

CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

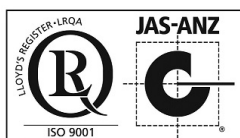
OREAS 452

Ferruginous Nickel-Cobalt Laterite Ore

Bulong Deposit, Western Australia



Accredited for compliance with ISO 17034



COA-2079-OREAS 452-R0

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Table 1. Certified Values, Uncertainty & Tolerance Intervals in OREAS 452.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
Al ₂ O ₃ , Aluminium(III) oxide (wt.%)	8.35	8.28	8.41	8.29	8.40
CaO, Calcium oxide (wt.%)	0.772	0.760	0.785	0.763	0.782
Co, Cobalt (ppm)	805	791	819	794	816
Cr ₂ O ₃ , Chromium(III) oxide (wt.%)	1.02	1.01	1.04	1.01	1.03
Cu, Copper (ppm)	107	92	123	IND	IND
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	34.34	34.09	34.59	34.20	34.48
K ₂ O, Potassium oxide (wt.%)	0.200	0.189	0.211	0.193	0.207
MgO, Magnesium oxide (wt.%)	3.68	3.63	3.72	3.65	3.70
MnO, Manganese oxide (wt.%)	0.303	0.297	0.308	0.299	0.306
Na ₂ O, Sodium oxide (wt.%)	0.888	0.852	0.924	0.872	0.904
Ni, Nickel (wt.%)	1.06	1.05	1.07	1.05	1.07
P ₂ O ₅ , Phosphorus(V) oxide (wt.%)	0.020	0.018	0.022	0.019	0.021
Pb, Lead (ppm)	< 50	IND	IND	IND	IND
SiO ₂ , Silicon dioxide (wt.%)	39.27	38.97	39.56	39.09	39.44
SO ₃ , Sulphur trioxide (wt.%)	0.336	0.305	0.367	0.326	0.346
TiO ₂ , Titanium dioxide (wt.%)	0.343	0.329	0.356	0.334	0.352
V ₂ O ₅ , Vanadium(V) oxide (ppm)	188	126	250	IND	IND
Zn, Zinc (ppm)	153	142	164	IND	IND
Thermogravimetry					
LOI ¹⁰⁰⁰ , Loss on ignition @1000 °C (wt.%)	9.22	8.98	9.47	9.15	9.30
Borate / Peroxide Fusion ICP					
Al, Aluminium (wt.%)	4.19	4.08	4.29	4.14	4.24
B, Boron (ppm)	55	43	67	49	60
Ba, Barium (ppm)	133	127	139	129	138
Be, Beryllium (ppm)	< 1	IND	IND	IND	IND
Ca, Calcium (wt.%)	0.535	0.485	0.585	0.516	0.554
Cd, Cadmium (ppm)	< 10	IND	IND	IND	IND
Ce, Cerium (ppm)	18.2	16.9	19.5	17.8	18.6
Co, Cobalt (ppm)	792	770	815	780	805
Cr, Chromium (wt.%)	0.673	0.649	0.696	0.663	0.682
Cs, Caesium (ppm)	0.48	0.36	0.61	0.44	0.53
Cu, Copper (ppm)	103	96	111	100	107
Dy, Dysprosium (ppm)	1.28	1.16	1.41	1.21	1.36
Er, Erbium (ppm)	0.74	0.65	0.83	0.68	0.79
Eu, Europium (ppm)	0.39	0.34	0.44	0.37	0.41
Fe, Iron (wt.%)	22.69	22.09	23.28	22.47	22.91
Ga, Gallium (ppm)	8.22	7.38	9.05	7.78	8.65
Gd, Gadolinium (ppm)	1.34	1.25	1.43	1.27	1.42

SI unit equivalents: ppm (parts per million; 1×10^{-6}) $\equiv$ mg/kg; wt.% (weight per cent) $\equiv$ % (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 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued					
Ho, Holmium (ppm)	0.25	0.22	0.29	0.24	0.27
In, Indium (ppm)	< 0.1	IND	IND	IND	IND
K, Potassium (wt.%)	0.167	0.143	0.191	0.153	0.181
La, Lanthanum (ppm)	4.34	4.07	4.61	4.19	4.49
Li, Lithium (ppm)	7.47	6.01	8.92	7.06	7.87
Lu, Lutetium (ppm)	0.12	0.09	0.14	IND	IND
Mg, Magnesium (wt.%)	2.10	2.03	2.16	2.06	2.13
Mn, Manganese (wt.%)	0.222	0.215	0.229	0.218	0.225
Mo, Molybdenum (ppm)	3.87	3.21	4.53	IND	IND
Nd, Neodymium (ppm)	5.08	4.65	5.52	4.91	5.25
Ni, Nickel (wt.%)	1.01	0.97	1.04	0.99	1.02
P, Phosphorus (wt.%)	0.010	0.008	0.011	0.009	0.010
Pb, Lead (ppm)	< 100	IND	IND	IND	IND
Pr, Praseodymium (ppm)	1.29	1.18	1.40	1.24	1.34
Rb, Rubidium (ppm)	6.19	5.19	7.19	5.58	6.81
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND
S, Sulphur (wt.%)	0.138	0.128	0.149	IND	IND
Sb, Antimony (ppm)	0.83	0.55	1.10	IND	IND
Sc, Scandium (ppm)	28.5	26.1	30.9	27.4	29.6
Se, Selenium (ppm)	< 20	IND	IND	IND	IND
Si, Silicon (wt.%)	17.90	17.36	18.43	17.63	18.16
Sm, Samarium (ppm)	1.31	1.18	1.44	1.23	1.39
Sr, Strontium (ppm)	34.9	32.2	37.7	33.4	36.4
Tb, Terbium (ppm)	0.21	0.19	0.23	0.20	0.22
Te, Tellurium (ppm)	< 1	IND	IND	IND	IND
Th, Thorium (ppm)	1.18	1.09	1.27	1.11	1.24
Ti, Titanium (wt.%)	0.199	0.191	0.207	0.196	0.202
Tm, Thulium (ppm)	0.11	0.09	0.12	IND	IND
U, Uranium (ppm)	0.54	0.49	0.58	0.49	0.58
V, Vanadium (ppm)	105	100	111	100	111
W, Tungsten (ppm)	2.55	1.76	3.34	2.25	2.85
Y, Yttrium (ppm)	5.00	4.52	5.47	4.79	5.21
Yb, Ytterbium (ppm)	0.80	0.71	0.89	0.76	0.84
Zn, Zinc (ppm)	156	145	166	150	161
Zr, Zirconium (ppm)	39.4	32.9	45.9	37.3	41.6
Infrared Combustion					
C, Carbon (wt.%)	0.186	0.166	0.206	0.175	0.198
S, Sulphur (wt.%)	0.124	0.110	0.137	0.117	0.130

SI unit equivalents: ppm (parts per million; 1×10^{-6}) $\equiv$ mg/kg; wt.% (weight per cent) $\equiv$ % (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 2. Indicative Values for in OREAS 452.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
As	ppm	16.7	Ge	ppm	< 100	Se	ppm	< 100
BaO	ppm	158	Hf	ppm	< 80	Sn	ppm	< 100
Bi	ppm	< 100	Hg	ppm	< 100	SrO	ppm	< 100
C	wt. %	0.100	In	ppm	< 100	Ta	ppm	< 100
Cd	ppm	< 100	Mo	ppm	< 100	Te	ppm	< 100
Cl	ppm	1591	Rb	ppm	< 100	Tl	ppm	< 100
Cs	ppm	< 100	Sb	ppm	< 100	W	ppm	< 100
Ga	ppm	< 100	Sc	ppm	20.0	ZrO ₂	ppm	72
Thermogravimetry								
H ₂ O-	wt. %	6.99						
Borate / Peroxide Fusion ICP								
Ag	ppm	< 10	Hf	ppm	0.98	Ta	ppm	0.20
As	ppm	7.66	Na	wt. %	0.551	Tl	ppm	< 0.5
Bi	ppm	0.18	Nb	ppm	2.24			
Ge	ppm	1.43	Sn	ppm	1.31			

SI unit equivalents: ppm (parts per million; 1×10^{-6}) $\equiv$ mg/kg; wt. % (weight per cent) $\equiv$ % (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 handling and correct use' should be read carefully.

Table 1 provides the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 2 shows indicative values including major and trace element characterisation, Table 3 provides some indicative physical properties, Table 4 shows indicative mineralogy by semi-quantitative XRD analysis and Table 5 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 (PDM³) are presented in the detailed certification data for this CRM (**OREAS 452-DataPack.1.0.260826_171146.xlsx**). The certified values and uncertainties in this Certificate are the sole authoritative figures. Any additional significant figures in the DataPack are provided for reference only and do not affect the certified results.

Results are also presented in scatter plots for Ni and Co by fusion XRF in Figures 1 to 2 respectively, together with $\pm 3SD$ (magenta) and $\pm 5\%$ (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.

INTENDED USE

OREAS 452 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 452 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 452 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological samples
- For the verification/ validation of analytical methods for analytes reported in Table 1
- For the calibration of instruments used in the determination of the concentration of analytes reported in Table 1. 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 Table 1)

SOURCE MATERIAL

OREAS 452 was prepared predominantly from nontronitic nickel-cobalt laterite ore sourced from Bulong, Western Australia, with minor additions of weathered basaltic material and other lateritic and saprolitic nickel materials to achieve the target nickel, cobalt and matrix composition.

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) at 1000 °C: ≥ 1 g
- Borate fusion /Sodium peroxide with ICP-OES and/or MS finish: ≥ 0.2 g
- C and S by infrared combustion furnace/CS analyser: ≥ 0.1 g

INSTRUCTIONS FOR HANDLING, STORAGE & CORRECT USE

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

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [11].

As per routine analysis at commercial laboratories, the certified values derived by borate fusion with XRF finish are on a dry sample basis.

Analytes by all other methods refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis for these methods.

Single-use Sachets

OREAS 452 is packaged in single-use, 10 g 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., 500 g 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 452 contains a 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 3 in this certificate.

Authoritative Source of Information

This Certificate of Analysis constitutes the primary and authoritative document for the certified values, associated expanded uncertainties, and their correct use. While the accompanying DataPack provides supporting information, including raw data and uncertainty estimates with additional significant figures, these extended figures are provided solely for transparency, convenience and statistical reference. Users must rely exclusively on the values stated in this Certificate, rounded to an appropriate number of significant figures, for all metrological and analytical purposes. Any discrepancy between values presented in the DataPack and those in this Certificate shall be resolved in favour of the information provided herein.

Notice on Certificate Updates

The version of the Certificate of Analysis (COA) available on the OREAS website is considered the official and most current version. As COAs may be revised following periodic reviews, re-evaluation of data, or the availability of new information, users are strongly advised to refer to the latest online version prior to each use.

It is the user's responsibility to ensure that the most recent and applicable certificate is used to support the traceability, validity, and fitness-for-purpose of the certified reference material (CRM). Any significant changes to the sections of this certificate will be clearly documented in the revised certificate.

QC Monitoring Using Multiples of the Standard Deviation (SD)

When applying SDs to monitor performance, it is important to recognise that laboratories differ in proficiency, and that different methods have differing levels of precision. Each laboratory has its own inherent SD (specific to an analyte–method–concentration combination), which is not directly comparable to SDs derived from a round robin.

As most data in this round robin came from world-class laboratories, the interlaboratory SDs are narrower than would be expected across a broader mix. To provide more realistic benchmarks, this report presents pooled interlaboratory SDs, incorporating both within-lab variation and between-lab bias. These should be treated as a starting point only, with QC managers refining them against their own control charts.

The performance gates shown in Table 5 are intended only to be used as an initial 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, SDs 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 differs from the certified value, the significance of this bias should be assessed against the combined standard uncertainty of the certified value and the laboratory's own measurement uncertainty. Where the observed difference lies within this combined uncertainty, the bias is generally not considered significant.

PERIOD OF VALIDITY

The certification of OREAS 452 remains valid, within the specified measurement uncertainties, until at least February 2038, 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.

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 452 was prepared in the following manner:

- Drying to constant mass at 105 °C
- Crushing and multi-stage milling of the ores to 100 % minus 30 microns
- Crushing and multi-stage milling of the weathered basalt to 98 % minus 75 microns
- Blending the ores and weathered basalt in appropriate proportions to achieve desired grades
- Homogenisation using OREAS' novel processing technologies
- Packaging in 10 g units in laminated foil pouches and 500 g units in plastic wide-mouth jars

PHYSICAL PROPERTIES

OREAS 452 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

Table 3. Physical properties of in OREAS 452.

Bulk Density (kg/m ³)	Moisture (wt.%)	Munsell Notation [‡]	Munsell Color [‡]
780	4.88	5YR 5/6	Light 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 4 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.

'Clay minerals' appear to be mainly vermicullite, smectite, and/or illite. 'Kandite group' appears to be mainly kaolinite. A trevorite reference pattern was used to refine and report the 'Fe or Cr spinel' phase. A trace of calcite may be present.

Table 4. Indicative mineralogy of in OREAS 452 by semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Fe or Cr spinels	3
Goethite	43
Clay minerals	27
Serpentine	10
Kandite group	6
Clinopyroxene	2
Plagioclase	6
Quartz	2

ANALYTICAL PROGRAM

Twenty-eight commercial analytical laboratories participated in the program to certify the elements reported in Table 1. The following methods were employed:

- Lithium borate fusion whole rock analysis package by X-ray fluorescence (up to 24 laboratories depending on the element)
- Thermogravimetry: Loss on Ignition (LOI) at 1000 °C (12 laboratories used a thermogravimetric analyser, 7 laboratories included LOI with their fusion package and 6 laboratories used a conventional muffle furnace)
- Lithium borate or sodium peroxide fusion with full suite ICP-OES and ICP-MS elemental packages (up to 24 laboratories depending on the element)
- Total C and S by IR combustion furnace (24 laboratories)

For the round robin program, six 1 kg test units were collected at predetermined intervals during the bagging stage, immediately after homogenisation. These units are considered representative of the entire prepared batch. Each participating laboratory received six test portions, obtained by subsampling 10 g from each of the six distinct 1 kg units.

Homogeneity was assessed by submitting 12 pulp samples to a single laboratory for analysis. Paired samples were drawn from each of the six test units, enabling an Analysis of Variance (ANOVA) to compare within-unit and between-unit variances. This statistical method provides a relative measure of homogeneity and tests the null hypothesis that all units derive from the same population distribution (refer to the 'Homogeneity Evaluation' section below).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. Alex Stewart International, Mendoza, Argentina
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, Perth, WA, Australia
12. Directorate of Mineral Research & Exploration, Ankara, Ankara, Turkey
13. ESAN Istanbul, Istanbul, Turkey
14. Inspectorate (BV), Lima, Peru
15. Inspectorate Griffith India, Gandhidham, Gujarat, India
16. Inspectorate Griffith India Pvt. Ltd., Bhubaneswar, Odisha, India
17. Intertek, Cupang, Muntinlupa, Philippines
18. Intertek, Perth, WA, Australia
19. Laboratoire LABOMINE SARL, Agadir, Souss-Massa, Morocco
20. Nagrom, Perth, WA, Australia
21. Ni Lab, Pouembout, New Caledonia
22. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
23. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
24. SGS Australia Mineral Services, Perth, WA, Australia
25. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
26. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
27. Société le Nickel (SLN), Noumea, New Caledonia
28. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

Please note: To maintain anonymity of participating laboratories, the alphabetical list above does not correspond to the Lab ID numbers shown in the scatter plots below.

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). Outlier evaluation was conducted in accordance with ISO 17034:2016 and ISO 33405:2024. While formal statistical tests were applied, professional statistical judgment was also exercised in determining the validity of potential outliers. Assessment of systematic bias and performance using independent control materials (CRMs) was incorporated to ensure compliance with the referenced standards and to establish metrological traceability of the certified values.

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 [14]. All known or suspected sources of bias have been investigated or taken into account.

Indicative (uncertified) values (Table 2) 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 5, '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) shown in Table 1 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 Ni by fusion XRF, where 99 % of the time ($1-\alpha=0.99$) at least 95 % of subsamples ($p=0.95$) will have concentrations lying between 1.05 and 1.07 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. ***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 precision error method outlined above, homogeneity was also evaluated using an ANOVA study. This involved sending 12 × 10 g pulp samples to the Bureau Veritas Geoanalytical (Perth, Australia) laboratory for analysis by lithium borate fusion with X-ray fluorescence finish (code XF201). The 12 samples consisted of paired samples from each of the six sampling units to enable an Analysis of Variance (ANOVA) by comparison of within- and between-unit variances across the six pairs. The samples were randomised prior to submission to minimise potential analytical sequence effects. The 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. An ANOVA constructed in this way 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 % confidence level (i.e., a significant difference likely does exist; meaning there is evidence of heterogeneity between the sample intervals).

Of the thirteen analytes evaluated by ANOVA, only Mn returned a statistically significant result ($p = 0.027$). However, the Mn data showed very low dispersion ($RSD = 0.22\%$), with XRF results ranging only from 0.232% to 0.234% and reported at 0.001% increments. This limited resolution reduces the ability to distinguish minor analytical variation from true between-unit variation. Round robin Mn results by fusion XRF and fusion-ICP also showed no systematic trend with sampling interval. The Mn result is therefore considered a statistical anomaly rather than evidence of meaningful heterogeneity. All other ANOVA results were non-significant. 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 452 is fit-for-purpose as a certified reference material (see 'Intended Use').

METROLOGICAL TRACEABILITY

The measurement scale for the certified values is mass fraction (mass/mass), with the underlying mass measurements metrologically traceable to the International System of Units (SI) through the SI unit of mass [13]. In accordance with common geoanalytical practice, data presented in the tables of this certificate are expressed as mass fractions in either weight percent (wt. %) or parts per million (ppm), with ppm equivalent to milligrams per kilogram (mg/kg).

Samples submitted to the interlaboratory certification program were selected to represent the entire CRM batch, with this representativeness maintained across laboratory sample sets. Each dataset was subject to the participating laboratory's internal quality-control procedures, including reference materials and other QC checks.

Participating laboratories were selected based on demonstrated analytical competence, including prior performance in ORE Pty Ltd interlaboratory comparison programs, and expertise in relevant methods, measurands and matrices. For measurands reported in Table 1, data were obtained from ISO/IEC 17025 accredited laboratories where applicable. Where accreditation was not held for a specific measurand, analytical competence and metrological traceability were supported using well-characterised, independently certified reference materials as control samples in the round robin study.

Consistent with ISO 33405:2024 [4], clause 9.2.5, and ISO 17034:2016 [8], clause 7.12.4, metrological traceability and validity of characterisation results were evaluated using appropriate supporting evidence. Agreement of control-sample results with certified values provided additional evidence of the validity of the results. Certified values were assigned from the means of accepted laboratory datasets using the statistical procedures described in this report.

Operationally Defined Measurands

The certified values assigned to OREAS reference materials are operationally defined. The value obtained is therefore dependent, to some degree, on the measurement approach and conditions under which the measurand is realised and cannot necessarily be defined independently of them. Accordingly, while the mass-fraction measurement scale is underpinned by mass measurements traceable to the SI, metrological traceability of the certified value is established with reference to the applicable operationally defined measurand.

In accordance with ISO 33405:2024 [4], clause 9.5, characterisation is based on results obtained from a network of competent laboratories working within the applicable operational definition, with concordance of the results assessed as part of the value-assignment process. Individual laboratory implementations may vary, and the resulting interlaboratory variation reflects, in part, differences inherent in the practical realisation of the measurand.

The certified value is therefore applicable to measurements consistent with the stated operational definition. Relevant interlaboratory variation is incorporated, as appropriate, into the uncertainty associated with the certified value. This approach provides the basis for metrological traceability appropriate to operationally defined measurands and supports the requirements of ISO 17034:2016 and ISO 33405:2024.

COMMUTABILITY

The certified values reported herein are derived from measurements performed using analytical methods involving sample pre-treatment steps, such as fusion or acid digestion. These processes convert the sample matrix into a chemically simplified and stable form, facilitating calibration traceable to primary standards via solution-based calibration protocols. Due to the established robustness and effectiveness of these pre-treatment methods, issues related to commutability are not expected to impact the suitability of this Certified Reference Material (CRM) for its intended use.

OREAS CRMs are prepared from natural ore materials, ensuring the presence of matrix and mineralogical characteristics representative of typical exploration and process samples. Consistent with ISO 17034:2016 and ISO Guide 30, users are advised to select CRMs with matrix and mineralisation styles closely matching those of their routine samples to minimize matrix effects and enhance analytical comparability. Detailed descriptions of the CRM's source material and mineralogical characteristics are provided in the 'Source Material' section to guide appropriate CRM selection.

PERFORMANCE GATES

Table 5 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 % $\pm 2DL$ [1].

Table 5. Performance Gates for OREAS 452.

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 ₂ O ₃ , wt. %	8.35	0.062	8.22	8.47	8.16	8.53	0.75%	1.49%	2.24%	7.93	8.76
CaO, wt. %	0.772	0.018	0.737	0.808	0.720	0.825	2.27%	4.54%	6.80%	0.734	0.811
Co, ppm	805	14	776	834	762	849	1.80%	3.60%	5.40%	765	845
Cr ₂ O ₃ , wt. %	1.02	0.022	0.98	1.07	0.96	1.09	2.19%	4.39%	6.58%	0.97	1.07
Cu, ppm	107	19	69	145	50	164	17.70%	35.40%	53.10%	102	113
Fe ₂ O ₃ , wt. %	34.34	0.217	33.91	34.77	33.69	34.99	0.63%	1.26%	1.89%	32.62	36.06
K ₂ O, wt. %	0.200	0.012	0.175	0.225	0.163	0.237	6.17%	12.34%	18.52%	0.190	0.210
MgO, wt. %	3.68	0.063	3.55	3.80	3.48	3.87	1.73%	3.45%	5.18%	3.49	3.86
MnO, wt. %	0.303	0.007	0.289	0.316	0.283	0.322	2.18%	4.37%	6.55%	0.287	0.318
Na ₂ O, wt. %	0.888	0.057	0.774	1.002	0.717	1.059	6.42%	12.84%	19.26%	0.844	0.932
Ni, wt. %	1.06	0.011	1.03	1.08	1.02	1.09	1.08%	2.16%	3.25%	1.00	1.11
P ₂ O ₅ , wt. %	0.020	0.001	0.017	0.023	0.016	0.024	6.70%	13.41%	20.11%	0.019	0.021
Pb, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
SiO ₂ , wt. %	39.27	0.252	38.76	39.77	38.51	40.02	0.64%	1.29%	1.93%	37.30	41.23
SO ₃ , wt. %	0.336	0.035	0.265	0.407	0.230	0.442	10.56%	21.11%	31.67%	0.319	0.353
TiO ₂ , wt. %	0.343	0.013	0.316	0.369	0.303	0.383	3.90%	7.79%	11.69%	0.326	0.360
V ₂ O ₅ , ppm	188	32	124	252	92	284	17.06%	34.12%	51.19%	179	197
Zn, ppm	153	11	130	176	119	188	7.50%	15.00%	22.50%	146	161
Thermogravimetry											
LOI ¹⁰⁰⁰ , wt. %	9.22	0.395	8.43	10.01	8.04	10.41	4.28%	8.56%	12.85%	8.76	9.69
Borate / Peroxide Fusion ICP											
Al, wt. %	4.19	0.153	3.88	4.49	3.73	4.65	3.65%	7.31%	10.96%	3.98	4.40
B, ppm	55	8	38	71	30	80	15.13%	30.26%	45.39%	52	58
Ba, ppm	133	7	119	148	112	155	5.37%	10.74%	16.11%	127	140
Be, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ca, wt. %	0.535	0.078	0.380	0.690	0.302	0.768	14.53%	29.06%	43.59%	0.508	0.562
Cd, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	18.2	1.14	15.9	20.5	14.8	21.6	6.29%	12.59%	18.88%	17.3	19.1
Co, ppm	792	36	719	865	683	902	4.61%	9.22%	13.82%	752	832
Cr, wt. %	0.673	0.036	0.600	0.745	0.563	0.782	5.42%	10.83%	16.25%	0.639	0.706
Cs, ppm	0.48	0.07	0.34	0.62	0.27	0.69	14.54%	29.08%	43.62%	0.46	0.51
Cu, ppm	103	9	85	122	76	131	8.95%	17.89%	26.84%	98	109
Dy, ppm	1.28	0.093	1.10	1.47	1.00	1.56	7.28%	14.56%	21.84%	1.22	1.35
Er, ppm	0.74	0.09	0.57	0.91	0.48	0.99	11.58%	23.17%	34.75%	0.70	0.78
Eu, ppm	0.39	0.032	0.33	0.46	0.30	0.49	8.12%	16.25%	24.37%	0.37	0.41
Fe, wt. %	22.69	0.871	20.94	24.43	20.07	25.30	3.84%	7.68%	11.52%	21.55	23.82
Ga, ppm	8.22	0.736	6.75	9.69	6.01	10.43	8.95%	17.91%	26.86%	7.81	8.63
Gd, ppm	1.34	0.072	1.20	1.49	1.13	1.56	5.36%	10.73%	16.09%	1.28	1.41
Ho, ppm	0.25	0.03	0.20	0.31	0.18	0.33	10.03%	20.06%	30.09%	0.24	0.27
In, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt. %	0.167	0.022	0.124	0.211	0.102	0.233	13.04%	26.08%	39.13%	0.159	0.176
La, ppm	4.34	0.306	3.73	4.95	3.42	5.26	7.06%	14.12%	21.18%	4.12	4.56
Li, ppm	7.47	1.07	5.32	9.61	4.25	10.68	14.35%	28.71%	43.06%	7.09	7.84

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

Table 5 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
Borate / Peroxide Fusion ICP continued											
Lu, ppm	0.12	0.01	0.09	0.14	0.08	0.16	10.61%	21.22%	31.82%	0.11	0.12
Mg, wt. %	2.10	0.071	1.95	2.24	1.88	2.31	3.41%	6.81%	10.22%	1.99	2.20
Mn, wt. %	0.222	0.011	0.200	0.244	0.189	0.255	4.97%	9.93%	14.90%	0.211	0.233
Mo, ppm	3.87	0.62	2.64	5.10	2.03	5.72	15.89%	31.77%	47.66%	3.68	4.07
Nd, ppm	5.08	0.404	4.28	5.89	3.87	6.29	7.95%	15.89%	23.84%	4.83	5.34
Ni, wt. %	1.01	0.061	0.88	1.13	0.82	1.19	6.07%	12.13%	18.20%	0.96	1.06
P, wt. %	0.010	0.002	0.006	0.013	0.004	0.015	18.91%	37.81%	56.72%	0.009	0.010
Pb, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pr, ppm	1.29	0.112	1.06	1.51	0.95	1.63	8.72%	17.44%	26.16%	1.22	1.35
Rb, ppm	6.19	0.91	4.38	8.00	3.48	8.91	14.62%	29.24%	43.86%	5.88	6.50
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	0.138	0.011	0.116	0.160	0.105	0.171	7.94%	15.88%	23.82%	0.131	0.145
Sb, ppm	0.83	0.15	0.52	1.13	0.37	1.29	18.48%	36.97%	55.45%	0.79	0.87
Sc, ppm	28.5	3.4	21.8	35.2	18.4	38.6	11.80%	23.60%	35.40%	27.1	29.9
Se, ppm	< 20	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Si, wt. %	17.90	0.617	16.66	19.13	16.04	19.75	3.45%	6.89%	10.34%	17.00	18.79
Sm, ppm	1.31	0.15	1.01	1.61	0.86	1.75	11.34%	22.68%	34.02%	1.24	1.37
Sr, ppm	34.9	3.27	28.4	41.5	25.1	44.7	9.35%	18.71%	28.06%	33.2	36.7
Tb, ppm	0.21	0.017	0.18	0.24	0.16	0.26	7.82%	15.65%	23.47%	0.20	0.22
Te, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	1.18	0.110	0.96	1.40	0.85	1.51	9.38%	18.75%	28.13%	1.12	1.23
Ti, wt. %	0.199	0.009	0.182	0.216	0.174	0.225	4.27%	8.54%	12.81%	0.189	0.209
Tm, ppm	0.11	0.01	0.08	0.13	0.07	0.14	11.24%	22.48%	33.71%	0.10	0.11
U, ppm	0.54	0.049	0.44	0.63	0.39	0.68	9.11%	18.22%	27.34%	0.51	0.56
V, ppm	105	6	94	117	89	122	5.24%	10.49%	15.73%	100	111
W, ppm	2.55	0.51	1.52	3.58	1.00	4.09	20.20%	40.39%	60.59%	2.42	2.68
Y, ppm	5.00	0.244	4.51	5.49	4.27	5.73	4.88%	9.76%	14.65%	4.75	5.25
Yb, ppm	0.80	0.064	0.67	0.93	0.61	0.99	8.05%	16.11%	24.16%	0.76	0.84
Zn, ppm	156	14	129	183	115	196	8.70%	17.41%	26.11%	148	163
Zr, ppm	39.4	5.1	29.2	49.7	24.1	54.8	12.96%	25.93%	38.89%	37.5	41.4
Infrared Combustion											
C, wt. %	0.186	0.035	0.117	0.256	0.082	0.290	18.59%	37.19%	55.78%	0.177	0.196
S, wt. %	0.124	0.016	0.092	0.155	0.076	0.171	12.86%	25.72%	38.58%	0.117	0.130

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

Figure 1. Ni by Borate Fusion XRF in OREAS 452

SPC.2079.RR.OREAS 452.2.Fusion XRF.Ni.Lab.260818.132017.SS

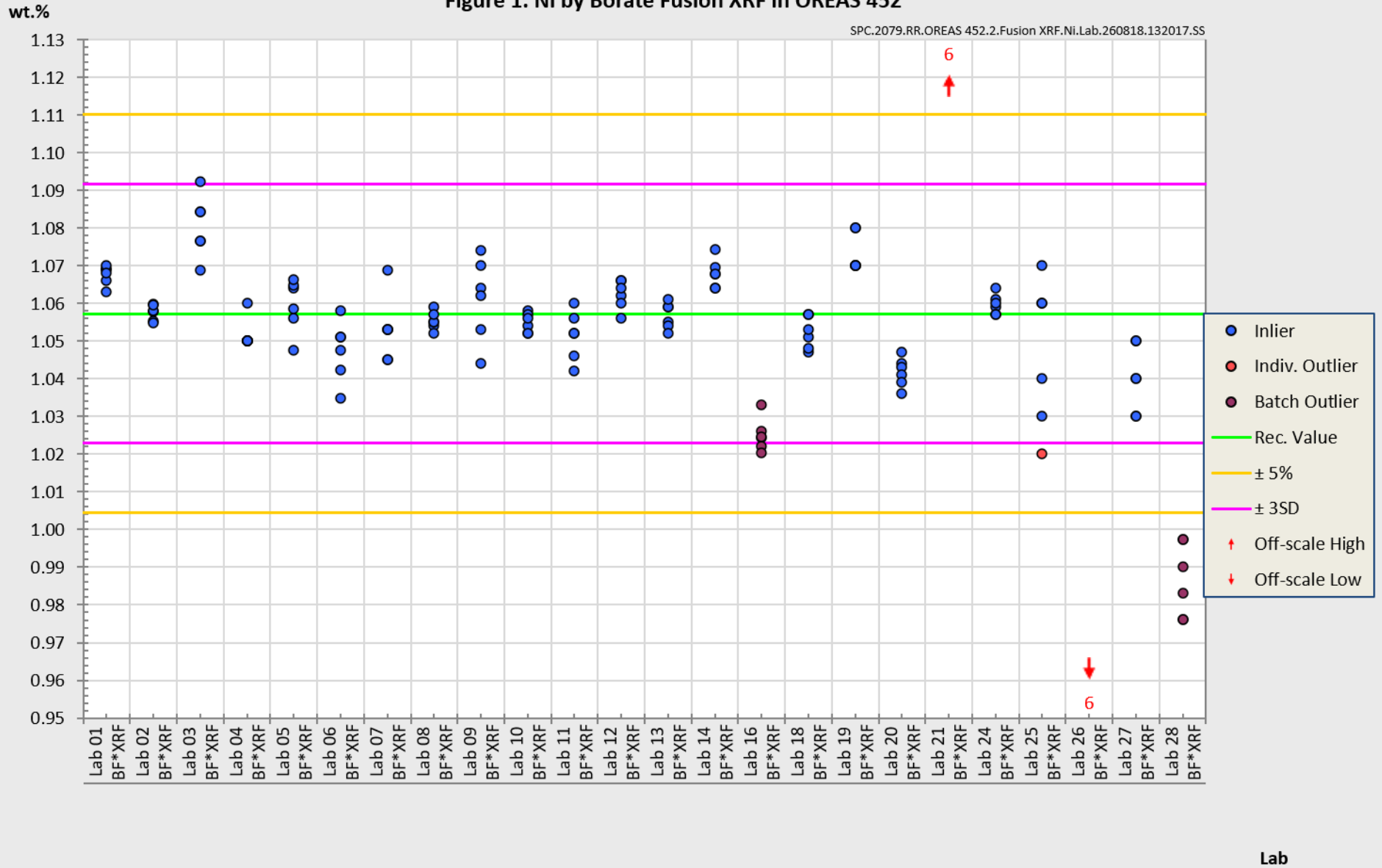
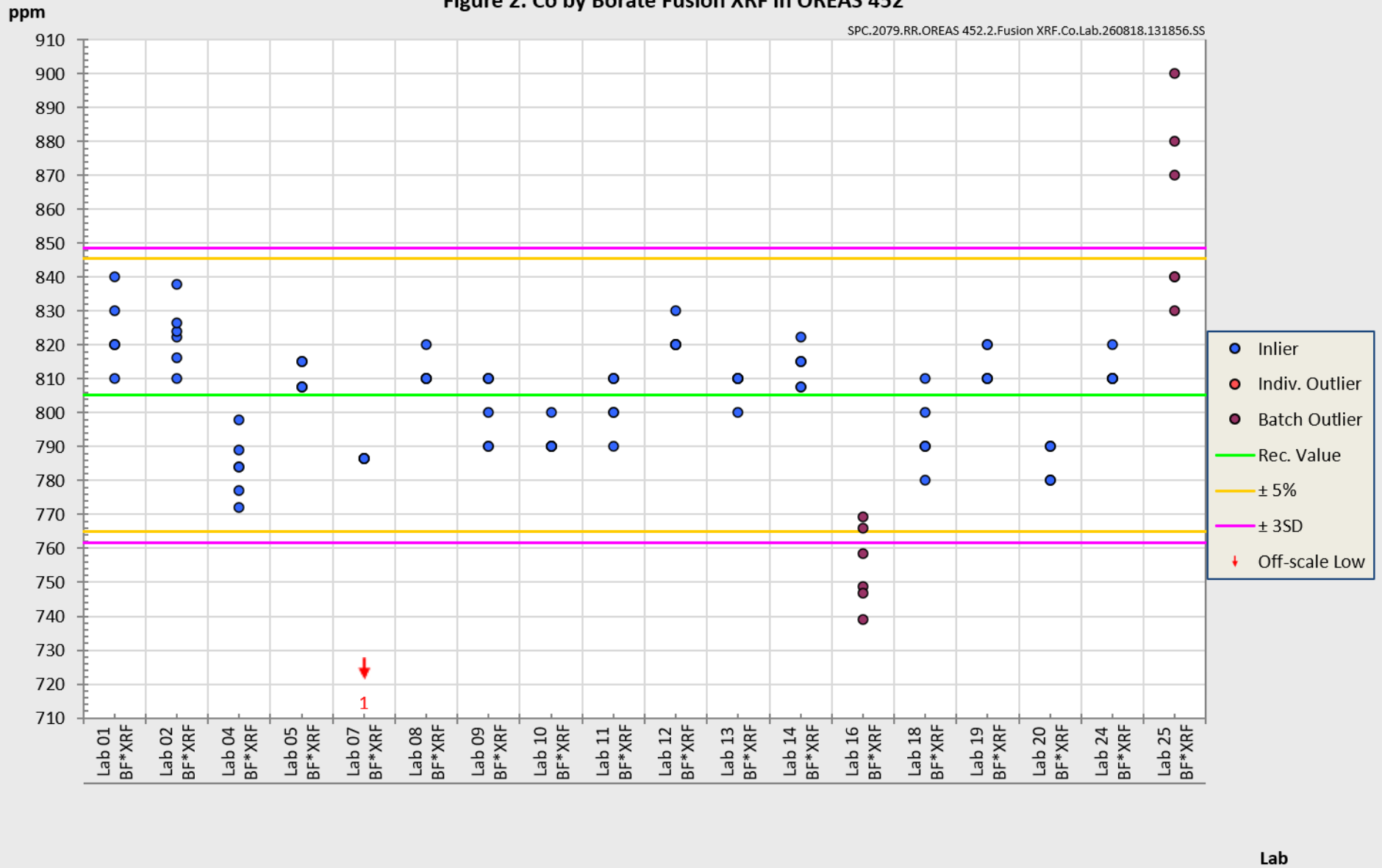


Figure 2. Co by Borate Fusion XRF in OREAS 452

SPC.2079.RR.OREAS 452.2.Fusion XRF.Co.Lab.260818.131856.SS



PREPARER AND SUPPLIER

Certified reference material OREAS 452 is prepared, certified and supplied by:

ORE Research & Exploration Pty Ltd
37A Hosie Street
Bayswater North VIC 3153
AUSTRALIA

Tel: +613-9729 0333
Web: www.oreas.com
Email: info@ore.com.au

CERTIFYING OFFICER

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

ACCREDITATION

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