

## **CERTIFICATE OF ANALYSIS FOR**

# OREAS 750b

(Pegmatite Li Ore, Western Australia)





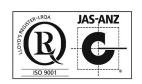


Table 1. Certified Values, Uncertainty & Tolerance Intervals for multi-elements by 4-acid digestion in OREAS 750b.

OREAS 750D.									
Constituent	Certified	95% Expande	d Uncertainty	95% Tolera	ance Limits				
Jonstituent	Value <sup>†</sup>	Low	High	Low	High				
4-Acid Digestion									
Ag, Silver (ppm)	0.094	0.071	0.117	IND	IND				
Al, Aluminium (wt.%)	3.91	3.81	4.01	3.84	3.98				
As, Arsenic (ppm)	2.88	2.40	3.36	2.55	3.21				
Ba, Barium (ppm)	370	361	379	364	376				
Be, Beryllium (ppm)	3.53	3.38	3.67	3.42	3.63				
Bi, Bismuth (ppm)	0.44	0.38	0.50	0.39	0.48				
Ca, Calcium (wt.%)	0.609	0.589	0.628	0.598	0.619				
Cd, Cadmium (ppm)	0.090	0.061	0.119	IND	IND				
Ce, Cerium (ppm)	29.8	27.6	31.9	28.8	30.7				
Co, Cobalt (ppm)	2.99	2.87	3.12	2.87	3.11				
Cr, Chromium (ppm)	17.9	15.6	20.3	16.9	19.0				
Cs, Caesium (ppm)	39.2	38.0	40.5	38.4	40.0				
Cu, Copper (ppm)	10.7	10.0	11.5	10.2	11.3				
Dy, Dysprosium (ppm)	1.52	1.37	1.67	1.42	1.62				
Er, Erbium (ppm)	0.59	0.53	0.65	0.55	0.63				
Eu, Europium (ppm)	0.52	0.45	0.58	0.49	0.54				
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	1.53	1.49	1.58	1.51	1.56				
Ga, Gallium (ppm)	16.8	16.2	17.5	16.4	17.3				
Gd, Gadolinium (ppm)	2.28	2.09	2.47	2.13	2.43				
Hf, Hafnium (ppm)	1.21	1.12	1.30	1.16	1.26				
Ho, Holmium (ppm)	0.24	0.21	0.27	0.22	0.26				
In, Indium (ppm)	0.021	0.017	0.025	0.018	0.024				
K, Potassium (wt.%)	1.15	1.12	1.18	1.13	1.17				
La, Lanthanum (ppm)	14.4	13.5	15.4	14.0	14.8				
Li, Lithium (wt.%)	0.245	0.238	0.251	0.241	0.249				
Li <sub>2</sub> O, Lithium oxide (wt.%)	0.527	0.513	0.541	0.519	0.535				
Lu, Lutetium (ppm)	0.065	0.054	0.077	IND	IND				
Mg, Magnesium (wt.%)	0.267	0.257	0.276	0.260	0.273				
Mn, Manganese (wt.%)	0.039	0.038	0.040	0.038	0.040				
Mo, Molybdenum (ppm)	1.60	1.47	1.73	1.51	1.69				
Na, Sodium (wt.%)	0.755	0.734	0.775	0.743	0.767				
Nb, Niobium (ppm)	28.4	27.3	29.6	27.4	29.4				
Nd, Neodymium (ppm)	13.5	12.4	14.6	13.1	13.9				
Ni, Nickel (ppm)	7.66	7.25	8.07	7.39	7.94				
P, Phosphorus (wt.%)	0.039	0.038	0.041	0.038	0.041				
1			1		I.				

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. IND = indeterminate (due to limited reading resolution of the methods employed).

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<sup>&</sup>lt;sup>†</sup>The operationally defined measurands meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

Table 1 continued.

	Certified	95% Expand	ed Uncertainty	95% Tolerance Limits		
Constituent	Value <sup>†</sup>	Low	High	Low	High	
4-Acid Digestion continue		LOW	T IIIgii	LOW	Tilgii	
Pb, Lead (ppm)	9.55	9.17	9.93	9.24	9.86	
Pr, Praseodymium (ppm)	3.65	3.34	3.96	3.41	3.89	
Rb, Rubidium (ppm)	288	276	299	282	293	
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND	
S, Sulphur (wt.%)	0.045	0.041	0.049	0.043	0.047	
Sb, Antimony (ppm)	0.39	0.35	0.42	0.35	0.42	
Sc, Scandium (ppm)	3.20	3.00	3.40	3.07	3.33	
Sm, Samarium (ppm)	2.69	2.47	2.91	2.57	2.81	
Sn, Tin (ppm)	25.8	24.3	27.2	24.8	26.7	
Sr, Strontium (ppm)	60	58	63	59	62	
Ta, Tantalum (ppm)	64	61	67	62	66	
Tb, Terbium (ppm)	0.31	0.28	0.33	0.29	0.32	
Te, Tellurium (ppm)	< 0.05	IND	IND	IND	IND	
Th, Thorium (ppm)	5.94	5.61	6.27	5.71	6.17	
Ti, Titanium (wt.%)	0.151	0.145	0.156	0.147	0.154	
TI, Thallium (ppm)	1.93	1.86	2.00	1.89	1.98	
Tm, Thulium (ppm)	0.071	0.054	0.088	IND	IND	
U, Uranium (ppm)	1.91	1.70	2.13	1.77	2.06	
V, Vanadium (ppm)	22.8	21.6	23.9	22.0	23.6	
W, Tungsten (ppm)	5.08	4.52	5.64	4.72	5.44	
Y, Yttrium (ppm)	6.13	5.81	6.45	5.93	6.33	
Yb, Ytterbium (ppm)	0.46	0.40	0.53	0.43	0.50	
Zn, Zinc (ppm)	60	57	62	58	61	
Zr, Zirconium (ppm)	27.8	26.0	29.7	26.5	29.1	

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.

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).

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<sup>&</sup>lt;sup>†</sup>The operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 750b.

	•			95 % Tolerance Limits			
Constituent	Certified Value	-	led Uncertainty		1		
Denote / Denoted a Fuel on 10		Low	High	Low	High		
Borate / Peroxide Fusion IC		0.00	4.44	2.05	4.00		
Al, Aluminium (wt.%)	4.02	3.92	4.11	3.95	4.08		
Ba, Barium (ppm)	371	358	384	363	378		
Be, Beryllium (ppm)	3.88	3.31	4.45	IND	IND		
Bi, Bismuth (ppm)	0.46	0.24	0.67	IND	IND		
Ca, Calcium (wt.%)	0.601	0.551	0.650	0.581	0.620		
Ce, Cerium (ppm)	29.6	28.0	31.1	28.3	30.8		
Co, Cobalt (ppm)	3.13	2.90	3.37	2.86	3.41		
Cs, Caesium (ppm)	39.1	37.7	40.4	38.2	39.9		
Dy, Dysprosium (ppm)	2.21	1.99	2.43	2.02	2.40		
Er, Erbium (ppm)	1.13	0.98	1.28	1.03	1.24		
Eu, Europium (ppm)	0.52	0.45	0.58	0.47	0.56		
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	1.56	1.50	1.61	1.52	1.59		
Ga, Gallium (ppm)	16.9	15.9	18.0	16.4	17.5		
Gd, Gadolinium (ppm)	2.64	2.34	2.95	2.53	2.76		
Ge, Germanium (ppm)	1.49	0.51	2.47	IND	IND		
Ho, Holmium (ppm)	0.40	0.35	0.44	0.37	0.43		
In, Indium (ppm)	< 0.2	IND	IND	IND	IND		
K, Potassium (wt.%)	1.18	1.14	1.23	1.16	1.21		
La, Lanthanum (ppm)	14.6	13.4	15.8	14.1	15.1		
Li, Lithium (wt.%)	0.246	0.237	0.255	0.241	0.251		
Li <sub>2</sub> O, Lithium oxide (wt.%)	0.529	0.510	0.548	0.518	0.540		
Lu, Lutetium (ppm)	0.16	0.12	0.19	IND	IND		
Mg, Magnesium (wt.%)	0.266	0.254	0.278	0.256	0.276		
Mn, Manganese (wt.%)	0.039	0.038	0.040	0.038	0.040		
Nb, Niobium (ppm)	29.3	27.5	31.1	27.9	30.7		
Nd, Neodymium (ppm)	13.2	12.2	14.2	12.7	13.7		
P, Phosphorus (wt.%)	0.039	0.038	0.040	0.038	0.040		
Pb, Lead (ppm)	9.64	8.14	11.14	IND	IND		
Pr, Praseodymium (ppm)	3.49	3.27	3.70	3.28	3.69		
Rb, Rubidium (ppm)	287	273	300	280	293		
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND		
Si, Silicon (wt.%)	40.13	38.92	41.34	39.53	40.73		
Sm, Samarium (ppm)	2.79	2.52	3.06	2.64	2.94		
Sn, Tin (ppm)	38.7	36.6	40.9	36.6	40.9		
Sr, Strontium (ppm)	62	57	67	60	64		
Ta, Tantalum (ppm)	69	64	73	66	71		
Tb, Terbium (ppm)	0.40	0.36	0.44	0.37	0.43		
Stunit equivalents: nnm (narts ne							

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.

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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 continued.

Comptitue of	Certified	95% Expande	ed Uncertainty	95% Tolerance Limits		
Constituent	Value	Low	High	Low	High	
Borate / Peroxide Fusion ICP continued						
Th, Thorium (ppm)	5.96	5.58	6.33	5.67	6.24	
Ti, Titanium (wt.%)	0.154	0.145	0.163	0.147	0.161	
TI, Thallium (ppm)	1.96	1.84	2.09	1.77	2.16	
Tm, Thulium (ppm)	0.16	0.14	0.18	IND	IND	
U, Uranium (ppm)	2.28	1.90	2.65	2.03	2.52	
V, Vanadium (ppm)	22.8	20.7	24.8	19.8	25.7	
W, Tungsten (ppm)	5.54	4.37	6.71	4.49	6.59	
Y, Yttrium (ppm)	11.4	10.4	12.3	10.4	12.3	
Yb, Ytterbium (ppm)	1.04	0.87	1.21	IND	IND	
Zn, Zinc (ppm)	57	49	64	54	59	
Borate Fusion XRF						
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	7.63	7.56	7.70	7.57	7.70	
BaO, Barium oxide (ppm)	407	337	478	379	436	
CaO, Calcium oxide (wt.%)	0.851	0.836	0.867	0.838	0.865	
Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)	< 100	IND	IND	IND	IND	
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	1.56	1.54	1.59	1.54	1.58	
K <sub>2</sub> O, Potassium oxide (wt.%)	1.39	1.38	1.41	1.38	1.41	
MgO, Magnesium oxide (wt.%)	0.452	0.431	0.473	0.443	0.462	
MnO, Manganese oxide (wt.%)	0.050	0.050	0.051	0.049	0.052	
Na <sub>2</sub> O, Sodium oxide (wt.%)	1.01	0.98	1.04	0.99	1.03	
Nb, Niobium (ppm)	< 50	IND	IND	IND	IND	
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.088	0.083	0.093	0.086	0.090	
Rb, Rubidium (ppm)	259	229	290	222	296	
SiO <sub>2</sub> , Silicon dioxide (wt.%)	85.46	84.85	86.08	84.85	86.08	
SO <sub>3</sub> , Sulphur trioxide (wt.%)	0.105	0.093	0.117	0.096	0.114	
SrO, Strontium oxide (ppm)	96	80	112	IND	IND	
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.257	0.248	0.265	0.249	0.264	
Thermogravimetry						
LOI <sup>1000</sup> , Loss On Ignition @1000°C (wt.%)	0.570	0.500	0.640	0.538	0.602	

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.

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).

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Table 3. Indicative Values for OREAS 750b.

Ag pp As pp B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	om	10 0.14 10 14.3	Hg Se	ppm ppm	< 1 1.34	Constituent	Unit	Value						
B pp Ge pp Peroxide Fusion Ag pp As pp B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp	om	: 10	Se											
Ge pp Peroxide Fusion Ag pp As pp B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	om 0 ICP om < om 2 om 1	: 10	Se											
Ag pp As pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp	iCP om < om 2 om 1	: 10		ppm	1.34									
Ag pp As pp B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	om < om 2 om 1		Hf											
As pp B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	om 2 om 1		Hf	Peroxide Fusion ICP										
B pp Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	om 1	4.3		ppm	3.67	Sb	ppm	0.48						
Cd pp Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp			Hg	ppm	< 5	Sc	ppm	< 5						
Cr pp Cu pp Borate Fusion XF Ag pp As pp Be pp	m -	7.6	Мо	ppm	2.11	Se	ppm	< 10						
Cu pp  Borate Fusion XF  Ag pp  As pp  Be pp	ן וווע <	: 10	Na	wt.%	0.740	Te	ppm	< 1						
Ag pp As pp Be pp	om 4	2.2	Ni	ppm	18.8	Zr	ppm	114						
Ag pp As pp Be pp	om 1	6.1	S	wt.%	0.042									
As pp Be pp	RF													
Be pp	om <i>0.</i>	097	Но	ppm	0.42	Та	ppm	55						
	om 3	7.2	In	ppm	< 0.1	Tb	ppm	0.38						
	om 3	2.50	La	ppm	33.4	Te	ppm	< 0.1						
Ві рр	om <	100	Lu	ppm	0.15	Th	ppm	17.0						
Ce pp	om 3	1.2	Мо	ppm	< 50	TI	ppm	1.97						
Со рр	om <	100	Nd	ppm	14.0	Tm	ppm	0.17						
Cs pp	om 4	0.9	NiO	ppm	28.7	U	ppm	5.33						
CuO pp	om 4	3.0	Pb	ppm	< 50	V <sub>2</sub> O <sub>5</sub>	ppm	50						
Dy pp	om 2	2.12	Pr	ppm	3.59	W	ppm	6.28						
Er pp	om <i>1</i>	.11	Re	ppm	< 0.1	Y	ppm	23.3						
Eu pp	om 0	.51	Sb	ppm	< 50	Yb	ppm	1.03						
Ga pp	om <i>1</i>	6.6	Sc	ppm	4.17	Zn	ppm	69						
Gd pp	om 2	2.65	Se	ppm	1.25	Zr	ppm	128						
H <sub>2</sub> O- wt.	.% 0.	107	Sm	ppm	2.88									
Hf pp	om <	: 80	Sn	ppm	27.4									
Aqua Regia Dige	Aqua Regia Digestion													
Cs pp	1													

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 laboratories all accredited to ISO 17025 for 4-acid digestion) and Table 2 (generated from data supplied by laboratories mostly accredited to ISO 17025) provide the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties 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 (PDM $^3$ ) are presented in the detailed certification data for this CRM (**OREAS 750b-DataPack.1.0.240809\_005633.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 750b was prepared from a blend of spodumene concentrate derived from the processing of lithium pegmatite ores sourced from the Greenbushes area of southwest Western Australia, Londonderry lithium-pegmatite ore (containing elevated levels of lithium, rubidium, caesium, tin and tantalum), barren granodiorite and quartz. The barren granodiorite was sourced from the mafic, S-Type, Late Devonian Bulla Granodiorite complex located in northern Melbourne, Australia.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 750b was prepared in the following manner:

- Drying of ore and barren materials to constant mass at 105 °C;
- Crushing and milling of the barren materials to >98 % minus 75 μm;
- Multi-stage milling of ore and spodumene concentrate materials to 100% minus 30 μm;
- Check analysis of ore and spodumene concentrate for contained Li concentration;
- Blending the ore, concentrate and barren materials in appropriate proportions to achieve the desired Li grade;

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- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10 g units in laminated foil pouches and 1 kg units in plastic wide-mouth jars.

#### PHYSICAL PROPERTIES

OREAS 750b was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

Table 4. Physical properties of OREAS 750b.

Bulk Density (kg/m³)	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
841	0.43	N8	Very Light Gray

<sup>&</sup>lt;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.

#### ANALYTICAL PROGRAM

Twenty-eight commercial analytical laboratories participated in the program to certify the elements reported in Table 1 and 2. The following methods were employed:

- Sodium peroxide fusion with full suite ICP-OES and ICP-MS elemental packages (up to 24 laboratories depending on the element) except for one laboratory that used lithium borate fusion with ICP-MS for Nb. Sn and Ta only;
- 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 26 laboratories depending on the element);
- Lithium borate fusion whole rock analysis package by X-ray fluorescence (up to 20 laboratories depending on the element);
- Thermogravimetry: Loss on Ignition (LOI) at 1000° C (8 laboratories used a thermogravimetric analyser, 8 laboratories included LOI with their fusion package and 5 laboratories used a conventional muffle furnace).

For the round robin program twelve 1 kg 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 15 g scoop splits from each of three separate 1 kg test units. This format enabled a nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance (see 'Homogeneity Evaluation' section below).

#### STATISTICAL ANALYSIS

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

For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per

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

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

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

**Standard Deviation** intervals (see Table 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 Tables 1 and 2 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 (Li<sub>2</sub>O) by 4-acid digestion with ICP, where 99 % of the time (1- $\alpha$ =0.99) at least 95 % of subsamples ( $\rho$ =0.95) will have concentrations lying between 0.519 and 0.535 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.* 

Based on the statistical analysis of the results of the interlaboratory certification program, it can be concluded that OREAS 750b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

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#### 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)  $\pm$  10 %.

i.e., Certified Value ± 10% ± 2DL [1].

Table 5. Performance Gates for OREAS 750b.

0	Certified		Absolute	Standard	Deviations	6	Relative	Standard D	eviations	5 % window	
Constituent	Value 1SD	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digest	ion										
Ag, ppm	0.094	0.019	0.055	0.133	0.036	0.152	20.60%	41.20%	61.80%	0.090	0.099
Al, wt.%	3.91	0.150	3.61	4.21	3.46	4.36	3.84%	7.67%	11.51%	3.71	4.10
As, ppm	2.88	0.51	1.87	3.90	1.36	4.40	17.61%	35.23%	52.84%	2.74	3.03
Ba, ppm	370	10	349	391	339	401	2.81%	5.62%	8.43%	351	388
Be, ppm	3.53	0.211	3.11	3.95	2.90	4.16	5.97%	11.94%	17.91%	3.35	3.70
Bi, ppm	0.44	0.06	0.32	0.55	0.27	0.61	13.11%	26.23%	39.34%	0.42	0.46
Ca, wt.%	0.609	0.023	0.563	0.654	0.541	0.676	3.71%	7.41%	11.12%	0.578	0.639
Cd, ppm	0.090	0.032	0.025	0.155	0.000	0.187	35.96%	71.93%	107.9%	0.086	0.095
Ce, ppm	29.8	2.12	25.5	34.0	23.4	36.1	7.12%	14.23%	21.35%	28.3	31.2
Co, ppm	2.99	0.147	2.70	3.29	2.55	3.44	4.92%	9.85%	14.77%	2.84	3.14
Cr, ppm	17.9	3.3	11.4	24.5	8.2	27.7	18.14%	36.27%	54.41%	17.1	18.8
Cs, ppm	39.2	1.34	36.5	41.9	35.2	43.2	3.42%	6.83%	10.25%	37.2	41.2
Cu, ppm	10.7	0.93	8.9	12.6	7.9	13.5	8.70%	17.39%	26.09%	10.2	11.3
Dy, ppm	1.52	0.145	1.23	1.81	1.09	1.96	9.51%	19.01%	28.52%	1.45	1.60
Er, ppm	0.59	0.047	0.50	0.68	0.45	0.73	7.87%	15.73%	23.60%	0.56	0.62
Eu, ppm	0.52	0.06	0.40	0.63	0.35	0.69	11.02%	22.04%	33.06%	0.49	0.54
Fe <sub>2</sub> O <sub>3</sub> , wt.%	1.53	0.054	1.43	1.64	1.37	1.69	3.50%	6.99%	10.49%	1.46	1.61
Ga, ppm	16.8	0.93	15.0	18.7	14.1	19.6	5.52%	11.04%	16.55%	16.0	17.7
Gd, ppm	2.28	0.121	2.04	2.52	1.92	2.64	5.29%	10.58%	15.88%	2.17	2.40
Hf, ppm	1.21	0.091	1.03	1.39	0.94	1.48	7.49%	14.97%	22.46%	1.15	1.27
Ho, ppm	0.24	0.013	0.21	0.27	0.20	0.28	5.48%	10.97%	16.45%	0.23	0.25
In, ppm	0.021	0.004	0.013	0.029	0.009	0.033	18.66%	37.32%	55.99%	0.020	0.022

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

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

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Table 5 continued.

			Absolute	Standard	Deviations		Relative	Standard D	eviations	5 % window	
Constituent	Certified Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digest	ion continu	ed	LOW	nigii	LOW	nigii					
K, wt.%	1.15	0.041	1.07	1.23	1.03	1.27	3.56%	7.12%	10.68%	1.09	1.21
La, ppm	14.4	1.14	12.1	16.7	11.0	17.8	7.91%	15.81%	23.72%	13.7	15.1
Li, wt.%	0.245	0.007	0.231	0.259	0.224	0.266	2.83%	5.67%	8.50%	0.233	0.257
Li <sub>2</sub> O, wt.%	0.527	0.015	0.497	0.557	0.482	0.572	2.83%	5.67%	8.50%	0.501	0.553
Lu, ppm	0.065	0.009	0.048	0.083	0.039	0.092	13.58%	27.16%	40.73%	0.062	0.069
Mg, wt.%	0.267	0.012	0.243	0.290	0.231	0.302	4.44%	8.87%	13.31%	0.253	0.280
Mn, wt.%	0.039	0.001	0.036	0.041	0.035	0.043	3.35%	6.70%	10.04%	0.037	0.041
Mo, ppm	1.60	0.088	1.42	1.78	1.34	1.86	5.49%	10.97%	16.46%	1.52	1.68
Na, wt.%	0.755	0.034	0.686	0.823	0.652	0.857	4.54%	9.07%	13.61%	0.717	0.792
Nb, ppm	28.4	1.51	25.4	31.5	23.9	33.0	5.31%	10.61%	15.92%	27.0	29.9
Nd, ppm	13.5	0.99	11.5	15.5	10.5	16.5	7.31%	14.62%	21.93%	12.8	14.2
Ni, ppm	7.66	0.329	7.00	8.32	6.67	8.65	4.30%	8.60%	12.89%	7.28	8.05
P, wt.%	0.039	0.002	0.035	0.044	0.033	0.046	5.67%	11.33%	17.00%	0.037	0.041
Pb, ppm	9.55	0.436	8.68	10.42	8.24	10.86	4.57%	9.14%	13.71%	9.07	10.03
Pr, ppm	3.65	0.229	3.19	4.11	2.96	4.34	6.28%	12.55%	18.83%	3.47	3.83
Rb, ppm	288	15	258	317	243	332	5.17%	10.34%	15.51%	273	302
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.045	0.004	0.037	0.053	0.032	0.058	9.42%	18.83%	28.25%	0.043	0.047
Sb, ppm	0.39	0.04	0.30	0.47	0.26	0.51	10.77%	21.54%	32.31%	0.37	0.41
Sc, ppm	3.20	0.244	2.71	3.69	2.47	3.93	7.61%	15.23%	22.84%	3.04	3.36
Sm, ppm	2.69	0.180	2.33	3.05	2.15	3.23	6.69%	13.39%	20.08%	2.55	2.82
Sn, ppm	25.8	1.81	22.1	29.4	20.3	31.2	7.03%	14.06%	21.09%	24.5	27.1
Sr, ppm	60	3.0	54	66	51	69	4.97%	9.94%	14.91%	57	63
Ta, ppm	64	5.5	53	75	48	81	8.48%	16.95%	25.43%	61	68
Tb, ppm	0.31	0.023	0.26	0.35	0.24	0.38	7.60%	15.20%	22.80%	0.29	0.32
Te, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	5.94	0.445	5.05	6.83	4.60	7.28	7.50%	14.99%	22.49%	5.64	6.24
Ti, wt.%	0.151	0.006	0.139	0.162	0.133	0.168	3.92%	7.85%	11.77%	0.143	0.158
TI, ppm	1.93	0.105	1.72	2.14	1.62	2.25	5.43%	10.86%	16.30%	1.83	2.03
Tm, ppm	0.071	0.009	0.053	0.089	0.044	0.098	12.46%	24.92%	37.38%	0.067	0.075
U, ppm	1.91	0.161	1.59	2.24	1.43	2.40	8.43%	16.85%	25.28%	1.82	2.01
V, ppm	22.8	0.94	20.9	24.7	20.0	25.6	4.13%	8.26%	12.39%	21.6	23.9
W, ppm	5.08	0.471	4.14	6.02	3.67	6.49	9.27%	18.54%	27.81%	4.83	5.34
Y, ppm	6.13	0.316	5.50	6.76	5.18	7.08	5.15%	10.31%	15.46%	5.83	6.44
Yb, ppm	0.46	0.05	0.36	0.57	0.31	0.62	11.17%	22.34%	33.51%	0.44	0.49
Zn, ppm	60	3.5	53	67	49	70	5.84%	11.69%	17.53%	57	63
Zr, ppm	27.8	2.28	23.3	32.4	21.0	34.7	8.19%	16.39%	24.58%	26.4	29.2
Borate / Perox			0.7:	4.00	0.00		0.4537	0.0007	40.0501	0.00	4.00
Al, wt.%	4.02	0.139	3.74	4.29	3.60	4.43	3.45%	6.90%	10.35%	3.82	4.22
Ba, ppm	371	12	348	394	336	406	3.12%	6.24%	9.37%	352	389
Be, ppm	3.88	0.62	2.63	5.13	2.01	5.75	16.06%	32.12%	48.18%	3.69	4.08
Bi, ppm	0.46	0.12	0.22	0.70	0.10	0.81	25.82%	51.65%	77.47%	0.44	0.48
Ca, wt.%	0.601	0.036	0.529	0.672	0.494	0.708	5.94%	11.87%	17.81%	0.571	0.631

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

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.

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

Table 5 continued.

			Ahsolute	Standard	Deviation		Relative	Standard D	eviations	5 % window	
Constituent	Certified			1	Г	1	TCIative		I I	3 70 W	I
	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate / Perox	kide Fusion	ICP conti	nued	T	T	T				T	T
Ce, ppm	29.6	1.75	26.1	33.1	24.3	34.8	5.91%	11.83%	17.74%	28.1	31.0
Co, ppm	3.13	0.296	2.54	3.73	2.25	4.02	9.45%	18.89%	28.34%	2.98	3.29
Cs, ppm	39.1	1.61	35.8	42.3	34.2	43.9	4.13%	8.26%	12.38%	37.1	41.0
Dy, ppm	2.21	0.122	1.97	2.46	1.85	2.58	5.52%	11.04%	16.56%	2.10	2.32
Er, ppm	1.13	0.084	0.97	1.30	0.88	1.38	7.40%	14.80%	22.20%	1.08	1.19
Eu, ppm	0.52	0.046	0.43	0.61	0.38	0.65	8.81%	17.62%	26.43%	0.49	0.54
Fe <sub>2</sub> O <sub>3</sub> , wt.%	1.56	0.043	1.47	1.64	1.43	1.68	2.74%	5.47%	8.21%	1.48	1.63
Ga, ppm	16.9	0.83	15.3	18.6	14.4	19.4	4.91%	9.82%	14.73%	16.1	17.8
Gd, ppm	2.64	0.256	2.13	3.16	1.88	3.41	9.69%	19.39%	29.08%	2.51	2.78
Ge, ppm	1.49	0.43	0.62	2.36	0.18	2.79	29.22%	58.43%	87.65%	1.41	1.56
Ho, ppm	0.40	0.029	0.34	0.46	0.31	0.49	7.36%	14.72%	22.08%	0.38	0.42
In, ppm	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	1.18	0.052	1.08	1.29	1.03	1.34	4.39%	8.79%	13.18%	1.12	1.24
La, ppm	14.6	0.94	12.7	16.5	11.8	17.5	6.46%	12.92%	19.39%	13.9	15.4
Li, wt.%	0.246	0.009	0.227	0.264	0.218	0.273	3.77%	7.53%	11.30%	0.233	0.258
Li <sub>2</sub> O, wt.%	0.529	0.020	0.489	0.569	0.469	0.589	3.77%	7.53%	11.30%	0.502	0.555
Lu, ppm	0.16	0.02	0.11	0.20	0.09	0.22	13.67%	27.34%	41.02%	0.15	0.17
Mg, wt.%	0.266	0.013	0.239	0.293	0.226	0.306	5.01%	10.03%	15.04%	0.253	0.279
Mn, wt.%	0.039	0.001	0.037	0.041	0.036	0.042	2.28%	4.55%	6.83%	0.037	0.041
Nb, ppm	29.3	1.23	26.9	31.8	25.6	33.0	4.18%	8.36%	12.54%	27.8	30.8
Nd, ppm	13.2	0.76	11.7	14.8	10.9	15.5	5.76%	11.52%	17.28%	12.6	13.9
P, wt.%	0.039	0.003	0.034	0.044	0.031	0.047	6.94%	13.89%	20.83%	0.037	0.041
Pb, ppm	9.64	0.925	7.79	11.49	6.87	12.42	9.60%	19.19%	28.79%	9.16	10.12
Pr, ppm	3.49	0.242	3.00	3.97	2.76	4.21	6.93%	13.86%	20.79%	3.31	3.66
Rb, ppm	287	12	263	310	252	321	4.05%	8.09%	12.14%	272	301
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Si, wt.%	40.13	1.355	37.42	42.84	36.06	44.19	3.38%	6.75%	10.13%	38.12	42.13
Sm, ppm	2.79	0.176	2.44	3.14	2.26	3.32	6.30%	12.61%	18.91%	2.65	2.93
Sn, ppm	38.7	2.18	34.4	43.1	32.2	45.3	5.62%	11.24%	16.87%	36.8	40.7
Sr, ppm	62	8	47	77	39	85	12.39%	24.77%	37.16%	59	65
Ta, ppm	69	3.9	61	76	57	80	5.65%	11.30%	16.96%	65	72
Tb, ppm	0.40	0.021	0.35	0.44	0.33	0.46	5.36%	10.71%	16.07%	0.38	0.42
Th, ppm	5.96	0.245	5.47	6.45	5.22	6.69	4.11%	8.22%	12.32%	5.66	6.25
Ti, wt.%	0.154	0.008	0.138	0.170	0.131	0.177	5.07%	10.14%	15.21%	0.146	0.162
TI, ppm	1.96	0.096	1.77	2.16	1.67	2.25	4.90%	9.80%	14.70%	1.87	2.06
Tm, ppm	0.16	0.013	0.14	0.19	0.12	0.20	7.88%	15.76%	23.65%	0.15	0.17
U, ppm	2.28	0.31	1.65	2.90	1.33	3.22	13.80%	27.61%	41.41%	2.16	2.39
V, ppm	22.8	2.6	17.6	28.0	15.0	30.6	11.42%	22.83%	34.25%	21.6	23.9
W, ppm	5.54	0.76	4.02	7.06	3.26	7.82	13.72%	27.44%	41.16%	5.26	5.82
Y, ppm	11.4	0.70	9.7	13.0	8.9	13.8	7.24%	14.49%	21.73%	10.8	11.9
	1.04	0.02	0.78	1.29	0.66	1.42	12.19%	24.38%	36.57%	0.99	1.09
Yb, ppm											
Zn, ppm	57	10	37	76	27	86	17.20%	34.39%	51.59%	54	59

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

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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		Absolute	Standard	Deviations	5	Relative Standard Deviations			5 % window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate Fusion XRF											
Al <sub>2</sub> O <sub>3</sub> , wt.%	7.63	0.117	7.40	7.87	7.28	7.98	1.54%	3.08%	4.62%	7.25	8.01
BaO, ppm	407	43	322	493	279	536	10.49%	20.98%	31.47%	387	428
CaO, wt.%	0.851	0.016	0.819	0.884	0.803	0.900	1.91%	3.83%	5.74%	0.809	0.894
Cr <sub>2</sub> O <sub>3</sub> , ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Fe <sub>2</sub> O <sub>3</sub> , wt.%	1.56	0.027	1.51	1.62	1.48	1.64	1.76%	3.52%	5.28%	1.48	1.64
K <sub>2</sub> O, wt.%	1.39	0.016	1.36	1.42	1.34	1.44	1.13%	2.26%	3.38%	1.32	1.46
MgO, wt.%	0.452	0.023	0.406	0.499	0.382	0.522	5.15%	10.30%	15.44%	0.430	0.475
MnO, wt.%	0.050	0.002	0.047	0.054	0.045	0.055	3.20%	6.40%	9.61%	0.048	0.053
Na <sub>2</sub> O, wt.%	1.01	0.037	0.94	1.09	0.90	1.12	3.67%	7.34%	11.00%	0.96	1.06
Nb, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
P <sub>2</sub> O <sub>5</sub> , wt.%	0.088	0.007	0.075	0.101	0.068	0.108	7.50%	14.99%	22.49%	0.084	0.092
Rb, ppm	259	21	218	300	198	321	7.92%	15.83%	23.75%	246	272
SiO <sub>2</sub> , wt.%	85.46	0.785	83.89	87.03	83.11	87.82	0.92%	1.84%	2.75%	81.19	89.74
SO <sub>3</sub> , wt.%	0.105	0.014	0.077	0.133	0.063	0.148	13.48%	26.96%	40.44%	0.100	0.110
SrO, ppm	96	19	57	134	38	153	19.97%	39.93%	59.90%	91	100
TiO <sub>2</sub> , wt.%	0.257	0.009	0.239	0.274	0.231	0.283	3.39%	6.78%	10.16%	0.244	0.269
Thermogravin	netry										
LOI <sup>1000</sup> , wt.%	0.570	0.106	0.358	0.782	0.252	0.887	18.57%	37.14%	55.71%	0.541	0.598

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

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.

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

#### PARTICIPATING LABORATORIES

- 1. Actlabs, Ancaster, Ontario, Canada
- 2. African Natural Resources & Mines Ltd, Suleja, Niger State, Nigeria
- 3. AGAT Laboratories, Calgary, Alberta, Canada
- 4. ALS, Johannesburg, South Africa
- 5. ALS, Lima, Peru
- 6. ALS, Loughrea, Galway, Ireland
- 7. ALS, Malaga, WA, Australia
- 8. ALS, Vancouver, BC, Canada
- 9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
- 10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 11. CERTIMIN, Lima, Peru
- 12. CRS Laboratories Oy, Kempele, Northern Ostrobothnia, Finland
- 13. Inspectorate (BV), Lima, Peru
- 14. Intertek, Cupang, Muntinlupa, Philippines
- 15. Intertek, Perth, WA, Australia
- 16. Intertek, Townsville, QLD, Australia
- 17. Labwest Minerals Analysis, Perth, WA, Australia
- 18. MSALABS, Vancouver, BC, Canada
- 19. Ontario Geological Survey, Sudbury, Ontario, Canada
- 20. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 21. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 22. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
- 23. SGS, Randfontein, Gauteng, South Africa
- 24. SGS Australia Mineral Services, Perth, WA, Australia
- 25. SGS Canada Inc., Vancouver, BC, Canada
- 26. SGS del Peru, Lima, Peru
- 27. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 28. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

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

#### PREPARER AND SUPPLIER

Certified reference material OREAS 750b is prepared, certified and supplied by:



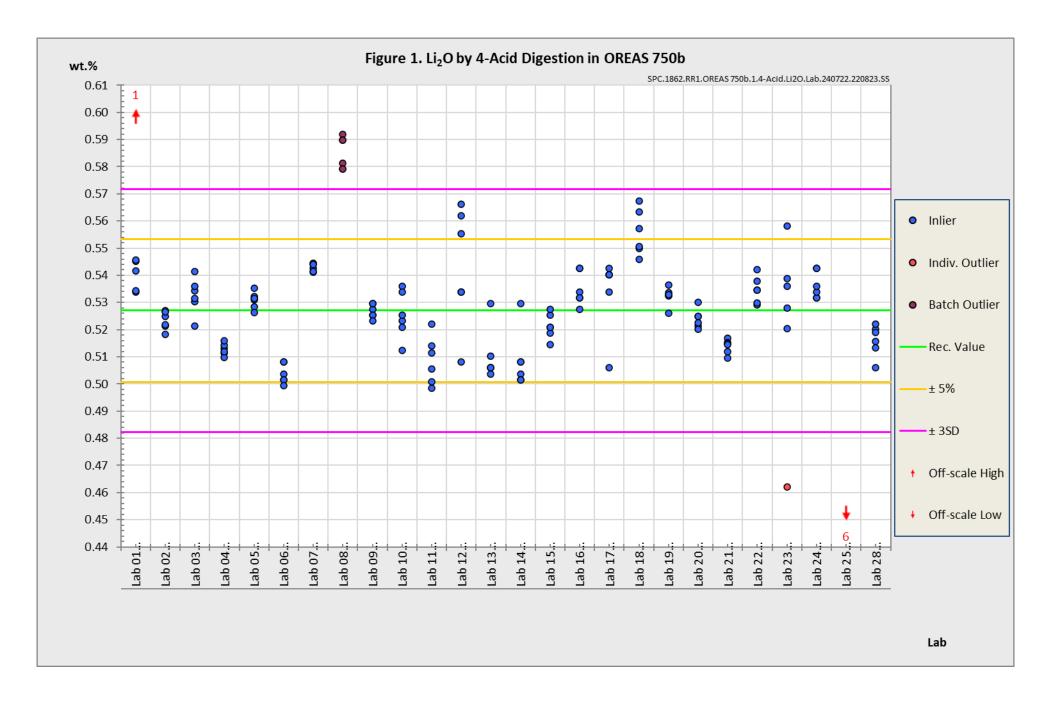
ORE Research & Exploration Pty Ltd 37A Hosie Street

Bayswater North VIC 3153

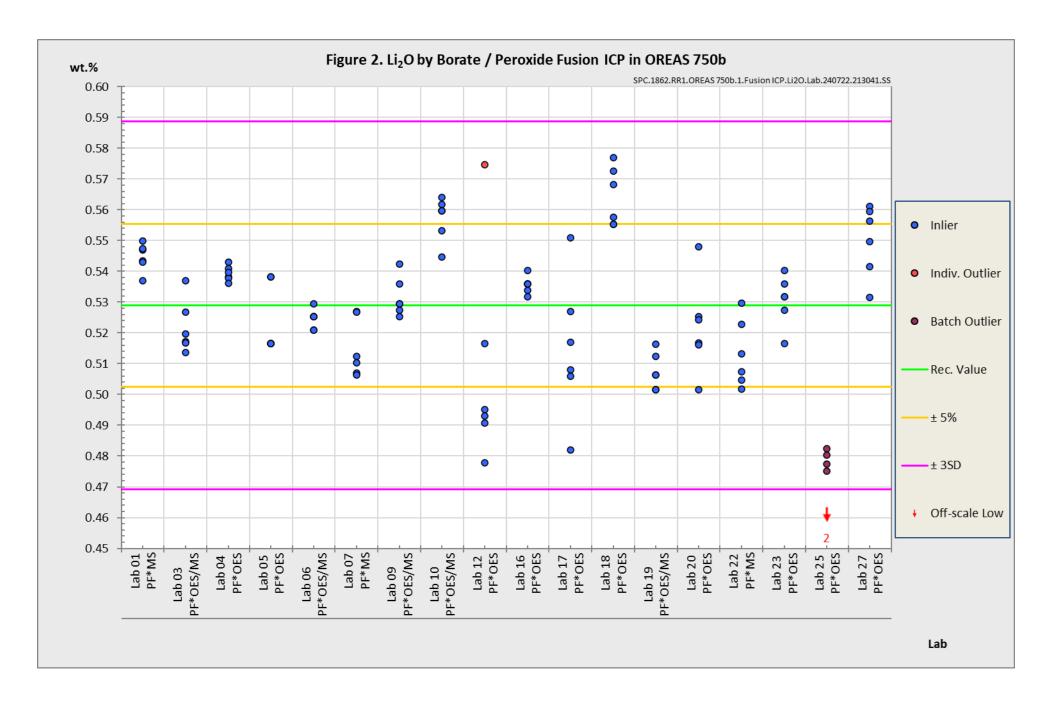
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#### METROLOGICAL TRACEABILITY

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)). 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 4-acid digestion (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

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

#### COMMUTABILITY

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

#### INTENDED USE

OREAS 750b 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 750b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 750b is intended for the following uses:

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

#### MINIMUM SAMPLE SIZE

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

- Lithium Borate / Sodium peroxide fusion with ICP-OES and/or MS finish: ≥0.2 g;
- Borate fusion with X-ray fluorescence finish: ≥0.2 g;
- Loss on Ignition (LOI) at 1000°C: ≥1 g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish: ≥0.25 g.

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 750b remains valid, within the specified measurement uncertainties, until at least February 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 750b is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

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

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 750b contains a non-hygroscopic\* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (~0.04 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 adsorped 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

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#### **DOCUMENT HISTORY**

Revision No.	Date	Changes applied
0	9 <sup>th</sup> September, 2024	First publication.

#### **CERTIFYING OFFICER**



9<sup>th</sup> September, 2024

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

# **QMS CERTIFICATION**

ORE Pty Ltd is accredited for compliance with ISO 17034:2016.





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**

- [1] Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).
- [2] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.
- [3] ISO Guide 33401:2024-01. Reference materials Contents of certificates, labels and accompanying documentation.
- [4] ISO Guide 33405:2024-05. Reference materials Approaches for characterization and assessment of homogeneity and stability.
- [5] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

- [6] ISO 16269:2014. Statistical interpretation of data Part 6: Determination of statistical tolerance intervals.
- [7] ISO/TR 16476:2016, Reference Materials Establishing and expressing metrological traceability of quantity values assigned to reference materials.
- [8] ISO 17025:2017, General requirements for the competence of testing and calibration laboratories.
- [9] ISO 17034:2016. General requirements for the competence of reference material producers.
- [10] Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), Minnesota (USA).
- [11] OREAS-BUP-70-09-11: Statistical Analysis OREAS Evaluation Method.
- [12] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.
- [13] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.
- [14] Thompson, A.; Taylor, B.N. (2008); Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Nov 2021).
- [15] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.