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CERTIFICATE OF ANALYSIS FOR

CARBONATITE SUPERGENE REE-Nb ORE (TREO ~0.53%) CERTIFIED REFERENCE MATERIAL

OREAS 460

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)	2209	89	2155	2263	2153	2265
Dy_2O_3, Dysprosium(III) oxide (ppm)	22.8	0.86	22.3	23.3	22.0	23.6
Er_2O_3, Erbium(III) oxide (ppm)	6.88	0.400	6.67	7.08	6.60	7.15
Eu_2O_3, Europium(III) oxide (ppm)	26.3	1.12	25.7	27.0	25.7	27.0
Gd_2O_3, Gadolinium(III) oxide (ppm)	58	3.4	56	60	56	60
Ho_2O_3, Holmium(III) oxide (ppm)	3.18	0.251	3.03	3.32	3.07	3.28
La_2O_3, Lanthanum(III) oxide (ppm)	1606	88	1556	1656	1576	1635
Lu_2O_3, Lutetium(III) oxide (ppm)	0.59	0.055	0.56	0.62	0.56	0.63
Nb_2O_5, Niobium(V) oxide (ppm)	998	56	962	1034	974	1021
Nd_2O_3, Neodymium(III) oxide (ppm)	911	54	879	943	896	927
Pr_6O_{11}, Praseodymium(III,IV) oxide (ppm)	294	10	288	301	286	303
Sm_2O_3, Samarium(III) oxide (ppm)	125	4	122	127	122	127
Tb_4O_7, Terbium(III,IV) oxide (ppm)	5.70	0.250	5.56	5.83	5.49	5.90
ThO_2, Thorium dioxide (ppm)	132	4	130	134	130	134
Tm_2O_3, Thulium(III) oxide (ppm)	0.80	0.062	0.77	0.83	0.75	0.85
U_3O_8, Uranium(V,VI) oxide (ppm)	4.97	0.227	4.85	5.09	4.81	5.13
Y_2O_3, Yttrium(III) oxide (ppm)	76	3.3	74	78	74	78
Yb_2O_3, Ytterbium(III) oxide (ppm)	4.45	0.299	4.28	4.61	4.27	4.63
ZrO_2, Zirconium dioxide (ppm)	638	29	619	658	612	664

Note: intervals may appear asymmetric due to rounding.

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 460.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO_2, Cerium(IV) oxide (ppm)	2213	87.6	2161	2266	2048	2379
Fe_2O_3, Iron(III) oxide (wt.%)	26.81	0.383	26.32	27.30	26.63	26.99
La_2O_3, Lanthanum(III) oxide (ppm)	1626	62.2	1583	1669	IND	IND
Nd_2O_3, Neodymium(III) oxide (ppm)	958	66.0	903	1013	IND	IND
Pr_6O_{11}, Praseodymium(III,IV) oxide (ppm)	289	63	226	353	IND	IND
Thermogravimetry						
LOI, Loss On Ignition @ 1000°C (wt.%)	2.54	0.209	2.32	2.77	2.47	2.61
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	6.74	0.135	6.65	6.82	6.64	6.83
Al_2O_3, Aluminium(III) oxide (wt.%)	12.73	0.255	12.57	12.88	12.54	12.91
Ba, Barium (ppm)	808	53	779	837	795	821
BaO, Barium oxide (ppm)	902	59	870	934	888	917
Be, Beryllium (ppm)	2.48	0.42	2.10	2.86	IND	IND
Bi, Bismuth (ppm)	1.48	0.33	1.21	1.76	IND	IND
Ca, Calcium (wt.%)	0.697	0.024	0.680	0.713	0.679	0.715
CaO, Calcium oxide (wt.%)	0.975	0.034	0.952	0.998	0.950	1.000
Ce, Cerium (ppm)	1798	72	1754	1842	1752	1844
CeO_2, Cerium(IV) oxide (ppm)	2209	89	2155	2263	2153	2265
Co, Cobalt (ppm)	9.44	1.48	7.78	11.09	9.03	9.84
Cr, Chromium (ppm)	393	21	380	407	382	404
Cr_2O_3, Chromium(III) oxide (ppm)	575	30	555	594	559	590
Cs, Cesium (ppm)	3.68	0.246	3.57	3.78	3.51	3.85
Dy, Dysprosium (ppm)	19.8	0.75	19.4	20.3	19.1	20.5
Dy_2O_3, Dysprosium(III) oxide (ppm)	22.8	0.86	22.3	23.3	22.0	23.6
Er, Erbium (ppm)	6.01	0.350	5.83	6.19	5.78	6.25
Er_2O_3, Erbium(III) oxide (ppm)	6.88	0.400	6.67	7.08	6.60	7.15
Eu, Europium (ppm)	22.7	0.96	22.2	23.3	22.2	23.3
Eu_2O_3, Europium(III) oxide (ppm)	26.3	1.12	25.7	27.0	25.7	27.0
Fe, Iron (wt.%)	18.90	0.628	18.49	19.32	18.53	19.27
Fe_2O_3, Iron(III) oxide (wt.%)	27.02	0.898	26.43	27.62	26.50	27.55
Gd, Gadolinium (ppm)	50	3.0	48	52	48	52
Gd_2O_3, Gadolinium(III) oxide (ppm)	58	3.4	56	60	56	60
Hf, Hafnium (ppm)	11.8	0.56	11.5	12.1	11.2	12.4
HfO_2, Hafnium dioxide (ppm)	13.9	0.66	13.6	14.3	13.2	14.6
Ho, Holmium (ppm)	2.77	0.219	2.65	2.90	2.68	2.86
Ho_2O_3, Holmium(III) oxide (ppm)	3.18	0.251	3.03	3.32	3.07	3.28
K, Potassium (wt.%)	1.23	0.086	1.17	1.29	1.20	1.26
K_2O, Potassium oxide (wt.%)	1.48	0.103	1.41	1.55	1.45	1.52
La, Lanthanum (ppm)	1369	75	1327	1412	1344	1394
La_2O_3, Lanthanum(III) oxide (ppm)	1606	88	1556	1656	1576	1635
Li, Lithium (ppm)	19.6	0.85	19.0	20.1	IND	IND
Lu, Lutetium (ppm)	0.52	0.049	0.50	0.55	0.49	0.55

Note: intervals may appear asymmetric due to rounding.

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)						
Lu_2O_3, Lutetium(III) oxide (ppm)	0.59	0.055	0.56	0.62	0.56	0.63
Mg, Magnesium (wt.%)	0.751	0.033	0.730	0.773	0.735	0.768
MgO, Magnesium oxide (wt.%)	1.25	0.055	1.21	1.28	1.22	1.27
Mn, Manganese (ppm)	361	30	335	387	350	372
MnO, Manganese oxide (ppm)	466	39	432	500	452	480
Mo, Molybdenum (ppm)	25.2	3.7	21.9	28.4	23.8	26.6
Nb, Niobium (ppm)	698	39	673	722	681	714
Nb_2O_5, Niobium(V) oxide (ppm)	998	56	962	1034	974	1021
Nd, Neodymium (ppm)	781	47	754	809	768	795
Nd_2O_3, Neodymium(III) oxide (ppm)	911	54	879	943	896	927
Ni, Nickel (ppm)	53	4.9	51	55	46	60
NiO, Nickel oxide (ppm)	68	6.3	65	71	59	77
P, Phosphorus (wt.%)	0.198	0.014	0.190	0.207	0.181	0.215
P_2O_5, Phosphorus(V) oxide (wt.%)	0.455	0.033	0.435	0.474	0.415	0.494
Pb, Lead (ppm)	67	5.4	64	70	58	77
PbO, Lead oxide (ppm)	72	5.8	69	75	62	83
Pr, Praseodymium (ppm)	244	8	239	249	237	251
Pr_6O_{11}, Praseodymium(III,IV) oxide (ppm)	294	10	288	301	286	303
Rb, Rubidium (ppm)	75	4.1	73	78	74	77
Sb, Antimony (ppm)	3.71	0.39	3.20	4.22	3.33	4.09
Si, Silicon (wt.%)	23.28	0.292	23.08	23.48	22.93	23.63
SiO_2, Silicon dioxide (wt.%)	49.80	0.624	49.37	50.24	49.05	50.55
Sm, Samarium (ppm)	107	3	106	109	105	110
Sm_2O_3, Samarium(III) oxide (ppm)	125	4	122	127	122	127
Sn, Tin (ppm)	16.0	2.1	14.7	17.4	IND	IND
SnO_2, Tin dioxide (ppm)	20.4	2.7	18.7	22.1	IND	IND
Sr, Strontium (ppm)	305	12	298	311	298	312
SrO, Strontium oxide (ppm)	360	14	353	368	352	369
Ta, Tantalum (ppm)	13.7	0.64	13.4	14.1	12.8	14.7
Ta_2O_5, Tantalum(V) oxide (ppm)	16.8	0.78	16.3	17.2	15.6	18.0
Tb, Terbium (ppm)	4.84	0.212	4.73	4.96	4.67	5.01
Tb_4O_7, Terbium(III,IV) oxide (ppm)	5.70	0.250	5.56	5.83	5.49	5.90
Th, Thorium (ppm)	116	3	114	118	114	118
ThO_2, Thorium dioxide (ppm)	132	4	130	134	130	134
Ti, Titanium (wt.%)	1.20	0.028	1.18	1.22	1.18	1.22
TiO_2, Titanium dioxide (wt.%)	2.00	0.046	1.97	2.03	1.97	2.04
Tm, Thulium (ppm)	0.70	0.054	0.68	0.73	0.66	0.75
Tm_2O_3, Thulium(III) oxide (ppm)	0.80	0.062	0.77	0.83	0.75	0.85
U, Uranium (ppm)	4.21	0.193	4.11	4.31	4.08	4.35
U_3O_8, Uranium(V,VI) oxide (ppm)	4.97	0.227	4.85	5.09	4.81	5.13
V, Vanadium (ppm)	255	13	248	263	246	264

Note: intervals may appear asymmetric due to rounding.

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)						
V_2O_5, Vanadium(V) oxide (ppm)	456	22	442	469	439	472
W, Tungsten (ppm)	< 6	IND	IND	IND	IND	IND
WO_3, Tungsten trioxide (ppm)	< 8	IND	IND	IND	IND	IND
Y, Yttrium (ppm)	60	2.6	58	61	58	61
Y_2O_3, Yttrium(III) oxide (ppm)	76	3.3	74	78	74	78
Yb, Ytterbium (ppm)	3.91	0.262	3.76	4.05	3.75	4.07
Yb_2O_3, Ytterbium(III) oxide (ppm)	4.45	0.299	4.28	4.61	4.27	4.63
Zn, Zinc (ppm)	121	23	93	148	IND	IND
ZnO, Zinc oxide (ppm)	150	29	116	184	IND	IND
Zr, Zirconium (ppm)	472	21	458	487	453	492
ZrO_2, Zirconium dioxide (ppm)	638	29	619	658	612	664
4-Acid Digestion						
Ag, Silver (ppm)	< 1	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	6.55	0.230	6.42	6.69	6.47	6.64
As, Arsenic (ppm)	53	3.4	51	56	52	55
Ba, Barium (ppm)	815	23.3	800	829	803	827
Be, Beryllium (ppm)	2.40	0.109	2.35	2.45	2.25	2.55
Bi, Bismuth (ppm)	1.44	0.077	1.40	1.49	1.36	1.53
Ca, Calcium (wt.%)	0.701	0.035	0.681	0.721	0.689	0.713
Cd, Cadmium (ppm)	< 0.1	IND	IND	IND	IND	IND
Ce, Cerium (ppm)	1853	132.8	1738	1969	1812	1895
Co, Cobalt (ppm)	10.0	0.55	9.7	10.4	9.6	10.4
Cr, Chromium (ppm)	347	28.2	327	367	335	359
Cs, Cesium (ppm)	3.78	0.273	3.62	3.94	3.65	3.91
Cu, Copper (ppm)	41.7	2.00	40.5	43.0	39.5	44.0
Dy, Dysprosium (ppm)	18.0	0.44	17.8	18.3	17.7	18.4
Er, Erbium (ppm)	4.65	0.216	4.51	4.80	4.51	4.79
Eu, Europium (ppm)	23.1	1.31	22.2	24.0	22.3	23.8
Fe, Iron (wt.%)	18.56	0.431	18.30	18.83	18.25	18.87
Ga, Gallium (ppm)	33.0	4.3	28.9	37.2	31.8	34.3
Gd, Gadolinium (ppm)	48.2	2.46	46.4	49.9	47.1	49.2
Hf, Hafnium (ppm)	6.09	0.580	5.67	6.50	5.85	6.32
Ho, Holmium (ppm)	2.37	0.133	2.28	2.45	2.28	2.45
In, Indium (ppm)	0.31	0.017	0.30	0.32	0.30	0.32
K, Potassium (wt.%)	1.25	0.035	1.22	1.27	1.22	1.27
La, Lanthanum (ppm)	1298	113.2	1228	1368	1268	1328
Li, Lithium (ppm)	18.3	1.48	17.5	19.2	17.6	19.1
Lu, Lutetium (ppm)	0.36	0.05	0.33	0.38	0.34	0.37
Mg, Magnesium (wt.%)	0.724	0.058	0.690	0.759	0.713	0.736
Mn, Manganese (ppm)	307	29.2	288	326	300	315
Mo, Molybdenum (ppm)	25.4	0.73	25.0	25.8	24.8	26.1
Na, Sodium (wt.%)	0.133	0.006	0.130	0.137	IND	IND

Note: intervals may appear asymmetric due to rounding.

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
4-Acid Digestion continued						
Nd, Neodymium (ppm)	792	26.5	775	809	770	813
Ni, Nickel (ppm)	62	4.0	59	64	58	65
P, Phosphorus (wt.%)	0.193	0.009	0.187	0.198	0.188	0.198
Pb, Lead (ppm)	65	2.4	63	67	63	67
Pr, Praseodymium (ppm)	240	11.2	233	246	232	248
Rb, Rubidium (ppm)	76	5.2	73	79	74	79
Re, Rhenium (ppm)	< 0.004	IND	IND	IND	IND	IND
S, Sulphur (ppm)	291	41	267	316	IND	IND
Sb, Antimony (ppm)	3.40	0.289	3.21	3.59	3.25	3.54
Sc, Scandium (ppm)	27.9	1.26	27.1	28.6	26.9	28.8
Se, Selenium (ppm)	< 5	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	101	4.8	98	104	99	104
Sn, Tin (ppm)	13.3	1.13	12.5	14.0	12.8	13.7
Sr, Strontium (ppm)	306	15.0	296	315	300	311
Ta, Tantalum (ppm)	11.5	1.6	10.1	12.8	11.0	11.9
Tb, Terbium (ppm)	4.60	0.172	4.50	4.71	4.47	4.73
Te, Tellurium (ppm)	0.21	0.04	0.18	0.24	0.15	0.26
Th, Thorium (ppm)	113	4.9	110	117	111	116
Ti, Titanium (wt.%)	0.731	0.140	0.640	0.821	0.705	0.756
Tl, Thallium (ppm)	0.38	0.029	0.36	0.39	0.35	0.40
Tm, Thulium (ppm)	0.50	0.048	0.47	0.53	0.47	0.53
U, Uranium (ppm)	3.85	0.166	3.76	3.95	3.71	4.00
V, Vanadium (ppm)	238	14.4	229	247	232	244
W, Tungsten (ppm)	3.14	0.287	2.92	3.36	2.86	3.42
Y, Yttrium (ppm)	49.3	2.59	47.8	50.8	47.5	51.1
Yb, Ytterbium (ppm)	2.64	0.189	2.54	2.75	2.52	2.77
Zn, Zinc (ppm)	117	6.7	113	121	115	119
Zr, Zirconium (ppm)	218	25	201	234	212	223

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 460.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al_2O_3	wt.%	*12.80*	Lu_2O_3	ppm	*< 20*	Ta_2O_5	ppm	*< 100*
BaO	ppm	*950*	MgO	wt.%	*1.24*	Tb_4O_7	ppm	*< 20*
CaO	wt.%	*1.02*	MnO	ppm	*488*	ThO_2	ppm	*166*
Cr_2O_3	ppm	*375*	Na_2O	wt.%	*0.204*	TiO_2	wt.%	*1.99*
Dy_2O_3	ppm	*16.7*	Nb_2O_5	ppm	*984*	Tm_2O_3	ppm	*< 10*
Er_2O_3	ppm	*< 10*	P_2O_5	wt.%	*0.471*	U_3O_8	ppm	*< 100*
Eu_2O_3	ppm	*< 100*	SiO_2	wt.%	*50.42*	V_2O_5	ppm	*477*
Gd_2O_3	ppm	*88*	Sm_2O_3	ppm	*138*	WO_3	ppm	*< 100*
HfO_2	ppm	*< 100*	SnO_2	ppm	*< 100*	Y_2O_3	ppm	*107*
Ho_2O_3	ppm	*< 10*	SO_3	wt.%	*0.068*	Yb_2O_3	ppm	*< 10*
K_2O	wt.%	*1.54*	SrO	ppm	*100*	ZrO_2	ppm	*567*

Table 2 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Thermogravimetry								
H_2O-	wt.%	*0.752*						
Borate/ Peroxide Fusion ICP								
Ag	ppm	*7.67*	Ga†	ppm	*33.8*	S	ppm	*321*
As	ppm	*75*	Ge	ppm	*1.63*	Sc	ppm	*34.5*
B	ppm	*122*	In	ppm	*0.31*	Se	ppm	*< 20*
Cd	ppm	*< 1*	Na	wt.%	*0.132*	Te	ppm	*< 1*
Cu	ppm	*43.7*	Re	ppm	*< 0.1*	Tl	ppm	*< 0.5*
4-Acid Digestion								
Ge	ppm	*1.79*	Nb	ppm	*616*			

†Reported Ga (Gallium) values are based on best consensus and are not considered accurate due to known spectral interferences from Ce (Cerium) when analysed by conventional ICP-MS methods. These interferences can significantly overestimate Ga concentrations. For accurate determination of Ga, analysis using ICP-MS with triple quadrupole (ICP-MS QQQ) is strongly recommended. Users should note that true Ga concentrations are expected to be substantially lower than the indicative value reported here.

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 460 is an ore grade, rare earth element (TREO = 0.53%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 460 were sourced from both a quarry north of Melbourne (weathered barren siltstone) and 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% H_2O-).

OREAS 460 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 112 certified values (and 50 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%: CeO_2 (46.7%), La_2O_3 (25.5%), Nd_2O_3 (18.5%), Pr_6O_{11} (5.32%), Sm_2O_3 (2.27%) and Eu_2O_3 (0.443%), together with minor components of HREE: Dy_2O_3 (0.124%) and Tb_4O_7 (0.068%).

COMMINUTION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 460 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 112 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-HNO_3-$HClO_4$-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 460. 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 112 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 50 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 (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 460 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 460.

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	2213	88	2038	2389	1951	2476	3.96%	7.92%	11.88%	2103	2324
Fe_2O_3, wt.%	26.81	0.383	26.04	27.58	25.66	27.96	1.43%	2.86%	4.29%	25.47	28.15
La_2O_3, ppm	1626	62	1502	1751	1440	1813	3.82%	7.65%	11.47%	1545	1708
Nd_2O_3, ppm	958	66	826	1090	760	1156	6.89%	13.78%	20.67%	910	1006
Pr_6O_{11}, ppm	289	63	164	415	101	478	21.76%	43.52%	65.27%	275	304
Thermogravimetry											
LOI, wt.%	2.54	0.209	2.13	2.96	1.92	3.17	8.21%	16.42%	24.64%	2.42	2.67
Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)											
Al, wt.%	6.74	0.135	6.47	7.01	6.33	7.14	2.01%	4.01%	6.02%	6.40	7.07
Al_2O_3, wt.%	12.73	0.255	12.22	13.24	11.96	13.49	2.01%	4.01%	6.02%	12.09	13.36
Ba, ppm	808	53	702	914	649	967	6.55%	13.10%	19.65%	768	848
BaO, ppm	902	59	784	1020	725	1079	6.55%	13.10%	19.65%	857	947
Be, ppm	2.48	0.42	1.64	3.32	1.21	3.74	17.01%	34.01%	51.02%	2.35	2.60
Bi, ppm	1.48	0.33	0.83	2.14	0.50	2.47	22.03%	44.05%	66.08%	1.41	1.56
Ca, wt.%	0.697	0.024	0.649	0.745	0.625	0.769	3.45%	6.91%	10.36%	0.662	0.732

Note: intervals may appear asymmetric due to rounding.

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
Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
CaO, wt.%	0.975	0.034	0.907	1.042	0.874	1.076	3.45%	6.91%	10.36%	0.926	1.024
Ce, ppm	1798	72	1654	1943	1581	2015	4.02%	8.03%	12.05%	1708	1888
CeO_2, ppm	2209	89	2031	2386	1943	2475	4.02%	8.03%	12.05%	2098	2319
Co, ppm	9.44	1.48	6.47	12.40	4.99	13.88	15.71%	31.42%	47.12%	8.96	9.91
Cr, ppm	393	21	351	435	331	456	5.30%	10.60%	15.91%	374	413
Cr_2O_3, ppm	575	30	514	636	483	666	5.30%	10.60%	15.91%	546	603
Cs, ppm	3.68	0.246	3.19	4.17	2.94	4.41	6.68%	13.36%	20.04%	3.49	3.86
Dy, ppm	19.8	0.75	18.3	21.3	17.6	22.1	3.78%	7.57%	11.35%	18.9	20.8
Dy_2O_3, ppm	22.8	0.86	21.1	24.5	20.2	25.4	3.78%	7.57%	11.35%	21.6	23.9
Er, ppm	6.01	0.350	5.31	6.71	4.96	7.06	5.82%	11.63%	17.45%	5.71	6.31
Er_2O_3, ppm	6.88	0.400	6.08	7.68	5.68	8.08	5.82%	11.63%	17.45%	6.53	7.22
Eu, ppm	22.7	0.96	20.8	24.7	19.9	25.6	4.24%	8.47%	12.71%	21.6	23.9
Eu_2O_3, ppm	26.3	1.12	24.1	28.6	23.0	29.7	4.24%	8.47%	12.71%	25.0	27.6
Fe, wt.%	18.90	0.628	17.65	20.16	17.02	20.79	3.32%	6.64%	9.97%	17.96	19.85
Fe_2O_3, wt.%	27.02	0.898	25.23	28.82	24.33	29.72	3.32%	6.64%	9.97%	25.67	28.38
Gd, ppm	50	3.0	44	56	41	59	5.91%	11.83%	17.74%	48	53
Gd_2O_3, ppm	58	3.4	51	65	47	68	5.91%	11.83%	17.74%	55	61
Hf, ppm	11.8	0.56	10.7	12.9	10.1	13.5	4.77%	9.54%	14.31%	11.2	12.4
HfO_2, ppm	13.9	0.66	12.6	15.3	11.9	15.9	4.77%	9.54%	14.31%	13.2	14.6
Ho, ppm	2.77	0.219	2.33	3.21	2.11	3.43	7.91%	15.82%	23.74%	2.63	2.91
Ho_2O_3, ppm	3.18	0.251	2.67	3.68	2.42	3.93	7.91%	15.82%	23.74%	3.02	3.34
K, wt.%	1.23	0.086	1.06	1.40	0.97	1.49	6.96%	13.93%	20.89%	1.17	1.29
K_2O, wt.%	1.48	0.103	1.28	1.69	1.17	1.79	6.96%	13.93%	20.89%	1.41	1.56
La, ppm	1369	75	1219	1519	1144	1595	5.48%	10.97%	16.45%	1301	1438
La_2O_3, ppm	1606	88	1430	1782	1342	1870	5.48%	10.97%	16.45%	1526	1686
Li, ppm	19.6	0.85	17.9	21.3	17.0	22.1	4.36%	8.71%	13.07%	18.6	20.5
Lu, ppm	0.52	0.049	0.42	0.62	0.38	0.67	9.35%	18.69%	28.04%	0.50	0.55
Lu_2O_3, ppm	0.59	0.055	0.48	0.70	0.43	0.76	9.35%	18.69%	28.04%	0.56	0.62
Mg, wt.%	0.751	0.033	0.685	0.818	0.652	0.851	4.42%	8.85%	13.27%	0.714	0.789
MgO, wt.%	1.25	0.055	1.14	1.36	1.08	1.41	4.42%	8.85%	13.27%	1.18	1.31
Mn, ppm	361	30	301	422	270	452	8.39%	16.77%	25.16%	343	379
MnO, ppm	466	39	388	544	349	584	8.39%	16.77%	25.16%	443	490

Note: intervals may appear asymmetric due to rounding.

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
Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
Mo, ppm	25.2	3.7	17.9	32.5	14.2	36.2	14.52%	29.04%	43.55%	23.9	26.4
Nb, ppm	698	39	620	776	581	815	5.59%	11.18%	16.76%	663	732
Nb_2O_5, ppm	998	56	886	1109	831	1165	5.59%	11.18%	16.76%	948	1048
Nd, ppm	781	47	688	875	641	921	5.97%	11.94%	17.91%	742	820
Nd_2O_3, ppm	911	54	802	1020	748	1075	5.97%	11.94%	17.91%	866	957
Ni, ppm	53	4.9	43	63	39	68	9.24%	18.48%	27.73%	51	56
NiO, ppm	68	6.3	55	80	49	87	9.24%	18.48%	27.73%	64	71
P, wt.%	0.198	0.014	0.169	0.227	0.155	0.242	7.30%	14.59%	21.89%	0.188	0.208
P_2O_5, wt.%	0.455	0.033	0.388	0.521	0.355	0.554	7.30%	14.59%	21.89%	0.432	0.477
Pb, ppm	67	5.4	57	78	51	83	7.96%	15.92%	23.89%	64	71
PbO, ppm	72	5.8	61	84	55	90	7.96%	15.92%	23.89%	69	76
Pr, ppm	244	8	227	261	218	269	3.48%	6.97%	10.45%	232	256
Pr_6O_{11}, ppm	294	10	274	315	264	325	3.48%	6.97%	10.45%	280	309
Rb, ppm	75	4.1	67	83	63	87	5.43%	10.86%	16.28%	71	79
Sb, ppm	3.71	0.39	2.93	4.49	2.53	4.88	10.55%	21.09%	31.64%	3.52	3.89
Si, wt.%	23.28	0.292	22.70	23.86	22.41	24.16	1.25%	2.51%	3.76%	22.12	24.44
SiO_2, wt.%	49.80	0.624	48.55	51.05	47.93	51.68	1.25%	2.51%	3.76%	47.31	52.29
Sm, ppm	107	3	101	114	97	118	3.15%	6.30%	9.45%	102	113
Sm_2O_3, ppm	125	4	117	132	113	136	3.15%	6.30%	9.45%	118	131
Sn, ppm	16.0	2.1	11.8	20.3	9.6	22.5	13.36%	26.71%	40.07%	15.2	16.8
SnO_2, ppm	20.4	2.7	14.9	25.8	12.2	28.5	13.36%	26.71%	40.07%	19.3	21.4
Sr, ppm	305	12	281	328	269	340	3.88%	7.76%	11.64%	290	320
SrO, ppm	360	14	332	388	318	402	3.88%	7.76%	11.64%	342	378
Ta, ppm	13.7	0.64	12.5	15.0	11.8	15.7	4.66%	9.32%	13.98%	13.1	14.4
Ta_2O_5, ppm	16.8	0.78	15.2	18.3	14.4	19.1	4.66%	9.32%	13.98%	15.9	17.6
Tb, ppm	4.84	0.212	4.42	5.27	4.21	5.48	4.38%	8.76%	13.14%	4.60	5.08
Tb_4O_7, ppm	5.70	0.250	5.20	6.19	4.95	6.44	4.38%	8.76%	13.14%	5.41	5.98
Th, ppm	116	3	109	122	106	126	2.81%	5.63%	8.44%	110	122
ThO_2, ppm	132	4	124	139	121	143	2.81%	5.63%	8.44%	125	138
Ti, wt.%	1.20	0.028	1.14	1.26	1.12	1.28	2.31%	4.63%	6.94%	1.14	1.26
TiO_2, wt.%	2.00	0.046	1.91	2.09	1.86	2.14	2.31%	4.63%	6.94%	1.90	2.10
Tm, ppm	0.70	0.054	0.59	0.81	0.54	0.86	7.70%	15.39%	23.09%	0.67	0.74

Note: intervals may appear asymmetric due to rounding.

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
Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
Tm_2O_3, ppm	0.80	0.062	0.68	0.93	0.62	0.99	7.70%	15.39%	23.09%	0.76	0.84
U, ppm	4.21	0.193	3.83	4.60	3.63	4.79	4.58%	9.16%	13.74%	4.00	4.42
U_3O_8, ppm	4.97	0.227	4.51	5.42	4.28	5.65	4.58%	9.16%	13.74%	4.72	5.22
V, ppm	255	13	230	280	218	293	4.92%	9.83%	14.75%	242	268
V_2O_5, ppm	456	22	411	500	388	523	4.92%	9.83%	14.75%	433	478
W, ppm	< 6	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
WO_3, ppm	< 8	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Y, ppm	60	2.6	55	65	52	67	4.34%	8.69%	13.03%	57	63
Y_2O_3, ppm	76	3.3	69	82	66	86	4.34%	8.69%	13.03%	72	80
Yb, ppm	3.91	0.262	3.38	4.43	3.12	4.69	6.71%	13.42%	20.14%	3.71	4.10
Yb_2O_3, ppm	4.45	0.299	3.85	5.05	3.55	5.35	6.71%	13.42%	20.14%	4.23	4.67
Zn, ppm	121	23	74	167	51	190	19.31%	38.62%	57.92%	115	127
ZnO, ppm	150	29	92	208	63	237	19.31%	38.62%	57.92%	143	158
Zr, ppm	472	21	429	515	408	537	4.54%	9.08%	13.62%	449	496
ZrO_2, ppm	638	29	580	696	551	725	4.54%	9.08%	13.62%	606	670
4-Acid Digestion											
Ag, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	6.55	0.230	6.09	7.02	5.86	7.25	3.51%	7.03%	10.54%	6.23	6.88
As, ppm	53	3.4	47	60	43	64	6.41%	12.81%	19.22%	51	56
Ba, ppm	815	23	768	861	745	885	2.85%	5.71%	8.56%	774	856
Be, ppm	2.40	0.109	2.18	2.62	2.07	2.72	4.52%	9.05%	13.57%	2.28	2.52
Bi, ppm	1.44	0.077	1.29	1.60	1.21	1.68	5.35%	10.71%	16.06%	1.37	1.52
Ca, wt.%	0.701	0.035	0.632	0.770	0.597	0.805	4.93%	9.87%	14.80%	0.666	0.736
Cd, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	1853	133	1588	2119	1455	2252	7.16%	14.33%	21.49%	1761	1946
Co, ppm	10.0	0.55	8.9	11.1	8.4	11.7	5.48%	10.96%	16.44%	9.5	10.5
Cr, ppm	347	28	291	403	262	432	8.13%	16.25%	24.38%	330	364
Cs, ppm	3.78	0.273	3.23	4.32	2.96	4.60	7.22%	14.44%	21.66%	3.59	3.97
Cu, ppm	41.7	2.00	37.7	45.7	35.7	47.7	4.78%	9.57%	14.35%	39.6	43.8
Dy, ppm	18.0	0.44	17.2	18.9	16.7	19.3	2.42%	4.84%	7.26%	17.1	18.9
Er, ppm	4.65	0.216	4.22	5.08	4.00	5.30	4.65%	9.30%	13.95%	4.42	4.88
Eu, ppm	23.1	1.31	20.4	25.7	19.1	27.0	5.69%	11.37%	17.06%	21.9	24.2

Note: intervals may appear asymmetric due to rounding.

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											
Fe, wt.%	18.56	0.431	17.70	19.42	17.27	19.85	2.32%	4.64%	6.96%	17.63	19.49
Ga, ppm	33.0	4.3	24.5	41.6	20.2	45.8	12.92%	25.83%	38.75%	31.4	34.7
Gd, ppm	48.2	2.46	43.2	53.1	40.8	55.6	5.11%	10.23%	15.34%	45.8	50.6
Hf, ppm	6.09	0.580	4.93	7.25	4.35	7.83	9.53%	19.07%	28.60%	5.78	6.39
Ho, ppm	2.37	0.133	2.10	2.63	1.97	2.77	5.64%	11.28%	16.92%	2.25	2.48
In, ppm	0.31	0.017	0.27	0.34	0.26	0.36	5.61%	11.21%	16.82%	0.29	0.32
K, wt.%	1.25	0.035	1.18	1.32	1.14	1.35	2.82%	5.63%	8.45%	1.18	1.31
La, ppm	1298	113	1072	1524	958	1638	8.72%	17.44%	26.16%	1233	1363
Li, ppm	18.3	1.48	15.4	21.3	13.9	22.8	8.09%	16.18%	24.27%	17.4	19.3
Lu, ppm	0.36	0.05	0.26	0.45	0.21	0.50	13.29%	26.58%	39.87%	0.34	0.37
Mg, wt.%	0.724	0.058	0.609	0.840	0.551	0.898	8.00%	16.01%	24.01%	0.688	0.761
Mn, ppm	307	29	249	366	220	395	9.50%	19.00%	28.50%	292	323
Mo, ppm	25.4	0.73	24.0	26.9	23.2	27.6	2.87%	5.73%	8.60%	24.2	26.7
Na, wt.%	0.133	0.006	0.121	0.145	0.116	0.151	4.41%	8.82%	13.22%	0.126	0.140
Nd, ppm	792	27	739	845	712	871	3.35%	6.69%	10.04%	752	831
Ni, ppm	62	4.0	54	70	50	74	6.41%	12.81%	19.22%	59	65
P, wt.%	0.193	0.009	0.174	0.211	0.165	0.221	4.81%	9.62%	14.43%	0.183	0.202
Pb, ppm	65	2.4	60	70	58	72	3.63%	7.26%	10.89%	62	68
Pr, ppm	240	11	217	262	206	273	4.69%	9.38%	14.07%	228	252
Rb, ppm	76	5.2	66	87	61	92	6.83%	13.65%	20.48%	73	80
Re, ppm	< 0.004	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, ppm	291	41	209	374	168	415	14.13%	28.25%	42.38%	277	306
Sb, ppm	3.40	0.289	2.82	3.98	2.53	4.26	8.51%	17.02%	25.52%	3.23	3.57
Sc, ppm	27.9	1.26	25.4	30.4	24.1	31.7	4.52%	9.04%	13.56%	26.5	29.3
Se, ppm	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	101	5	92	111	87	116	4.72%	9.43%	14.15%	96	106
Sn, ppm	13.3	1.13	11.0	15.5	9.9	16.6	8.48%	16.96%	25.44%	12.6	13.9
Sr, ppm	306	15	275	336	260	351	4.93%	9.85%	14.78%	290	321
Ta, ppm	11.5	1.6	8.2	14.7	6.6	16.3	14.10%	28.19%	42.29%	10.9	12.0
Tb, ppm	4.60	0.172	4.26	4.95	4.09	5.12	3.73%	7.45%	11.18%	4.37	4.83
Te, ppm	0.21	0.04	0.13	0.28	0.09	0.32	18.11%	36.21%	54.32%	0.20	0.22
Th, ppm	113	5	104	123	99	128	4.28%	8.57%	12.85%	108	119

Note: intervals may appear asymmetric due to rounding.

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											
Ti, wt.%	0.731	0.140	0.451	1.010	0.312	1.149	19.11%	38.21%	57.32%	0.694	0.767
Tl, ppm	0.38	0.029	0.32	0.43	0.29	0.46	7.57%	15.14%	22.71%	0.36	0.40
Tm, ppm	0.50	0.048	0.41	0.60	0.36	0.65	9.50%	19.00%	28.51%	0.48	0.53
U, ppm	3.85	0.166	3.52	4.18	3.36	4.35	4.30%	8.60%	12.90%	3.66	4.04
V, ppm	238	14	209	267	195	281	6.07%	12.15%	18.22%	226	250
W, ppm	3.14	0.287	2.56	3.71	2.28	4.00	9.15%	18.29%	27.44%	2.98	3.30
Y, ppm	49.3	2.59	44.1	54.5	41.5	57.1	5.25%	10.50%	15.75%	46.8	51.8
Yb, ppm	2.64	0.189	2.27	3.02	2.08	3.21	7.15%	14.30%	21.45%	2.51	2.78
Zn, ppm	117	7	103	130	97	137	5.76%	11.52%	17.28%	111	123
Zr, ppm	218	25	167	268	142	293	11.61%	23.21%	34.82%	207	229

Note: intervals may appear asymmetric due to rounding

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 ($\rho=0.95$) will have concentrations lying between 1576 and 1635 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 460 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 112 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 460 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, Booysens, 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 460 has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd
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It is available in unit sizes of 10g in laminated foil pouches or 1kg in plastic jars.

INTENDED USE

OREAS 460 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 460 has been prepared from a blend of barren weathered siltstone and low grade/waste REE bearing ore (TREO = 0.53%). 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% H_2O-).

OREAS 460 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.

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.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
1	24th September, 2025	Ga reclassified from Certified Value to Indicative Value.
0	28th April, 2015	First publication.

LEGAL NOTICE

CERTIFYING OFFICER

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

REFERENCES

ISO Guide 30 (1992), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2000), Reference materials – Contents of certificates and labels.

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.

Jaireth, S., Hoatson D.M., Miezitis, Y. Ore Geology Reviews 62 (2014) 72-128 - Geological setting and resources of the major rare-earth-element deposits in Australia.