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## CERTIFICATE OF ANALYSIS FOR

### Li Concentrate

(Spodumene Concentrate, Greenbushes, Western Australia)

## CERTIFIED REFERENCE MATERIAL

# OREAS 999

### Summary Statistics for Key Analytes.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Peroxide Fusion ICP</b>						
Li, Lithium (wt.%)	2.67	0.103	2.63	2.72	2.62	2.73
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.76	0.222	5.65	5.86	5.63	5.88
<b>4-Acid Digestion</b>						
Li, Lithium (wt.%)	2.65	0.057	2.62	2.68	2.59	2.71
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.70	0.122	5.64	5.76	5.58	5.83

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.  
Note: intervals may appear asymmetric due to rounding.



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Table 1. Certified Values, SDs, 95% Confidence & Tolerance Limits for OREAS 999.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Peroxide Fusion ICP</b>						
Al, Aluminium (wt.%)	12.23	0.411	12.01	12.46	11.94	12.52
Ba, Barium (ppm)	39.1	2.64	37.5	40.6	35.6	42.5
Be, Beryllium (ppm)	51	2.1	49	52	48	53
Bi, Bismuth (ppm)	2.21	0.185	2.07	2.34	2.06	2.35
Ca, Calcium (wt.%)	0.481	0.059	0.454	0.508	0.458	0.504
Ce, Cerium (ppm)	4.15	0.360	3.75	4.55	3.80	4.50
Co, Cobalt (ppm)	5.24	0.343	4.94	5.54	4.97	5.51
Cr, Chromium (ppm)	112	17	104	120	103	120
Cs, Cesium (ppm)	93	3.6	91	95	91	95
Cu, Copper (ppm)	25.5	3.4	21.6	29.4	22.7	28.2
Dy, Dysprosium (ppm)	0.80	0.050	0.79	0.82	0.76	0.85
Er, Erbium (ppm)	0.27	0.03	0.24	0.30	0.23	0.30
Fe, Iron (wt.%)	1.73	0.067	1.70	1.76	1.69	1.77
Ga, Gallium (ppm)	88	6.4	83	92	85	90
Gd, Gadolinium (ppm)	1.08	0.16	1.02	1.14	0.98	1.17
Ge, Germanium (ppm)	4.31	0.45	4.06	4.56	3.77	4.85
Ho, Holmium (ppm)	0.11	0.011	0.11	0.12	IND	IND
K, Potassium (wt.%)	0.522	0.048	0.499	0.544	0.502	0.542
La, Lanthanum (ppm)	1.84	0.25	1.64	2.03	1.69	1.98
Li, Lithium (wt.%)	2.67	0.103	2.63	2.72	2.62	2.73
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.76	0.222	5.65	5.86	5.63	5.88
Mg, Magnesium (wt.%)	0.473	0.020	0.462	0.484	0.461	0.485
Mn, Manganese (wt.%)	0.147	0.006	0.144	0.150	0.144	0.150
Mo, Molybdenum (ppm)	2.27	0.38	2.06	2.48	IND	IND
Nb, Niobium (ppm)	74	6.2	70	78	71	76
Nd, Neodymium (ppm)	1.90	0.22	1.74	2.05	1.66	2.14
Ni, Nickel (ppm)	52	4.2	51	54	47	57
P, Phosphorus (wt.%)	0.016	0.005	0.014	0.019	IND	IND
Pr, Praseodymium (ppm)	0.52	0.06	0.46	0.57	0.49	0.55
Rb, Rubidium (ppm)	423	20	411	434	413	432
Sb, Antimony (ppm)	1.02	0.17	0.88	1.16	IND	IND
Si, Silicon (wt.%)	30.30	0.680	29.81	30.79	29.63	30.96
Sm, Samarium (ppm)	1.02	0.099	0.97	1.06	IND	IND
Sn, Tin (ppm)	84	5.3	80	87	80	87
Sr, Strontium (ppm)	23.0	3.3	20.5	25.4	21.3	24.6
Ta, Tantalum (ppm)	45.5	2.08	44.2	46.8	44.2	46.8

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

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 1 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Peroxide Fusion ICP continued</b>						
Tb, Terbium (ppm)	0.18	0.02	0.17	0.19	IND	IND
Th, Thorium (ppm)	3.01	0.158	2.92	3.11	2.87	3.15
Ti, Titanium (wt.%)	0.034	0.004	0.032	0.036	0.033	0.036
Tl, Thallium (ppm)	4.26	0.253	4.06	4.46	4.07	4.45
U, Uranium (ppm)	2.51	0.226	2.30	2.72	2.36	2.66
W, Tungsten (ppm)	6.94	0.391	6.78	7.10	6.38	7.51
Y, Yttrium (ppm)	4.19	0.43	3.90	4.48	3.92	4.46
Zn, Zinc (ppm)	77	4.3	75	79	71	83
Zr, Zirconium (ppm)	32.9	3.4	29.6	36.2	31.3	34.5
<b>Borate Fusion XRF</b>						
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	23.39	0.114	23.34	23.44	23.27	23.51
CaO, Calcium oxide (wt.%)	0.670	0.013	0.664	0.676	0.663	0.677
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	2.49	0.033	2.47	2.51	2.47	2.50
K <sub>2</sub> O, Potassium oxide (wt.%)	0.612	0.009	0.607	0.616	0.602	0.621
MgO, Magnesium oxide (wt.%)	0.796	0.012	0.791	0.801	0.788	0.805
MnO, Manganese oxide (wt.%)	0.192	0.005	0.189	0.194	0.188	0.195
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.945	0.024	0.933	0.956	0.928	0.961
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.038	0.004	0.035	0.040	0.036	0.040
SiO <sub>2</sub> , Silicon dioxide (wt.%)	64.23	0.446	64.03	64.43	64.01	64.44
SO <sub>3</sub> , Sulphur trioxide (wt.%)	0.042	0.006	0.037	0.046	IND	IND
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.060	0.007	0.057	0.063	0.056	0.065
<b>Thermogravimetry</b>						
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	0.764	0.114	0.704	0.824	0.720	0.808
<b>4-Acid Digestion</b>						
Al, Aluminium (wt.%)	10.77	1.42	10.14	11.40	10.36	11.18
As, Arsenic (ppm)	5.36	1.07	4.82	5.89	4.96	5.75
Ba, Barium (ppm)	39.6	2.34	38.5	40.8	38.2	41.1
Be, Beryllium (ppm)	49.8	3.49	48.3	51.3	48.1	51.5
Bi, Bismuth (ppm)	2.11	0.131	2.05	2.17	1.99	2.23
Ca, Calcium (wt.%)	0.450	0.033	0.437	0.463	0.434	0.466
Co, Cobalt (ppm)	4.95	0.262	4.82	5.08	4.72	5.18
Cr, Chromium (ppm)	81	21	72	89	76	85
Cs, Cesium (ppm)	88	11	83	93	85	91
Cu, Copper (ppm)	25.4	2.34	24.4	26.4	24.3	26.6
Dy, Dysprosium (ppm)	0.67	0.13	0.57	0.78	0.62	0.72
Er, Erbium (ppm)	0.23	0.05	0.19	0.28	IND	IND

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

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 1 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>4-Acid Digestion continued</b>						
Fe, Iron (wt.%)	1.62	0.097	1.58	1.66	1.57	1.67
Ga, Gallium (ppm)	82	5.4	80	84	79	85
Hf, Hafnium (ppm)	1.98	0.171	1.91	2.06	1.93	2.04
Ho, Holmium (ppm)	0.090	0.018	0.076	0.103	IND	IND
K, Potassium (wt.%)	0.500	0.041	0.482	0.519	0.486	0.514
La, Lanthanum (ppm)	1.68	0.34	1.49	1.87	1.57	1.78
Li, Lithium (wt.%)	2.65	0.057	2.62	2.68	2.59	2.71
Li <sub>2</sub> O, Lithium oxide (wt.%)	5.70	0.122	5.64	5.76	5.58	5.83
Mg, Magnesium (wt.%)	0.410	0.066	0.380	0.440	0.395	0.425
Mn, Manganese (wt.%)	0.143	0.006	0.141	0.145	0.140	0.146
Mo, Molybdenum (ppm)	2.06	0.140	2.00	2.12	1.92	2.19
Na, Sodium (wt.%)	0.693	0.023	0.682	0.703	0.672	0.713
Nb, Niobium (ppm)	75	4.7	73	77	73	78
Nd, Neodymium (ppm)	1.87	0.28	1.66	2.07	1.74	1.99
Ni, Nickel (ppm)	47.5	1.74	46.8	48.2	46.4	48.6
P, Phosphorus (wt.%)	0.016	0.002	0.015	0.017	0.015	0.017
Pb, Lead (ppm)	5.17	0.304	4.99	5.35	4.78	5.56
S, Sulphur (wt.%)	0.020	0.002	0.019	0.020	0.017	0.022
Sb, Antimony (ppm)	1.11	0.095	1.07	1.15	1.05	1.17
Sc, Scandium (ppm)	1.83	0.37	1.64	2.02	1.74	1.92
Sn, Tin (ppm)	63	5.5	61	66	61	66
Sr, Strontium (ppm)	16.9	2.9	15.5	18.4	16.0	17.9
Ta, Tantalum (ppm)	49.0	4.00	47.3	50.7	47.0	51.0
Tb, Terbium (ppm)	0.15	0.02	0.13	0.17	IND	IND
Ti, Titanium (wt.%)	0.034	0.001	0.034	0.035	0.034	0.035
Tl, Thallium (ppm)	4.26	0.188	4.17	4.34	4.09	4.42
U, Uranium (ppm)	2.12	0.37	1.94	2.31	2.01	2.23
V, Vanadium (ppm)	14.8	1.18	14.2	15.3	13.8	15.7
W, Tungsten (ppm)	6.97	0.546	6.71	7.23	6.65	7.29
Yb, Ytterbium (ppm)	0.24	0.022	0.22	0.25	0.21	0.26
Zn, Zinc (ppm)	71	4.1	70	73	69	74
Zr, Zirconium (ppm)	20.0	2.3	18.9	21.1	18.7	21.3

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

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 2. Indicative Values for OREAS 999.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Peroxide Fusion ICP</b>								
Ag	ppm	3.84	In	ppm	< 0.2	Sc	ppm	< 5
As	ppm	5.68	Lu	ppm	0.054	Se	ppm	2.64
B	ppm	< 50	Na	wt.%	0.628	Te	ppm	0.41
Cd	ppm	< 0.2	Pb	ppm	6.66	Tm	ppm	0.037
Eu	ppm	0.071	Re	ppm	< 0.1	V	ppm	14.8
Hf	ppm	2.09	S	wt.%	0.021	Yb	ppm	0.26
<b>Borate Fusion XRF</b>								
As <sub>2</sub> O <sub>3</sub>	ppm	< 10	MoO <sub>3</sub>	ppm	< 10	SrO	ppm	48.0
BaO	ppm	81	Nb <sub>2</sub> O <sub>5</sub>	ppm	223	Ta <sub>2</sub> O <sub>5</sub>	ppm	70
Cl	ppm	325	NiO	ppm	61	V <sub>2</sub> O <sub>5</sub>	ppm	< 100
CoO	ppm	17.5	PbO	ppm	23.2	WO <sub>3</sub>	ppm	11.7
Cr <sub>2</sub> O <sub>3</sub>	ppm	153	Sb <sub>2</sub> O <sub>3</sub>	ppm	< 10	ZnO	ppm	118
CuO	ppm	33.3	SnO <sub>2</sub>	ppm	90	ZrO <sub>2</sub>	ppm	77
<b>Thermogravimetry</b>								
H <sub>2</sub> O-	wt.%	0.330						
<b>4-Acid Digestion</b>								
Ag	ppm	0.137	Hg	ppm	< 2	Si	wt.%	31.71
B	ppm	7.43	In	ppm	0.022	Sm	ppm	0.83
Cd	ppm	0.076	Lu	ppm	0.028	Te	ppm	< 0.05
Ce	ppm	2.64	Pr	ppm	0.46	Th	ppm	2.20
Eu	ppm	0.060	Rb	ppm	338	Tm	ppm	0.028
Gd	ppm	0.79	Re	ppm	< 0.002	Y	ppm	2.58
Ge	ppm	0.12	Se	ppm	< 1			

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

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.

## 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.

## SOURCE MATERIAL

OREAS 999 is a spodumene concentrate product derived from the processing of lithium pegmatite ores sourced from the Eastern Goldfields Region of Western Australia.

## COMMINUTION AND HOMOGENISATION PROCEDURES

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

- Drying to constant mass at 105°C;
- Milling 100% minus 30 microns;
- Homogenisation;
- Packaging in 10g units in laminated foil pouches and 500g units in plastic wide-mouth jars.

## ANALYTICAL PROGRAM

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

- Sodium peroxide fusion with full suite elemental package by ICP-OES and/or MS finish (20 laboratories);
- Lithium borate fusion whole rock analysis package by X-ray fluorescence (19 laboratories);
- Thermogravimetry: Moisture at 105°C (2 laboratories as a part of their fusion package) and Loss on Ignition (LOI) at 1000°C (8 laboratories used a thermogravimetric analyser, 4 laboratories used conventional muffle furnace and 9 laboratories included LOI with their fusion package);
- 4-acid digestion for full suite elemental package by ICP-OES and MS finish (up to 24 laboratories depending on the element).

For the round robin program twelve 300g 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 10g scoop splits from each of three separate 300g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance.

Table 1 presents the 101 certified values (including Li in both elemental and oxide form for peroxide fusion and 4-acid digestion) together with their associated 1SD's, 95% confidence and tolerance limits, Table 2 shows 57 indicative values and Table 3 provides performance gate intervals for the certified values based on their pooled 1SD's. Tabulated results of all elements 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 999 DataPack-1.0.190208\_104741.xlsx**).

Results are also presented in scatter plots for Li<sub>2</sub>O (wt.%) by peroxide fusion ICP and 4-acid digestion in Figure 1 and 2 respectively, together with ±3SD (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.

## STATISTICAL ANALYSIS

**Certified Values, Standard Deviations, Confidence and Tolerance Limits** have been determined for each analytical method following removal of individual and laboratory outliers (Table 1). Certified Values are the mean of means after outlier filtering. The 95% Confidence Limit is a measure of the reliability of the certified value, i.e. the narrower the Confidence Interval the greater the certainty in the Certified Value. It should not be used as a control limit for laboratory performance.

**95% Confidence Limits** are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. *95% Confidence Limits should not be used as control limits for laboratory performance.*

**Indicative (uncertified) values** (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient ( $< 5$ ) to support certification; ii) interlaboratory consensus is poor; or iii) a significant proportion of results are outlying or reported as less than detection limits.

**Standard Deviation** values (1SDs) are reported in Table 1. They 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.**

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 inter-lab 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.

Table 3 shows **Performance Gates** 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. 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)  $\pm$  10%.

*i.e. Certified Value  $\pm$  10%  $\pm$  2DL (adapted from Govett, 1983)*

**Table 3. Performance Gates for OREAS 999.**

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>Peroxide Fusion ICP</b>											
Al, wt. %	12.23	0.411	11.41	13.06	11.00	13.47	3.36%	6.72%	10.08%	11.62	12.84
Ba, ppm	39.1	2.64	33.8	44.3	31.2	47.0	6.75%	13.49%	20.24%	37.1	41.0
Be, ppm	51	2.1	46	55	44	57	4.12%	8.24%	12.36%	48	53
Bi, ppm	2.21	0.185	1.84	2.58	1.65	2.76	8.40%	16.81%	25.21%	2.10	2.32
Ca, wt. %	0.481	0.059	0.363	0.599	0.304	0.658	12.25%	24.51%	36.76%	0.457	0.505
Ce, ppm	4.15	0.360	3.43	4.87	3.07	5.23	8.66%	17.32%	25.99%	3.94	4.36
Co, ppm	5.24	0.343	4.56	5.93	4.21	6.27	6.54%	13.08%	19.63%	4.98	5.50
Cr, ppm	112	17	78	145	62	162	14.90%	29.79%	44.69%	106	117
Cs, ppm	93	3.6	86	100	82	104	3.90%	7.80%	11.70%	89	98
Cu, ppm	25.5	3.4	18.7	32.3	15.3	35.7	13.29%	26.58%	39.87%	24.2	26.8
Dy, ppm	0.80	0.050	0.71	0.90	0.66	0.95	6.17%	12.34%	18.51%	0.76	0.84
Er, ppm	0.27	0.03	0.20	0.33	0.17	0.36	11.86%	23.72%	35.58%	0.25	0.28
Fe, wt. %	1.73	0.067	1.60	1.87	1.53	1.93	3.85%	7.69%	11.54%	1.65	1.82
Ga, ppm	88	6.4	75	101	68	107	7.32%	14.63%	21.95%	83	92
Gd, ppm	1.08	0.16	0.77	1.39	0.61	1.55	14.55%	29.10%	43.65%	1.03	1.13
Ge, ppm	4.31	0.45	3.41	5.22	2.95	5.68	10.52%	21.04%	31.56%	4.10	4.53
Ho, ppm	0.11	0.011	0.09	0.14	0.08	0.15	9.95%	19.89%	29.84%	0.11	0.12
K, wt. %	0.522	0.048	0.426	0.617	0.378	0.665	9.16%	18.32%	27.49%	0.496	0.548
La, ppm	1.84	0.25	1.35	2.33	1.10	2.57	13.35%	26.70%	40.05%	1.74	1.93
Li, wt. %	2.67	0.103	2.47	2.88	2.36	2.98	3.86%	7.73%	11.59%	2.54	2.81
Li <sub>2</sub> O, wt. %	5.76	0.222	5.31	6.20	5.09	6.42	3.86%	7.73%	11.59%	5.47	6.05
Mg, wt. %	0.473	0.020	0.432	0.514	0.412	0.534	4.32%	8.64%	12.96%	0.449	0.497
Mn, wt. %	0.147	0.006	0.136	0.158	0.130	0.163	3.79%	7.59%	11.38%	0.139	0.154
Mo, ppm	2.27	0.38	1.51	3.02	1.13	3.40	16.68%	33.35%	50.03%	2.15	2.38
Nb, ppm	74	6.2	61	86	55	92	8.42%	16.84%	25.25%	70	77
Nd, ppm	1.90	0.22	1.46	2.34	1.24	2.55	11.54%	23.09%	34.63%	1.80	1.99
Ni, ppm	52	4.2	44	61	40	65	7.99%	15.97%	23.96%	50	55
P, wt. %	0.016	0.005	0.006	0.026	0.001	0.031	31.10%	62.20%	93.30%	0.015	0.017
Pr, ppm	0.52	0.06	0.39	0.65	0.32	0.71	12.40%	24.79%	37.19%	0.49	0.54
Rb, ppm	423	20	383	462	364	482	4.66%	9.31%	13.97%	402	444

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt. %  $\equiv$  1000 ppb, parts per billion.

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 3. Performance Gates 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>Peroxide Fusion ICP continued</b>											
Sb, ppm	1.02	0.17	0.69	1.36	0.53	1.52	16.22%	32.43%	48.65%	0.97	1.08
Si, wt. %	30.30	0.680	28.94	31.66	28.26	32.34	2.24%	4.49%	6.73%	28.78	31.81
Sm, ppm	1.02	0.099	0.82	1.21	0.72	1.31	9.75%	19.50%	29.25%	0.96	1.07
Sn, ppm	84	5.3	73	94	68	99	6.30%	12.61%	18.91%	79	88
Sr, ppm	23.0	3.3	16.3	29.7	13.0	33.0	14.54%	29.09%	43.63%	21.8	24.1
Ta, ppm	45.5	2.08	41.4	49.7	39.3	51.8	4.56%	9.12%	13.68%	43.3	47.8
Tb, ppm	0.18	0.02	0.14	0.22	0.12	0.24	11.11%	22.22%	33.33%	0.17	0.19
Th, ppm	3.01	0.158	2.70	3.33	2.54	3.48	5.24%	10.47%	15.71%	2.86	3.16
Ti, wt. %	0.034	0.004	0.026	0.043	0.022	0.047	11.99%	23.98%	35.97%	0.033	0.036
Tl, ppm	4.26	0.253	3.76	4.77	3.50	5.02	5.94%	11.88%	17.83%	4.05	4.48
U, ppm	2.51	0.226	2.06	2.96	1.83	3.19	9.02%	18.04%	27.07%	2.38	2.64
W, ppm	6.94	0.391	6.16	7.73	5.77	8.12	5.63%	11.27%	16.90%	6.60	7.29
Y, ppm	4.19	0.43	3.33	5.05	2.90	5.48	10.25%	20.50%	30.75%	3.98	4.40
Zn, ppm	77	4.3	68	85	64	90	5.58%	11.15%	16.73%	73	81
Zr, ppm	32.9	3.4	26.1	39.7	22.8	43.0	10.27%	20.54%	30.81%	31.3	34.5
<b>Borate Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	23.39	0.114	23.16	23.62	23.05	23.74	0.49%	0.98%	1.47%	22.22	24.56
CaO, wt. %	0.670	0.013	0.643	0.696	0.630	0.709	1.98%	3.96%	5.93%	0.636	0.703
Fe <sub>2</sub> O <sub>3</sub> , wt. %	2.49	0.033	2.42	2.55	2.39	2.59	1.31%	2.62%	3.94%	2.37	2.61
K <sub>2</sub> O, wt. %	0.612	0.009	0.593	0.630	0.583	0.640	1.55%	3.09%	4.64%	0.581	0.642
MgO, wt. %	0.796	0.012	0.772	0.820	0.760	0.832	1.50%	2.99%	4.49%	0.756	0.836
MnO, wt. %	0.192	0.005	0.182	0.201	0.177	0.206	2.50%	5.01%	7.51%	0.182	0.201
Na <sub>2</sub> O, wt. %	0.945	0.024	0.897	0.993	0.873	1.017	2.54%	5.08%	7.62%	0.897	0.992
P <sub>2</sub> O <sub>5</sub> , wt. %	0.038	0.004	0.031	0.045	0.027	0.049	9.65%	19.30%	28.95%	0.036	0.040
SiO <sub>2</sub> , wt. %	64.23	0.446	63.34	65.12	62.89	65.56	0.69%	1.39%	2.08%	61.02	67.44
SO <sub>3</sub> , wt. %	0.042	0.006	0.029	0.054	0.023	0.060	14.63%	29.26%	43.89%	0.040	0.044
TiO <sub>2</sub> , wt. %	0.060	0.007	0.047	0.073	0.041	0.080	10.81%	21.63%	32.44%	0.057	0.063
<b>Thermogravimetry</b>											
LOI <sup>1000</sup> , wt. %	0.764	0.114	0.536	0.993	0.421	1.107	14.96%	29.91%	44.87%	0.726	0.802
<b>4-Acid Digestion</b>											
Al, wt. %	10.77	1.42	7.92	13.62	6.50	15.04	13.22%	26.44%	39.66%	10.23	11.31
As, ppm	5.36	1.07	3.22	7.49	2.15	8.56	19.94%	39.89%	59.83%	5.09	5.62
Ba, ppm	39.6	2.34	34.9	44.3	32.6	46.6	5.90%	11.81%	17.71%	37.6	41.6
Be, ppm	49.8	3.49	42.8	56.8	39.3	60.2	7.01%	14.01%	21.02%	47.3	52.3
Bi, ppm	2.11	0.131	1.85	2.37	1.72	2.51	6.18%	12.36%	18.55%	2.01	2.22
Ca, wt. %	0.450	0.033	0.384	0.516	0.351	0.548	7.31%	14.63%	21.94%	0.427	0.472
Co, ppm	4.95	0.262	4.43	5.48	4.17	5.74	5.29%	10.58%	15.88%	4.71	5.20
Cr, ppm	81	21	40	122	19	142	25.49%	50.97%	76.46%	77	85
Cs, ppm	88	11	66	110	56	121	12.31%	24.61%	36.92%	84	93
Cu, ppm	25.4	2.34	20.8	30.1	18.4	32.5	9.19%	18.38%	27.56%	24.2	26.7
Dy, ppm	0.67	0.13	0.42	0.93	0.30	1.05	18.72%	37.45%	56.17%	0.64	0.71

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt. %  $\equiv$  1000 ppb, parts per billion.

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 3. Performance Gates 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>											
Er, ppm	0.23	0.05	0.14	0.33	0.10	0.37	19.56%	39.12%	58.68%	0.22	0.25
Fe, wt.%	1.62	0.097	1.42	1.81	1.33	1.91	5.99%	11.99%	17.98%	1.54	1.70
Ga, ppm	82	5.4	71	93	66	98	6.58%	13.16%	19.74%	78	86
Hf, ppm	1.98	0.171	1.64	2.33	1.47	2.50	8.62%	17.23%	25.85%	1.89	2.08
Ho, ppm	0.090	0.018	0.055	0.125	0.037	0.142	19.54%	39.07%	58.61%	0.085	0.094
K, wt.%	0.500	0.041	0.418	0.582	0.377	0.623	8.20%	16.40%	24.60%	0.475	0.525
La, ppm	1.68	0.34	1.00	2.36	0.66	2.69	20.24%	40.48%	60.71%	1.59	1.76
Li, wt.%	2.65	0.057	2.54	2.76	2.48	2.82	2.14%	4.27%	6.41%	2.52	2.78
Li <sub>2</sub> O, wt.%	5.70	0.122	5.46	5.95	5.34	6.07	2.14%	4.27%	6.41%	5.42	5.99
Mg, wt.%	0.410	0.066	0.277	0.543	0.211	0.609	16.20%	32.41%	48.61%	0.389	0.430
Mn, wt.%	0.143	0.006	0.132	0.154	0.126	0.160	3.96%	7.92%	11.89%	0.136	0.150
Mo, ppm	2.06	0.140	1.78	2.34	1.64	2.48	6.81%	13.63%	20.44%	1.95	2.16
Na, wt.%	0.693	0.023	0.646	0.739	0.622	0.763	3.38%	6.76%	10.14%	0.658	0.727
Nb, ppm	75	4.7	66	85	61	89	6.20%	12.39%	18.59%	72	79
Nd, ppm	1.87	0.28	1.31	2.42	1.03	2.70	14.85%	29.70%	44.55%	1.77	1.96
Ni, ppm	47.5	1.74	44.0	51.0	42.3	52.7	3.65%	7.31%	10.96%	45.1	49.9
P, wt.%	0.016	0.002	0.013	0.019	0.011	0.021	10.59%	21.18%	31.76%	0.015	0.017
Pb, ppm	5.17	0.304	4.56	5.78	4.26	6.08	5.88%	11.76%	17.65%	4.91	5.43
S, wt.%	0.020	0.002	0.016	0.023	0.015	0.024	8.00%	16.00%	24.00%	0.019	0.021
Sb, ppm	1.11	0.095	0.92	1.30	0.83	1.39	8.51%	17.03%	25.54%	1.05	1.17
Sc, ppm	1.83	0.37	1.09	2.57	0.72	2.94	20.19%	40.37%	60.56%	1.74	1.92
Sn, ppm	63	5.5	52	74	47	80	8.62%	17.23%	25.85%	60	67
Sr, ppm	16.9	2.9	11.2	22.7	8.3	25.6	16.97%	33.93%	50.90%	16.1	17.8
Ta, ppm	49.0	4.00	41.0	57.0	37.0	61.0	8.17%	16.33%	24.50%	46.6	51.5
Tb, ppm	0.15	0.02	0.10	0.20	0.08	0.23	16.31%	32.62%	48.94%	0.14	0.16
Ti, wt.%	0.034	0.001	0.031	0.037	0.030	0.039	4.31%	8.63%	12.94%	0.033	0.036
Tl, ppm	4.26	0.188	3.88	4.63	3.69	4.82	4.43%	8.86%	13.29%	4.04	4.47
U, ppm	2.12	0.37	1.38	2.86	1.01	3.23	17.43%	34.87%	52.30%	2.02	2.23
V, ppm	14.8	1.18	12.4	17.1	11.2	18.3	7.97%	15.94%	23.91%	14.0	15.5
W, ppm	6.97	0.546	5.88	8.07	5.34	8.61	7.83%	15.66%	23.49%	6.62	7.32
Yb, ppm	0.24	0.022	0.19	0.28	0.17	0.30	9.50%	19.00%	28.50%	0.22	0.25
Zn, ppm	71	4.1	63	80	59	84	5.72%	11.45%	17.17%	68	75
Zr, ppm	20.0	2.3	15.5	24.6	13.2	26.9	11.39%	22.79%	34.18%	19.0	21.0

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

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.

**Tolerance Limits** (ISO Guide 3207) 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 oxide (Li<sub>2</sub>O) by peroxide fusion ICP, where 99% of the time (1- $\alpha$ =0.99) at least 95% of subsamples ( $\rho$ =0.95) will have concentrations lying between 5.63 and 5.88 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 Guide 35). *Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.*

### **ANOVA Study**

The homogeneity of OREAS 999 has also 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 that no evidence exists that between-unit variance is greater than within-unit variance.

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 999 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 999 is sufficiently homogenous and is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## **PARTICIPATING LABORATORIES**

1. AGAT Laboratories, Mississauga, Ontario, Canada
2. Alex Stewart International, Mendoza, Argentina
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Perth, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
10. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
11. Bureau Veritas Geoanalytical, Perth, WA, Australia
12. Inspectorate (BV), Lima, Peru
13. Intertek Genalysis, Perth, WA, Australia
14. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
15. Nagrom, Perth, WA, Australia
16. Ontario Geological Survey, Sudbury, Ontario, Canada
17. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
18. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
19. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
20. SGS, Randfontein, Gauteng, South Africa
21. SGS Australia Mineral Services, Perth, WA, Australia
22. SGS Canada Inc., Vancouver, BC, Canada
23. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
24. UIS Analytical Services, Centurion, South Africa

***Please Note: Above numbered alphabetical list of participating laboratories does not reflect the Lab ID numbering on the scatter plots below***

Figure 1. Li<sub>2</sub>O by PF ICP in OREAS 999

SPC.1422.RR1.OREAS 999.4.PF ICP.Li2O.Lab.190208.121819.SN

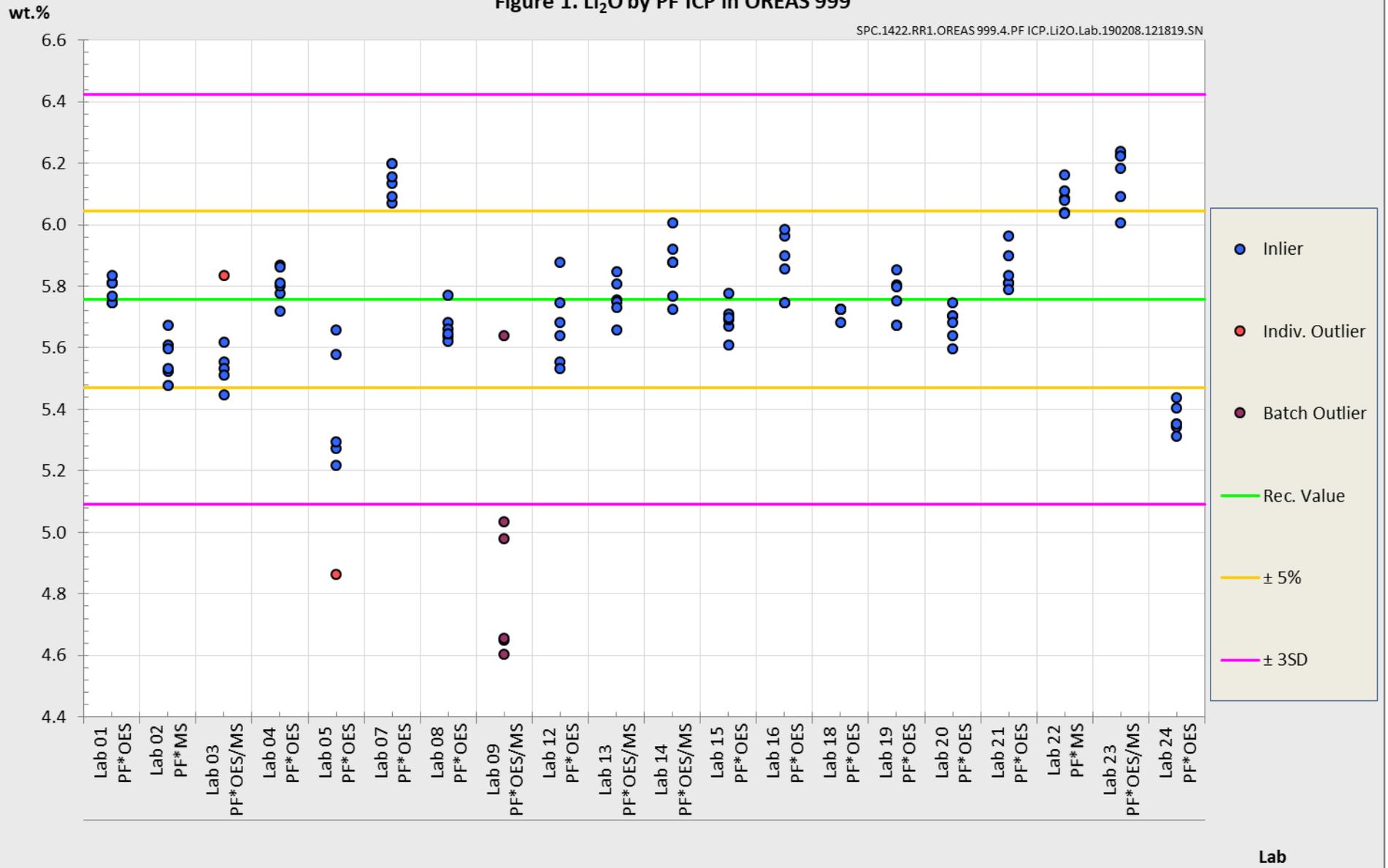
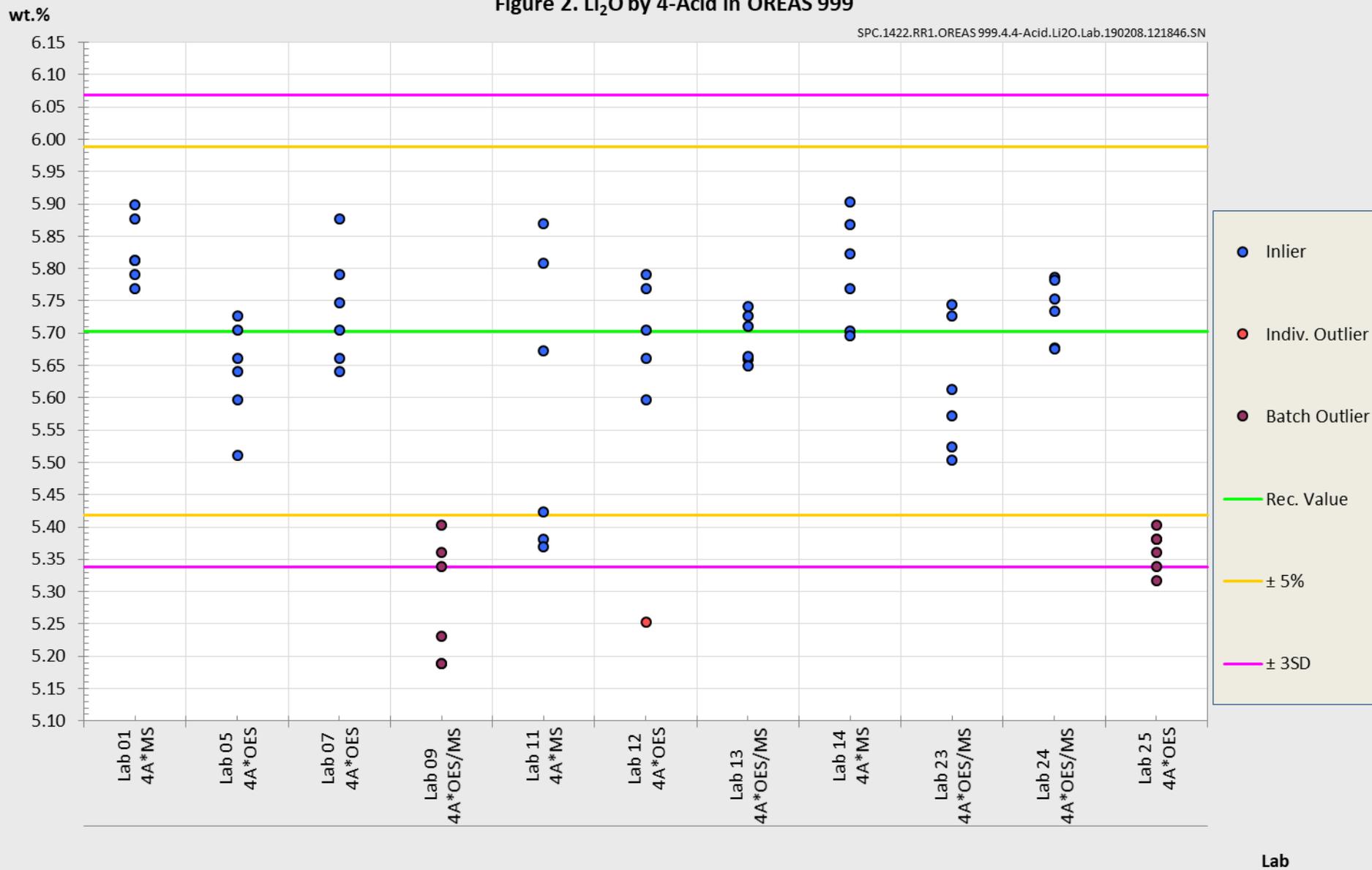


Figure 2. Li<sub>2</sub>O by 4-Acid in OREAS 999

SPC.1422.RR1.OREAS 999.4.4-Acid.Li2O.Lab.190208.121846.SN



## PREPARER AND SUPPLIER

Certified reference material OREAS 999 was prepared, certified and supplied by:



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

Tel: +613-9729 0333  
Fax: +613-9729 8338  
Web: [www.ore.com.au](http://www.ore.com.au)  
Email: [info@ore.com.au](mailto:info@ore.com.au)

It is packaged in 10g units in laminated foil packets and in 500g units in wide-mouth plastic jars.

## METROLOGICAL TRACEABILITY

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' 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 that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

Guide ISO/TR 16476:2016, 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 method is possible. In this case, certification takes place on the basis of agreement among independent measurement results** (see ISO Guide 35:2006, Clause 10)."*

## COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) 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 their field samples.

## INTENDED USE

OREAS 999 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 999 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 999 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 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.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 999 was sourced from Li concentrate (spodumene) and is low in reactive sulphides. In its unopened state and under normal conditions of storage it has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

## INSTRUCTIONS FOR CORRECT USE

The certified values for lithium borate fusion XRF and for LOI are on a 'dry sample' basis whilst all other certified values are reported on a 'sample as received' basis.

## HANDLING INSTRUCTIONS

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

## 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
1	22 <sup>nd</sup> February 2019	Updated 'SOURCE MATERIAL' section. Table of content got 'QMS ACCREDITED' link added.
0	11 <sup>th</sup> February 2019	First publication.

## QMS ACCREDITED

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



## CERTIFYING OFFICER

A handwritten signature in black ink, appearing to read 'S. Hamlyn'.

22<sup>nd</sup> February, 2019

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

## REFERENCES

- Govett, G.J.S. (1983), ed. Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).
- ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.
- ISO Guide 31 (2015), Reference materials – Contents of certificates and labels.
- ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.
- ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.