

**CERTIFICATE OF ANALYSIS FOR**

**CERTIFIED REFERENCE MATERIAL**  
**OREAS 999b**

**Spodumene Concentrate**

**(Eastern Goldfields Region, Western Australia)**



Accredited for compliance with ISO 17034



COA-1965-OREAS 999b-R1  
BUP-70-10-01 Ver:2.0

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**Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 999b.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Borate / Peroxide Fusion ICP</b>					
Al, Aluminium (wt.%)	12.91	12.63	13.19	12.72	13.11
B, Boron (ppm)	221	185	257	210	232
Ba, Barium (ppm)	105	101	110	102	109
Be, Beryllium (ppm)	28.0	25.4	30.7	27.0	29.1
Ca, Calcium (wt.%)	0.227	0.200	0.253	0.208	0.245
Cd, Cadmium (ppm)	< 10	IND	IND	IND	IND
Ce, Cerium (ppm)	3.20	2.82	3.57	2.81	3.59
Co, Cobalt (ppm)	2.62	1.52	3.72	2.30	2.94
Cr, Chromium (ppm)	55	44	65	49	61
Cs, Caesium (ppm)	228	217	239	219	237
Er, Erbium (ppm)	< 0.1	IND	IND	IND	IND
Fe, Iron (wt.%)	0.745	0.709	0.780	0.728	0.761
Ga, Gallium (ppm)	81	76	86	79	83
Ge, Germanium (ppm)	5.16	4.24	6.08	IND	IND
Ho, Holmium (ppm)	< 0.1	IND	IND	IND	IND
K, Potassium (wt.%)	0.772	0.714	0.830	0.749	0.795
La, Lanthanum (ppm)	1.42	1.08	1.76	IND	IND
Li, Lithium (wt.%)	2.68	2.59	2.77	2.63	2.73
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.77	5.58	5.95	5.66	5.88
Mg, Magnesium (wt.%)	0.179	0.170	0.189	0.174	0.185
Mn, Manganese (wt.%)	0.138	0.131	0.144	0.134	0.141
Nb, Niobium (ppm)	244	226	261	237	250
P, Phosphorus (wt.%)	0.111	0.102	0.121	0.101	0.122
Pr, Praseodymium (ppm)	0.40	0.33	0.48	IND	IND
Rb, Rubidium (ppm)	462	438	486	450	474
Sc, Scandium (ppm)	< 5	IND	IND	IND	IND
Si, Silicon (wt.%)	30.34	29.66	31.03	29.71	30.98
Sn, Tin (ppm)	385	363	407	374	397
Sr, Strontium (ppm)	76	72	81	73	80
Ta, Tantalum (ppm)	729	686	773	701	757
Tb, Terbium (ppm)	< 0.1	IND	IND	IND	IND
Th, Thorium (ppm)	1.11	0.95	1.26	IND	IND
Ti, Titanium (wt.%)	0.020	0.019	0.021	0.019	0.021
Tl, Thallium (ppm)	4.41	4.08	4.73	4.15	4.66
Tm, Thulium (ppm)	< 0.1	IND	IND	IND	IND

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

Note: intervals may appear asymmetric due to rounding.

†These operationally defined measurands meet 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 1 continued.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Borate / Peroxide Fusion ICP continued</b>					
U, Uranium (ppm)	4.22	3.77	4.68	3.98	4.47
W, Tungsten (ppm)	10.0	7.6	12.5	9.0	11.0
Y, Yttrium (ppm)	1.29	1.01	1.56	IND	IND
Zn, Zinc (ppm)	121	109	133	114	127
<b>4-Acid Digestion</b>					
Al, Aluminium (wt.%)	10.00	8.64	11.36	9.60	10.41
Ba, Barium (ppm)	96	91	101	93	98
Be, Beryllium (ppm)	27.1	25.1	29.1	26.2	28.0
Bi, Bismuth (ppm)	0.61	0.56	0.66	0.58	0.64
Ca, Calcium (wt.%)	0.204	0.190	0.219	0.198	0.211
Cd, Cadmium (ppm)	0.22	0.19	0.26	0.20	0.25
Co, Cobalt (ppm)	2.03	1.90	2.16	1.94	2.12
Cr, Chromium (ppm)	29.0	25.2	32.8	26.7	31.3
Cs, Caesium (ppm)	224	210	238	219	229
Cu, Copper (ppm)	8.77	7.99	9.54	8.30	9.23
Eu, Europium (ppm)	0.11	0.07	0.14	IND	IND
Fe, Iron (wt.%)	0.673	0.651	0.695	0.654	0.692
Ga, Gallium (ppm)	75	71	79	73	77
Hf, Hafnium (ppm)	3.76	3.43	4.08	3.61	3.90
K, Potassium (wt.%)	0.708	0.683	0.733	0.692	0.724
Li, Lithium (wt.%)	2.65	2.59	2.71	2.63	2.67
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.70	5.57	5.84	5.66	5.75
Mg, Magnesium (wt.%)	0.122	0.103	0.141	0.117	0.126
Mn, Manganese (wt.%)	0.132	0.127	0.138	0.130	0.135
Mo, Molybdenum (ppm)	6.45	6.17	6.72	6.25	6.64
Na, Sodium (wt.%)	0.625	0.608	0.641	0.613	0.637
Nb, Niobium (ppm)	240	223	257	231	248
Ni, Nickel (wt.%)	0.002	0.002	0.002	0.002	0.002
P, Phosphorus (wt.%)	0.104	0.094	0.113	0.100	0.108
Pb, Lead (ppm)	3.82	3.22	4.42	3.55	4.10
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.022	0.021	0.024	0.021	0.023
Sb, Antimony (ppm)	0.14	0.11	0.16	0.12	0.15
Sc, Scandium (ppm)	0.52	0.38	0.65	IND	IND
Se, Selenium (ppm)	< 1	IND	IND	IND	IND

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

Note: intervals may appear asymmetric due to rounding.

<sup>†</sup>These operationally defined measurands meet 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 1 continued.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
Sm, Samarium (ppm)	0.29	0.19	0.38	IND	IND
Sn, Tin (ppm)	231	217	246	222	241
Sr, Strontium (ppm)	58	52	64	56	60
Ta, Tantalum (ppm)	678	595	760	657	698
Tb, Terbium (ppm)	0.048	0.029	0.067	IND	IND
Te, Tellurium (ppm)	< 0.05	IND	IND	IND	IND
Ti, Titanium (wt.%)	0.019	0.018	0.020	0.018	0.020
Tl, Thallium (ppm)	4.23	4.04	4.43	4.11	4.36
U, Uranium (ppm)	2.61	2.08	3.15	2.46	2.77
W, Tungsten (ppm)	9.22	8.61	9.84	8.88	9.56
Yb, Ytterbium (ppm)	0.095	0.081	0.108	IND	IND
Zn, Zinc (ppm)	120	116	123	117	122
Zr, Zirconium (ppm)	16.8	15.8	17.8	16.1	17.5
<b>Borate Fusion XRF</b>					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	24.33	23.99	24.67	24.23	24.43
CaO, Calcium oxide (wt.%)	0.315	0.306	0.324	0.306	0.325
Co, Cobalt (ppm)	< 100	IND	IND	IND	IND
Cu, Copper (ppm)	< 50	IND	IND	IND	IND
Fe, Iron (wt.%)	0.741	0.722	0.760	0.731	0.751
K <sub>2</sub> O, Potassium oxide (wt.%)	0.864	0.845	0.883	0.851	0.877
Mg, Magnesium (wt.%)	0.189	0.176	0.202	0.180	0.197
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.849	0.793	0.905	0.826	0.873
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.263	0.252	0.274	0.258	0.268
SiO <sub>2</sub> , Silicon dioxide (wt.%)	64.27	63.44	65.09	63.99	64.55
Sn, Tin (ppm)	377	307	447	344	410
V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)	< 180	IND	IND	IND	IND
Zn, Zinc (ppm)	112	85	139	IND	IND
<b>Thermogravimetry</b>					
LOI <sup>1000</sup> , Loss on ignition @1000 °C (wt.%)	1.19	1.08	1.30	1.15	1.24

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

Note: intervals may appear asymmetric due to rounding.

<sup>†</sup>These operationally defined measurands meet 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. Indicative Values for OREAS 999b.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Borate / Peroxide Fusion ICP</b>								
Ag	ppm	1.87	In	ppm	0.096	Sb	ppm	0.15
As	ppm	< 100	Lu	ppm	< 0.05	Se	ppm	< 20
Bi	ppm	0.64	Mo	ppm	7.84	Sm	ppm	0.30
Cu	ppm	13.9	Na	wt.%	0.653	Te	ppm	< 1
Dy	ppm	0.28	Nd	ppm	1.49	V	ppm	9.92
Eu	ppm	0.12	Ni	wt.%	0.003	Yb	ppm	< 0.1
Gd	ppm	0.26	Pb	ppm	34.7	Zr	ppm	18.4
Hf	ppm	4.87	Re	ppm	< 0.1			
Hg	ppm	0.11	S	wt.%	0.026			
<b>4-Acid Digestion</b>								
Ag	ppm	0.140	Ge	ppm	0.75	Pr	ppm	0.27
As	ppm	0.79	Hg	ppm	0.050	Pt	ppb	9.03
B	ppm	5.00	Ho	ppm	0.043	Rb	ppm	372
Ce	ppm	1.49	In	ppm	0.84	Th	ppm	0.54
Dy	ppm	0.18	La	ppm	1.41	Tm	ppm	0.020
Er	ppm	0.10	Lu	ppm	0.022	V	ppm	5.99
Gd	ppm	0.22	Nd	ppm	0.97	Y	ppm	0.70
<b>Borate Fusion XRF</b>								
As	ppm	59	Hg	ppm	< 100	Se	ppm	< 100
BaO	ppm	106	In	ppm	< 100	SO <sub>3</sub>	wt.%	0.048
Bi	ppm	< 100	La	ppm	< 90	SrO	ppm	99
Cd	ppm	< 100	MnO	wt.%	0.181	Ta	ppm	< 100
Ce	ppm	< 80	Mo	ppm	< 50	Te	ppm	< 100
Cl	ppm	773	Nb	ppm	252	TiO <sub>2</sub>	wt.%	0.037
Cr <sub>2</sub> O <sub>3</sub>	ppm	78	Ni	wt.%	< 0.005	Tl	ppm	< 100
Cs	ppm	< 100	Pb	ppm	60	TOT_XRF	wt.%	94.29
Ga	ppm	< 100	Rb	ppm	473	W	ppm	24.7
Ge	ppm	< 100	Sb	ppm	< 50	Y	ppm	56
HfO <sub>2</sub>	ppm	< 100	Sc	ppm	< 100	Zr	ppm	71
<b>Thermogravimetry</b>								
H <sub>2</sub> O-	wt.%	0.477						
<b>Infrared Combustion</b>								
C	wt.%	0.645	S	wt.%	0.049			

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  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 correct use' should be read carefully.

Table 1 (generated from data supplied by ISO 17025 accredited laboratories for the analytical methods employed) provides the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 2 shows indicative values, Table 3 provides some indicative physical properties, Table 4 shows indicative mineralogy by semi-quantitative XRD analysis and Table 5 presents 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<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 999b-DataPack.1.0.250509\_002420.xlsx**). Results are also presented in scatter plots for Li<sub>2</sub>O (wt.%) by 4-acid digestion and borate/peroxide fusion with ICP in Figures 1 and 2 respectively, together with  $\pm 3$  SD (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.

## SOURCE MATERIAL

OREAS 999b is a spodumene concentrate product derived from the processing of lithium pegmatite ores sourced from the Eastern Goldfields Region of Western Australia. The Spodumene concentrate is primarily composed of spodumene (LiAlSi<sub>2</sub>O<sub>6</sub>), a lithium-bearing pyroxene. Minor gangue minerals include quartz, albite, muscovite, and mica group minerals.

## COMMINTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 999b was prepared in the following manner:

- Drying of spodumene concentrate to constant mass at 105 °C;
- Milling of spodumene concentrate materials to 100 % minus 30 µm;
- 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 999b 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 OREAS 999b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
795	0.45	N8	Very Light Gray

<sup>‡</sup>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. A presence of some amorphous material is very likely. Some cookeite might be present and reported under chlorite

**Table 4. Indicative mineralogy of OREAS 999b based on semi-quantitative XRD analysis.**

Mineral / Mineral Group	% (mass ratio)
Chlorite	< 1
Annite - biotite - phlogopite	1
Muscovite	3
Ca amphibole	< 1
Spodumene	75
Plagioclase	8
Tourmaline	< 1
Quartz	12
Dolomite - ankerite	< 1
Anatase	< 1

## ANALYTICAL PROGRAM

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

- Sodium peroxide/borate fusion with full suite ICP-OES and ICP-MS elemental packages (up to 15 laboratories depending on the element);
- 4-acid (HNO<sub>3</sub>-HF-HClO<sub>4</sub>-HCl) digestion with full suite ICP-OES and ICP-MS elemental packages (up to 22 laboratories depending on the element);
- Lithium borate fusion whole rock analysis package by X-ray fluorescence (up to 14 laboratories depending on the element);
- Thermogravimetry: Loss on Ignition (LOI) at 1000 °C (10 laboratories used a thermogravimetric analyser, 3 laboratories included LOI with their fusion package and 2 laboratories used a conventional muffle furnace).

For the round robin program six 600 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 one 20 g subsample from each of 600 g test units. Homogeneity was evaluated by submitting 12 x 20 g pulp samples to a single laboratory for analysis by peroxide fusion with ICP-OES finish. Paired samples were taken from each of the odd-numbered sampling units, allowing for an Analysis of Variance (ANOVA) to compare within- and between-unit variances across the six pairs. This statistical approach provides a relative measure of homogeneity and tests the null hypothesis that all sampling units originate from the same population distribution (see 'Homogeneity Evaluation' section 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).

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 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 lithium ( $\text{Li}_2\text{O}$ ) by 4-acid digestion with ICP, where 99 % of the time ( $1-\alpha=0.99$ ) at least 95 % of subsamples ( $p=0.95$ ) will have concentrations lying between 5.66 and 5.75 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 x 20 g pulp samples to the ALS Brisbane laboratory for analysis by peroxide fusion with ICP-OES finish (code ME-ICP89). 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 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). 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 999b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## 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/mltrule.htm](http://www.westgard.com/mltrule.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 ( $\text{DL}$ )  $\pm 10\ %$ .

*i.e., Certified Value  $\pm 10\ % \pm 2\text{DL}$  [1].*

**Table 5. Performance Gates for OREAS 999b.**

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
<b>Borate / Peroxide Fusion ICP</b>											
Al, wt.%	12.91	0.182	12.55	13.28	12.37	13.46	1.41%	2.81%	4.22%	12.27	13.56
B, ppm	221	26	170	273	144	298	11.64%	23.28%	34.92%	210	232
Ba, ppm	105	2	101	110	98	113	2.33%	4.67%	7.00%	100	111
Be, ppm	28.0	2.69	22.6	33.4	19.9	36.1	9.60%	19.20%	28.80%	26.6	29.4
Ca, wt.%	0.227	0.025	0.177	0.276	0.152	0.301	10.96%	21.92%	32.88%	0.215	0.238
Cd, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	3.20	0.32	2.56	3.84	2.24	4.16	10.03%	20.06%	30.10%	3.04	3.36
Co, ppm	2.62	0.72	1.17	4.07	0.45	4.79	27.62%	55.23%	82.85%	2.49	2.75
Cr, ppm	55	10	35	74	26	84	17.85%	35.69%	53.54%	52	58
Cs, ppm	228	10	208	248	199	257	4.29%	8.59%	12.88%	217	239
Er, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Fe, wt.%	0.745	0.035	0.674	0.815	0.639	0.850	4.71%	9.42%	14.13%	0.707	0.782
Ga, ppm	81	3.8	73	89	69	92	4.72%	9.45%	14.17%	77	85
Ge, ppm	5.16	0.357	4.45	5.88	4.09	6.23	6.92%	13.83%	20.75%	4.90	5.42
Ho, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	0.772	0.064	0.644	0.900	0.580	0.964	8.29%	16.58%	24.87%	0.733	0.811
La, ppm	1.42	0.20	1.01	1.82	0.81	2.03	14.27%	28.53%	42.80%	1.35	1.49
Li, wt.%	2.68	0.098	2.48	2.87	2.39	2.97	3.64%	7.28%	10.92%	2.55	2.81
Li <sub>2</sub> O, wt.%	5.77	0.210	5.35	6.19	5.14	6.40	3.64%	7.28%	10.92%	5.48	6.06
Mg, wt.%	0.179	0.008	0.164	0.194	0.157	0.202	4.18%	8.36%	12.54%	0.170	0.188
Mn, wt.%	0.138	0.006	0.126	0.149	0.120	0.155	4.14%	8.27%	12.41%	0.131	0.144
Nb, ppm	244	15	214	274	199	289	6.14%	12.27%	18.41%	232	256
P, wt.%	0.111	0.009	0.094	0.129	0.085	0.138	7.84%	15.69%	23.53%	0.106	0.117
Pr, ppm	0.40	0.05	0.31	0.49	0.27	0.54	11.19%	22.38%	33.57%	0.38	0.42
Rb, ppm	462	21	419	505	398	526	4.65%	9.29%	13.94%	439	485
Sc, ppm	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Si, wt.%	30.34	0.635	29.07	31.62	28.44	32.25	2.09%	4.19%	6.28%	28.83	31.86
Sn, ppm	385	26	334	437	308	463	6.72%	13.44%	20.16%	366	405
Sr, ppm	76	3.8	69	84	65	88	4.96%	9.93%	14.89%	73	80
Ta, ppm	729	50	628	830	578	880	6.92%	13.85%	20.77%	693	766
Tb, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	1.11	0.072	0.96	1.25	0.89	1.32	6.52%	13.04%	19.57%	1.05	1.16
Ti, wt.%	0.020	0.001	0.017	0.023	0.016	0.024	7.04%	14.08%	21.12%	0.019	0.021
Tl, ppm	4.41	0.279	3.85	4.97	3.57	5.24	6.33%	12.66%	18.99%	4.19	4.63
Tm, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
U, ppm	4.22	0.314	3.60	4.85	3.28	5.17	7.44%	14.87%	22.31%	4.01	4.44
W, ppm	10.0	1.5	7.1	12.9	5.7	14.4	14.54%	29.08%	43.62%	9.5	10.5
Y, ppm	1.29	0.115	1.06	1.52	0.94	1.63	8.93%	17.85%	26.78%	1.22	1.35
Zn, ppm	121	15	90	151	75	166	12.65%	25.31%	37.96%	115	127
<b>4-Acid Digestion</b>											
Al, wt.%	10.00	1.92	6.16	13.84	4.24	15.76	19.19%	38.38%	57.57%	9.50	10.50
Ba, ppm	96	5.5	85	107	79	112	5.75%	11.50%	17.24%	91	101
Be, ppm	27.1	3.0	21.0	33.2	18.0	36.2	11.23%	22.45%	33.68%	25.7	28.5

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  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
<b>4-Acid Digestion continued</b>											
Bi, ppm	0.61	0.047	0.52	0.70	0.47	0.75	7.66%	15.31%	22.97%	0.58	0.64
Ca, wt.%	0.204	0.022	0.159	0.249	0.137	0.272	11.00%	21.99%	32.99%	0.194	0.215
Cd, ppm	0.22	0.03	0.17	0.28	0.15	0.30	11.60%	23.20%	34.79%	0.21	0.23
Co, ppm	2.03	0.125	1.78	2.28	1.66	2.41	6.17%	12.33%	18.50%	1.93	2.13
Cr, ppm	29.0	6.1	16.9	41.1	10.8	47.2	20.91%	41.83%	62.74%	27.5	30.4
Cs, ppm	224	21	183	265	162	285	9.19%	18.39%	27.58%	213	235
Cu, ppm	8.77	1.05	6.67	10.86	5.62	11.91	11.97%	23.94%	35.91%	8.33	9.20
Eu, ppm	0.11	0.02	0.06	0.15	0.04	0.17	21.14%	42.29%	63.43%	0.10	0.11
Fe, wt.%	0.673	0.026	0.621	0.725	0.595	0.751	3.88%	7.77%	11.65%	0.639	0.707
Ga, ppm	75	3.9	67	83	63	87	5.23%	10.45%	15.68%	71	79
Hf, ppm	3.76	0.344	3.07	4.45	2.72	4.79	9.16%	18.32%	27.48%	3.57	3.95
K, wt.%	0.708	0.029	0.651	0.765	0.622	0.794	4.05%	8.10%	12.15%	0.672	0.743
Li, wt.%	2.65	0.069	2.51	2.79	2.44	2.86	2.60%	5.20%	7.81%	2.52	2.78
Li <sub>2</sub> O, wt.%	5.70	0.148	5.41	6.00	5.26	6.15	2.60%	5.20%	7.81%	5.42	5.99
Mg, wt.%	0.122	0.034	0.054	0.190	0.020	0.224	27.94%	55.89%	83.83%	0.116	0.128
Mn, wt.%	0.132	0.008	0.116	0.149	0.108	0.157	6.25%	12.50%	18.75%	0.126	0.139
Mo, ppm	6.45	0.279	5.89	7.00	5.61	7.28	4.32%	8.64%	12.97%	6.12	6.77
Na, wt.%	0.625	0.024	0.577	0.673	0.552	0.697	3.86%	7.72%	11.58%	0.594	0.656
Nb, ppm	240	25	189	290	163	316	10.62%	21.24%	31.85%	228	252
Ni, wt. %	0.002	0.000	0.001	0.002	0.001	0.002	11.81%	23.63%	35.44%	0.002	0.002
P, wt. %	0.104	0.009	0.085	0.122	0.076	0.132	9.05%	18.10%	27.15%	0.098	0.109
Pb, ppm	3.82	0.65	2.53	5.11	1.88	5.76	16.94%	33.88%	50.82%	3.63	4.01
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	0.022	0.003	0.016	0.028	0.013	0.031	13.72%	27.43%	41.15%	0.021	0.023
Sb, ppm	0.14	0.02	0.09	0.18	0.06	0.21	17.43%	34.87%	52.30%	0.13	0.14
Sc, ppm	0.52	0.10	0.31	0.72	0.21	0.82	19.59%	39.19%	58.78%	0.49	0.54
Se, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	0.29	0.04	0.20	0.37	0.16	0.42	15.08%	30.15%	45.23%	0.27	0.30
Sn, ppm	231	14	204	259	190	273	5.95%	11.89%	17.84%	220	243
Sr, ppm	58	9	40	76	31	85	15.73%	31.46%	47.19%	55	61
Ta, ppm	678	134	409	946	275	1081	19.82%	39.64%	59.47%	644	711
Tb, ppm	0.048	0.009	0.030	0.066	0.022	0.075	18.33%	36.66%	55.00%	0.046	0.050
Te, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ti, wt. %	0.019	0.001	0.017	0.021	0.016	0.023	6.04%	12.09%	18.13%	0.018	0.020
Tl, ppm	4.23	0.224	3.78	4.68	3.56	4.90	5.30%	10.59%	15.89%	4.02	4.44
U, ppm	2.61	0.98	0.66	4.57	0.00	5.55	37.45%	74.89%	112.34	2.48	2.75
W, ppm	9.22	0.796	7.63	10.81	6.84	11.61	8.63%	17.26%	25.89%	8.76	9.68
Yb, ppm	0.095	0.009	0.077	0.112	0.069	0.120	9.07%	18.14%	27.21%	0.090	0.099
Zn, ppm	120	5	110	129	105	134	3.97%	7.95%	11.92%	114	126
Zr, ppm	16.8	1.45	13.9	19.7	12.4	21.1	8.64%	17.28%	25.91%	16.0	17.6
<b>Borate Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	24.33	0.278	23.77	24.89	23.50	25.16	1.14%	2.29%	3.43%	23.11	25.55
CaO, wt. %	0.315	0.006	0.304	0.326	0.299	0.332	1.78%	3.55%	5.33%	0.300	0.331

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  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
<b>Borate Fusion XRF continued</b>											
Co, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Fe, wt.%	0.741	0.022	0.697	0.785	0.675	0.807	2.96%	5.91%	8.87%	0.704	0.778
K <sub>2</sub> O, wt.%	0.864	0.018	0.828	0.899	0.811	0.917	2.04%	4.09%	6.13%	0.821	0.907
Mg, wt.%	0.189	0.012	0.165	0.212	0.153	0.224	6.26%	12.52%	18.77%	0.179	0.198
Na <sub>2</sub> O, wt.%	0.849	0.057	0.736	0.963	0.679	1.020	6.68%	13.36%	20.03%	0.807	0.892
P <sub>2</sub> O <sub>5</sub> , wt.%	0.263	0.008	0.247	0.279	0.239	0.286	2.98%	5.97%	8.95%	0.250	0.276
SiO <sub>2</sub> , wt.%	64.27	0.966	62.33	66.20	61.37	67.16	1.50%	3.01%	4.51%	61.05	67.48
Sn, ppm	377	34	308	445	274	480	9.10%	18.21%	27.31%	358	396
V <sub>2</sub> O <sub>5</sub> , ppm	< 180	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Zn, ppm	112	16	80	144	64	160	14.16%	28.32%	42.47%	106	118
<b>Thermogravimetry</b>											
LOI <sup>1000</sup> , wt.%	1.19	0.16	0.88	1.51	0.72	1.67	13.19%	26.39%	39.58%	1.14	1.25

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  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.

## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Lima, Peru
4. ALS, Loughrea, Galway, Ireland
5. American Assay Laboratories, Sparks, Nevada, USA
6. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
7. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
8. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
9. Bureau Veritas Minerals, Ankara, Central Anatolia, Turkey
10. CERTIMIN, Lima, Peru
11. Inspectorate (BV), Lima, Peru
12. Inspectorate Griffith India, Gandhidham, Gujarat, India
13. Intertek, Cupang, Muntinlupa, Philippines
14. Intertek, Perth, WA, Australia
15. Labwest Minerals Analysis, Perth, WA, Australia
16. MSALABS, Vancouver, BC, Canada
17. Paragon Geochemical Laboratories, Sparks, Nevada, USA
18. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
19. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
20. SGS, Ankara, Anatolia, Turkey
21. SGS Australia Mineral Services, Perth, WA, Australia
22. SGS Canada Inc., Vancouver, BC, Canada
23. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
24. Skyline Assayers & Laboratories, Tucson, Arizona, USA
25. 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 999b is prepared, certified and supplied by:

ORE Research & Exploration Pty Ltd      Tel: +613-9729 0333  
37A Hosie Street      Web: [www.oreas.com](http://www.oreas.com)  
Bayswater North VIC 3153      Email: [info@ore.com.au](mailto:info@ore.com.au)  
AUSTRALIA

**Figure 1. Li<sub>2</sub>O by Borate / Peroxide Fusion ICP in OREAS 999b**

SPC.1965.RR1.OREAS 999b.3.Fusion ICP.Li2O.Lab.250507.172949.SN

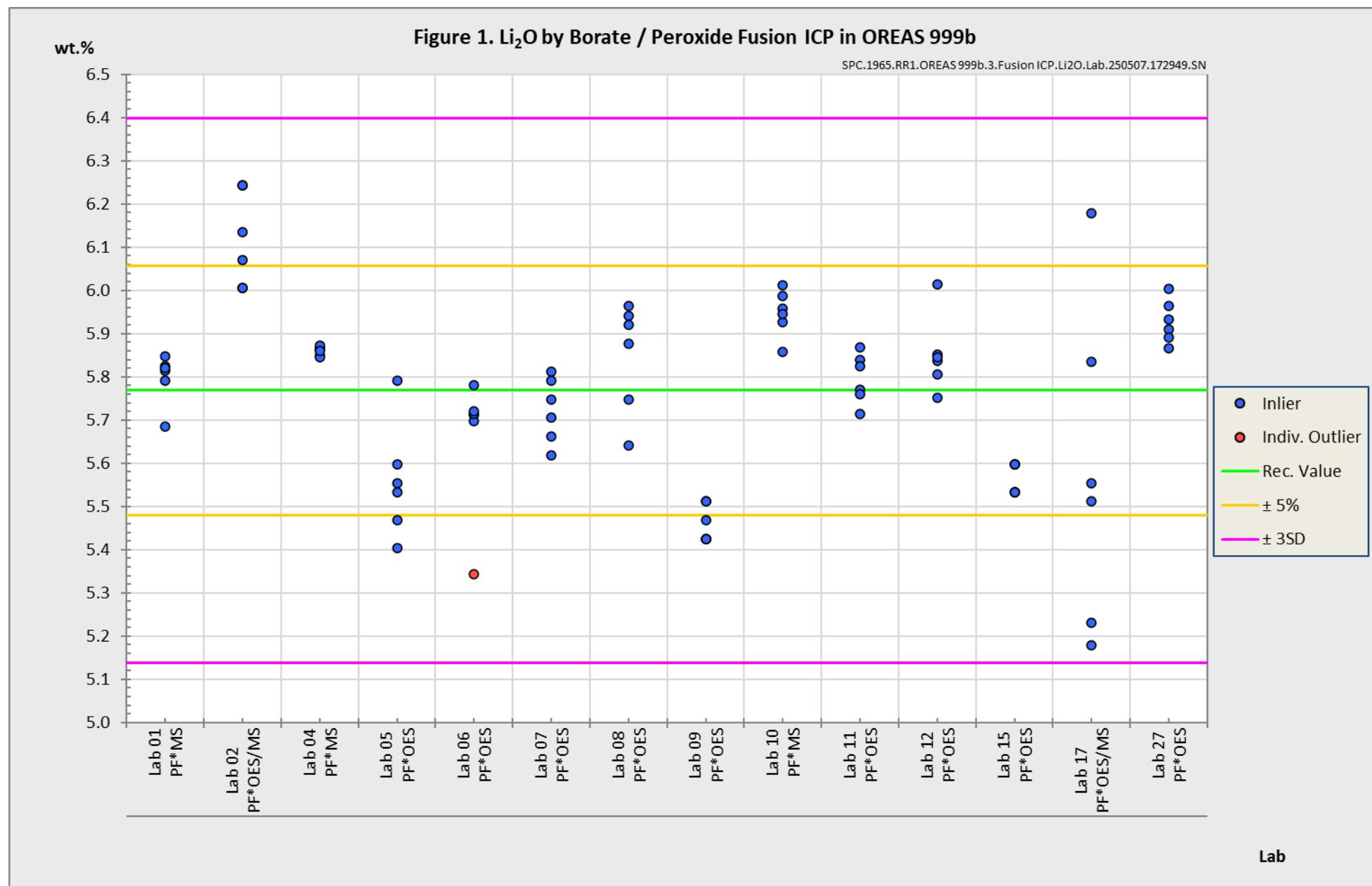
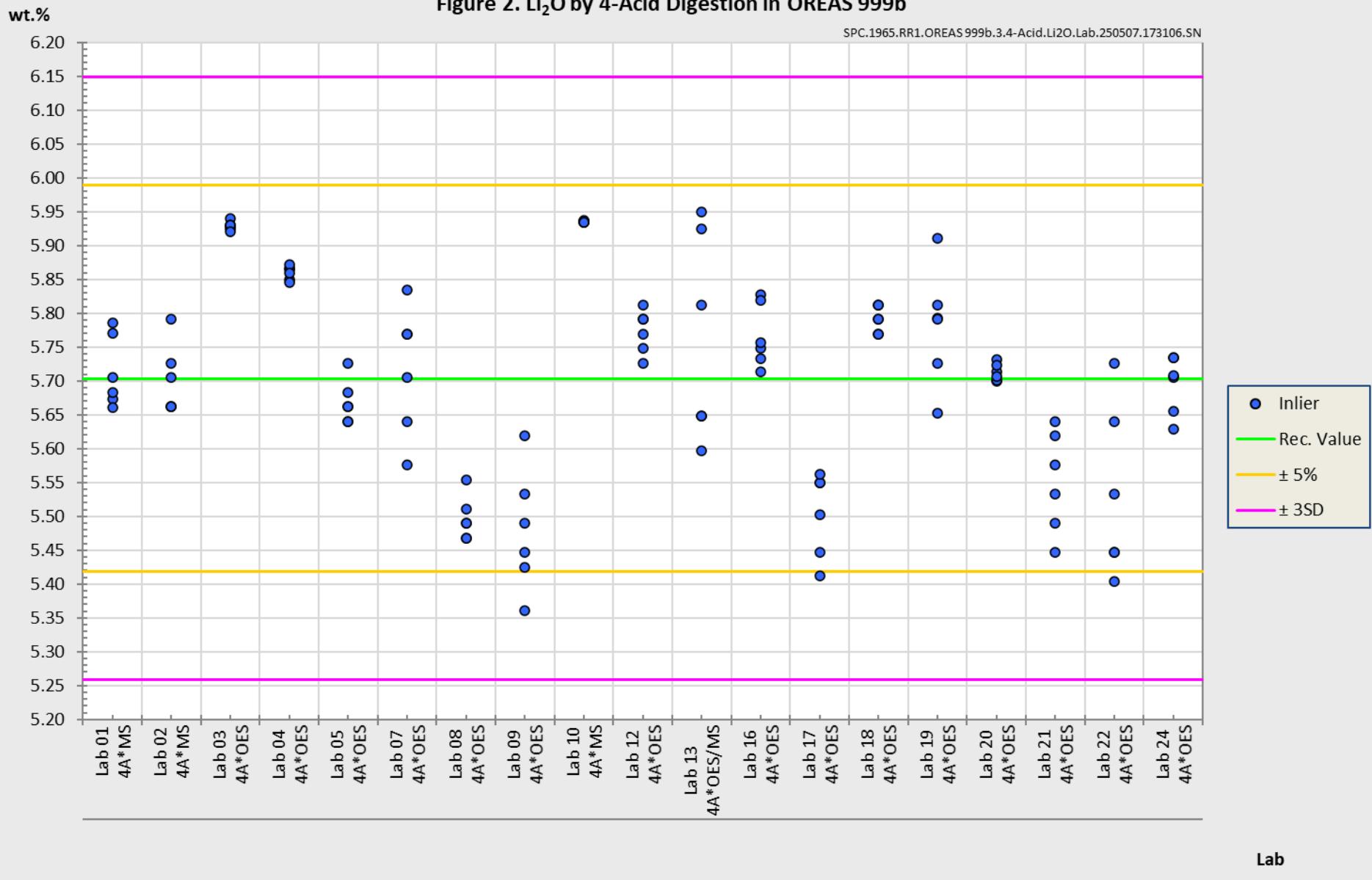


Figure 2. Li<sub>2</sub>O by 4-Acid Digestion in OREAS 999b

SPC.1965.RR1.OREAS 999b.3.4-Acid.Li2O.Lab.250507.173106.SN



## 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 all methods (Table 1). 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 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 999b 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 999b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 999b is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 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).

## 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:

- Sodium peroxide / Lithium Borate fusion with ICP-OES and/or MS finish:  $\geq 0.2$  g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25$  g;
- Borate fusion with X-ray fluorescence finish:  $\geq 0.2$  g;
- Loss on Ignition (LOI) at  $1000^{\circ}\text{C}$ :  $\geq 1$  g;

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 999b 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 999b 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., 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 999b 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 3 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<sub>2</sub>O- 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.

### **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 5 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.

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## DOCUMENT HISTORY

Revision No.	Date	Changes applied
1	12 <sup>th</sup> January, 2026	Revised the mineralogy description on page 8 by removing unnecessary details.
0	23 <sup>rd</sup> May, 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.



## REFERENCES

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