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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 408

Hematite Iron Ore, Hawks Nest, South Australia

Accredited for compliance with ISO 17034

ISO 9001

Table 1. Certified Values, Uncertainty & Tolerance Intervals for multi-elements by Borate Fusion XRF, Thermogravimetry and 4-Acid Digestion in OREAS 408.

Constituent	Certified Value[†]	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	0.998	0.969	1.026	0.979	1.017
As, Arsenic (ppm)	35.6	26.5	44.7	IND	IND
CaO, Calcium oxide (wt.%)	0.311	0.302	0.320	0.303	0.319
Cl, Chlorine (ppm)	207	154	261	IND	IND
Fe, Iron (wt.%)	63.22	62.98	63.46	63.01	63.43
Fe(Calc), Iron normalised to 100% (wt.%)	63.24	63.10	63.37	62.96	63.51
K_2O, Potassium oxide (wt.%)	0.027	0.025	0.030	0.026	0.029
MgO, Magnesium oxide (wt.%)	0.068	0.058	0.079	0.063	0.074
Mn, Manganese (wt.%)	0.081	0.077	0.085	0.079	0.083
P, Phosphorus (wt.%)	0.015	0.015	0.016	0.015	0.016
S, Sulphur (wt.%)	0.016	0.015	0.018	0.015	0.017
SiO_2, Silicon dioxide (wt.%)	7.01	6.92	7.09	6.94	7.07
Zn, Zinc (ppm)	81	67	96	IND	IND
Thermogravimetry					
LOI^{1000}, Loss on ignition @1000 °C (wt.%)	0.985	0.887	1.084	0.942	1.029
LOI^{371}, Loss on ignition @371 °C (wt.%)	0.352	0.322	0.383	0.327	0.378
LOI^{425}, Loss on ignition @425 °C (wt.%)	0.414	0.359	0.468	0.381	0.447
LOI^{650}, Loss on ignition @650 °C (wt.%)	0.727	0.700	0.755	0.691	0.764
4-Acid Digestion					
Ag, Silver (ppm)	0.284	0.258	0.310	0.265	0.303
Al, Aluminium (wt.%)	0.524	0.505	0.542	0.511	0.536
As, Arsenic (ppm)	30.5	29.0	32.0	29.1	32.0
Ba, Barium (ppm)	28.3	26.6	30.1	26.6	30.0
Be, Beryllium (ppm)	2.43	2.31	2.55	2.36	2.50
Bi, Bismuth (ppm)	1.19	1.13	1.25	1.13	1.25
Ca, Calcium (wt.%)	0.216	0.206	0.227	0.209	0.224
Ce, Cerium (ppm)	15.9	15.1	16.7	15.5	16.3
Co, Cobalt (ppm)	32.6	30.9	34.2	31.7	33.4
Cr, Chromium (ppm)	34.1	31.0	37.3	32.1	36.2
Cs, Caesium (ppm)	1.65	1.57	1.73	1.60	1.69
Cu, Copper (ppm)	21.2	20.1	22.3	20.4	22.0
Dy, Dysprosium (ppm)	3.01	2.74	3.28	2.88	3.13
Er, Erbium (ppm)	1.83	1.67	2.00	IND	IND
Fe, Iron (wt.%)	60.12	57.52	62.72	59.09	61.15
Ga, Gallium (ppm)	4.40	4.11	4.69	4.24	4.55
Gd, Gadolinium (ppm)	2.71	2.54	2.89	2.59	2.83

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

[†] The operationally defined measurands meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

Note 1: intervals may appear asymmetric due to rounding. IND = indeterminate (due to limited reading resolution of the methods employed).

Table 1 continued.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Ge, Germanium (ppm)	0.53	0.42	0.65	0.50	0.57
In, Indium (ppm)	0.26	0.24	0.27	0.25	0.27
K, Potassium (wt.%)	0.025	0.022	0.028	0.023	0.027
La, Lanthanum (ppm)	6.36	5.97	6.75	6.20	6.52
Li, Lithium (ppm)	175	167	183	171	179
Mg, Magnesium (wt.%)	0.039	0.035	0.043	0.038	0.041
Mn, Manganese (wt.%)	0.074	0.071	0.077	0.073	0.075
Mo, Molybdenum (ppm)	5.74	5.37	6.10	5.55	5.92
Na, Sodium (wt.%)	0.030	0.028	0.031	0.028	0.032
Nb, Niobium (ppm)	1.95	1.83	2.07	1.82	2.09
Nd, Neodymium (ppm)	9.86	9.36	10.36	9.50	10.21
Ni, Nickel (ppm)	27.3	25.6	28.9	26.1	28.4
P, Phosphorus (wt.%)	0.015	0.014	0.016	0.014	0.016
Pb, Lead (ppm)	24.0	21.8	26.1	23.0	24.9
Pr, Praseodymium (ppm)	2.11	1.97	2.25	2.04	2.18
Rb, Rubidium (ppm)	3.21	3.03	3.38	3.09	3.32
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.019	0.019	0.020	0.018	0.021
Sb, Antimony (ppm)	9.42	8.71	10.13	8.93	9.90
Sc, Scandium (ppm)	1.41	1.30	1.53	IND	IND
Se, Selenium (ppm)	< 1	IND	IND	IND	IND
Sm, Samarium (ppm)	2.38	2.10	2.66	2.22	2.54
Sn, Tin (ppm)	12.9	12.0	13.9	12.3	13.5
Sr, Strontium (ppm)	33.9	32.5	35.4	33.0	34.9
Ta, Tantalum (ppm)	3.76	3.27	4.24	3.51	4.00
Te, Tellurium (ppm)	< 0.01	IND	IND	IND	IND
Th, Thorium (ppm)	0.81	0.76	0.86	0.76	0.86
Ti, Titanium (wt.%)	0.010	0.010	0.011	0.010	0.010
Tl, Thallium (ppm)	0.18	0.17	0.19	IND	IND
U, Uranium (ppm)	3.21	3.06	3.35	3.12	3.30
V, Vanadium (ppm)	15.4	13.6	17.1	14.5	16.2
W, Tungsten (ppm)	19.1	17.7	20.5	18.3	19.9
Y, Yttrium (ppm)	18.4	17.6	19.2	17.9	18.9
Zn, Zinc (ppm)	73	70	76	71	76
Zr, Zirconium (ppm)	7.37	7.13	7.60	7.14	7.59

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: intervals may appear asymmetric due to rounding.

†The operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 408.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Classical Wet Chemistry					
FeO, Iron(II) oxide (wt.%)	1.13	1.02	1.24	1.07	1.19
Satmagan 135					
Fe_3O_4, Iron(II,III) oxide (wt.%)	1.98	1.68	2.28	IND	IND
Gas / Liquid Pycnometry					
SG, Specific Gravity (Unity)	4.76	4.59	4.92	4.71	4.80

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: intervals may appear asymmetric due to rounding.
IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95 % Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 3. Indicative Values for OREAS 408.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Ba	ppm	*39.8*	Na_2O	wt.%	*0.040*	TiO_2	wt.%	*0.018*
Co	ppm	*56*	Ni	ppm	*36.8*	V	ppm	*10.5*
Cr	ppm	*40.9*	Pb	ppm	*35.4*	Zr	ppm	*14.6*
Cu	ppm	*41.5*	Sn	ppm	*25.7*			
HfO_2	ppm	*< 100*	Sr	ppm	*36.5*			
4-Acid Digestion								
B	ppm	*< 100*	Hg	ppm	*0.092*	Tm	ppm	*0.26*
Cd	ppm	*0.055*	Ho	ppm	*0.60*	Yb	ppm	*1.76*
Eu	ppm	*0.67*	Lu	ppm	*0.27*			
Hf	ppm	*0.27*	Tb	ppm	*0.44*			
Davis Tube Recovery								
MassRec	wt.%	*0.513*						
Aqua Regia Digestion								
Ag	ppm	*0.213*	K	wt.%	*0.016*	Se	ppm	*< 1*
Al	wt.%	*0.362*	La	ppm	*5.09*	Sn	ppm	*13.5*
As	ppm	*32.0*	Li	ppm	*16.1*	Sr	ppm	*29.0*
Ba	ppm	*24.5*	Mg	wt.%	*0.030*	Ta	ppm	*< 0.05*
Be	ppm	*1.91*	Mn	wt.%	*0.071*	Te	ppm	*0.053*
Bi	ppm	*1.17*	Mo	ppm	*5.85*	Th	ppm	*0.76*
Ca	wt.%	*0.203*	Na	wt.%	*0.020*	Ti	wt.%	*0.007*
Cd	ppm	*0.055*	Nb	ppm	*0.20*	Tl	ppm	*0.17*
Ce	ppm	*12.8*	Ni	ppm	*31.7*	U	ppm	*3.14*
Co	ppm	*36.2*	P	wt.%	*0.014*	V	ppm	*38.5*
Cr	ppm	*34.2*	Pb	ppm	*23.7*	W	ppm	*19.4*
Cs	ppm	*1.39*	Rb	ppm	*1.97*	Y	ppm	*16.6*
Cu	ppm	*22.3*	Re	ppm	*< 0.05*	Zn	ppm	*66*
Ga	ppm	*4.22*	S	wt.%	*0.020*	Zr	ppm	*4.10*
Hf	ppm	*0.12*	Sb	ppm	*10.3*			
In	ppm	*0.28*	Sc	ppm	*1.00*			

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

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INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself. In evaluating laboratory performance with this CRM, the section headed ‘Instructions for correct use’ should be read carefully.

Table 1 (generated from data supplied by laboratories all accredited to ISO 17025 for borate fusion with XRF, multi-temperature LOI and 4-acid digestion) and Table 2 (generated from data supplied by laboratories mostly accredited to ISO 17025) provide the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties, Table 5 provides indicative mineralogy based on semi-quantitative XRD analysis and Table 6 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 408-DataPack.1.0.250327_131032.xlsx**). Results are also presented in scatter plot for Fe by borate fusion XRF in Figure 1, together with ±3 SD (magenta) and ±5 % (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

SOURCE MATERIAL

OREAS 408 is a certified reference material composed of hematite iron ore, obtained from the Hawks Nest Magnetite Project under an exploration license with Peak Iron Mines. The CRM contains approximately 86 % hematite and 3 % magnetite. The project site is situated approximately 120 kilometres south of Coober Pedy in South Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 408 was prepared in the following manner:

- Drying to constant mass at 105 °C;
- Homogenisation using OREAS’ novel processing technologies;
- Packaging in 10 g units in laminated foil pouches and 1 kg units in plastic jars.

PHYSICAL PROPERTIES

OREAS 408 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

Table 4. Physical properties of OREAS 408.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
816	0.25	10R 3/4	Dark Reddish Brown

‡The Munsell Rock Color Chart helps geologists and archeologists communicate with colour more effectively by cross-referencing ISCC-NBS colour names with unique Munsell alpha-numeric colour notations for rock colour samples.

MINERALOGY

The semi-quantitative XRD results shown in Table 5 below were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. A trace of illite, kutnohorite and maghemite might be present. A presence of some amorphous material is very likely. 'Kandite group' appears to be mainly kaolinite.

Table 5. Indicative mineralogy of OREAS 408 based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Kandite group	< 1
Quartz	8
Calcite	< 1
Siderite	< 1
Molybdenite and/or boehmite	< 1
Hematite	86
Magnetite	3
Goethite	2

ANALYTICAL PROGRAM

Twenty-four commercial analytical laboratories participated in the certification program for the analytes listed in Tables 1 and 2. The following methods were undertaken:

- Lithium borate fusion extended iron ore suite package with X-ray fluorescence (up to 23 laboratories depending on the analyte);
- Thermogravimetry: Loss on ignition (LOI) by multi-point TGA at 371 °C, 425 °C, 650 °C and 1000 °C on dry sample basis (up to 24 laboratories depending on the analyte).
- 4-acid digestion (HNO_3-HF-$HClO_4$-HCl) with full suite ICP-OES and ICP-MS elemental packages (up to 16 laboratories depending on the element);
- Acid digestion (HF/H_2SO_4) with titration to determine FeO (Fe^{2+} expressed as FeO);
- Satmagan 135 analysis to determine magnetic susceptibility (reported as the theoretical Fe_3O_4 content);

- Davis Tube Recovery (DTR)* using a 20-30 g sample size to determine magnetic content and reported as the ‘Mass Recovered’ (MassRec);
- Gas/liquid pycnometer analysis to determine Specific Gravity.

*See ‘Appendix’ for specified methodology.

For the round robin program, six 800 g test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking two 10 g scoop splits from each of three separate 800 g test units (either the odd or even test units). This format enabled a nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance. A further homogeneity evaluation was also undertaken at one laboratory involving a nested ANOVA within 12 x 10 g samples (duplicates of each of the six 800 g test units; see ‘Homogeneity Evaluation’ section below for further details).

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Tables 1 and 2) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration).

For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

95% Expanded Uncertainty provides a 95 % probability that the true value of the analyte under consideration lies between the upper and lower limits and is calculated according to the method outlined in [5] and [15]. All known or suspected sources of bias have been investigated or taken into account.

Indicative (uncertified) values (Table 3) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor. This data is intended for ‘informational purposes’ only.

Standard Deviation intervals (see Table 6, ‘Performance Gates’) provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

The SD for each analyte’s certified value is calculated from the same filtered data set used to determine the certified value, i.e., after removal of all individual, lab dataset (batch) and

3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e., the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. ***The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.***

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) [6] shown in Tables 1 to 3 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for Fe by borate fusion XRF method, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 63.01 and 63.43 wt.%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO 33405:2024). ***Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.***

Analysis of Variance (ANOVA) Study

In addition to the analysis of precision errors method outlined above, homogeneity was also evaluated using a nested ANOVA both i) within the interlaboratory program and ii) at one laboratory that was sent paired samples of each of the six test units. ANOVA enables a relative measure of homogeneity and permits a test of the null hypothesis that all ‘units’ are drawn from the same population distribution. ANOVA tests that no statistically significant difference exists in the variance between-units to that of the variance within-units. A *p*-value < 0.05 would indicate rejection of the null hypothesis at the 95 % significance level (i.e., a significant difference likely does exist; meaning there is evidence of heterogeneity between the six 800 g test units taken for homogeneity evaluation). No outliers were removed prior to the calculation of *p*-values because the presence of outliers could indicate a degree of heterogeneity.

i) Nested ANOVA within the interlaboratory program

The six samples received by each participating laboratory were obtained by taking two 10 g scoop splits from each of three separate 800 g test units (either the odd or even test units). The homogeneity of OREAS 408 has been evaluated in an ANOVA study for all certified analytes occurring at least 20 times the lower limit of detection. No significant *p*-values were found indicating a lack of evidence that between-unit variance is greater than within-unit variance.

ii) Nested ANOVA within one laboratory

This involved sending 12 x 10 g pulp samples to the Bureau Veritas minerals laboratory in Perth, Western Australia for Al_2O_3, CaO, Fe, K_2O, MgO, Mn, Na_2O, P, S, SiO_2, TiO_2 analysis by borate fusion with XRF and LOI at 1000 °C. The 12 samples consisted of paired subsamples from each of the six 800 g test units to enable an Analysis of Variance (ANOVA) by comparison of within- and between-unit variances across the 12 pairs. Any statistically significant result must be checked to determine whether it is also technically significant. The variation is tightly constrained and the magnitude of the effect of between-unit variation is negligible compared to typical measurement error. All *p*-values are consequently found to be insignificant, and the Null Hypothesis is therefore retained.

It is important to note that ANOVA provides a relative measure of homogeneity and that a CRM having poor absolute homogeneity can still pass these tests if the within-unit heterogeneity is large and similar across all units. Based on the statistical analysis of the results of the interlaboratory certification program, it can be concluded that OREAS 408 is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

PERFORMANCE GATES

Table 6 below shows intervals calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned (also see ‘Intended Use’ section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit www.westgard.com/mltirule.htm). A second method utilises a 5 % window calculated directly from the certified value.

Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5 % window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5 % method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL) ± 10 %.

i.e., Certified Value ±10 % ±2DL [1].

Table 6. Performance Gates for OREAS 408.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate Fusion XRF											
Al_2O_3, wt.%	0.998	0.026	0.945	1.050	0.919	1.076	2.63%	5.27%	7.90%	0.948	1.048
As, ppm	35.6	5.1	25.4	45.7	20.3	50.8	14.29%	28.58%	42.86%	33.8	37.3
CaO, wt.%	0.311	0.009	0.292	0.330	0.283	0.339	3.01%	6.02%	9.03%	0.295	0.327
Cl, ppm	207	48	111	304	62	353	23.34%	46.68%	70.02%	197	218
Fe, wt.%	63.22	0.302	62.62	63.82	62.31	64.13	0.48%	0.96%	1.43%	60.06	66.38
Fe(Calc), wt.%	63.24	0.136	62.96	63.51	62.83	63.65	0.22%	0.43%	0.65%	60.07	66.40
K_2O, wt.%	0.027	0.003	0.021	0.034	0.018	0.037	11.30%	22.60%	33.91%	0.026	0.029
MgO, wt.%	0.068	0.009	0.050	0.087	0.040	0.097	13.73%	27.47%	41.20%	0.065	0.072
Mn, wt.%	0.081	0.004	0.074	0.088	0.070	0.092	4.43%	8.87%	13.30%	0.077	0.085
P, wt.%	0.015	0.001	0.013	0.018	0.012	0.019	6.83%	13.67%	20.50%	0.015	0.016
S, wt.%	0.016	0.002	0.011	0.021	0.009	0.024	15.19%	30.38%	45.57%	0.016	0.017
SiO_2, wt.%	7.01	0.081	6.84	7.17	6.76	7.25	1.16%	2.31%	3.47%	6.66	7.36
Zn, ppm	81	9	64	99	55	108	10.64%	21.28%	31.92%	77	86

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 1: intervals may appear asymmetric due to rounding.

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 windows.

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Thermogravimetry											
LOI^{1000}, wt.%	0.985	0.111	0.764	1.207	0.653	1.318	11.24%	22.49%	33.73%	0.936	1.035
LOI^{371}, wt.%	0.352	0.030	0.293	0.412	0.263	0.442	8.46%	16.91%	25.37%	0.335	0.370
LOI^{425}, wt.%	0.414	0.034	0.346	0.481	0.312	0.515	8.18%	16.37%	24.55%	0.393	0.434
LOI^{650}, wt.%	0.727	0.033	0.662	0.793	0.629	0.825	4.49%	8.98%	13.46%	0.691	0.764
4-Acid Digestion											
Ag, ppm	0.284	0.022	0.240	0.328	0.218	0.350	7.77%	15.54%	23.31%	0.270	0.298
Al, wt.%	0.524	0.022	0.479	0.569	0.457	0.591	4.28%	8.55%	12.83%	0.498	0.550
As, ppm	30.5	1.21	28.1	32.9	26.9	34.2	3.95%	7.90%	11.85%	29.0	32.1
Ba, ppm	28.3	1.51	25.3	31.3	23.8	32.8	5.33%	10.66%	15.99%	26.9	29.7
Be, ppm	2.43	0.175	2.08	2.78	1.91	2.96	7.19%	14.38%	21.56%	2.31	2.55
Bi, ppm	1.19	0.062	1.07	1.31	1.01	1.37	5.17%	10.34%	15.52%	1.13	1.25
Ca, wt.%	0.216	0.010	0.197	0.235	0.188	0.245	4.41%	8.82%	13.23%	0.205	0.227
Ce, ppm	15.9	0.80	14.3	17.5	13.5	18.3	5.05%	10.10%	15.15%	15.1	16.7
Co, ppm	32.6	1.64	29.3	35.9	27.7	37.5	5.05%	10.10%	15.14%	31.0	34.2
Cr, ppm	34.1	4.9	24.2	44.0	19.3	49.0	14.49%	28.99%	43.48%	32.4	35.9
Cs, ppm	1.65	0.062	1.53	1.77	1.46	1.84	3.75%	7.51%	11.26%	1.57	1.73
Cu, ppm	21.2	0.91	19.4	23.0	18.5	23.9	4.28%	8.56%	12.83%	20.1	22.3
Dy, ppm	3.01	0.180	2.65	3.37	2.47	3.55	5.99%	11.97%	17.96%	2.86	3.16
Er, ppm	1.83	0.095	1.64	2.02	1.54	2.12	5.21%	10.42%	15.63%	1.74	1.92
Fe, wt.%	60.12	3.414	53.29	66.95	49.88	70.36	5.68%	11.36%	17.04%	57.11	63.13
Ga, ppm	4.40	0.335	3.73	5.07	3.39	5.40	7.61%	15.23%	22.84%	4.18	4.62
Gd, ppm	2.71	0.100	2.51	2.91	2.41	3.01	3.70%	7.40%	11.10%	2.58	2.85
Ge, ppm	0.53	0.10	0.33	0.73	0.23	0.83	18.88%	37.75%	56.63%	0.50	0.56
In, ppm	0.26	0.013	0.23	0.28	0.22	0.30	5.20%	10.40%	15.60%	0.24	0.27
K, wt.%	0.025	0.006	0.013	0.037	0.007	0.043	24.34%	48.68%	73.03%	0.024	0.026
La, ppm	6.36	0.378	5.60	7.11	5.22	7.49	5.94%	11.89%	17.83%	6.04	6.68
Li, ppm	175	12	152	198	140	210	6.64%	13.29%	19.93%	166	184
Mg, wt.%	0.039	0.007	0.025	0.054	0.018	0.061	18.06%	36.11%	54.17%	0.037	0.041
Mn, wt.%	0.074	0.006	0.063	0.085	0.057	0.091	7.61%	15.21%	22.82%	0.070	0.077
Mo, ppm	5.74	0.480	4.78	6.70	4.30	7.18	8.37%	16.74%	25.11%	5.45	6.02
Na, wt.%	0.030	0.002	0.027	0.033	0.025	0.035	5.31%	10.63%	15.94%	0.028	0.031
Nb, ppm	1.95	0.105	1.74	2.16	1.64	2.27	5.37%	10.74%	16.11%	1.85	2.05
Nd, ppm	9.86	0.373	9.11	10.61	8.74	10.98	3.78%	7.57%	11.35%	9.37	10.35
Ni, ppm	27.3	1.94	23.4	31.1	21.4	33.1	7.11%	14.22%	21.33%	25.9	28.6
P, wt.%	0.015	0.001	0.012	0.017	0.011	0.019	9.31%	18.62%	27.93%	0.014	0.015
Pb, ppm	24.0	3.0	17.9	30.0	14.9	33.0	12.60%	25.20%	37.80%	22.8	25.2
Pr, ppm	2.11	0.099	1.91	2.31	1.81	2.40	4.68%	9.35%	14.03%	2.00	2.21
Rb, ppm	3.21	0.186	2.84	3.58	2.65	3.76	5.79%	11.58%	17.37%	3.05	3.37
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.019	0.001	0.017	0.022	0.016	0.023	5.77%	11.54%	17.30%	0.019	0.020
Sb, ppm	9.42	0.760	7.90	10.94	7.14	11.70	8.07%	16.14%	24.20%	8.95	9.89
Sc, ppm	1.41	0.097	1.22	1.61	1.12	1.71	6.90%	13.80%	20.69%	1.34	1.48
Se, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 1: intervals may appear asymmetric due to rounding.

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 windows.

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion continued											
Sm, ppm	2.38	0.171	2.04	2.72	1.87	2.89	7.18%	14.35%	21.53%	2.26	2.50
Sn, ppm	12.9	0.96	11.0	14.8	10.1	15.8	7.39%	14.78%	22.17%	12.3	13.6
Sr, ppm	33.9	1.20	31.5	36.3	30.3	37.5	3.54%	7.08%	10.62%	32.2	35.6
Ta, ppm	3.76	0.59	2.57	4.94	1.97	5.54	15.82%	31.64%	47.46%	3.57	3.94
Te, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	0.81	0.044	0.72	0.90	0.68	0.94	5.39%	10.77%	16.16%	0.77	0.85
Ti, wt.%	0.010	0.001	0.009	0.011	0.008	0.012	5.92%	11.83%	17.75%	0.010	0.011
Tl, ppm	0.18	0.009	0.16	0.20	0.15	0.21	5.21%	10.42%	15.63%	0.17	0.19
U, ppm	3.21	0.136	2.94	3.48	2.80	3.61	4.23%	8.45%	12.68%	3.05	3.37
V, ppm	15.4	2.6	10.2	20.5	7.7	23.0	16.67%	33.33%	50.00%	14.6	16.1
W, ppm	19.1	1.82	15.5	22.7	13.6	24.6	9.52%	19.05%	28.57%	18.1	20.1
Y, ppm	18.4	0.68	17.1	19.8	16.4	20.4	3.67%	7.35%	11.02%	17.5	19.3
Zn, ppm	73	4.0	65	81	61	85	5.44%	10.87%	16.31%	69	77
Zr, ppm	7.37	0.202	6.96	7.77	6.76	7.97	2.74%	5.47%	8.21%	7.00	7.73
Classical Wet Chemistry											
FeO, wt.%	1.13	0.18	0.77	1.49	0.59	1.67	15.88%	31.75%	47.63%	1.07	1.19
Satmagan 135											
Fe_3O_4, wt.%	1.98	0.26	1.45	2.51	1.19	2.77	13.33%	26.66%	39.99%	1.88	2.08
Gas / Liquid Pycnometry											
SG, Unity	4.76	0.175	4.41	5.11	4.23	5.28	3.68%	7.36%	11.04%	4.52	4.99

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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 windows.

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. African Natural Resources & Mines Ltd, Suleja, Niger State, Nigeria
3. AGAT Laboratories, Calgary, Alberta, Canada
4. ALS, Brisbane, QLD, Australia
5. ALS, Lima, Peru
6. ALS, Loughrea, Galway, Ireland
7. ALS, Malaga, WA, Australia
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
11. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
12. Bureau Veritas Geoanalytical, Perth, WA, Australia
13. Bureau Veritas MET, Whyalla, SA, Australia
14. Inspectorate Griffith India, Gandhidham, Gujarat, India
15. Inspectorate Griffith India Pvt. Ltd., Bhubaneswar, Odisha, India
16. Intertek, Cupang, Muntinlupa, Philippines
17. Intertek, Perth, WA, Australia
18. MSALABS, Vancouver, BC, Canada
19. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
20. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
21. SGS Australia Mineral Services, Perth, WA, Australia
22. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
23. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
24. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

Please note: To preserve anonymity, the above numbered alphabetical list of participating laboratories does not correspond with the Lab ID numbering on the scatter plots below.

PREPARER AND SUPPLIER

Certified reference material OREAS 408 is prepared and certified by:

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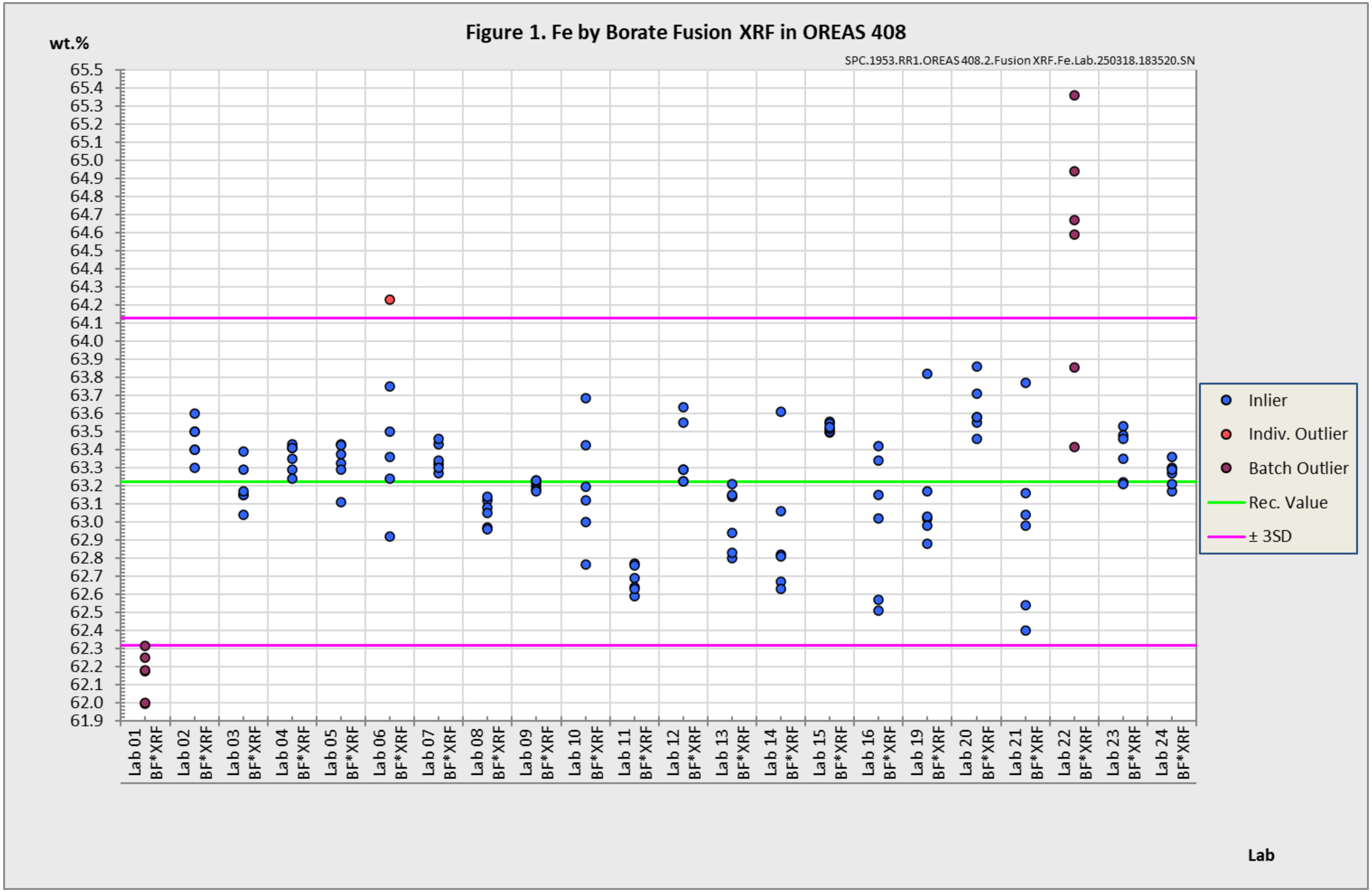

Figure 1. Fe by Borate Fusion XRF in OREAS 408

METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified values are metrologically traceable to the international measurement scale (SI) of mass (either as a % mass fraction or as milligrams per kilogram (mg/kg)) [14]. In line with popular use, all data within tables in this certificate are expressed as the mass fraction in either weight percent (wt.%) or parts per million (ppm).

The analytical samples sent to participating laboratories were selected in a manner to be representative of the entire prepared batch of CRM. This representativeness was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [8], each analytical data set received from the participating laboratories has been validated by its assayer through the inclusion of internal reference materials and QC checks during and post analysis.

The participating laboratories were chosen on the basis of their competence (from past performance in interlaboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. These laboratories are accredited to ISO 17025 for borate fusion with XRF, thermogravimetry and 4-acid digestion (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016 [7], section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *“Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation.* ***In other cases, only a comparison among different laboratories using the same procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand*** *(ISO Guide 33405:2024-05, 9.2.4c) [4].”* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (fusion/digestion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine ‘field’ samples in the relevant measurement process. Care should be taken to ensure ‘matrix matching’ as close as practically achievable. The matrix and mineralisation style of the CRM is described in the ‘Source Material’ section and users should select appropriate CRMs matching these attributes to the field samples being analysed.

INTENDED USE

OREAS 408 is intended to cover all activities needed to produce a measurement result. This includes extraction, possible separation steps and the actual measurement process (the signal producing step). OREAS 408 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 408 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 and 2 in geological samples;
- For the validation/ verification of analytical methods for analytes reported in Tables 1 and 2;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 and 2. When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. Users can determine an approximation of the standard uncertainty by calculating one fourth of the width of the Expanded Uncertainty interval given in this certificate (Expanded Uncertainty intervals are provided in Tables 1 and 2).

MINIMUM SAMPLE SIZE

To relate analytical determinations to the values in this certificate, the minimum mass of sample used should match the typical mass that the laboratories used in the interlaboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Borate fusion with X-ray fluorescence finish: ≥0.2 g;
- Loss on Ignition (LOI) by multi-point TGA: ≥1 g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish: ≥0.25 g;
- FeO by classical wet chemistry ≥ 1.0 g;
- Magnetic Susceptibility by Satmagan 135 ≥ 1.0 g;
- Davis Tube Recovery (DTR) ≥ 20 g;
- Specific gravity by pycnometer: ≥3.0 g.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 408 remains valid, within the specified measurement uncertainties, until at least August 2039, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

Store in a clean and cool dry place away from direct sunlight.

Long-term stability will be monitored at appropriate intervals and purchasers notified if any changes are observed. The period of validity may well be indefinite and will be reassessed prior to expiry with the aim of extending the validity if possible.

Single-use sachets

OREAS 408 is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

Repeat-use packaging (e.g., 1 kg plastic jars)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 408 contains a non-hygroscopic* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (~0.02 wt.% S).

*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorbed moisture (weakly held H_2O- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2mm thick) layer and left exposed for a period of 2 hours.

INSTRUCTIONS FOR HANDLING & CORRECT USE

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [12].

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised.

With the exception of the borate fusion with XRF (where the certified values are reported on a dry sample basis), all analyses were performed on the samples as received and the certified values are reported as such in line with conventional instrumental method procedures.

QC monitoring using multiples of the Standard Deviation (SD)

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include interlaboratory bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

The performance gates shown in Table 6 are intended only to be used as a preliminary guide as to what a laboratory may be able to achieve. Over a period of time monitoring your own laboratory's data for this CRM, SD's should be calculated directly from your own laboratory's process. This will enable you to establish more specific performance gates that are fit for purpose for your application as well as the ability to monitor bias. If your long-term trend analysis shows an average value that is within the 95 % expanded uncertainty then generally there is no cause for concern in regard to bias.

LEGAL NOTICE

Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the use of this material and information.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	14th April, 2025	First publication.

CERTIFYING OFFICER

Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager - ORE P/L

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016 (Accreditation number 20483).

ORE Pty Ltd is ISO 9001:2015 certified by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.

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[4] ISO 33405:2024-05. Reference materials – Approaches for characterization and assessment of homogeneity and stability.

[5] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[6] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.

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[11] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.

[12] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.

[13] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.

[14] Thompson, A.; Taylor, B.N. (2008); Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Nov 2021).

[15] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.

APPENDIX

*Davis Tube Recovery (preferred) settings:

1. Stroke frequency 60/minute or 90/minute.
2. Stroke length 38mm.
3. Magnetic field strength 3000 gauss.
4. Tube angle 45 degrees.
5. Tube diameter 38mm.
6. Washing time 20 minutes or until clear.