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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 465b

Carbonatite Supergene REE-Nb Ore (TREO + Y_2O_3 ~8.84%)

(Mount Weld Mine, Western Australia)

Accredited for compliance with ISO 17034

ISO 9001

Table 1. Certified Values, Uncertainty & Tolerance Intervals for multi-elements by 4-acid digestion in OREAS 465b.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion					
Ag, Silver (ppm)	6.09	5.56	6.61	5.85	6.32
Al, Aluminium (wt.%)	5.53	5.26	5.79	5.39	5.67
Ba, Barium (wt.%)	0.344	0.318	0.370	0.335	0.354
Be, Beryllium (ppm)	12.3	11.4	13.2	12.0	12.7
Bi, Bismuth (ppm)	14.7	13.8	15.6	14.2	15.2
Ca, Calcium (wt.%)	0.643	0.597	0.688	0.626	0.660
Cd, Cadmium (ppm)	0.87	0.70	1.03	0.79	0.95
Ce, Cerium (wt.%)	3.47	3.06	3.88	3.35	3.59
Co, Cobalt (ppm)	17.2	16.3	18.2	16.7	17.7
Cr, Chromium (ppm)	288	262	314	281	295
Cs, Caesium (ppm)	< 0.05	IND	IND	IND	IND
Cu, Copper (ppm)	114	108	121	111	117
Dy, Dysprosium (ppm)	196	146	245	190	201
Er, Erbium (ppm)	38.0	30.0	45.9	35.4	40.6
Fe, Iron (wt.%)	29.18	28.13	30.22	28.57	29.78
Ho, Holmium (ppm)	22.6	19.6	25.7	21.6	23.6
In, Indium (ppm)	3.00	2.88	3.12	2.89	3.11
K, Potassium (wt.%)	0.012	0.010	0.014	0.012	0.013
Li, Lithium (ppm)	3.13	2.67	3.60	2.93	3.33
Mg, Magnesium (wt.%)	0.276	0.257	0.295	0.270	0.282
Mn, Manganese (wt.%)	0.214	0.200	0.227	0.208	0.220
Mo, Molybdenum (ppm)	96	89	102	93	98
Na, Sodium (wt.%)	0.060	0.048	0.071	0.058	0.062
Nb, Niobium (wt.%)	0.352	0.287	0.416	0.340	0.363
Nd, Neodymium (wt.%)	1.08	0.95	1.20	1.04	1.11
Ni, Nickel (ppm)	45.8	42.8	48.7	44.3	47.2
P, Phosphorus (wt.%)	2.63	2.31	2.96	2.53	2.74
Pb, Lead (ppm)	462	432	491	450	473
Pr, Praseodymium (wt.%)	0.342	0.273	0.411	0.335	0.349
Rb, Rubidium (ppm)	0.42	0.33	0.51	0.39	0.46
S, Sulphur (wt.%)	0.151	0.128	0.174	0.143	0.159
Sb, Antimony (ppm)	0.84	0.59	1.09	0.68	1.00
Sn, Tin (ppm)	69	52	85	64	73
Sr, Strontium (wt.%)	0.378	0.350	0.406	0.367	0.389
Te, Tellurium (ppm)	0.51	0.40	0.61	0.45	0.56

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

Note: intervals may appear asymmetric due to rounding.

†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[†]	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Tl, Thallium (ppm)	0.094	0.085	0.103	0.083	0.105
Tm, Thulium (ppm)	3.18	2.52	3.83	3.05	3.30
U, Uranium (ppm)	12.4	11.9	13.0	11.9	12.9
V, Vanadium (ppm)	420	395	444	409	430
W, Tungsten (ppm)	3.89	3.35	4.43	3.56	4.23
Zn, Zinc (ppm)	950	915	985	932	968

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

Note: intervals may appear asymmetric due to rounding.

[†] These operationally defined measurands meet 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 465b.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)					
Al, Aluminium (wt.%)	5.90	5.75	6.04	5.79	6.01
Al_2O_3, Aluminium(III) oxide (wt.%)	11.14	10.87	11.41	10.93	11.35
Ba, Barium (wt.%)	0.386	0.371	0.402	0.378	0.395
BaO, Barium oxide (wt.%)	0.432	0.414	0.449	0.422	0.441
Be, Beryllium (ppm)	13.5	11.6	15.3	12.1	14.9
Bi, Bismuth (ppm)	15.0	13.3	16.7	14.1	16.0
Ca, Calcium (wt.%)	0.672	0.636	0.709	0.638	0.707
CaO, Calcium oxide (wt.%)	0.946	0.889	1.004	0.898	0.995
Cd, Cadmium (ppm)	< 10	IND	IND	IND	IND
Ce, Cerium (wt.%)	3.56	3.44	3.68	3.48	3.63
CeO_2, Cerium(IV) oxide (wt.%)	4.37	4.22	4.52	4.28	4.46
Co, Cobalt (ppm)	19.4	17.5	21.2	17.9	20.8
Cr, Chromium (ppm)	350	321	379	333	366
Cr_2O_3, Chromium(III) oxide (ppm)	511	469	553	487	535
Cs, Caesium (ppm)	< 0.1	IND	IND	IND	IND
Cu, Copper (ppm)	142	117	167	134	150
Dy, Dysprosium (ppm)	198	190	206	192	203
Dy_2O_3, Dysprosium(III) oxide (ppm)	227	218	236	221	233
Er, Erbium (ppm)	46.1	43.9	48.3	45.0	47.1
Er_2O_3, Erbium(III) oxide (ppm)	53	50	55	52	54
Eu, Europium (ppm)	265	255	276	258	272
Eu_2O_3, Europium(III) oxide (ppm)	307	295	319	299	315
Fe, Iron (wt.%)	30.54	29.65	31.44	29.77	31.31

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
Note: intervals may appear asymmetric due to rounding.
IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)					
Fe_2O_3, Iron(III) oxide (wt.%)	43.67	42.39	44.95	42.57	44.77
Gd, Gadolinium (ppm)	557	528	585	546	567
Gd_2O_3, Gadolinium(III) oxide (ppm)	641	610	673	626	656
Ho, Holmium (ppm)	25.0	24.0	26.0	24.2	25.9
Ho_2O_3, Holmium(III) oxide (ppm)	28.7	27.5	29.8	27.7	29.7
In, Indium (ppm)	3.05	2.75	3.36	2.85	3.26
La, Lanthanum (wt.%)	2.10	2.03	2.16	2.05	2.14
La_2O_3, Lanthanum(III) oxide (wt.%)	2.46	2.39	2.53	2.41	2.51
Lu, Lutetium (ppm)	1.77	1.59	1.95	1.69	1.85
Lu_2O_3, Lutetium(III) oxide (ppm)	2.01	1.82	2.21	1.92	2.11
Mg, Magnesium (wt.%)	0.274	0.248	0.300	0.264	0.285
MgO, Magnesium oxide (wt.%)	0.455	0.411	0.498	0.437	0.472
Mn, Manganese (wt.%)	0.234	0.223	0.246	0.230	0.239
MnO, Manganese oxide (wt.%)	0.303	0.287	0.318	0.297	0.309
Mo, Molybdenum (ppm)	97	87	108	94	101
Nb, Niobium (wt.%)	0.423	0.405	0.441	0.412	0.434
Nd, Neodymium (wt.%)	1.09	1.05	1.13	1.07	1.12
Nd_2O_3, Neodymium(III) oxide (wt.%)	1.27	1.22	1.32	1.25	1.30
Ni, Nickel (ppm)	52	35	69	IND	IND
P, Phosphorus (wt.%)	3.21	2.92	3.51	3.12	3.30
P_2O_5, Phosphorus(V) oxide (wt.%)	7.36	6.69	8.04	7.15	7.57
Pb, Lead (ppm)	490	452	527	457	523
Pr, Praseodymium (wt.%)	0.339	0.325	0.353	0.330	0.348
Pr_6O_{11}, Praseodymium(III,IV) oxide (wt.%)	0.409	0.393	0.426	0.399	0.420
S, Sulphur (wt.%)	0.171	0.145	0.198	IND	IND
Sc, Scandium (ppm)	161	137	184	154	167
Si, Silicon (wt.%)	1.48	1.37	1.59	1.43	1.54
SiO_2, Silicon dioxide (wt.%)	3.17	2.93	3.40	3.05	3.28
Sm, Samarium (wt.%)	0.122	0.116	0.129	0.118	0.126
Sm_2O_3, Samarium(III) oxide (wt.%)	0.142	0.134	0.149	0.137	0.146
Sn, Tin (ppm)	131	125	136	127	135
Sr, Strontium (wt.%)	0.444	0.422	0.466	0.430	0.458
Ta, Tantalum (ppm)	67	64	71	65	70
Tb, Terbium (ppm)	52	49	54	50	54
Tb_4O_7, Terbium(III,IV) oxide (ppm)	61	58	63	58	63
Th, Thorium (ppm)	772	745	799	753	791
Ti, Titanium (wt.%)	5.18	4.95	5.41	5.08	5.28

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
Note: intervals may appear asymmetric due to rounding.

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

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)					
TiO_2, Titanium dioxide (wt.%)	8.64	8.25	9.03	8.48	8.80
Tm, Thulium (ppm)	4.03	3.80	4.25	3.87	4.19
Tm_2O_3, Thulium(III) oxide (ppm)	4.59	4.35	4.84	4.39	4.79
U, Uranium (ppm)	12.7	12.2	13.3	12.3	13.1
V, Vanadium (ppm)	450	426	474	435	464
V_2O_5, Vanadium(V) oxide (ppm)	803	761	846	777	829
W, Tungsten (ppm)	5.62	5.17	6.06	5.08	6.16
Y, Yttrium (ppm)	481	460	502	470	492
Y_2O_3, Yttrium(III) oxide (ppm)	605	580	631	590	621
Yb, Ytterbium (ppm)	17.7	16.6	18.7	17.0	18.3
Yb_2O_3, Ytterbium(III) oxide (ppm)	19.9	18.7	21.0	19.1	20.6
Zn, Zinc (ppm)	968	914	1022	934	1002
Zr, Zirconium (wt.%)	0.162	0.145	0.179	0.154	0.169
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	10.88	10.65	11.12	10.77	10.99
BaO, Barium oxide (wt.%)	0.451	0.415	0.488	0.439	0.464
CaO, Calcium oxide (wt.%)	0.958	0.912	1.005	0.946	0.971
CeO_2, Cerium(IV) oxide (wt.%)	4.32	4.24	4.40	4.28	4.36
Cr_2O_3, Chromium(III) oxide (ppm)	534	387	681	IND	IND
Eu_2O_3, Europium(III) oxide (ppm)	263	151	374	IND	IND
Fe_2O_3, Iron(III) oxide (wt.%)	44.30	43.01	45.59	44.05	44.55
Gd_2O_3, Gadolinium(III) oxide (ppm)	735	619	852	IND	IND
Ho_2O_3, Holmium(III) oxide (ppm)	< 100	IND	IND	IND	IND
K_2O, Potassium oxide (wt.%)	0.018	0.015	0.022	IND	IND
La_2O_3, Lanthanum(III) oxide (wt.%)	2.51	2.48	2.54	2.48	2.54
Lu_2O_3, Lutetium(III) oxide (ppm)	< 100	IND	IND	IND	IND
MgO, Magnesium oxide (wt.%)	0.467	0.438	0.496	0.453	0.481
MnO, Manganese oxide (wt.%)	0.318	0.300	0.335	0.311	0.325
Nb_2O_5, Niobium(V) oxide (wt.%)	0.599	0.576	0.621	0.587	0.610
Nd_2O_3, Neodymium(III) oxide (wt.%)	1.26	1.23	1.29	1.24	1.27
P_2O_5, Phosphorus(V) oxide (wt.%)	7.47	7.19	7.74	7.40	7.53
Pr_6O_{11}, Praseodymium(III,IV) oxide (wt.%)	0.408	0.384	0.432	0.394	0.423
SiO_2, Silicon dioxide (wt.%)	3.00	2.91	3.10	2.94	3.07
Sm_2O_3, Samarium(III) oxide (wt.%)	0.143	0.131	0.154	IND	IND
SrO, Strontium oxide (wt.%)	0.539	0.498	0.581	0.529	0.550
Tb_4O_7, Terbium(III,IV) oxide (ppm)	< 100	IND	IND	IND	IND
ThO_2, Thorium dioxide (ppm)	859	806	911	847	871

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

Note: intervals may appear asymmetric due to rounding.

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

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
TiO_2, Titanium dioxide (wt.%)	8.90	8.70	9.09	8.82	8.97
Tm_2O_3, Thulium(III) oxide (ppm)	< 100	IND	IND	IND	IND
U_3O_8, Uranium(V,VI) oxide (ppm)	< 100	IND	IND	IND	IND
V_2O_5, Vanadium(V) oxide (ppm)	892	700	1084	IND	IND
Y_2O_3, Yttrium(III) oxide (ppm)	588	472	703	IND	IND
Yb_2O_3, Ytterbium(III) oxide (ppm)	< 100	IND	IND	IND	IND
ZrO_2, Zirconium dioxide (wt.%)	0.232	0.211	0.254	0.224	0.241
Thermogravimetry					
LOI^{1000}, Loss on ignition @1000 °C (wt.%)	11.66	11.37	11.94	11.58	11.73

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

Note: intervals may appear asymmetric due to rounding.

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

Table 3. Indicative Values for OREAS 465b.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
As	ppm	*28.3*	La	wt.%	*1.78*	Tb	ppm	*56*
Eu	ppm	*227*	Lu	ppm	*1.72*	Th	ppm	*416*
Ga	ppm	*51*	Re	ppm	*0.006*	Ti	wt.%	*3.29*
Gd	ppm	*500*	Sc	ppm	*120*	Y	ppm	*355*
Ge	ppm	*10.7*	Se	ppm	*3.32*	Yb	ppm	*14.5*
Hf	ppm	*3.72*	Sm	wt.%	*0.127*	Zr	ppm	*216*
Hg	ppm	*0.50*	Ta	ppm	*40.6*			
Borate / Peroxide Fusion ICP								
Hf	ppm	*37.9*	Ga	ppm	*172*	Rb	ppm	*1.01*
K	wt.%	*0.085*	Ge	ppm	*92*	Re	ppm	*0.011*
Na	wt.%	*0.060*	HfO_2	ppm	*44.7*	Sb	ppm	*1.50*
Ag	ppm	*< 1*	K_2O	wt.%	*0.102*	Se	ppm	*503*
As	ppm	*201*	Li	ppm	*6.70*	Te	ppm	*< 1*
B	ppm	*28.9*	Na_2O	wt.%	*0.081*	Tl	ppm	*< 0.5*
Borate Fusion XRF								
As_2O_3	ppm	*< 100*	F	ppm	*< 5000*	Sb_2O_3	ppm	*< 100*
Bi_2O_3	ppm	*131*	Ga_2O_3	ppm	*< 100*	Sc_2O_3	ppm	*218*
Cl	ppm	*92*	HfO_2	ppm	*< 100*	SnO_2	ppm	*131*
Co_3O_4	ppm	*< 100*	MoO_3	ppm	*217*	SO_3	wt.%	*0.443*
Cs_2O	ppm	*< 100*	Na_2O	wt.%	*0.091*	Ta_2O_5	ppm	*73*
CuO	ppm	*168*	NiO	ppm	*137*	TOT_XRF	wt.%	*100.14*
Dy_2O_3	ppm	*196*	PbO	ppm	*500*	WO_3	ppm	*100*
Er_2O_3	ppm	*98*	Rb_2O	ppm	*< 100*	ZnO	wt.%	*0.128*
Thermogravimetry								
H_2O-	wt.%	*1.19*						

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

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

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

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, Table 5 shows indicative mineralogy by semi-quantitative XRD analysis and Table 6 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 465b-DataPack.1.1.251215_132957.xlsx**).

OREAS 465b belongs to a CRM suite covering 0.11 – 9.88% TREO + Y_2O_3, with method-specific certification for REEs, major elements, and trace elements using fusion with XRF, fusion with ICP-OES and/or ICP-MS, and 4-acid digestion with ICP-OES and/or ICP-MS.

Results are also presented in scatter plots for CeO_2, La_2O_3, Nd_2O_3 and Pr_6O_{11} by borate / peroxide fusion with ICP in Figures 1 to 4 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.

SOURCE MATERIAL

OREAS 465b is a certified reference material (CRM) prepared from a blend of rare earth element (REE)–bearing ores sourced from Lynas Corporation’s Mount Weld Central Lanthanide Deposit. The Mount Weld deposit is a Proterozoic, carbonatite-hosted, deeply weathered monazite-rich REE deposit located ~35 km south of Laverton in Western Australia. The REE-bearing ores are predominantly comprised of secondary REE phosphates such as monazite and other alteration products formed through intense tropical weathering.

The Mt Weld carbonatite has a thick weathering/regolith layer (10 to >70 m) of laterite overlying the unweathered carbonatite that contains high-grade REO deposits and concentrations of niobium, zirconium, and other 'rare' metals. A zone of supergene-enrichment contains abundant insoluble phosphates, aluminophosphates, clays, crandallite group minerals, iron and manganese-bearing oxides that contain elevated concentrations of

REE, Ba, Cr, Nb, Sr, Ta, Ti, Th, U, V, Y and Zr, including economic accumulations of REE, niobium-tantalum and phosphatic minerals. Extreme lateritic weathering prevailed in the supergene zone over a protracted period of time and resulted in the degradation of the residual magmatic REE-bearing minerals. The majority of the REOs are contained within secondary, low Th phosphate minerals with low levels of deleterious elements (e.g. F and Ca). The Central lanthanide deposit contains an indicative mix of predominantly LREE and shows the following proportions when summed to 100%: CeO_2 (48.9%), La_2O_3 (28.4%), Nd_2O_3 (14.2%), Pr_6O_{11} (4.62%), Sm_2O_3 (1.62%) and Eu_2O_3 (0.297%), together with minor components of HREE: Dy_2O_3 (0.257%) and Tb_4O_7 (0.0687%).

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 465b was prepared in the following manner:

- Drying to constant mass at 105 °C;
- Crushing and multi-stage milling to >99.5% minus 75 microns;
- Blending the ores and barren materials in appropriate proportions to achieve desired grades;
- 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 465b 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 465b.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
995	1.31	10R 4/6	Moderate Reddish Brown

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

MINERALOGY

The semi-quantitative XRD results shown in Table 5 below were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors.

'Clay mineral' appears to be mainly montmorillonite and/or illite. 'Crandallite group' appears to be mainly florencite. The samples might comprise svanbergite and, if present, is reported under 'Crandallite group'. A trace amount of magnesite might be present and, if present, is reported under 'Ilmenite". Some amorphous material might be present in the sample.

Table 5. Indicative mineralogy of OREAS 465b by semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Crandallite group	34
Monazite	3
Hematite	12
Ilmenite	3
Goethite	45
Anatase	1
Dolomite - ankerite	1

ANALYTICAL PROGRAM

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

- 4-acid (HNO_3-HF-$HClO_4$-HCl) digestion with full suite ICP-OES and ICP-MS elemental packages (up to 20 laboratories depending on the element);
- Sodium peroxide/borate fusion with full suite ICP-OES and ICP-MS elemental packages (up to 18 laboratories depending on the element);
- Lithium borate fusion whole rock analysis package with X-ray fluorescence (up to 13 laboratories depending on the element);
- Thermogravimetry: Loss on Ignition (LOI) at 1000 °C (9 laboratories used a thermogravimetric analyser, 5 laboratories included LOI with their fusion package and 3 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 a 20 g subsample from six different 600 g test units (either from the odd or even numbered test units). Homogeneity was evaluated using 12 subsamples analysed by sodium peroxide fusion with ICP finish. ANOVA was applied to triplicates from four test units to compare within- and between-unit variances, providing a statistical measure of batch homogeneity (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 cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

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

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

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

The SD for each analyte’s certified value is calculated from the same filtered data set used to determine the certified value, i.e., after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e., the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. ***The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.***

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) 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 CeO_2 by fusion ICP, where 99 % of the time ($1-\alpha=0.99$) at least 95 % of subsamples ($\rho=0.95$) will have concentrations lying between 4.28 and 4.46 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 borate fusion with ICP finish (code ME-MS81h). The 12 samples consisted of triplicate samples from four of the six sampling units to enable an Analysis of Variance (ANOVA) by comparison of within- and between-unit variances across the four triplicates. The ANOVA enables a relative measure of homogeneity and permits a test of the null hypothesis that all ‘units’ are drawn from the same population distribution. An ANOVA constructed in this way tests that no statistically significant difference exists in the variance between-units to that of the variance within-units. A p-value < 0.05 would indicate rejection of the null hypothesis at the 95 % confidence level (i.e., a significant difference likely does exist; meaning there is evidence of heterogeneity between the sample intervals).

Of the twelve analytes evaluated by ANOVA, only Er and Gd displayed significant p-values less than 0.05. Any statistically significant result must be checked to determine whether it is also technically significant. The RSD for Er across the 12 samples is 2.87%, and for Gd, it is 1.27%. This shows that the variation is tightly constrained and the magnitude of the effect of between-unit variation is negligible compared to typical measurement error. All p-values are consequently found to be insignificant, and the Null Hypothesis is therefore retained.

PERFORMANCE GATES

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

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

i.e., Certified Value ±10 % ±2DL [1].

Table 6. Performance Gates for OREAS 465b.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion											
Ag, ppm	6.09	0.467	5.15	7.02	4.68	7.49	7.67%	15.35%	23.02%	5.78	6.39
Al, wt.%	5.53	0.386	4.75	6.30	4.37	6.69	6.99%	13.97%	20.96%	5.25	5.80
Ba, wt.%	0.344	0.029	0.286	0.403	0.256	0.432	8.51%	17.02%	25.53%	0.327	0.361
Be, ppm	12.3	1.4	9.6	15.1	8.2	16.4	11.09%	22.17%	33.26%	11.7	12.9
Bi, ppm	14.7	0.90	12.9	16.5	12.0	17.4	6.10%	12.19%	18.29%	14.0	15.5
Ca, wt.%	0.643	0.061	0.520	0.766	0.459	0.827	9.54%	19.08%	28.62%	0.611	0.675
Cd, ppm	0.87	0.23	0.41	1.33	0.18	1.55	26.39%	52.78%	79.17%	0.82	0.91
Ce, wt.%	3.47	0.58	2.31	4.62	1.73	5.20	16.68%	33.35%	50.03%	3.29	3.64
Co, ppm	17.2	1.23	14.8	19.7	13.5	20.9	7.11%	14.23%	21.34%	16.4	18.1
Cr, ppm	288	43	202	374	159	417	14.91%	29.81%	44.72%	274	302
Cs, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, ppm	114	6	103	125	97	131	4.91%	9.82%	14.73%	108	120
Dy, ppm	196	73	50	341	0	414	37.24%	74.47%	111.71 %	186	205
Er, ppm	38.0	10.1	17.8	58.2	7.7	68.2	26.57%	53.13%	79.70%	36.1	39.9
Fe, wt.%	29.18	0.998	27.18	31.17	26.18	32.17	3.42%	6.84%	10.26%	27.72	30.63

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

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

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion continued											
Ho, ppm	22.6	3.2	16.2	29.1	12.9	32.3	14.31%	28.62%	42.93%	21.5	23.8
In, ppm	3.00	0.092	2.82	3.19	2.72	3.28	3.07%	6.14%	9.22%	2.85	3.15
K, wt.%	0.012	0.004	0.005	0.020	0.001	0.024	31.22%	62.45%	93.67%	0.012	0.013
Li, ppm	3.13	0.51	2.11	4.16	1.59	4.67	16.38%	32.75%	49.13%	2.98	3.29
Mg, wt.%	0.276	0.027	0.222	0.330	0.195	0.357	9.81%	19.62%	29.43%	0.262	0.290
Mn, wt.%	0.214	0.015	0.184	0.244	0.168	0.259	7.08%	14.15%	21.23%	0.203	0.225
Mo, ppm	96	9.0	77	114	68	123	9.44%	18.87%	28.31%	91	100
Na, wt.%	0.060	0.012	0.036	0.083	0.024	0.095	19.82%	39.63%	59.45%	0.057	0.063
Nb, wt.%	0.352	0.086	0.179	0.524	0.093	0.611	24.55%	49.10%	73.65%	0.334	0.369
Nd, wt.%	1.08	0.14	0.80	1.36	0.66	1.49	12.86%	25.72%	38.57%	1.02	1.13
Ni, ppm	45.8	3.95	37.9	53.7	33.9	57.6	8.63%	17.25%	25.88%	43.5	48.0
P, wt.%	2.63	0.40	1.83	3.43	1.43	3.83	15.22%	30.44%	45.65%	2.50	2.76
Pb, ppm	462	30	402	522	372	552	6.49%	12.99%	19.48%	439	485
Pr, wt.%	0.342	0.056	0.230	0.454	0.174	0.510	16.35%	32.69%	49.04%	0.325	0.359
Rb, ppm	0.42	0.08	0.26	0.59	0.18	0.67	19.24%	38.49%	57.73%	0.40	0.45
S, wt.%	0.151	0.032	0.088	0.215	0.056	0.246	20.99%	41.98%	62.97%	0.144	0.159
Sb, ppm	0.84	0.25	0.34	1.34	0.09	1.59	29.67%	59.35%	89.02%	0.80	0.88
Sn, ppm	69	27	15	123	0	150	39.30%	78.60%	117.89%	65	72
Sr, wt.%	0.378	0.029	0.320	0.436	0.291	0.465	7.67%	15.35%	23.02%	0.359	0.397
Te, ppm	0.51	0.08	0.35	0.66	0.27	0.74	15.58%	31.15%	46.73%	0.48	0.53
Tl, ppm	0.094	0.006	0.082	0.106	0.076	0.112	6.30%	12.60%	18.90%	0.090	0.099
Tm, ppm	3.18	0.90	1.38	4.97	0.49	5.87	28.23%	56.46%	84.69%	3.02	3.34
U, ppm	12.4	0.70	11.0	13.8	10.3	14.5	5.67%	11.33%	17.00%	11.8	13.0
V, ppm	420	37	346	493	309	530	8.79%	17.57%	26.36%	399	441
W, ppm	3.89	0.63	2.63	5.15	2.00	5.78	16.16%	32.33%	48.49%	3.70	4.09
Zn, ppm	950	52	846	1054	795	1105	5.45%	10.91%	16.36%	902	997
Borate / Peroxide Fusion ICP											
Al, wt.%	5.90	0.114	5.67	6.13	5.55	6.24	1.94%	3.88%	5.82%	5.60	6.19
Al_2O_3, wt.%	11.14	0.216	10.71	11.57	10.49	11.79	1.94%	3.88%	5.82%	10.58	11.70
Ba, wt.%	0.386	0.016	0.355	0.418	0.340	0.433	4.02%	8.04%	12.06%	0.367	0.406
BaO, wt.%	0.432	0.017	0.397	0.466	0.379	0.484	4.02%	8.04%	12.06%	0.410	0.453
Be, ppm	13.5	1.10	11.3	15.7	10.2	16.8	8.19%	16.38%	24.57%	12.8	14.1
Bi, ppm	15.0	0.74	13.5	16.5	12.8	17.3	4.96%	9.92%	14.88%	14.3	15.8
Ca, wt.%	0.672	0.037	0.599	0.746	0.562	0.783	5.48%	10.96%	16.44%	0.639	0.706
CaO, wt.%	0.946	0.050	0.845	1.047	0.795	1.097	5.33%	10.65%	15.98%	0.899	0.994
Cd, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, wt.%	3.56	0.141	3.28	3.84	3.14	3.98	3.95%	7.90%	11.86%	3.38	3.74
CeO_2, wt.%	4.37	0.173	4.02	4.72	3.85	4.89	3.95%	7.90%	11.86%	4.15	4.59
Co, ppm	19.4	1.22	16.9	21.8	15.7	23.0	6.32%	12.65%	18.97%	18.4	20.3
Cr, ppm	350	27	296	404	269	430	7.68%	15.37%	23.05%	332	367
Cr_2O_3, ppm	511	39	433	590	393	629	7.68%	15.37%	23.05%	486	537
Cs, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, ppm	142	24	93	191	69	215	17.17%	34.34%	51.52%	135	149

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

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

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate / Peroxide Fusion ICP continued											
Dy, ppm	198	8	182	213	175	221	3.91%	7.82%	11.74%	188	208
Dy_2O_3, ppm	227	9	209	245	200	254	3.91%	7.82%	11.74%	216	238
Er, ppm	46.1	2.61	40.9	51.3	38.3	53.9	5.66%	11.33%	16.99%	43.8	48.4
Er_2O_3, ppm	53	3.0	47	59	44	62	5.66%	11.33%	16.99%	50	55
Eu, ppm	265	13	240	291	227	304	4.81%	9.61%	14.42%	252	279
Eu_2O_3, ppm	307	15	278	337	263	352	4.81%	9.61%	14.42%	292	323
Fe, wt.%	30.54	0.751	29.04	32.05	28.29	32.80	2.46%	4.92%	7.38%	29.02	32.07
Fe_2O_3, wt.%	43.67	1.074	41.52	45.82	40.45	46.89	2.46%	4.92%	7.38%	41.48	45.85
Gd, ppm	557	35	486	627	450	663	6.35%	12.70%	19.06%	529	584
Gd_2O_3, ppm	641	41	559	723	519	764	6.37%	12.75%	19.12%	609	673
Ho, ppm	25.0	1.10	22.8	27.2	21.7	28.3	4.40%	8.80%	13.20%	23.8	26.3
Ho_2O_3, ppm	28.7	1.26	26.2	31.2	24.9	32.5	4.40%	8.80%	13.20%	27.2	30.1
In, ppm	3.05	0.158	2.74	3.37	2.58	3.53	5.17%	10.34%	15.51%	2.90	3.21
La, wt.%	2.10	0.057	1.98	2.21	1.93	2.27	2.71%	5.42%	8.13%	1.99	2.20
La_2O_3, wt.%	2.46	0.067	2.33	2.59	2.26	2.66	2.71%	5.42%	8.13%	2.34	2.58
Lu, ppm	1.77	0.104	1.56	1.98	1.46	2.08	5.88%	11.77%	17.65%	1.68	1.86
Lu_2O_3, ppm	2.01	0.114	1.79	2.24	1.67	2.35	5.64%	11.28%	16.93%	1.91	2.11
Mg, wt.%	0.274	0.031	0.212	0.337	0.180	0.368	11.41%	22.82%	34.23%	0.260	0.288
MgO, wt.%	0.455	0.052	0.351	0.558	0.299	0.610	11.41%	22.82%	34.23%	0.432	0.477
Mn, wt.%	0.234	0.010	0.214	0.255	0.203	0.265	4.40%	8.79%	13.19%	0.223	0.246
MnO, wt.%	0.303	0.013	0.276	0.329	0.263	0.343	4.40%	8.79%	13.19%	0.288	0.318
Mo, ppm	97	6.8	84	111	77	118	6.96%	13.92%	20.89%	93	102
Nb, wt.%	0.423	0.023	0.377	0.470	0.353	0.493	5.51%	11.01%	16.52%	0.402	0.444
Nd, wt.%	1.09	0.058	0.98	1.21	0.92	1.27	5.33%	10.66%	15.99%	1.04	1.15
Nd_2O_3, wt.%	1.27	0.068	1.14	1.41	1.07	1.48	5.33%	10.66%	15.99%	1.21	1.34
Ni, ppm	52	9	34	70	25	80	17.58%	35.16%	52.75%	49	55
P, wt.%	3.21	0.201	2.81	3.61	2.61	3.81	6.24%	12.48%	18.72%	3.05	3.37
P_2O_5, wt.%	7.36	0.459	6.44	8.28	5.98	8.74	6.24%	12.48%	18.72%	6.99	7.73
Pb, ppm	490	21	448	531	427	552	4.24%	8.48%	12.72%	465	514
Pr, wt.%	0.339	0.017	0.305	0.373	0.287	0.390	5.06%	10.11%	15.17%	0.322	0.356
Pr_6O_{11}, wt.%	0.409	0.021	0.368	0.451	0.347	0.471	5.06%	10.11%	15.17%	0.389	0.430
S, wt.%	0.171	0.011	0.150	0.193	0.139	0.204	6.28%	12.57%	18.85%	0.163	0.180
Sc, ppm	161	24	113	209	89	233	14.94%	29.89%	44.83%	153	169
Si, wt.%	1.48	0.063	1.35	1.61	1.29	1.67	4.26%	8.52%	12.78%	1.41	1.55
SiO_2, wt.%	3.17	0.135	2.90	3.44	2.76	3.57	4.26%	8.52%	12.78%	3.01	3.33
Sm, wt.%	0.122	0.008	0.107	0.137	0.099	0.145	6.25%	12.51%	18.76%	0.116	0.128
Sm_2O_3, wt.%	0.142	0.009	0.124	0.159	0.115	0.168	6.25%	12.51%	18.76%	0.135	0.149
Sn, ppm	131	4	122	139	118	144	3.33%	6.66%	9.98%	124	137
Sr, wt.%	0.444	0.029	0.386	0.502	0.357	0.531	6.50%	13.01%	19.51%	0.422	0.466
Ta, ppm	67	4.3	59	76	55	80	6.32%	12.64%	18.97%	64	71
Tb, ppm	52	2.3	47	56	45	58	4.43%	8.87%	13.30%	49	54
Tb_4O_7, ppm	61	2.7	55	66	53	69	4.43%	8.87%	13.30%	58	64
Th, ppm	772	29	714	830	685	859	3.77%	7.54%	11.30%	733	810

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

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

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate / Peroxide Fusion ICP continued											
Ti, wt.%	5.18	0.236	4.71	5.65	4.47	5.89	4.55%	9.11%	13.66%	4.92	5.44
TiO_2, wt.%	8.64	0.394	7.85	9.43	7.46	9.82	4.55%	9.11%	13.66%	8.21	9.07
Tm, ppm	4.03	0.253	3.52	4.53	3.27	4.79	6.29%	12.57%	18.86%	3.83	4.23
Tm_2O_3, ppm	4.59	0.278	4.04	5.15	3.76	5.43	6.05%	12.09%	18.14%	4.36	4.82
U, ppm	12.7	0.43	11.9	13.6	11.4	14.0	3.35%	6.71%	10.06%	12.1	13.3
V, ppm	450	28	393	506	365	534	6.27%	12.53%	18.80%	427	472
V_2O_5, ppm	803	50	702	904	652	954	6.27%	12.53%	18.80%	763	843
W, ppm	5.62	1.01	3.60	7.63	2.60	8.64	17.93%	35.87%	53.80%	5.34	5.90
Y, ppm	481	27	428	534	401	561	5.54%	11.09%	16.63%	457	505
Y_2O_3, ppm	605	30	546	665	516	695	4.95%	9.89%	14.84%	575	636
Yb, ppm	17.7	0.88	15.9	19.4	15.0	20.3	5.01%	10.02%	15.03%	16.8	18.5
Yb_2O_3, ppm	19.9	0.93	18.0	21.7	17.1	22.7	4.68%	9.37%	14.05%	18.9	20.9
Zn, ppm	968	49	870	1066	821	1115	5.06%	10.13%	15.19%	920	1017
Zr, wt.%	0.162	0.021	0.119	0.205	0.097	0.226	13.28%	26.57%	39.85%	0.154	0.170
Borate Fusion XRF											
Al_2O_3, wt.%	10.88	0.252	10.38	11.39	10.13	11.64	2.32%	4.64%	6.95%	10.34	11.43
BaO, wt.%	0.451	0.020	0.410	0.492	0.390	0.513	4.54%	9.08%	13.61%	0.429	0.474
CaO, wt.%	0.958	0.060	0.838	1.079	0.778	1.139	6.26%	12.52%	18.79%	0.911	1.006
CeO_2, wt.%	4.32	0.117	4.09	4.55	3.97	4.67	2.70%	5.40%	8.10%	4.10	4.54
Cr_2O_3, ppm	534	91	352	716	261	807	17.03%	34.05%	51.08%	507	561
Eu_2O_3, ppm	263	60	143	382	83	442	22.75%	45.51%	68.26%	249	276
Fe_2O_3, wt.%	44.30	1.581	41.14	47.46	39.56	49.04	3.57%	7.14%	10.71%	42.09	46.52
Gd_2O_3, ppm	735	104	527	944	423	1048	14.16%	28.32%	42.48%	699	772
Ho_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K_2O, wt.%	0.018	0.004	0.011	0.026	0.007	0.030	20.09%	40.18%	60.27%	0.018	0.019
La_2O_3, wt.%	2.51	0.031	2.45	2.57	2.42	2.60	1.24%	2.47%	3.71%	2.38	2.63
Lu_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
MgO, wt.%	0.467	0.035	0.397	0.537	0.362	0.572	7.49%	14.97%	22.46%	0.444	0.490
MnO, wt.%	0.318	0.014	0.289	0.346	0.275	0.361	4.53%	9.06%	13.58%	0.302	0.334
Nb_2O_5, wt.%	0.599	0.022	0.555	0.643	0.533	0.665	3.68%	7.36%	11.04%	0.569	0.629
Nd_2O_3, wt.%	1.26	0.037	1.18	1.33	1.15	1.37	2.94%	5.87%	8.81%	1.19	1.32
P_2O_5, wt.%	7.47	0.337	6.79	8.14	6.46	8.48	4.51%	9.03%	13.54%	7.09	7.84
Pr_6O_{11}, wt.%	0.408	0.026	0.356	0.461	0.330	0.487	6.43%	12.85%	19.28%	0.388	0.429
SiO_2, wt.%	3.00	0.091	2.82	3.19	2.73	3.28	3.05%	6.09%	9.14%	2.85	3.15
Sm_2O_3, wt.%	0.143	0.011	0.121	0.165	0.110	0.176	7.70%	15.39%	23.09%	0.136	0.150
SrO, wt.%	0.539	0.034	0.472	0.607	0.438	0.641	6.27%	12.55%	18.82%	0.512	0.566
Tb_4O_7, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
ThO_2, ppm	859	48	764	954	716	1001	5.54%	11.08%	16.62%	816	902
TiO_2, wt.%	8.90	0.242	8.41	9.38	8.17	9.62	2.72%	5.44%	8.16%	8.45	9.34
Tm_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
U_3O_8, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
V_2O_5, ppm	892	121	651	1133	530	1253	13.51%	27.03%	40.54%	847	936
Y_2O_3, ppm	588	69	450	725	382	794	11.68%	23.36%	35.04%	558	617

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

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

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate Fusion XRF continued											
Yb_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
ZrO_2, wt.%	0.232	0.014	0.205	0.260	0.191	0.274	5.99%	11.99%	17.98%	0.221	0.244
Thermogravimetry											
LOI^{1000}, wt.%	11.66	0.489	10.68	12.64	10.19	13.12	4.19%	8.39%	12.58%	11.07	12.24

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

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

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

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Lima, Peru
4. ALS, Loughrea, Galway, Ireland
5. ALS, Malaga, WA, Australia
6. ALS, Vancouver, BC, Canada
7. American Assay Laboratories, Sparks, Nevada, USA
8. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
9. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
10. Bureau Veritas Minerals, Ankara, Central Anatolia, Turkey
11. CRS Laboratories Oy, Kempele, Northern Ostrobothnia, Finland
12. Inspectorate (BV), Lima, Peru
13. Intertek, Perth, WA, Australia
14. Intertek Genalysis, Adelaide, SA, Australia
15. Ontario Geological Survey, Sudbury, Ontario, Canada
16. Paragon Geochemical Laboratories, Sparks, Nevada, USA
17. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
18. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
19. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
20. SGS, Ankara, Anatolia, Turkey
21. SGS, Randfontein, Gauteng, South Africa
22. SGS Australia Mineral Services, Perth, WA, Australia
23. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
24. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
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 465b is prepared, certified and supplied by:

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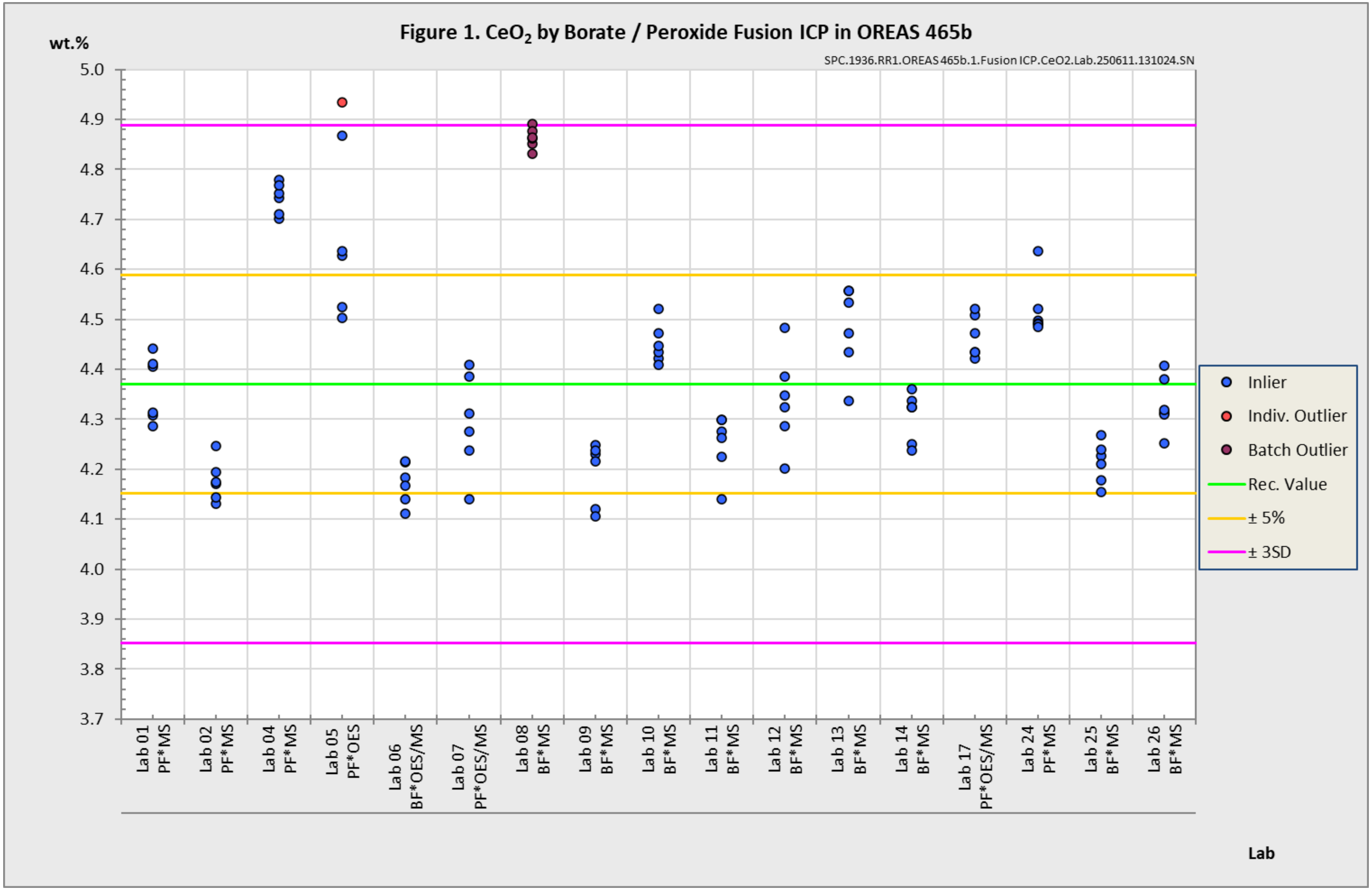

Figure 1. CeO_2 by Borate / Peroxide Fusion ICP in OREAS 465b

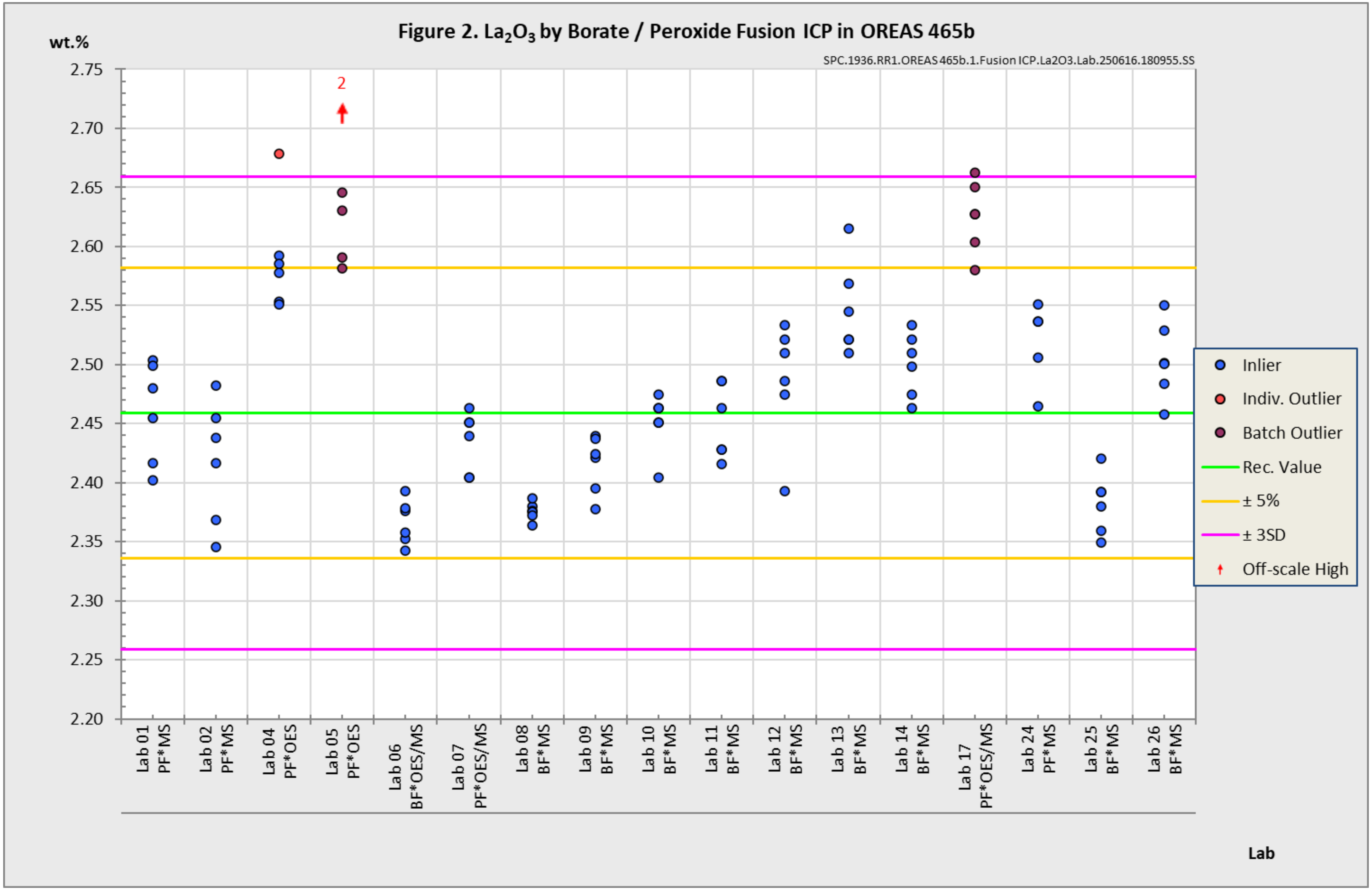
Figure 2. La_2O_3 by Borate / Peroxide Fusion ICP in OREAS 465b
wt.%
SPC.1936.RR1.OREAS465b.1.Fusion ICP.La2O3.Lab.250616.180955.SS
2.75
2.70
2.65
2.60
2.55
2.50
2.45
2.40
2.35
2.30
2.25
2.20
2
Lab 01 PF*MS
Lab 02 PF*MS
Lab 04 PF*OES
Lab 05 PF*OES
Lab 06 BF*OES/MS
Lab 07 PF*OES/MS
Lab 08 BF*MS
Lab 09 BF*MS
Lab 10 BF*MS
Lab 11 BF*MS
Lab 12 BF*MS
Lab 13 BF*MS
Lab 14 BF*MS
Lab 17 PF*OES/MS
Lab 24 PF*MS
Lab 25 BF*MS
Lab 26 BF*MS
Lab
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale High

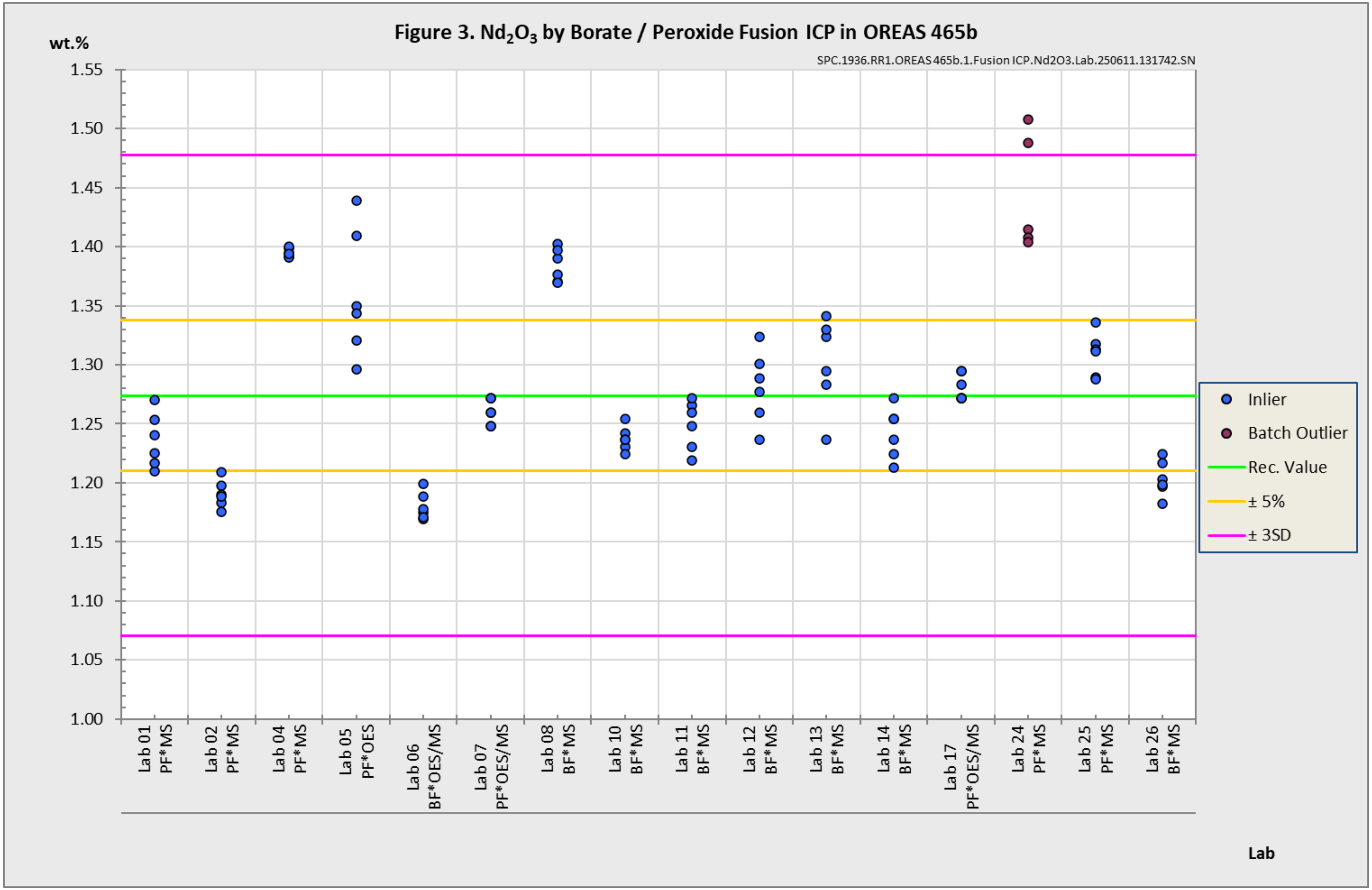
Figure 3. Nd_2O_3 by Borate / Peroxide Fusion ICP in OREAS 465b
SPC.1936.RR1.OREAS 465b.1.Fusion ICP.Nd2O3.Lab.250611.131742.SN
wt.%
1.55
1.50
1.45
1.40
1.35
1.30
1.25
1.20
1.15
1.10
1.05
1.00
Lab 01 PF*MS
Lab 02 PF*MS
Lab 04 PF*MS
Lab 05 PF*OES
Lab 06 BF*OES/MS
Lab 07 PF*OES/MS
Lab 08 BF*MS
Lab 10 BF*MS
Lab 11 BF*MS
Lab 12 BF*MS
Lab 13 BF*MS
Lab 14 BF*MS
Lab 17 PF*OES/MS
Lab 24 PF*MS
Lab 25 PF*MS
Lab 26 BF*MS
Lab
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD

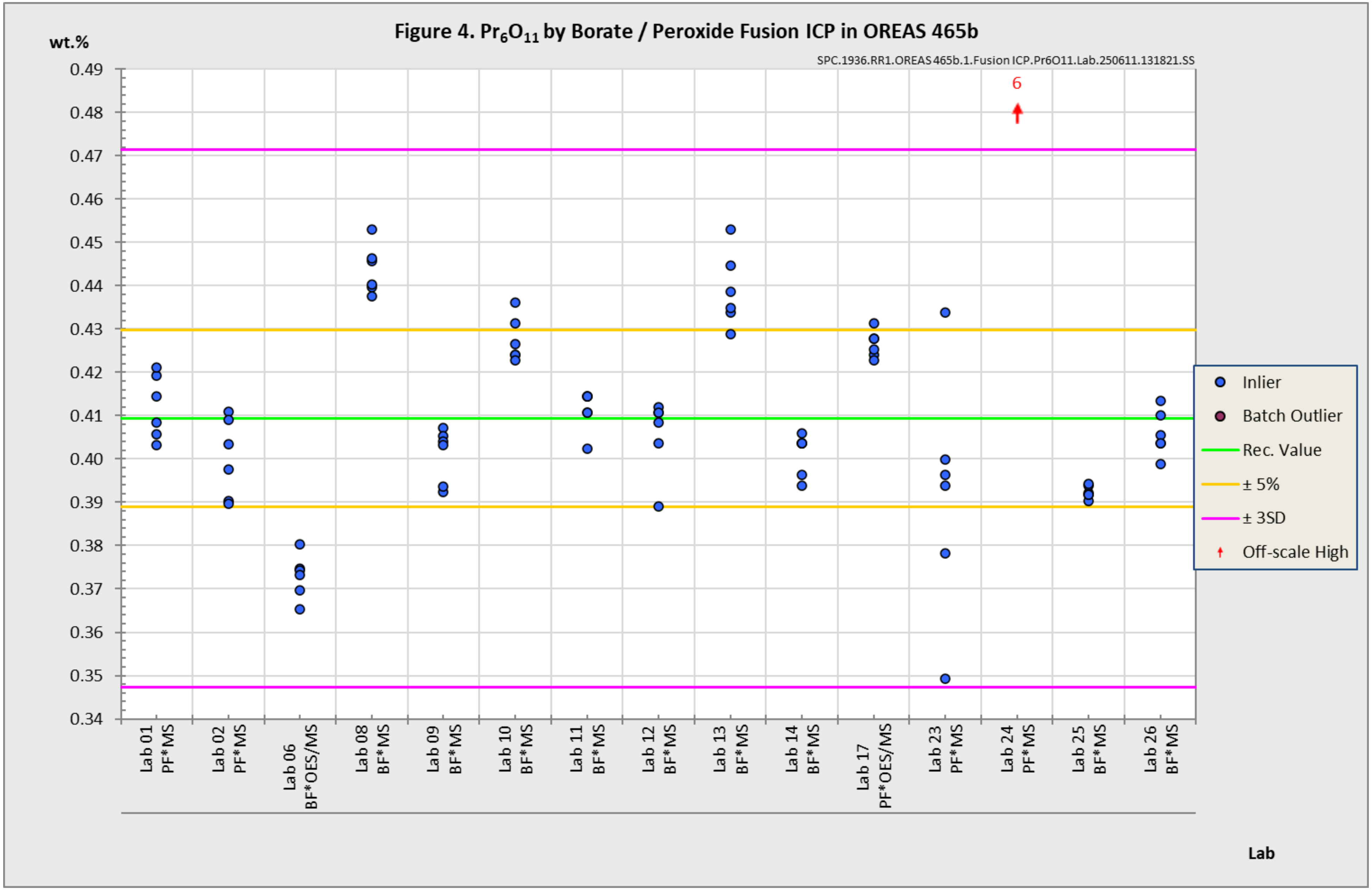
Figure 4. Pr6O11 by Borate / Peroxide Fusion ICP in OREAS 465b
wt.%
SPC.1936.RR1.OREAS465b.1.Fusion ICP.Pr6O11.Lab.250611.131821.SS
0.49
0.48
0.47
0.46
0.45
0.44
0.43
0.42
0.41
0.40
0.39
0.38
0.37
0.36
0.35
0.34
6
Lab 01 PF*MS
Lab 02 PF*MS
Lab 06 BF*OES/MS
Lab 08 BF*MS
Lab 09 BF*MS
Lab 10 BF*MS
Lab 11 BF*MS
Lab 12 BF*MS
Lab 13 BF*MS
Lab 14 BF*MS
Lab 17 PF*OES/MS
Lab 23 PF*MS
Lab 24 PF*MS
Lab 25 BF*MS
Lab 26 BF*MS
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale High
Lab

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 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 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 465b 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 465b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 465b 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/ validation 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:

- 4-acid digestion with ICP-OES and/or MS finish: ≥ 0.25 g
- Sodium peroxide / Lithium Borate 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.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 465b 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 10 g sachets

OREAS 465b is available 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 465b contains a pre-equilibrated 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 (~0.15 wt.% S).

*A pre-equilibrated 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_2O- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will 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.

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

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

QC monitoring using multiples of the Standard Deviation (SD)

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

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

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

LEGAL NOTICE

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

DOCUMENT HISTORY

Revision No.	Date	Changes applied
1	15th December, 2025	Updated certified values for selected REE elements (4-acid digestion).
0	15th July, 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

[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 33401:2024-01. Reference materials – Contents of certificates, labels and accompanying documentation.

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

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

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

[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.