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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 466

Carbonatite Supergene REE-Nb Ore (TREO + Y_2O_3 ~0.11%)

(Mount Weld Mine, Western Australia)

Accredited for compliance with ISO 17034

ISO 9001

COA-1936-OREAS 466-R2 BUP-70-10-01 Ver:2.0	15-Dec-2025

Table 1. Certified Values, Uncertainty & Tolerance Intervals for multi-elements by 4-acid digestion in OREAS 466.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion					
Ag, Silver (ppm)	0.322	0.258	0.386	0.287	0.357
Al, Aluminium (wt.%)	7.91	7.69	8.13	7.77	8.04
As, Arsenic (ppm)	481	463	500	472	491
Ba, Barium (ppm)	702	679	725	689	714
Be, Beryllium (ppm)	2.92	2.80	3.03	2.81	3.02
Bi, Bismuth (ppm)	22.1	20.5	23.7	20.9	23.2
Ca, Calcium (wt.%)	0.226	0.215	0.236	0.219	0.232
Ce, Cerium (ppm)	339	325	354	332	347
Co, Cobalt (ppm)	2.92	2.74	3.10	2.81	3.04
Cr, Chromium (ppm)	132	123	141	127	137
Cs, Caesium (ppm)	10.8	10.3	11.2	10.4	11.1
Cu, Copper (ppm)	34.2	32.9	35.4	33.2	35.2
Eu, Europium (ppm)	4.26	3.96	4.55	4.13	4.38
Fe, Iron (wt.%)	4.97	4.81	5.13	4.86	5.08
Ga, Gallium (ppm)	22.4	21.3	23.4	21.8	22.9
Gd, Gadolinium (ppm)	11.4	10.5	12.3	11.0	11.8
Ge, Germanium (ppm)	0.42	0.32	0.52	0.38	0.46
In, Indium (ppm)	0.12	0.11	0.14	0.11	0.13
K, Potassium (wt.%)	2.39	2.33	2.45	2.34	2.45
La, Lanthanum (ppm)	229	215	243	221	237
Li, Lithium (ppm)	43.6	42.3	44.9	42.5	44.6
Mg, Magnesium (wt.%)	0.412	0.389	0.435	0.400	0.424
Mn, Manganese (wt.%)	0.009	0.009	0.010	0.009	0.009
Mo, Molybdenum (ppm)	6.41	6.07	6.75	6.15	6.68
Na, Sodium (wt.%)	0.164	0.153	0.175	0.158	0.169
Nb, Niobium (ppm)	109	104	115	105	114
Nd, Neodymium (ppm)	152	146	159	148	156
Ni, Nickel (ppm)	18.7	17.6	19.7	17.9	19.4
P, Phosphorus (wt.%)	0.055	0.053	0.057	0.054	0.056
Pb, Lead (ppm)	22.9	21.4	24.4	21.5	24.2
Pr, Praseodymium (ppm)	44.1	42.0	46.3	42.9	45.4
Rb, Rubidium (ppm)	140	134	147	135	145
S, Sulphur (wt.%)	0.012	0.009	0.014	IND	IND
Sb, Antimony (ppm)	5.45	5.24	5.65	5.20	5.69
Sc, Scandium (ppm)	18.8	17.8	19.8	18.2	19.4

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).

Table 1 continued.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Sm, Samarium (ppm)	20.8	19.5	22.2	19.8	21.9
Sn, Tin (ppm)	17.5	16.6	18.5	16.8	18.3
Sr, Strontium (ppm)	107	103	111	104	110
Ta, Tantalum (ppm)	2.57	2.37	2.76	2.43	2.70
Tb, Terbium (ppm)	1.26	1.16	1.37	1.24	1.29
Te, Tellurium (ppm)	0.30	0.22	0.38	0.25	0.35
Th, Thorium (ppm)	32.4	30.6	34.2	31.2	33.6
Ti, Titanium (wt.%)	0.448	0.413	0.484	0.426	0.471
Tl, Thallium (ppm)	0.79	0.73	0.84	0.76	0.82
U, Uranium (ppm)	3.04	2.88	3.19	2.92	3.15
V, Vanadium (ppm)	128	123	132	124	131
W, Tungsten (ppm)	90	83	97	86	94
Zn, Zinc (ppm)	41.8	39.9	43.8	40.1	43.6
Zr, Zirconium (ppm)	163	155	171	158	168

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).

Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 466.

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.%)	8.34	8.18	8.51	8.24	8.45
Al_2O_3, Aluminium(III) oxide (wt.%)	15.77	15.46	16.08	15.58	15.96
As, Arsenic (ppm)	472	431	514	453	491
B, Boron (wt.%)	0.138	0.121	0.156	0.132	0.145
Ba, Barium (ppm)	719	692	746	706	732
BaO, Barium oxide (ppm)	803	773	832	788	817
Be, Beryllium (ppm)	3.12	2.28	3.97	2.55	3.70
Bi, Bismuth (ppm)	22.0	19.3	24.6	19.5	24.4
Ca, Calcium (wt.%)	0.245	0.231	0.259	0.233	0.257
CaO, Calcium oxide (wt.%)	0.343	0.324	0.362	0.326	0.359
Cd, Cadmium (ppm)	< 10	IND	IND	IND	IND
Ce, Cerium (wt.%)	0.036	0.034	0.038	0.035	0.036
CeO_2, Cerium(IV) oxide (wt.%)	0.044	0.041	0.046	0.043	0.045
Co, Cobalt (ppm)	3.26	2.59	3.92	2.80	3.71

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)					
Cr, Chromium (ppm)	170	148	192	153	187
Cr_2O_3, Chromium(III) oxide (ppm)	249	217	281	224	273
Cs, Caesium (ppm)	10.9	10.2	11.5	10.4	11.4
Cu, Copper (ppm)	35.6	30.5	40.6	28.4	42.7
Dy, Dysprosium (ppm)	7.64	6.96	8.32	7.25	8.03
Dy_2O_3, Dysprosium(III) oxide (ppm)	8.72	7.96	9.48	8.28	9.16
Er, Erbium (ppm)	3.65	3.29	4.02	3.46	3.85
Er_2O_3, Erbium(III) oxide (ppm)	4.24	3.79	4.69	4.01	4.47
Eu, Europium (ppm)	4.42	4.09	4.74	4.19	4.64
Eu_2O_3, Europium(III) oxide (ppm)	5.11	4.74	5.49	4.86	5.37
Fe, Iron (wt.%)	5.17	4.98	5.37	5.09	5.25
Fe_2O_3, Iron(III) oxide (wt.%)	7.40	7.12	7.68	7.28	7.51
Ga, Gallium (ppm)	22.2	20.2	24.2	20.9	23.6
Gd, Gadolinium (ppm)	12.1	11.4	12.9	11.7	12.6
Gd_2O_3, Gadolinium(III) oxide (ppm)	14.0	13.2	14.8	13.4	14.5
Hf, Hafnium (ppm)	7.50	6.73	8.27	7.04	7.96
HfO_2, Hafnium dioxide (ppm)	8.84	7.94	9.75	8.31	9.38
Ho, Holmium (ppm)	1.37	1.25	1.48	1.29	1.44
Ho_2O_3, Holmium(III) oxide (ppm)	1.56	1.43	1.70	1.48	1.65
K, Potassium (wt.%)	2.41	2.29	2.52	2.36	2.45
K_2O, Potassium oxide (wt.%)	2.90	2.76	3.04	2.85	2.95
La, Lanthanum (ppm)	238	225	251	231	245
La_2O_3, Lanthanum(III) oxide (ppm)	279	264	294	271	287
Li, Lithium (ppm)	44.4	41.4	47.4	42.3	46.5
Lu, Lutetium (ppm)	0.47	0.41	0.53	0.42	0.52
Lu_2O_3, Lutetium(III) oxide (ppm)	0.53	0.44	0.62	0.47	0.59
Mg, Magnesium (wt.%)	0.486	0.457	0.514	0.474	0.497
MgO, Magnesium oxide (wt.%)	0.805	0.758	0.852	0.786	0.824
Mn, Manganese (wt.%)	0.010	0.009	0.011	0.010	0.010
MnO, Manganese oxide (wt.%)	0.013	0.012	0.014	0.012	0.013
Mo, Molybdenum (ppm)	6.72	5.57	7.87	IND	IND
Nb, Niobium (ppm)	113	107	119	110	116
Nd, Neodymium (ppm)	155	147	163	151	159
Nd_2O_3, Neodymium(III) oxide (ppm)	181	172	190	176	186
Ni, Nickel (ppm)	24.6	11.6	37.7	IND	IND
P, Phosphorus (wt.%)	0.056	0.048	0.064	0.050	0.062

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)					
P_2O_5, Phosphorus(V) oxide (wt.%)	0.128	0.111	0.146	0.115	0.142
Pr, Praseodymium (ppm)	44.4	41.8	46.9	43.0	45.7
Pr_6O_{11}, Praseodymium(III,IV) oxide (ppm)	54	51	57	52	55
Rb, Rubidium (ppm)	138	130	146	132	143
Sc, Scandium (ppm)	19.2	15.4	22.9	17.8	20.6
Si, Silicon (wt.%)	30.70	28.72	32.67	30.10	31.29
SiO_2, Silicon dioxide (wt.%)	65.66	61.44	69.88	64.39	66.94
Sm, Samarium (ppm)	21.0	19.6	22.4	20.0	21.9
Sm_2O_3, Samarium(III) oxide (ppm)	24.3	22.7	26.0	23.2	25.4
Sn, Tin (ppm)	20.5	19.2	21.8	18.8	22.2
Sr, Strontium (ppm)	110	103	117	106	114
Ta, Tantalum (ppm)	2.94	2.69	3.19	2.71	3.17
Tb, Terbium (ppm)	1.48	1.36	1.61	1.41	1.55
Tb_4O_7, Terbium(III,IV) oxide (ppm)	1.74	1.60	1.89	1.66	1.82
Th, Thorium (ppm)	31.9	30.3	33.5	31.1	32.7
Ti, Titanium (wt.%)	0.587	0.566	0.607	0.572	0.601
TiO_2, Titanium dioxide (wt.%)	0.978	0.945	1.012	0.955	1.002
Tm, Thulium (ppm)	0.51	0.44	0.57	0.47	0.54
Tm_2O_3, Thulium(III) oxide (ppm)	0.58	0.50	0.65	0.53	0.62
U, Uranium (ppm)	3.44	3.15	3.74	3.31	3.58
V, Vanadium (ppm)	140	132	148	136	144
V_2O_5, Vanadium(V) oxide (ppm)	250	235	265	243	257
W, Tungsten (ppm)	94	88	101	91	98
Y, Yttrium (ppm)	34.6	32.8	36.5	33.4	35.9
Y_2O_3, Yttrium(III) oxide (ppm)	44.0	41.6	46.3	42.4	45.6
Yb, Ytterbium (ppm)	3.29	2.96	3.63	3.07	3.52
Yb_2O_3, Ytterbium(III) oxide (ppm)	3.72	3.30	4.13	3.47	3.97
Zn, Zinc (ppm)	47.4	39.2	55.7	43.7	51.1
Zr, Zirconium (ppm)	283	265	300	269	296
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	15.81	15.60	16.02	15.71	15.91
BaO, Barium oxide (ppm)	755	663	847	IND	IND
CaO, Calcium oxide (wt.%)	0.318	0.298	0.338	0.302	0.335
CeO_2, Cerium(IV) oxide (wt.%)	0.044	0.034	0.053	IND	IND
Cr_2O_3, Chromium(III) oxide (ppm)	197	151	243	IND	IND
Dy_2O_3, Dysprosium(III) oxide (ppm)	< 100	IND	IND	IND	IND
Er_2O_3, Erbium(III) oxide (ppm)	< 100	IND	IND	IND	IND
Eu_2O_3, Europium(III) oxide (ppm)	< 100	IND	IND	IND	IND

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 Fusion XRF continued					
Fe_2O_3, Iron(III) oxide (wt.%)	7.29	7.18	7.40	7.22	7.36
Ho_2O_3, Holmium(III) oxide (ppm)	< 100	IND	IND	IND	IND
K_2O, Potassium oxide (wt.%)	2.87	2.82	2.93	2.85	2.89
La_2O_3, Lanthanum(III) oxide (wt.%)	0.030	0.029	0.031	IND	IND
Lu_2O_3, Lutetium(III) oxide (ppm)	< 100	IND	IND	IND	IND
MgO, Magnesium oxide (wt.%)	0.824	0.803	0.844	0.812	0.835
MnO, Manganese oxide (wt.%)	0.011	0.006	0.017	IND	IND
Na_2O, Sodium oxide (wt.%)	0.236	0.201	0.271	0.224	0.248
Nb_2O_5, Niobium(V) oxide (ppm)	147	117	178	IND	IND
P_2O_5, Phosphorus(V) oxide (wt.%)	0.126	0.119	0.134	0.124	0.129
SiO_2, Silicon dioxide (wt.%)	66.40	65.89	66.92	66.16	66.65
Sm_2O_3, Samarium(III) oxide (ppm)	< 100	IND	IND	IND	IND
SnO_2, Tin dioxide (ppm)	< 100	IND	IND	IND	IND
SrO, Strontium oxide (ppm)	112	83	141	IND	IND
Tb_4O_7, Terbium(III,IV) oxide (ppm)	< 100	IND	IND	IND	IND
TiO_2, Titanium dioxide (wt.%)	0.986	0.967	1.004	0.974	0.997
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
Yb_2O_3, Ytterbium(III) oxide (ppm)	< 100	IND	IND	IND	IND
ZrO_2, Zirconium dioxide (ppm)	363	292	434	340	386
Thermogravimetry					
LOI^{1000}, Loss on ignition @1000 °C (wt.%)	4.00	3.82	4.19	3.93	4.07

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 466.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
Cd	ppm	*0.040*	Hg	ppm	*0.47*	Se	ppm	*0.90*
Dy	ppm	*5.57*	Ho	ppm	*0.87*	Tm	ppm	*0.27*
Er	ppm	*2.19*	Lu	ppm	*0.28*	Y	ppm	*20.3*
Hf	ppm	*4.70*	Re	ppm	*0.002*	Yb	ppm	*1.83*
Borate / Peroxide Fusion ICP								
Na	wt.%	*0.197*	Na_2O	wt.%	*0.266*	Sb	ppm	*5.78*
Ag	ppm	*< 1*	Pb	ppm	*25.7*	Se	ppm	*13.7*
Ge	ppm	*2.33*	Re	ppm	*0.004*	Te	ppm	*< 1*
In	ppm	*0.20*	S	wt.%	*0.011*	Tl	ppm	*0.77*
Borate Fusion XRF								
As_2O_3	ppm	*600*	HfO_2	ppm	*< 100*	SO_3	wt.%	*0.013*
Bi_2O_3	ppm	*< 100*	MoO_3	ppm	*< 100*	Ta_2O_5	ppm	*< 100*
Cl	ppm	*< 100*	Nd_2O_3	wt.%	*0.017*	Th	ppm	*49.1*
Co_3O_4	ppm	*< 100*	Ni	ppm	*30.3*	TOT_XRF	wt.%	*99.34*
Cs_2O	ppm	*< 100*	PbO	ppm	*< 100*	V_2O_5	ppm	*244*
Cu	ppm	*37.5*	Pr_6O_{11}	ppm	*112*	WO_3	ppm	*121*
F	wt.%	*< 0.5*	Rb_2O	ppm	*133*	Y	ppm	*65*
Ga_2O_3	ppm	*< 100*	Sb_2O_3	ppm	*< 100*	ZnO	ppm	*59*
Gd_2O_3	ppm	*110*	Sc_2O_3	ppm	*< 100*			
Thermogravimetry								
H_2O-	wt.%	*0.642*						

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.

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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, 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 466-DataPack.1.2.251215_134657.xlsx**).

OREAS 466 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 466 is a certified reference material (CRM) prepared from a blend of barren siliclastic sedimentary rock sourced from Victoria, Australia with a small addition of rare earth element (REE)–bearing waste rock 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 (40.6%), La_2O_3 (27.9%), Nd_2O_3 (16.8%), Pr_6O_{11} (4.98%), Sm_2O_3 (2.26%) and Eu_2O_3 (0.475%), together with minor components of HREE: Dy_2O_3 (0.811%) and Tb_4O_7 (0.162%).

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 466 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 500 g units in plastic wide-mouth jars.

PHYSICAL PROPERTIES

OREAS 466 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 466.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
792	1.94	5YR 6/4	Light Brown

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

MINERALOGY

The semi-quantitative XRD results shown in Table 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. *Muscovite may be underestimated due to overlapping pattern with illite. 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". A trace amount of maghemite might be present in sample. Some amorphous material might be present in the sample.

Table 5. Indicative mineralogy of OREAS 466 by semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Crandallite group	< 1
Monazite	< 1
Hematite	2
Goethite	3
Clay mineral	12
Kaolinite	5
*Muscovite	19
Tourmaline	6
Quartz	53
K-feldspar and/or rutile	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 12 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 0.043 and 0.045 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 Ta displayed significant p-value less than 0.05. Any statistically significant result must be checked to determine whether it is also technically significant. The RSD for Ta across the 12 samples is 12.64%.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 466.

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	0.322	0.040	0.242	0.403	0.201	0.443	12.52%	25.04%	37.56%	0.306	0.338
Al, wt.%	7.91	0.281	7.34	8.47	7.06	8.75	3.56%	7.12%	10.67%	7.51	8.30
As, ppm	481	18	446	517	429	534	3.66%	7.33%	10.99%	457	506
Ba, ppm	702	26	649	754	623	780	3.73%	7.47%	11.20%	667	737
Be, ppm	2.92	0.130	2.66	3.18	2.53	3.31	4.47%	8.94%	13.41%	2.77	3.06
Bi, ppm	22.1	1.67	18.7	25.4	17.1	27.1	7.56%	15.11%	22.67%	21.0	23.2
Ca, wt.%	0.226	0.008	0.210	0.241	0.203	0.248	3.37%	6.74%	10.12%	0.214	0.237
Ce, ppm	339	20	300	379	281	398	5.77%	11.54%	17.31%	322	356
Co, ppm	2.92	0.123	2.68	3.17	2.56	3.29	4.19%	8.39%	12.58%	2.78	3.07
Cr, ppm	132	15	103	161	88	176	11.10%	22.20%	33.31%	125	138
Cs, ppm	10.8	0.58	9.6	11.9	9.0	12.5	5.37%	10.73%	16.10%	10.2	11.3

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.

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											
Cu, ppm	34.2	1.40	31.4	37.0	30.0	38.4	4.10%	8.20%	12.30%	32.5	35.9
Eu, ppm	4.26	0.171	3.91	4.60	3.74	4.77	4.02%	8.04%	12.05%	4.04	4.47
Fe, wt.%	4.97	0.170	4.63	5.31	4.46	5.48	3.43%	6.86%	10.30%	4.72	5.22
Ga, ppm	22.4	1.30	19.8	25.0	18.5	26.3	5.82%	11.65%	17.47%	21.2	23.5
Gd, ppm	11.4	0.88	9.6	13.2	8.8	14.0	7.69%	15.38%	23.08%	10.8	12.0
Ge, ppm	0.42	0.08	0.25	0.59	0.17	0.67	19.91%	39.81%	59.72%	0.40	0.44
In, ppm	0.12	0.012	0.10	0.14	0.09	0.16	9.62%	19.23%	28.85%	0.11	0.13
K, wt.%	2.39	0.086	2.22	2.57	2.14	2.65	3.61%	7.21%	10.82%	2.27	2.51
La, ppm	229	17	196	262	179	279	7.27%	14.55%	21.82%	218	241
Li, ppm	43.6	1.28	41.0	46.1	39.7	47.4	2.94%	5.87%	8.81%	41.4	45.7
Mg, wt.%	0.412	0.030	0.351	0.473	0.321	0.503	7.35%	14.71%	22.06%	0.391	0.433
Mn, wt.%	0.009	0.001	0.008	0.010	0.008	0.011	5.70%	11.40%	17.10%	0.009	0.010
Mo, ppm	6.41	0.356	5.70	7.13	5.35	7.48	5.55%	11.10%	16.64%	6.09	6.73
Na, wt.%	0.164	0.013	0.137	0.191	0.123	0.204	8.21%	16.42%	24.62%	0.156	0.172
Nb, ppm	109	9	92	127	84	135	7.82%	15.63%	23.45%	104	115
Nd, ppm	152	8	136	168	128	176	5.21%	10.42%	15.63%	144	160
Ni, ppm	18.7	0.95	16.8	20.6	15.8	21.5	5.06%	10.12%	15.18%	17.7	19.6
P, wt.%	0.055	0.002	0.050	0.060	0.048	0.062	4.34%	8.68%	13.01%	0.052	0.058
Pb, ppm	22.9	2.05	18.8	27.0	16.7	29.0	8.96%	17.93%	26.89%	21.7	24.0
Pr, ppm	44.1	1.20	41.7	46.5	40.5	47.7	2.71%	5.42%	8.13%	41.9	46.3
Rb, ppm	140	6	128	152	122	158	4.27%	8.55%	12.82%	133	147
S, wt.%	0.012	0.003	0.005	0.018	0.002	0.021	26.99%	53.98%	80.97%	0.011	0.012
Sb, ppm	5.45	0.198	5.05	5.84	4.85	6.04	3.64%	7.28%	10.92%	5.17	5.72
Sc, ppm	18.8	1.38	16.1	21.6	14.7	23.0	7.33%	14.66%	21.99%	17.9	19.8
Sm, ppm	20.8	0.76	19.3	22.3	18.5	23.1	3.65%	7.29%	10.94%	19.8	21.9
Sn, ppm	17.5	0.82	15.9	19.2	15.1	20.0	4.68%	9.36%	14.03%	16.7	18.4
Sr, ppm	107	5	97	117	92	122	4.67%	9.35%	14.02%	102	112
Ta, ppm	2.57	0.27	2.02	3.11	1.74	3.39	10.68%	21.36%	32.04%	2.44	2.69
Tb, ppm	1.26	0.105	1.05	1.48	0.95	1.58	8.34%	16.68%	25.02%	1.20	1.33
Te, ppm	0.30	0.06	0.18	0.42	0.12	0.48	20.07%	40.14%	60.21%	0.28	0.31
Th, ppm	32.4	2.00	28.4	36.4	26.4	38.4	6.18%	12.36%	18.53%	30.8	34.0
Ti, wt.%	0.448	0.043	0.363	0.534	0.320	0.577	9.58%	19.15%	28.73%	0.426	0.471
Tl, ppm	0.79	0.059	0.67	0.90	0.61	0.96	7.43%	14.86%	22.29%	0.75	0.83
U, ppm	3.04	0.221	2.59	3.48	2.37	3.70	7.28%	14.56%	21.84%	2.88	3.19
V, ppm	128	4	120	136	116	140	3.13%	6.26%	9.40%	121	134
W, ppm	90	6.7	77	103	70	110	7.42%	14.84%	22.25%	86	95
Zn, ppm	41.8	2.19	37.5	46.2	35.3	48.4	5.22%	10.45%	15.67%	39.7	43.9
Zr, ppm	163	9	144	181	135	190	5.62%	11.24%	16.85%	155	171
Borate / Peroxide Fusion ICP											
Al, wt.%	8.34	0.171	8.00	8.69	7.83	8.86	2.05%	4.11%	6.16%	7.93	8.76
Al_2O_3, wt.%	15.77	0.324	15.12	16.42	14.80	16.74	2.05%	4.11%	6.16%	14.98	16.56
As, ppm	472	34	403	541	369	576	7.29%	14.59%	21.88%	449	496
B, wt.%	0.138	0.011	0.116	0.161	0.105	0.172	8.11%	16.21%	24.32%	0.131	0.145

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											
Ba, ppm	719	16	687	751	671	767	2.24%	4.48%	6.72%	683	755
BaO, ppm	803	18	767	839	749	857	2.24%	4.48%	6.72%	762	843
Be, ppm	3.12	0.38	2.36	3.89	1.97	4.27	12.28%	24.56%	36.83%	2.97	3.28
Bi, ppm	22.0	1.06	19.8	24.1	18.8	25.1	4.83%	9.67%	14.50%	20.9	23.1
Ca, wt.%	0.245	0.017	0.210	0.280	0.193	0.297	7.06%	14.12%	21.19%	0.233	0.257
CaO, wt.%	0.343	0.024	0.294	0.391	0.270	0.416	7.06%	14.12%	21.19%	0.326	0.360
Cd, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, wt.%	0.036	0.002	0.031	0.040	0.028	0.043	6.87%	13.74%	20.60%	0.034	0.037
CeO_2, wt.%	0.044	0.003	0.038	0.050	0.035	0.053	6.87%	13.74%	20.60%	0.042	0.046
Co, ppm	3.26	0.38	2.50	4.02	2.12	4.40	11.66%	23.32%	34.97%	3.09	3.42
Cr, ppm	170	23	124	216	101	239	13.51%	27.03%	40.54%	162	179
Cr_2O_3, ppm	249	34	182	316	148	350	13.51%	27.03%	40.54%	236	261
Cs, ppm	10.9	0.34	10.2	11.6	9.9	11.9	3.13%	6.26%	9.38%	10.3	11.4
Cu, ppm	35.6	3.08	29.4	41.7	26.3	44.8	8.65%	17.30%	25.95%	33.8	37.3
Dy, ppm	7.64	0.435	6.77	8.51	6.34	8.95	5.69%	11.39%	17.08%	7.26	8.02
Dy_2O_3, ppm	8.72	0.516	7.69	9.75	7.17	10.27	5.92%	11.84%	17.77%	8.28	9.16
Er, ppm	3.65	0.201	3.25	4.06	3.05	4.26	5.50%	11.00%	16.49%	3.47	3.84
Er_2O_3, ppm	4.24	0.299	3.64	4.84	3.35	5.14	7.04%	14.08%	21.12%	4.03	4.45
Eu, ppm	4.42	0.202	4.01	4.82	3.81	5.02	4.56%	9.13%	13.69%	4.20	4.64
Eu_2O_3, ppm	5.11	0.233	4.65	5.58	4.41	5.81	4.56%	9.13%	13.69%	4.86	5.37
Fe, wt.%	5.17	0.140	4.89	5.45	4.75	5.59	2.72%	5.43%	8.15%	4.91	5.43
Fe_2O_3, wt.%	7.40	0.201	6.99	7.80	6.79	8.00	2.72%	5.43%	8.15%	7.03	7.77
Ga, ppm	22.2	1.16	19.9	24.6	18.7	25.7	5.23%	10.46%	15.69%	21.1	23.3
Gd, ppm	12.1	0.49	11.2	13.1	10.7	13.6	4.07%	8.14%	12.20%	11.5	12.7
Gd_2O_3, ppm	14.0	0.53	12.9	15.0	12.4	15.6	3.78%	7.56%	11.34%	13.3	14.7
Hf, ppm	7.50	0.436	6.63	8.37	6.19	8.81	5.82%	11.64%	17.46%	7.13	7.88
HfO_2, ppm	8.84	0.515	7.82	9.87	7.30	10.39	5.82%	11.64%	17.46%	8.40	9.29
Ho, ppm	1.37	0.066	1.23	1.50	1.17	1.56	4.85%	9.70%	14.55%	1.30	1.43
Ho_2O_3, ppm	1.56	0.076	1.41	1.72	1.34	1.79	4.85%	9.70%	14.55%	1.49	1.64
K, wt.%	2.41	0.064	2.28	2.53	2.21	2.60	2.68%	5.35%	8.03%	2.29	2.53
K_2O, wt.%	2.90	0.078	2.74	3.05	2.67	3.13	2.68%	5.35%	8.03%	2.75	3.04
La, ppm	238	15	209	267	194	282	6.13%	12.25%	18.38%	226	250
La_2O_3, ppm	279	17	245	313	228	330	6.13%	12.25%	18.38%	265	293
Li, ppm	44.4	2.34	39.7	49.1	37.4	51.4	5.27%	10.54%	15.81%	42.2	46.6
Lu, ppm	0.47	0.036	0.40	0.54	0.36	0.58	7.64%	15.28%	22.92%	0.45	0.49
Lu_2O_3, ppm	0.53	0.049	0.43	0.63	0.38	0.68	9.16%	18.33%	27.49%	0.50	0.56
Mg, wt.%	0.486	0.024	0.437	0.534	0.413	0.558	4.99%	9.99%	14.98%	0.461	0.510
MgO, wt.%	0.805	0.040	0.725	0.886	0.685	0.926	4.99%	9.99%	14.98%	0.765	0.845
Mn, wt.%	0.010	0.000	0.009	0.011	0.009	0.011	3.52%	7.04%	10.56%	0.010	0.011
MnO, wt.%	0.013	0.000	0.012	0.014	0.012	0.014	3.52%	7.04%	10.56%	0.012	0.014
Mo, ppm	6.72	0.538	5.65	7.80	5.11	8.34	8.00%	16.00%	24.01%	6.39	7.06
Nb, ppm	113	6	101	125	95	131	5.27%	10.54%	15.80%	107	119
Nd, ppm	155	10	136	174	126	184	6.16%	12.33%	18.49%	147	163

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.

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											
Nd_2O_3, ppm	181	11	159	203	148	214	6.16%	12.33%	18.49%	172	190
Ni, ppm	24.6	6.4	11.8	37.5	5.3	43.9	26.10%	52.20%	78.31%	23.4	25.9
P, wt.%	0.056	0.005	0.047	0.065	0.042	0.070	8.11%	16.22%	24.33%	0.053	0.059
P_2O_5, wt.%	0.128	0.010	0.108	0.149	0.097	0.160	8.11%	16.22%	24.33%	0.122	0.135
Pr, ppm	44.4	2.50	39.4	49.4	36.8	51.9	5.65%	11.29%	16.94%	42.1	46.6
Pr_6O_{11}, ppm	54	3.0	48	60	45	63	5.65%	11.29%	16.94%	51	56
Rb, ppm	138	7	124	151	117	158	4.99%	9.99%	14.98%	131	145
Sc, ppm	19.2	3.2	12.8	25.6	9.5	28.8	16.75%	33.50%	50.25%	18.2	20.1
Si, wt.%	30.70	1.609	27.48	33.91	25.87	35.52	5.24%	10.48%	15.73%	29.16	32.23
SiO_2, wt.%	65.66	3.442	58.78	72.55	55.34	75.99	5.24%	10.48%	15.73%	62.38	68.95
Sm, ppm	21.0	1.29	18.4	23.6	17.1	24.8	6.14%	12.27%	18.41%	19.9	22.0
Sm_2O_3, ppm	24.3	1.49	21.3	27.3	19.9	28.8	6.14%	12.27%	18.41%	23.1	25.5
Sn, ppm	20.5	1.32	17.9	23.1	16.5	24.5	6.43%	12.85%	19.28%	19.5	21.5
Sr, ppm	110	6	98	122	92	128	5.40%	10.79%	16.19%	104	115
Ta, ppm	2.94	0.173	2.60	3.29	2.42	3.46	5.87%	11.74%	17.61%	2.79	3.09
Tb, ppm	1.48	0.091	1.30	1.66	1.21	1.75	6.12%	12.25%	18.37%	1.41	1.56
Tb_4O_7, ppm	1.74	0.107	1.53	1.96	1.42	2.06	6.12%	12.25%	18.37%	1.66	1.83
Th, ppm	31.9	1.65	28.6	35.2	27.0	36.9	5.17%	10.34%	15.52%	30.3	33.5
Ti, wt.%	0.587	0.016	0.555	0.618	0.539	0.634	2.69%	5.38%	8.07%	0.557	0.616
TiO_2, wt.%	0.978	0.026	0.926	1.031	0.899	1.057	2.69%	5.38%	8.07%	0.929	1.027
Tm, ppm	0.51	0.030	0.45	0.57	0.42	0.60	5.91%	11.82%	17.73%	0.48	0.53
Tm_2O_3, ppm	0.58	0.035	0.51	0.65	0.47	0.68	6.15%	12.29%	18.44%	0.55	0.60
U, ppm	3.44	0.223	3.00	3.89	2.78	4.11	6.47%	12.93%	19.40%	3.27	3.62
V, ppm	140	5	130	150	125	155	3.60%	7.20%	10.79%	133	147
V_2O_5, ppm	250	9	232	268	223	277	3.60%	7.20%	10.79%	237	262
W, ppm	94	5.1	84	105	79	110	5.36%	10.72%	16.08%	90	99
Y, ppm	34.6	1.98	30.7	38.6	28.7	40.6	5.73%	11.45%	17.18%	32.9	36.4
Y_2O_3, ppm	44.0	2.52	38.9	49.0	36.4	51.5	5.73%	11.45%	17.18%	41.8	46.2
Yb, ppm	3.29	0.176	2.94	3.65	2.77	3.82	5.34%	10.68%	16.02%	3.13	3.46
Yb_2O_3, ppm	3.72	0.256	3.20	4.23	2.95	4.49	6.90%	13.79%	20.69%	3.53	3.90
Zn, ppm	47.4	4.02	39.4	55.5	35.4	59.5	8.48%	16.96%	25.44%	45.1	49.8
Zr, ppm	283	12	258	307	245	320	4.39%	8.77%	13.16%	269	297
Borate Fusion XRF											
Al_2O_3, wt.%	15.81	0.253	15.30	16.32	15.05	16.57	1.60%	3.20%	4.80%	15.02	16.60
BaO, ppm	755	70	614	896	544	966	9.32%	18.64%	27.95%	717	793
CaO, wt.%	0.318	0.020	0.279	0.358	0.260	0.377	6.15%	12.30%	18.44%	0.303	0.334
CeO_2, wt.%	0.044	0.007	0.029	0.059	0.021	0.066	17.12%	34.24%	51.36%	0.041	0.046
Cr_2O_3, ppm	197	50	97	297	47	347	25.43%	50.86%	76.29%	187	207
Dy_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Er_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Eu_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Fe_2O_3, wt.%	7.29	0.100	7.09	7.49	6.99	7.59	1.37%	2.74%	4.11%	6.92	7.65
Ho_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

SI unit equivalents: ppm (parts per million; 1×10^{-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											
K_2O, wt.%	2.87	0.060	2.75	2.99	2.69	3.05	2.08%	4.16%	6.25%	2.73	3.02
La_2O_3, wt.%	0.030	0.000	0.029	0.031	0.029	0.031	1.26%	2.51%	3.77%	0.029	0.032
Lu_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
MgO, wt.%	0.824	0.022	0.780	0.867	0.759	0.888	2.62%	5.25%	7.87%	0.782	0.865
MnO, wt.%	0.011	0.003	0.006	0.017	0.003	0.020	24.93%	49.87%	74.80%	0.011	0.012
Na_2O, wt.%	0.236	0.031	0.173	0.299	0.142	0.330	13.32%	26.63%	39.95%	0.224	0.248
Nb_2O_5, ppm	147	32	83	212	51	244	21.83%	43.66%	65.48%	140	155
P_2O_5, wt.%	0.126	0.005	0.116	0.137	0.110	0.143	4.28%	8.56%	12.84%	0.120	0.133
SiO_2, wt.%	66.40	0.514	65.37	67.43	64.86	67.95	0.77%	1.55%	2.32%	63.08	69.72
Sm_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
SnO_2, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
SrO, ppm	112	22	68	157	46	179	19.69%	39.37%	59.06%	107	118
Tb_4O_7, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
TiO_2, wt.%	0.986	0.015	0.955	1.016	0.940	1.032	1.55%	3.10%	4.65%	0.936	1.035
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
Yb_2O_3, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
ZrO_2, ppm	363	39	285	441	246	480	10.76%	21.52%	32.29%	345	381
Thermogravimetry											
LOI^{1000}, wt.%	4.00	0.307	3.39	4.62	3.08	4.92	7.68%	15.36%	23.05%	3.80	4.20

SI unit equivalents: ppm (parts per million; 1×10^{-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 466 is prepared, certified and supplied by:

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AUSTRALIA	

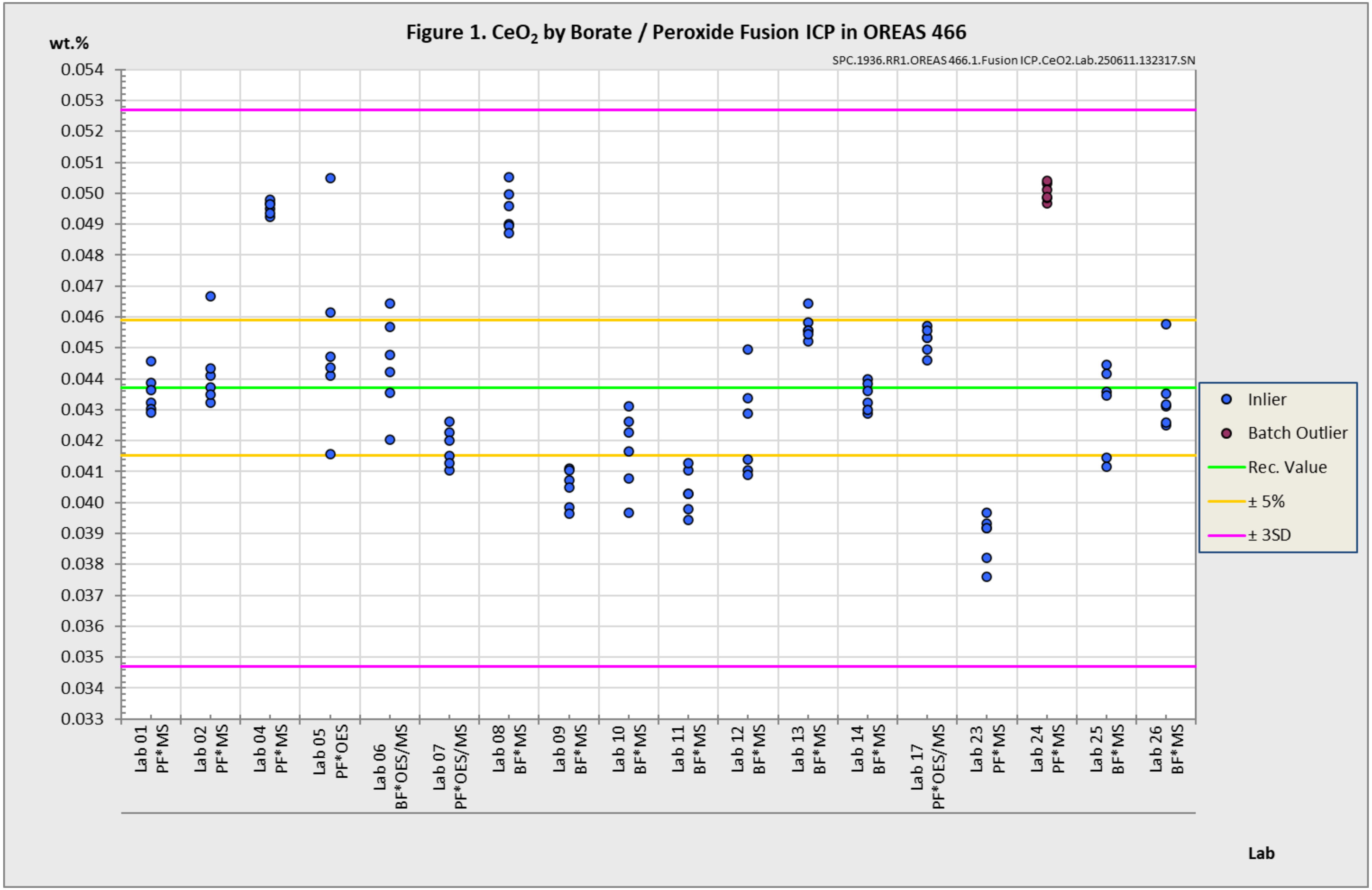
Figure 1. CeO$_2$ by Borate / Peroxide Fusion ICP in OREAS 466
wt.%
SPC.1936.RR1.OREAS466.1.Fusion ICP.CeO2.Lab.250611.132317.SN
0.054
0.053
0.052
0.051
0.050
0.049
0.048
0.047
0.046
0.045
0.044
0.043
0.042
0.041
0.040
0.039
0.038
0.037
0.036
0.035
0.034
0.033
Lab 01 PF*MS
Lab 02 PF*MS
Lab 04 PF*MS
Lab 05 PF*OES
Lab 06 BF*OES/MS
Lab 07 PF*OES/MS
Lab 08 BF*MS
Lab 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
Lab
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD

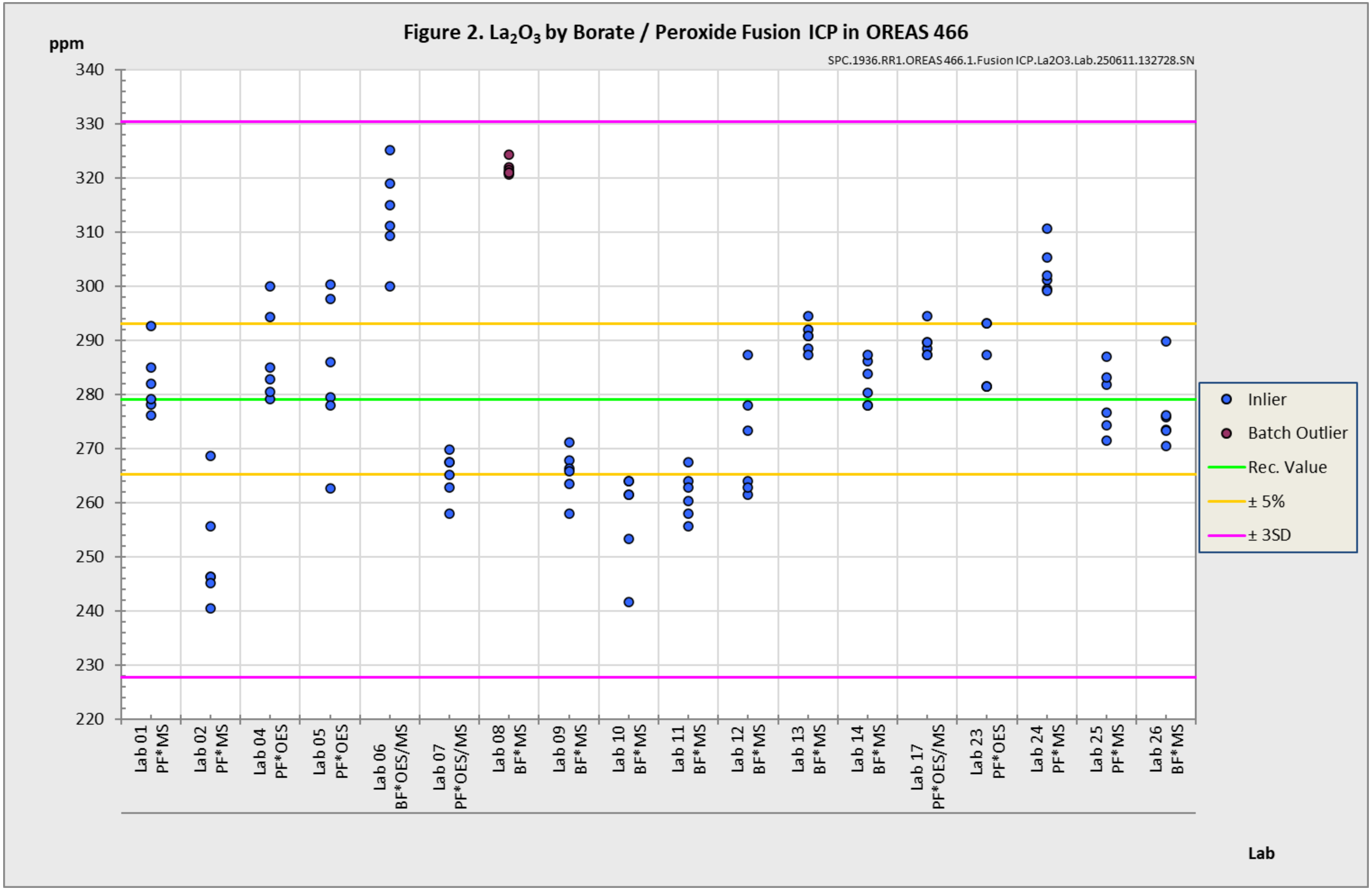
Figure 2. La2O3 by Borate / Peroxide Fusion ICP in OREAS 466
SPC.1936.RR1.OREAS 466.1.Fusion ICP.La2O3.Lab.250611.132728.SN
ppm
340
330
320
310
300
290
280
270
260
250
240
230
220
Lab 01 PF*MS
Lab 02 PF*MS
Lab 04 PF*OES
Lab 05 PF*OES
Lab 06 BF*OES/MS
Lab 07 PF*OES/MS
Lab 08 BF*MS
Lab 09 BF*MS
Lab 10 BF*MS
Lab 11 BF*MS
Lab 12 BF*MS
Lab 13 BF*MS
Lab 14 BF*MS
Lab 17 PF*OES/MS
Lab 23 PF*OES
Lab 24 PF*MS
Lab 25 PF*MS
Lab 26 BF*MS
Lab
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD

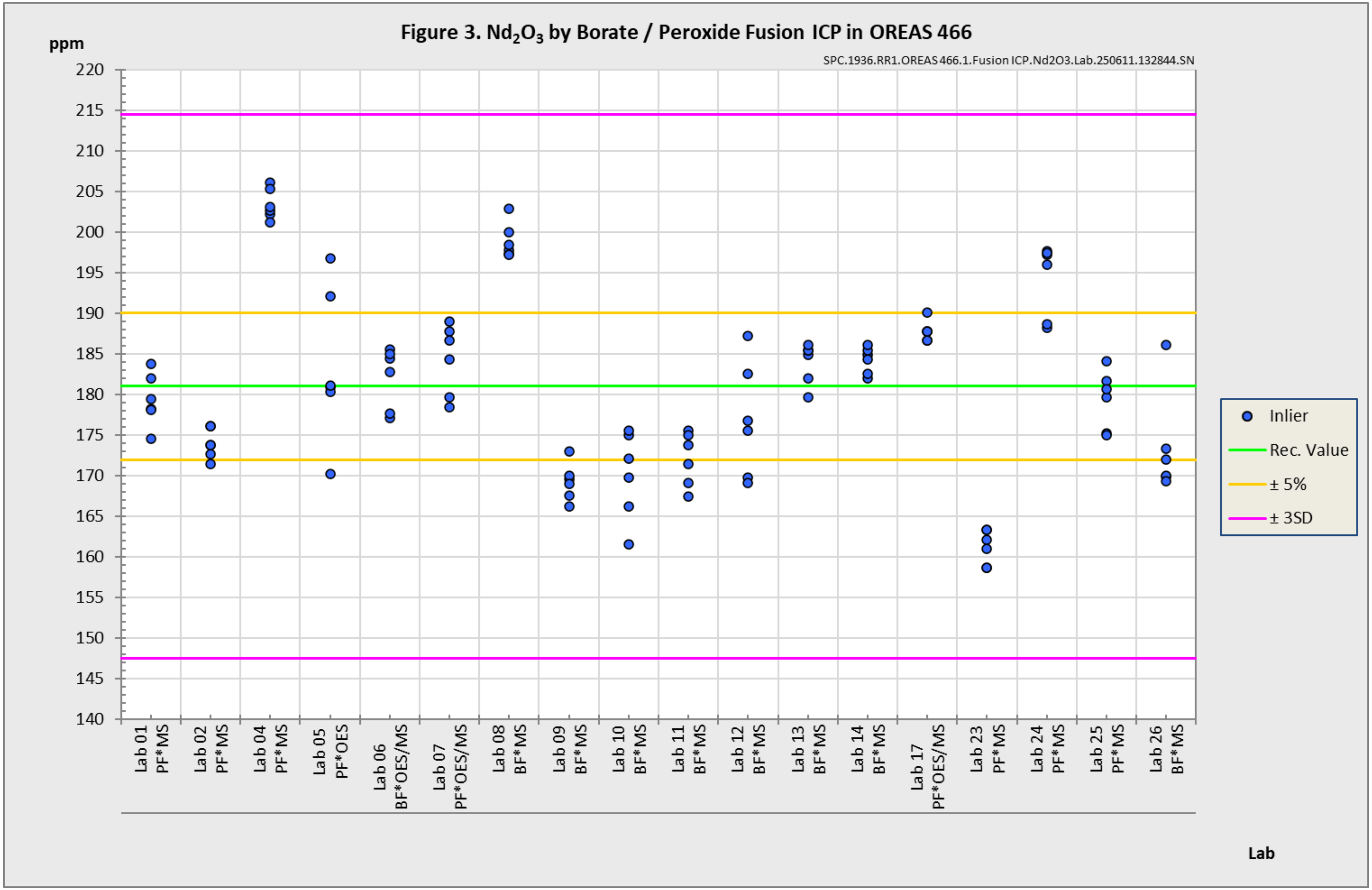

Figure 3. Nd_2O_3 by Borate / Peroxide Fusion ICP in OREAS 466

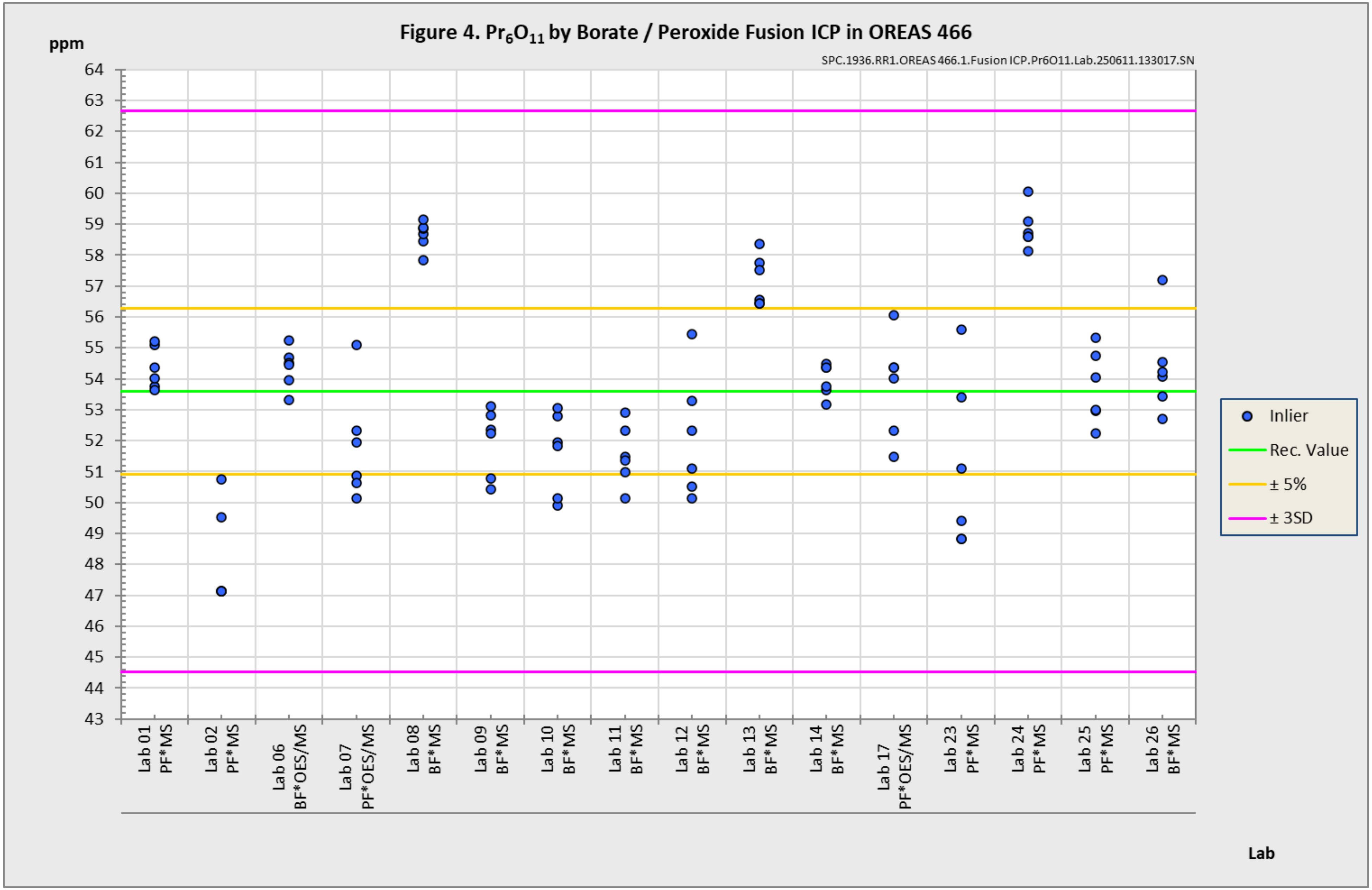
Figure 4. Pr_6O_{11} by Borate / Peroxide Fusion ICP in OREAS 466
SPC.1936.RR1.OREAS 466.1.Fusion ICP.Pr6O11.Lab.250611.133017.SN
ppm
64
63
62
61
60
59
58
57
56
55
54
53
52
51
50
49
48
47
46
45
44
43
Lab 01 PF*MS
Lab 02 PF*MS
Lab 06 BF*OES/MS
Lab 07 PF*OES/MS
Lab 08 BF*MS
Lab 09 BF*MS
Lab 10 BF*MS
Lab 11 BF*MS
Lab 12 BF*MS
Lab 13 BF*MS
Lab 14 BF*MS
Lab 17 PF*OES/MS
Lab 23 PF*MS
Lab 24 PF*MS
Lab 25 PF*MS
Lab 26 BF*MS
Lab
Inlier
Rec. Value
± 5%
± 3SD

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 466 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 466 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 466 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 466 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 466 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., 500 g plastic jars)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 466 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 (~0.01 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
2	15th December, 2025	Updated certified values for selected REE elements (4-acid digestion).
1	21st July, 2025	Updated 4-Acid Digestion results for several analytes.
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.