2
P	0.040	0.0006	1.6
S	0.028	0.0007	2.6
MgO	2.617	0.0115	0.4
K ₂ O	0.978	0.0036	0.4
Na ₂ O	1.751	0.0116	0.7
Ba	0.021	0.0021	10.2
Cl	1.982	0.1129	5.7
Cr	0.012	0.0003	2.8
Cu	0.005	0.0004	7.3
Ni	0.008	0.0004	5
Sr	0.008	0.0004	5.4
V	0.013	0.0003	2.5
Zn	0.007	0.0004	5
Zr	0.018	0.0006	3.1
LOI425	3.32	0.051	1.5
LOI650	10.04	0.026	0.3
LOI1000	11.01	0.037	0.3
LOI425 to 650	6.72	0.050	0.7
LOI650 to 1000	0.98	0.040	4.1

Material Characterisation and Certification Methodology

The process of characterisation was undertaken in accordance with ISO Guide 35:2017 and ISO17034:2016 following examination of grouped laboratory results for potential technical failures. Where required, further investigation of outliers was conducted. Laboratory results deemed technical outliers were removed from the analysis pool prior to the determination of statistical parameters. The certifying officer, in some cases, may use their judgment in identifying or eliminating outliers outside of these statistical parameters.

- Certified value was determined by average of laboratory averages for analytes with no outlier laboratory results, or median of laboratory medians for those with outlier laboratory results.
- Standard deviation (s) is the measure of spread of analyte determinations and includes inter-laboratory bias, method uncertainty, and material homogeneity uncertainty. Approximately 95% of determinations using the same analytical method are expected to be between two standard deviations either side of the certified value. The standard deviation is calculated from the validated laboratory group data less outlier laboratory and individual determinations.
- Within laboratory standard deviation (s_w) is the average spread of determination values across the reporting laboratories, less outlier laboratory and individual determinations. This is calculated by single factor ANOVA of the participating laboratory groups
- Confidence Interval (CI) is an estimate of the true (unknowable) analyte concentration in the material at the 95% confidence interval. For example, a 95% CI could be interpreted as there is a 0.95 probability that the true value is between certified value $\pm CI$. The narrower the interval, the more precise the certified value. The 95% CI should not be used for determination of quality control gates.
- Standard Uncertainty (U_{CRM}) is the sum of variance from characterisation, homogeneity and stability studies. The uncertainty of characterisation is derived from the standard deviation of average of laboratory averages divided by the square root of the number of laboratories. Uncertainty of material homogeneity (u_{hom}) is the sum of ANOVA within and between sample uncertainty derived from the homogeneity study. An allowance for stability has been included in accordance with ISO Guide 35.
- Coverage Factor (k) is the students t-distribution value for two tailed test at 95%.
- Expanded Uncertainty (U_{CRM}) is the product of coverage factor and standard uncertainty, and represents the 95% confidence interval of the true unknowable analyte concentration of the batch combined with the bias from individual samples.

The certified value of any elemental or oxide concentration may not be negative even though in some cases the uncertainty error bounds define a range less than 0%. These cases are due to low concentrations of some analytes relative to the analytical detection limits and increments of precision.

Analytes have been categorised as Certified or Informational based on:

- Confirmation of sufficient between-unit variance demonstrating material homogeneity.
- Minimum number of participating ISO17025 accredited laboratories for that particular analyte.
- Sufficient agreement between participating laboratories.

Participating Laboratories

Laboratories used in the certification process are listed in Table 5 in alphabetical order, along with batch number.

Table 5: PBS-310 Participating Laboratories

Laboratory Name	Location	Job #
ALS Geochemistry	Malaga, Western Australia	PH23159331
ALS Geochemistry	Stafford, Queensland	BR23166476
Bureau Veritas Minerals	Canning Vale, Western Australia	u348453
Bureau Veritas Minerals	Wingfield, South Australia	aa061064
Bureau Veritas Minerals	Whyalla, South Australia	WH094078
Intertek Genalysis	Maddington, Western Australia	1771.0/2310942
Intertek Genalysis (Utama Services Jakarta)	Jakarta, Indonesia	231959
Nagrom	Kelmscott, Western Australia	KM-2306-068182
SGS Geochemistry	Perth Airport, Western Australia	WM214396
Spectrolab	Geraldton, Western Australia	26086

Intended Use

This pulverised reference material is intended for monitoring and testing the accuracy and precision of XRF and TGA (LOI) analysis. This intended use may include a quality control program within a minerals or mine site laboratory.

The estimate of material and measurement uncertainties reported in this certificate are the product of the participating laboratories, not any individual laboratory. Commercial laboratories typically have different measurement uncertainties to site-based laboratories. Application of the grouped uncertainties reported in this certificate to a specific laboratory for ongoing QC may lead to many false reports of out-of-control processes, or alternatively non reporting of out-of-control processes.

It is recommended that the centre line and control limits of a Shewhart chart used for ongoing monitoring of a particular laboratory are derived from averaged values and variation from replicate analysis of this CRM after removal of outliers.

Preparer and supplier of reference material

This reference material has been prepared and is certified by:

Independent Mineral Standards Pty Ltd
 16-18 Durham Rd
 Bayswater, WA 6053
 Australia

www.imstandards.com.au

Minimum Sample Mass

This reference material has been certified using XRF fusion, and TGA (LOI) analysis. Uncertainty statements are only applicable if a minimum sample mass of 0.6g for XRF and 1.0g for TGA is used.

Period of Validity

This Certificate is valid 10 years from the date of original issue.

Commutability

This reference material is not commutable to any other analytical methods than as stated by its intended use.

Metrological Traceability

Metrological traceability of the assigned property values and their uncertainties has been established through an unbroken chain to the SI unit kilogram for Certified Values in Table 1. This is achieved through the use of assay laboratories accredited for ISO17025 for both the analytical method and analyte used in homogeneity, characterisation and stability studies.

Metrological traceability of the informational values and their uncertainties listed in Table 2 has not been established as values from laboratories and methods not accredited to ISO 17025 have been included in the characterisation studies. The values were incorporated to ensure sufficient number of participating laboratories to determine an uncertainty.

Stability and Storage Instructions

Jars should be stored in a cool dry location, and mixed by shaking the sealed container before opening for first use. 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 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

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

Certifying officer

Bruce Armstrong

Certification date

23rd August 2023

References

ISO Guide 35:2017, Reference materials – General and statistical principles for certification.

ISO17034:2016, General Requirements for the competence of reference material producers.