

CERTIFICATE OF ANALYSIS FOR

CARBONATITE SUPERGENE REE-Nb ORE (TREO 1.54%)

CERTIFIED REFERENCE MATERIAL

OREAS 462

Summary Statistics for Key Analytes (additional certified values are available in Table 1).

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate / Peroxide Fusion ICP						
CeO ₂ , Cerium(IV) oxide (ppm)	6082	256	5955	6209	5895	6269
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	59	2.6	57	60	57	60
Er ₂ O ₃ , Erbium(III) oxide (ppm)	14.0	0.64	13.6	14.3	13.4	14.5
Eu ₂ O ₃ , Europium(III) oxide (ppm)	91	3.3	89	93	89	94
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	190	8	186	195	185	196
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	7.44	0.527	7.15	7.73	7.09	7.79
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	4450	86	4416	4484	4336	4564
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.72	0.049	0.69	0.74	0.65	0.78
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2070	56	2033	2107	2008	2132
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	2985	149	2901	3070	2899	3072
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	888	41	868	909	865	911
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	428	10	423	432	416	440
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	16.9	0.63	16.6	17.2	16.4	17.4
ThO ₂ , Thorium dioxide (ppm)	283	10	278	289	276	291
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.37	0.118	1.32	1.43	1.26	1.49
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	7.25	0.314	7.11	7.39	6.84	7.66
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	168	8	164	173	164	173
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	6.28	0.411	6.08	6.47	5.97	6.58
ZrO ₂ , Zirconium dioxide (ppm)	801	36	779	823	769	834

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.



Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 462.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO ₂ , Cerium(IV) oxide (ppm)	6182	151.3	6054	6309	5988	6376
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	48.69	0.752	47.67	49.72	48.45	48.94
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	4517	91.8	4443	4592	4377	4657
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	3048	75.0	2979	3116	2944	3151
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	845	119	714	976	IND	IND
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	< 500	IND	IND	IND	IND	IND
Thermogravimetry						
LOI, Loss On Ignition @1000°C (wt.%)	0.771	0.120	0.610	0.932	0.737	0.805
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	5.79	0.195	5.65	5.93	5.68	5.91
Al ₂ O ₃ , Aluminium(III) oxide (wt.%)	10.95	0.369	10.68	11.21	10.73	11.16
Ba, Barium (ppm)	1025	52	996	1053	998	1052
BaO, Barium oxide (ppm)	1144	58	1112	1176	1114	1174
Be, Beryllium (ppm)	3.76	0.327	3.61	3.90	IND	IND
Bi, Bismuth (ppm)	2.58	0.42	2.13	3.04	IND	IND
Ca, Calcium (wt.%)	1.28	0.064	1.24	1.32	1.25	1.31
CaO, Calcium oxide (wt.%)	1.79	0.090	1.73	1.85	1.75	1.83
Ce, Cerium (ppm)	4951	208	4848	5054	4799	5104
CeO ₂ , Cerium(IV) oxide (ppm)	6082	256	5955	6209	5895	6269
Co, Cobalt (ppm)	12.5	2.0	10.4	14.5	11.7	13.2
Cr, Chromium (ppm)	609	31	591	627	592	626
Cr ₂ O ₃ , Chromium(III) oxide (ppm)	890	45	864	916	865	915
Cs, Cesium (ppm)	0.44	0.07	0.39	0.48	0.40	0.48
Dy, Dysprosium (ppm)	51	2.2	50	52	50	53
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	59	2.6	57	60	57	60
Er, Erbium (ppm)	12.2	0.56	11.9	12.5	11.8	12.7
Er ₂ O ₃ , Erbium(III) oxide (ppm)	14.0	0.64	13.6	14.3	13.4	14.5
Eu, Europium (ppm)	79	2.8	77	80	77	81
Eu ₂ O ₃ , Europium(III) oxide (ppm)	91	3.3	89	93	89	94
Fe, Iron (wt.%)	33.84	1.411	32.82	34.85	33.02	34.66
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	48.38	2.017	46.93	49.82	47.20	49.55
Ga, Gallium (ppm)	52	9	43	62	50	55
Gd, Gadolinium (ppm)	165	7	161	169	161	170
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	190	8	186	195	185	196
Hf, Hafnium (ppm)	14.0	0.78	13.7	14.3	13.3	14.6
HfO ₂ , Hafnium dioxide (ppm)	16.5	0.92	16.1	16.8	15.7	17.2
Ho, Holmium (ppm)	6.50	0.460	6.25	6.74	6.19	6.80
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	7.44	0.527	7.15	7.73	7.09	7.79
K, Potassium (wt.%)	0.120	0.017	0.106	0.134	IND	IND
K ₂ O, Potassium oxide (wt.%)	0.145	0.021	0.128	0.162	IND	IND

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

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

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)						
La, Lanthanum (ppm)	3794	73	3765	3824	3698	3891
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	4450	86	4416	4484	4336	4564
Li, Lithium (ppm)	11.3	0.84	10.3	12.3	IND	IND
Lu, Lutetium (ppm)	0.63	0.043	0.61	0.65	0.58	0.68
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.72	0.049	0.69	0.74	0.65	0.78
Mg, Magnesium (wt.%)	1.08	0.032	1.06	1.11	1.06	1.10
MgO, Magnesium oxide (wt.%)	1.80	0.053	1.76	1.84	1.76	1.83
Mn, Manganese (ppm)	933	68	881	985	913	952
MnO, Manganese oxide (ppm)	1204	87	1137	1271	1179	1230
Mo, Molybdenum (ppm)	51	3.3	49	54	49	54
Nb, Niobium (ppm)	1447	39	1422	1473	1404	1491
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2070	56	2033	2107	2008	2132
Nd, Neodymium (ppm)	2560	128	2487	2632	2486	2634
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	2985	149	2901	3070	2899	3072
Ni, Nickel (ppm)	66	9	59	74	52	81
NiO, Nickel oxide (ppm)	85	11	75	94	66	103
P, Phosphorus (wt.%)	0.492	0.010	0.486	0.499	0.479	0.506
P ₂ O ₅ , Phosphorus(V) oxide (wt.%)	1.13	0.024	1.11	1.14	1.10	1.16
Pb, Lead (ppm)	114	6	109	120	110	119
PbO, Lead oxide (ppm)	123	6	118	129	118	128
Pr, Praseodymium (ppm)	735	34	718	752	716	754
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	888	41	868	909	865	911
Rb, Rubidium (ppm)	6.58	0.377	6.40	6.75	6.26	6.90
S, Sulphur (ppm)	587	89	523	651	IND	IND
Sb, Antimony (ppm)	2.57	0.38	2.29	2.85	2.27	2.87
Si, Silicon (wt.%)	13.18	0.249	12.97	13.38	12.95	13.40
SiO ₂ , Silicon dioxide (wt.%)	28.19	0.532	27.74	28.63	27.70	28.67
Sm, Samarium (ppm)	369	9	365	373	358	379
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	428	10	423	432	416	440
Sn, Tin (ppm)	29.2	3.3	27.2	31.2	27.4	31.1
SnO ₂ , Tin dioxide (ppm)	37.1	4.2	34.6	39.6	34.8	39.4
Sr, Strontium (ppm)	765	22	754	775	742	787
SrO, Strontium oxide (ppm)	904	26	892	916	878	931
Ta, Tantalum (ppm)	25.7	1.65	24.6	26.7	24.1	27.2
Ta ₂ O ₅ , Tantalum(V) oxide (ppm)	31.3	2.02	30.1	32.6	29.4	33.3
Tb, Terbium (ppm)	14.4	0.54	14.1	14.7	14.0	14.8
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	16.9	0.63	16.6	17.2	16.4	17.4
Th, Thorium (ppm)	249	9	244	254	243	256
ThO ₂ , Thorium dioxide (ppm)	283	10	278	289	276	291
Ti, Titanium (wt.%)	1.91	0.074	1.86	1.96	1.86	1.95

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

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed).

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		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.%)	3.18	0.123	3.10	3.26	3.11	3.25
Tm, Thulium (ppm)	1.20	0.103	1.15	1.25	1.10	1.30
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.37	0.118	1.32	1.43	1.26	1.49
U, Uranium (ppm)	6.15	0.266	6.03	6.27	5.80	6.50
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	7.25	0.314	7.11	7.39	6.84	7.66
V, Vanadium (ppm)	380	18	372	389	364	396
V ₂ O ₅ , Vanadium(V) oxide (ppm)	679	32	664	694	650	708
W, Tungsten (ppm)	3.60	0.66	3.05	4.14	IND	IND
WO ₃ , Tungsten trioxide (ppm)	4.53	0.84	3.85	5.21	IND	IND
Y, Yttrium (ppm)	133	6	129	136	129	136
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	168	8	164	173	164	173
Yb, Ytterbium (ppm)	5.51	0.361	5.34	5.68	5.24	5.78
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	6.28	0.411	6.08	6.47	5.97	6.58
Zn, Zinc (ppm)	269	26	233	304	IND	IND
ZnO, Zinc oxide (ppm)	335	32	291	379	IND	IND
Zr, Zirconium (ppm)	593	27	577	610	569	617
ZrO ₂ , Zirconium dioxide (ppm)	801	36	779	823	769	834
4-Acid Digestion						
Ag, Silver (ppm)	< 2	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	5.71	0.132	5.63	5.79	5.60	5.81
As, Arsenic (ppm)	30.8	1.28	29.9	31.7	29.4	32.2
Ba, Barium (ppm)	1037	37.7	1014	1059	1016	1058
Be, Beryllium (ppm)	3.57	0.324	3.36	3.77	3.41	3.72
Bi, Bismuth (ppm)	2.69	0.173	2.59	2.79	2.57	2.82
Ca, Calcium (wt.%)	1.25	0.087	1.19	1.30	1.22	1.27
Cd, Cadmium (ppm)	< 0.2	IND	IND	IND	IND	IND
Ce, Cerium (ppm)	4984	180.4	4827	5142	4897	5071
Co, Cobalt (ppm)	13.0	0.87	12.5	13.6	12.7	13.4
Cr, Chromium (ppm)	502	82	445	558	486	517
Cs, Cesium (ppm)	0.45	0.06	0.41	0.49	0.43	0.48
Cu, Copper (ppm)	61	1.9	60	62	59	62
Dy, Dysprosium (ppm)	51	1.2	50	51	50	52
Er, Erbium (ppm)	10.8	0.42	10.6	11.1	10.5	11.2
Eu, Europium (ppm)	80	4.4	77	83	78	82
Fe, Iron (wt.%)	33.05	0.992	32.43	33.68	32.53	33.58
Ga, Gallium (ppm)	58	7	49	68	56	61
Gd, Gadolinium (ppm)	164	5.5	161	167	160	168
Hf, Hafnium (ppm)	7.40	0.83	6.79	8.01	7.00	7.80
Ho, Holmium (ppm)	6.18	0.323	5.98	6.38	5.99	6.37
In, Indium (ppm)	0.75	0.039	0.73	0.77	0.73	0.77

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

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

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
4-Acid Digestion continued						
K, Potassium (wt.%)	0.118	0.005	0.115	0.122	IND	IND
La, Lanthanum (ppm)	3669	193.5	3516	3822	3578	3760
Li, Lithium (ppm)	10.8	1.4	10.1	11.6	10.4	11.3
Lu, Lutetium (ppm)	0.53	0.10	0.47	0.59	0.50	0.56
Mg, Magnesium (wt.%)	1.01	0.12	0.93	1.08	0.99	1.03
Mn, Manganese (ppm)	840	63.3	790	890	820	860
Mo, Molybdenum (ppm)	53	3.5	51	56	52	55
Na, Sodium (wt.%)	0.178	0.012	0.171	0.185	IND	IND
Nd, Neodymium (ppm)	2595	125.6	2523	2668	2535	2656
Ni, Nickel (ppm)	74	3.1	72	76	71	77
P, Phosphorus (wt.%)	0.454	0.049	0.423	0.485	0.443	0.465
Pb, Lead (ppm)	119	5.3	115	122	116	121
Pr, Praseodymium (ppm)	736	29.8	720	752	720	752
Rb, Rubidium (ppm)	6.45	0.601	6.12	6.78	6.20	6.69
Re, Rhenium (ppm)	< 0.01	IND	IND	IND	IND	IND
S, Sulphur (ppm)	548	70	504	592	IND	IND
Sb, Antimony (ppm)	2.17	0.25	2.01	2.34	2.06	2.29
Sc, Scandium (ppm)	51	3.7	49	54	50	53
Sm, Samarium (ppm)	362	17.4	352	372	353	371
Sn, Tin (ppm)	23.9	1.37	22.9	24.9	23.0	24.8
Sr, Strontium (ppm)	738	58.5	701	774	727	748
Ta, Tantalum (ppm)	21.8	2.4	19.7	23.9	20.9	22.7
Tb, Terbium (ppm)	14.7	0.62	14.3	15.1	14.3	15.1
Te, Tellurium (ppm)	0.33	0.06	0.28	0.39	0.29	0.37
Th, Thorium (ppm)	246	7.9	241	250	237	254
Ti, Titanium (wt.%)	0.986	0.104	0.908	1.064	0.934	1.037
Tl, Thallium (ppm)	0.083	0.010	0.076	0.090	IND	IND
Tm, Thulium (ppm)	1.01	0.090	0.96	1.06	0.96	1.06
U, Uranium (ppm)	5.79	0.364	5.60	5.99	5.52	6.06
V, Vanadium (ppm)	353	26.0	334	372	347	358
W, Tungsten (ppm)	2.31	0.33	2.07	2.54	2.15	2.46
Y, Yttrium (ppm)	125	4.8	122	128	122	128
Yb, Ytterbium (ppm)	4.36	0.352	4.14	4.59	4.20	4.53
Zn, Zinc (ppm)	244	25	227	261	239	248
Zr, Zirconium (ppm)	270	31	249	291	260	281

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

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

Table 2. Indicative Values for OREAS 462.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al ₂ O ₃	wt. %	11.09	Lu ₂ O ₃	ppm	< 20	Tb ₄ O ₇	ppm	20.0
BaO	ppm	1250	MgO	wt. %	1.77	ThO ₂	ppm	298
CaO	wt. %	1.84	MnO	ppm	1233	TiO ₂	wt. %	3.19
Cr ₂ O ₃	ppm	618	Na ₂ O	wt. %	0.257	Tm ₂ O ₃	ppm	< 10
Dy ₂ O ₃	ppm	60	Nb ₂ O ₅	ppm	2078	U ₃ O ₈	ppm	< 100
Er ₂ O ₃	ppm	20.0	P ₂ O ₅	wt. %	1.15	V ₂ O ₅	ppm	745
Eu ₂ O ₃	ppm	83	SiO ₂	wt. %	28.52	WO ₃	ppm	< 100
Gd ₂ O ₃	ppm	188	SnO ₂	ppm	< 100	Y ₂ O ₃	ppm	112
HfO ₂	ppm	< 100	SO ₃	wt. %	0.139	Yb ₂ O ₃	ppm	10.0
Ho ₂ O ₃	ppm	10.0	SrO	ppm	733	ZrO ₂	ppm	717
K ₂ O	wt. %	0.149	Ta ₂ O ₅	ppm	< 100			
Thermogravimetry								
H ₂ O-	wt. %	0.667						
Borate / Peroxide Fusion ICP								
Ag	ppm	9.5	Ge	ppm	2.67	Se	ppm	< 20
As	ppm	99	In	ppm	0.78	Te	ppm	< 1
B	ppm	150	Na	wt. %	0.167	Tl	ppm	< 0.5
Cd	ppm	< 1	Re	ppm	< 0.1			
Cu	ppm	72	Sc	ppm	74			
4-Acid Digestion								
Ge	ppm	6.00	Nb	ppm	1231	Se	ppm	8.24

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

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

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.

SOURCE MATERIALS

OREAS 462 is an ore grade, rare earth element (TREO = 1.54%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 462 were sourced from Lynas Corporation's Mount Weld Project (the 'Central Lanthanide Deposit') which is located 35 kilometres south of Laverton in Western Australia. The Mount Weld source materials (waste, low and medium grade REE ores) were found to be highly hygroscopic to the extent that significant analytical errors would likely result during analysis unless strict moisture handling procedures were adhered. To avoid this complication, the hygroscopic property was destroyed by roasting the materials at 900°C for 2 hours. Following re-equilibration of the materials to laboratory atmosphere the hygroscopic moisture content was deemed acceptable (~0.5% H₂O-).

OREAS 462 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 113 certified values (and 49 indicative values) including REE's, majors and traces by fusion XRF, fusion ICP and 4-acid digestion.

The following summary of the mineralogy and supergene enrichment processes that operated in the host lateritic rocks is from Duncan and Willett (1990), Lottermoser (1990) and Lawrence (2006) as cited by S. Jaireth *et al* in 'Ore Geology Reviews 62 (2014) 72-128'.

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, Y, U, Th, Nb, Ta, Zr, Ti, V, Cr, Ba and Sr, 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%: CeO₂ (46.7%), La₂O₃ (25.5%), Nd₂O₃ (18.5%), Pr₆O₁₁ (5.32%), Sm₂O₃ (2.27%) and Eu₂O₃ (0.443%), together with minor components of HREE: Dy₂O₃ (0.124%) and Tb₄O₇ (0.068%).

COMMUNITION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 462 were prepared in the following manner:

- drying of materials to constant mass at 105°C;
- destruction of the hygroscopic property of the Mount Weld materials by roasting at 900°C for 2 hours;
- crushing and milling of materials to >99.5% minus 75 microns;
- preliminary homogenisation and check assaying of each material;
- blending in appropriate proportions to achieve the desired grades;
- packaging into 10g units sealed in laminated foil pouches and into 1kg units sealed in plastic jars.

ANALYTICAL PROGRAM

Twenty one commercial analytical laboratories participated in the program to certify the 113 elements reported in Table 1. The following methods were employed:

- REE Suite XRF package (up to 7 laboratories depending on the element);
- Thermogravimetry for Loss On Ignition (LOI) at 1000°C (7 laboratories);
- Borate/peroxide fusion for full elemental suite ICP-OES and ICP-MS (up to 15 laboratories depending on the element);

- 4-acid digestion (HF-HNO₃-HClO₄-HCl) for full elemental suite ICP-OES and ICP-MS finish (up to 14 laboratories depending on the element).

Samples for the round robin program were taken at nine predetermined sampling intervals immediately following final homogenisation and are considered representative of the entire batch of OREAS 462. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate sampling lots. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance. Table 1 presents the 113 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 49 indicative values. Table 3 provides performance gate intervals for the certified values of each method group based on their pooled 1SD's. Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM³) are presented in the detailed certification data for this CRM (**OREAS 462 Datapack.xlsx**).

STATISTICAL ANALYSIS

Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers. 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.

Certified Values are the means of accepted laboratory means after outlier filtering. Indicative (uncertified) values (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) inter-laboratory consensus is poor; or iii) a significant proportion of results are outlying.

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

Standard Deviation values (1SDs) are reported in Table 1 and 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. The SD values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

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

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

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

Table 3 shows **Performance Gates** calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned. A second method utilises a 5% window calculated directly from the certified value. Standard deviation is also shown in relative per cent 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.

Table 3. Performance Gates for OREAS 462.

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											
CeO ₂ , ppm	6182	151	5879	6484	5728	6636	2.45%	4.90%	7.34%	5873	6491
Fe ₂ O ₃ , wt. %	48.69	0.752	47.19	50.20	46.44	50.95	1.54%	3.09%	4.63%	46.26	51.13
La ₂ O ₃ , ppm	4517	92	4334	4701	4242	4793	2.03%	4.06%	6.10%	4292	4743
Nd ₂ O ₃ , ppm	3048	75	2898	3198	2823	3273	2.46%	4.92%	7.38%	2895	3200
Pr ₆ O ₁₁ , ppm	845	119	607	1084	488	1203	14.10%	28.21%	42.31%	803	888
Sm ₂ O ₃ , ppm	< 500	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Thermogravimetry											
LOI, wt. %	0.771	0.120	0.530	1.011	0.410	1.132	15.61%	31.22%	46.83%	0.732	0.809

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

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

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

Table 3 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 (majors and REE's shown in both oxide and elemental format)											
Al, wt.%	5.79	0.195	5.40	6.18	5.21	6.38	3.37%	6.74%	10.11%	5.50	6.08
Al ₂ O ₃ , wt.%	10.95	0.369	10.21	11.69	9.84	12.05	3.37%	6.74%	10.11%	10.40	11.50
Ba, ppm	1025	52	920	1129	868	1182	5.11%	10.21%	15.32%	974	1076
BaO, ppm	1144	58	1027	1261	969	1319	5.11%	10.21%	15.32%	1087	1201
Be, ppm	3.76	0.327	3.10	4.41	2.77	4.74	8.71%	17.42%	26.13%	3.57	3.94
Bi, ppm	2.58	0.42	1.74	3.43	1.32	3.85	16.28%	32.57%	48.85%	2.45	2.71
Ca, wt.%	1.28	0.064	1.15	1.41	1.09	1.47	5.01%	10.03%	15.04%	1.21	1.34
CaO, wt.%	1.79	0.090	1.61	1.97	1.52	2.06	5.01%	10.03%	15.04%	1.70	1.88
Ce, ppm	4951	208	4535	5367	4327	5575	4.20%	8.40%	12.60%	4704	5199
CeO ₂ , ppm	6082	256	5571	6593	5315	6849	4.20%	8.40%	12.60%	5778	6386
Co, ppm	12.5	2.0	8.4	16.5	6.4	18.5	16.21%	32.43%	48.64%	11.8	13.1
Cr, ppm	609	31	547	671	515	702	5.10%	10.21%	15.31%	578	639
Cr ₂ O ₃ , ppm	890	45	799	980	753	1026	5.10%	10.21%	15.31%	845	934
Cs, ppm	0.44	0.07	0.30	0.57	0.24	0.64	15.17%	30.35%	45.52%	0.42	0.46
Dy, ppm	51	2.2	47	56	44	58	4.39%	8.78%	13.17%	49	54
Dy ₂ O ₃ , ppm	59	2.6	54	64	51	67	4.39%	8.78%	13.17%	56	62
Er, ppm	12.2	0.56	11.1	13.3	10.5	13.9	4.58%	9.16%	13.74%	11.6	12.8
Er ₂ O ₃ , ppm	14.0	0.64	12.7	15.3	12.1	15.9	4.58%	9.16%	13.74%	13.3	14.7
Eu, ppm	79	2.8	73	84	70	87	3.59%	7.17%	10.76%	75	83
Eu ₂ O ₃ , ppm	91	3.3	85	98	81	101	3.59%	7.17%	10.76%	87	96
Fe, wt.%	33.84	1.411	31.01	36.66	29.60	38.07	4.17%	8.34%	12.51%	32.14	35.53
Fe ₂ O ₃ , wt.%	48.38	2.017	44.34	52.41	42.32	54.43	4.17%	8.34%	12.51%	45.96	50.79
Ga, ppm	52	9	35	69	27	78	16.38%	32.76%	49.15%	50	55
Gd, ppm	165	7	151	180	143	187	4.44%	8.88%	13.31%	157	173
Gd ₂ O ₃ , ppm	190	8	174	207	165	216	4.44%	8.88%	13.31%	181	200
Hf, ppm	14.0	0.78	12.4	15.5	11.6	16.3	5.62%	11.23%	16.85%	13.3	14.7
HfO ₂ , ppm	16.5	0.92	14.6	18.3	13.7	19.2	5.62%	11.23%	16.85%	15.6	17.3
Ho, ppm	6.50	0.460	5.57	7.42	5.11	7.88	7.09%	14.18%	21.26%	6.17	6.82
Ho ₂ O ₃ , ppm	7.44	0.527	6.39	8.49	5.86	9.02	7.09%	14.18%	21.26%	7.07	7.81
K, wt.%	0.120	0.017	0.086	0.154	0.069	0.172	14.24%	28.48%	42.72%	0.114	0.126
K ₂ O, wt.%	0.145	0.021	0.104	0.186	0.083	0.207	14.24%	28.48%	42.72%	0.138	0.152
La, ppm	3794	73	3648	3941	3574	4015	1.93%	3.87%	5.80%	3605	3984
La ₂ O ₃ , ppm	4450	86	4278	4622	4192	4708	1.93%	3.87%	5.80%	4228	4673
Li, ppm	11.3	0.84	9.6	13.0	8.8	13.8	7.46%	14.92%	22.38%	10.7	11.9
Lu, ppm	0.63	0.043	0.54	0.72	0.50	0.76	6.87%	13.74%	20.62%	0.60	0.66
Lu ₂ O ₃ , ppm	0.72	0.049	0.62	0.81	0.57	0.86	6.87%	13.74%	20.62%	0.68	0.75
Mg, wt.%	1.08	0.032	1.02	1.15	0.99	1.18	2.94%	5.88%	8.83%	1.03	1.14
MgO, wt.%	1.80	0.053	1.69	1.90	1.64	1.95	2.94%	5.88%	8.83%	1.71	1.89
Mn, ppm	933	68	798	1068	730	1135	7.24%	14.48%	21.72%	886	979
MnO, ppm	1204	87	1030	1379	943	1466	7.24%	14.48%	21.72%	1144	1265
Mo, ppm	51	3.3	45	58	42	61	6.32%	12.64%	18.96%	49	54

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 3 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 (majors and REE's shown in both oxide and elemental format)											
Nb, ppm	1447	39	1369	1525	1330	1564	2.69%	5.39%	8.08%	1375	1520
Nb ₂ O ₅ , ppm	2070	56	1959	2182	1903	2238	2.69%	5.39%	8.08%	1967	2174
Nd, ppm	2560	128	2304	2815	2176	2943	5.00%	9.99%	14.99%	2432	2688
Nd ₂ O ₃ , ppm	2985	149	2687	3284	2538	3433	5.00%	9.99%	14.99%	2836	3135
Ni, ppm	66	9	48	84	39	93	13.54%	27.08%	40.62%	63	70
NiO, ppm	85	11	62	107	50	119	13.54%	27.08%	40.62%	80	89
P, wt. %	0.492	0.010	0.472	0.513	0.461	0.524	2.12%	4.24%	6.36%	0.468	0.517
P ₂ O ₅ , wt. %	1.13	0.024	1.08	1.18	1.06	1.20	2.12%	4.24%	6.36%	1.07	1.18
Pb, ppm	114	6	103	126	97	131	4.95%	9.89%	14.84%	109	120
PbO, ppm	123	6	111	135	105	141	4.95%	9.89%	14.84%	117	129
Pr, ppm	735	34	668	803	634	836	4.59%	9.18%	13.77%	698	772
Pr ₆ O ₁₁ , ppm	888	41	807	970	766	1011	4.59%	9.18%	13.77%	844	933
Rb, ppm	6.58	0.377	5.82	7.33	5.45	7.71	5.73%	11.45%	17.18%	6.25	6.91
S, ppm	587	89	410	764	321	853	15.09%	30.17%	45.26%	558	616
Sb, ppm	2.57	0.38	1.81	3.33	1.42	3.71	14.86%	29.72%	44.57%	2.44	2.70
Si, wt. %	13.18	0.249	12.68	13.67	12.43	13.92	1.89%	3.78%	5.66%	12.52	13.84
SiO ₂ , wt. %	28.19	0.532	27.12	29.25	26.59	29.78	1.89%	3.78%	5.66%	26.78	29.60
Sm, ppm	369	9	351	387	342	396	2.41%	4.82%	7.23%	350	387
Sm ₂ O ₃ , ppm	428	10	407	448	397	459	2.41%	4.82%	7.23%	406	449
Sn, ppm	29.2	3.3	22.6	35.8	19.3	39.1	11.31%	22.62%	33.93%	27.8	30.7
SnO ₂ , ppm	37.1	4.2	28.7	45.5	24.5	49.7	11.31%	22.62%	33.93%	35.2	38.9
Sr, ppm	765	22	722	808	700	829	2.82%	5.64%	8.46%	726	803
SrO, ppm	904	26	853	955	828	981	2.82%	5.64%	8.46%	859	949
Ta, ppm	25.7	1.65	22.4	29.0	20.7	30.6	6.43%	12.86%	19.29%	24.4	26.9
Ta ₂ O ₅ , ppm	31.3	2.02	27.3	35.4	25.3	37.4	6.43%	12.86%	19.29%	29.8	32.9
Tb, ppm	14.4	0.54	13.3	15.5	12.8	16.0	3.74%	7.47%	11.21%	13.7	15.1
Tb ₄ O ₇ , ppm	16.9	0.63	15.7	18.2	15.0	18.8	3.74%	7.47%	11.21%	16.1	17.8
Th, ppm	249	9	231	267	222	276	3.58%	7.15%	10.73%	237	262
ThO ₂ , ppm	283	10	263	304	253	314	3.58%	7.15%	10.73%	269	298
Ti, wt. %	1.91	0.074	1.76	2.06	1.69	2.13	3.88%	7.75%	11.63%	1.81	2.00
TiO ₂ , wt. %	3.18	0.123	2.94	3.43	2.81	3.55	3.88%	7.75%	11.63%	3.02	3.34
Tm, ppm	1.20	0.103	1.00	1.41	0.89	1.51	8.57%	17.14%	25.71%	1.14	1.26
Tm ₂ O ₃ , ppm	1.37	0.118	1.14	1.61	1.02	1.73	8.57%	17.14%	25.71%	1.31	1.44
U, ppm	6.15	0.266	5.61	6.68	5.35	6.95	4.33%	8.67%	13.00%	5.84	6.45
U ₃ O ₈ , ppm	7.25	0.314	6.62	7.88	6.31	8.19	4.33%	8.67%	13.00%	6.89	7.61
V, ppm	380	18	344	416	326	434	4.75%	9.49%	14.24%	361	399
V ₂ O ₅ , ppm	679	32	614	743	582	775	4.75%	9.49%	14.24%	645	713
W, ppm	3.60	0.66	2.27	4.92	1.60	5.59	18.49%	36.98%	55.47%	3.42	3.78
WO ₃ , ppm	4.53	0.84	2.86	6.21	2.02	7.05	18.49%	36.98%	55.47%	4.31	4.76
Y, ppm	133	6	120	145	113	152	4.84%	9.67%	14.51%	126	139
Y ₂ O ₃ , ppm	168	8	152	185	144	193	4.84%	9.67%	14.51%	160	177

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 3 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 (majors and REE's shown in both oxide and elemental format)											
Yb, ppm	5.51	0.361	4.79	6.23	4.43	6.60	6.55%	13.10%	19.65%	5.24	5.79
Yb ₂ O ₃ , ppm	6.28	0.411	5.45	7.10	5.04	7.51	6.55%	13.10%	19.65%	5.96	6.59
Zn, ppm	269	26	217	321	191	347	9.65%	19.31%	28.96%	255	282
ZnO, ppm	335	32	270	399	238	432	9.65%	19.31%	28.96%	318	351
Zr, ppm	593	27	540	647	513	674	4.53%	9.06%	13.59%	564	623
ZrO ₂ , ppm	801	36	729	874	693	910	4.53%	9.06%	13.59%	761	842
4-Acid Digestion											
Ag, ppm	< 2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt. %	5.71	0.132	5.44	5.97	5.31	6.10	2.31%	4.62%	6.92%	5.42	5.99
As, ppm	30.8	1.28	28.2	33.3	26.9	34.6	4.15%	8.31%	12.46%	29.2	32.3
Ba, ppm	1037	38	961	1112	924	1150	3.63%	7.26%	10.90%	985	1089
Be, ppm	3.57	0.324	2.92	4.22	2.59	4.54	9.10%	18.19%	27.29%	3.39	3.75
Bi, ppm	2.69	0.173	2.35	3.04	2.18	3.21	6.41%	12.81%	19.22%	2.56	2.83
Ca, wt. %	1.25	0.087	1.07	1.42	0.99	1.51	6.96%	13.91%	20.87%	1.19	1.31
Cd, ppm	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	4984	180	4624	5345	4443	5525	3.62%	7.24%	10.86%	4735	5234
Co, ppm	13.0	0.87	11.3	14.8	10.4	15.6	6.71%	13.42%	20.13%	12.4	13.7
Cr, ppm	502	82	338	665	257	746	16.27%	32.54%	48.81%	476	527
Cs, ppm	0.45	0.06	0.32	0.58	0.26	0.64	14.11%	28.22%	42.33%	0.43	0.47
Cu, ppm	61	1.9	57	65	55	67	3.19%	6.38%	9.57%	58	64
Dy, ppm	51	1.2	48	53	47	54	2.40%	4.79%	7.19%	48	53
Er, ppm	10.8	0.42	10.0	11.7	9.6	12.1	3.86%	7.73%	11.59%	10.3	11.4
Eu, ppm	80	4.4	71	89	67	93	5.51%	11.03%	16.54%	76	84
Fe, wt. %	33.05	0.992	31.07	35.04	30.08	36.03	3.00%	6.00%	9.01%	31.40	34.71
Ga, ppm	58	7	44	73	37	80	12.16%	24.32%	36.47%	55	61
Gd, ppm	164	6	153	175	147	181	3.38%	6.76%	10.14%	156	172
Hf, ppm	7.40	0.83	5.73	9.06	4.90	9.89	11.25%	22.50%	33.75%	7.03	7.77
Ho, ppm	6.18	0.323	5.53	6.83	5.21	7.15	5.23%	10.47%	15.70%	5.87	6.49
In, ppm	0.75	0.039	0.67	0.83	0.63	0.87	5.19%	10.38%	15.58%	0.71	0.79
Fe, wt. %	33.05	0.992	31.07	35.04	30.08	36.03	3.00%	6.00%	9.01%	31.40	34.71
Ga, ppm	58	7	44	73	37	80	12.16%	24.32%	36.47%	55	61
Gd, ppm	164	6	153	175	147	181	3.38%	6.76%	10.14%	156	172
Hf, ppm	7.40	0.83	5.73	9.06	4.90	9.89	11.25%	22.50%	33.75%	7.03	7.77
Ho, ppm	6.18	0.323	5.53	6.83	5.21	7.15	5.23%	10.47%	15.70%	5.87	6.49
In, ppm	0.75	0.039	0.67	0.83	0.63	0.87	5.19%	10.38%	15.58%	0.71	0.79
K, wt. %	0.118	0.005	0.108	0.129	0.102	0.135	4.55%	9.10%	13.64%	0.113	0.124
La, ppm	3669	193	3282	4056	3089	4249	5.27%	10.55%	15.82%	3486	3852
Li, ppm	10.8	1.4	8.0	13.6	6.6	15.0	12.92%	25.85%	38.77%	10.3	11.4
Lu, ppm	0.53	0.10	0.34	0.72	0.24	0.82	18.26%	36.53%	54.79%	0.50	0.56
Mg, wt. %	1.01	0.12	0.77	1.24	0.65	1.36	11.81%	23.62%	35.43%	0.96	1.06
Mn, ppm	840	63	713	967	650	1030	7.54%	15.08%	22.62%	798	882

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

Note 1: intervals may appear asymmetric due to rounding.

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

Table 3 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											
Mo, ppm	53	3.5	46	60	43	64	6.56%	13.12%	19.69%	51	56
Na, wt. %	0.178	0.012	0.154	0.202	0.141	0.214	6.82%	13.64%	20.46%	0.169	0.187
Nd, ppm	2595	126	2344	2847	2218	2972	4.84%	9.68%	14.52%	2466	2725
Ni, ppm	74	3.1	68	80	64	83	4.22%	8.44%	12.66%	70	78
P, wt. %	0.454	0.049	0.356	0.552	0.307	0.601	10.80%	21.60%	32.40%	0.431	0.476
Pb, ppm	119	5	108	129	103	134	4.49%	8.99%	13.48%	113	124
Pr, ppm	736	30	676	796	646	826	4.06%	8.11%	12.17%	699	773
Rb, ppm	6.45	0.601	5.24	7.65	4.64	8.25	9.32%	18.65%	27.97%	6.12	6.77
Re, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, ppm	548	70	408	688	337	759	12.81%	25.62%	38.43%	521	575
Sb, ppm	2.17	0.25	1.67	2.68	1.42	2.93	11.58%	23.17%	34.75%	2.06	2.28
Sc, ppm	51	3.7	44	59	40	63	7.26%	14.52%	21.78%	49	54
Sm, ppm	362	17	327	397	310	414	4.80%	9.61%	14.41%	344	380
Sn, ppm	23.9	1.37	21.1	26.6	19.8	28.0	5.76%	11.51%	17.27%	22.7	25.1
Sr, ppm	738	59	620	855	562	913	7.93%	15.87%	23.80%	701	774
Ta, ppm	21.8	2.4	17.1	26.6	14.7	28.9	10.90%	21.81%	32.71%	20.7	22.9
Tb, ppm	14.7	0.62	13.5	15.9	12.8	16.6	4.22%	8.44%	12.66%	14.0	15.4
Te, ppm	0.33	0.06	0.21	0.46	0.15	0.52	18.31%	36.63%	54.94%	0.32	0.35
Th, ppm	246	8	230	261	222	269	3.22%	6.44%	9.66%	233	258
Ti, wt. %	0.986	0.104	0.777	1.195	0.673	1.299	10.58%	21.16%	31.74%	0.937	1.035
Tl, ppm	0.083	0.010	0.063	0.103	0.053	0.113	12.09%	24.18%	36.27%	0.079	0.087
Tm, ppm	1.01	0.090	0.83	1.19	0.74	1.28	8.90%	17.79%	26.69%	0.96	1.06
U, ppm	5.79	0.364	5.06	6.52	4.70	6.88	6.29%	12.57%	18.86%	5.50	6.08
V, ppm	353	26	301	405	274	431	7.39%	14.77%	22.16%	335	370
W, ppm	2.31	0.33	1.64	2.97	1.31	3.30	14.36%	28.72%	43.07%	2.19	2.42
Y, ppm	125	5	116	135	111	140	3.82%	7.64%	11.46%	119	132
Yb, ppm	4.36	0.352	3.66	5.07	3.31	5.42	8.08%	16.15%	24.23%	4.15	4.58
Zn, ppm	244	25	194	293	169	318	10.16%	20.33%	30.49%	231	256
Zr, ppm	270	31	208	332	178	363	11.44%	22.88%	34.32%	257	284

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

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

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

Tolerance Limits (ISO Guide 3207) were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for La_2O_3 by fusion ICP, where 99% of the time ($1-\alpha=0.99$) at least 95% of subsamples ($p=0.95$) will have concentrations lying between 4336 and 4564 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

The homogeneity of OREAS 462 has also been evaluated in an ANOVA study for all certified analytes. This study tests the null hypothesis that no statistically significant difference exists between the *between-unit variance* and the *within-unit variance* (i.e. p-values <0.05 indicate rejection of the null hypothesis). Of the 113 certified values, no failures were observed indicating no evidence to reject the null hypothesis.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 462 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

1. ALS, Brisbane, QLD, Australia
2. ALS, Lima, Peru
3. ALS, Loughrea, Galway, Ireland
4. ALS, Perth, WA, Australia
5. ALS, Vancouver, BC, Canada
6. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
7. Bureau Veritas Geoanalytical, Perth, WA, Australia
8. Intertek Genalysis, Adelaide, SA, Australia
9. Intertek Genalysis, Perth, WA, Australia
10. Intertek Testing Services, Cupang, Muntinlupa, Philippines
11. Intertek Testing Services, Shunyi, Beijing, China
12. Nagrom, Perth, WA, Australia
13. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
14. SGS Australia Mineral Services, Perth (Newburn), WA, Australia
15. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
16. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
17. SGS Mineral Services, Townsville, QLD, Australia
18. SGS South Africa Pty Ltd, Booyens, Gauteng, South Africa
19. SGS Vostok Limited, Chita, Russian Federation
20. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
21. UIS Analytical Services, Centurion, South Africa

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 462 has been prepared, certified and is supplied by:

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It is available in unit sizes of 10g in laminated foil pouches or 1kg in plastic jars.

INTENDED USE

OREAS 462 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological samples;
- for the verification of analytical methods for analytes reported in Table 1;
- for the calibration of instruments used in the determination of the concentration of analytes reported in Table 1.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 462 has been prepared from ore grade/waste REE bearing ore (TREO = 1.54%). The source materials (waste, low and medium grade REE ores) were found to be highly hygroscopic and this property was destroyed by roasting the materials at 900°C for 2 hours. Following re-equilibration of the materials to laboratory atmosphere the hygroscopic moisture content was deemed acceptable (~0.5% H₂O-).

OREAS 462 has been packaged in single-use, 10g units in laminated foil pouches and 1kg units in plastic jars. In its unopened state and under normal conditions of storage the CRM has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

INSTRUCTIONS FOR CORRECT USE

The certified values derived by 4-acid digestion and by fusion with ICP-OES/MS refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis.

In contrast the certified values derived by lithium borate fusion XRF and for LOI at 1000°C are on a dry sample basis. This is standard laboratory protocol for fusion XRF determinations and requires the removal of hygroscopic moisture by drying in air to constant mass at 105°C. If the reference material is not dried prior to analysis, the certified values should be corrected to the moisture-bearing basis.

TRACEABILITY

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis. The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs) for a particular analytical method, analyte, or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified and non-certified (indicative) values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

HANDLING INSTRUCTIONS

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

LEGAL NOTICE

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

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DOCUMENT HISTORY

Revision No.	Date	Changes applied
2	20 th February, 2024	Added missing Certified Values to Table 1 (V ₂ O ₅ , W, WO ₃ and Y).
1	28 th April, 2015	Revised 'Borate / Peroxide Fusion ICP' Certified Values and associated statistics due to the omission of data from one laboratory (borate fusion with ICP-OES data added).
0	30 th March, 2015	First publication.

QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2008 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.



CERTIFYING OFFICER

A handwritten signature in blue ink, appearing to read 'Craig Hamlyn'.

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

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