

CERTIFICATE OF ANALYSIS FOR

CARBONATITE SUPERGENE REE-Nb ORE (TREO 1.06%)

CERTIFIED REFERENCE MATERIAL

OREAS 461

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)	4311	162	4222	4401	4193	4430
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	40.0	1.66	39.1	40.9	38.6	41.3
Er ₂ O ₃ , Erbium(III) oxide (ppm)	10.1	0.34	9.9	10.3	9.7	10.4
Eu ₂ O ₃ , Europium(III) oxide (ppm)	54	2.5	53	56	52	56
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	116	9	110	121	112	119
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	5.22	0.350	5.02	5.43	4.99	5.45
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	3155	185	3051	3259	3090	3220
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.59	0.050	0.56	0.62	0.54	0.63
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	1854	140	1762	1947	1803	1906
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	1900	63	1863	1937	1848	1952
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	590	25	577	604	572	609
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	255	9	251	259	247	263
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	10.7	0.36	10.5	10.9	10.3	11.0
ThO ₂ , Thorium dioxide (ppm)	239	8	234	244	234	243
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.02	0.098	0.96	1.07	0.96	1.08
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	5.65	0.230	5.53	5.77	5.47	5.83
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	116	6	112	119	112	119
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	5.00	0.282	4.86	5.15	4.72	5.29
ZrO ₂ , Zirconium dioxide (ppm)	815	36	790	839	786	843

Note: intervals may appear asymmetric due to rounding.



Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 461.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO ₂ , Cerium(IV) oxide (ppm)	4376	54.9	4331	4421	IND	IND
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	46.09	0.773	45.06	47.12	45.78	46.40
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	3191	64.1	3149	3232	3053	3328
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	1928	132.7	1801	2055	IND	IND
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	585	103	484	686	IND	IND
ThO ₂ , Thorium dioxide (ppm)	265	52	194	335	IND	IND
Thermogravimetry						
LOI, Loss On Ignition @1000°C (wt.%)	0.996	0.175	0.801	1.192	0.940	1.053
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	5.99	0.208	5.84	6.13	5.88	6.09
Al ₂ O ₃ , Aluminium(III) oxide (wt.%)	11.31	0.393	11.03	11.59	11.12	11.50
Ba, Barium (ppm)	929	55	898	960	912	946
BaO, Barium oxide (ppm)	1037	62	1003	1071	1018	1056
Be, Beryllium (ppm)	2.30	0.38	1.91	2.69	IND	IND
Bi, Bismuth (ppm)	2.49	0.39	2.09	2.88	IND	IND
Ca, Calcium (wt.%)	1.26	0.057	1.22	1.30	1.23	1.29
CaO, Calcium oxide (wt.%)	1.76	0.080	1.71	1.81	1.72	1.80
Ce, Cerium (ppm)	3510	132	3437	3583	3414	3606
CeO ₂ , Cerium(IV) oxide (ppm)	4311	162	4222	4401	4193	4430
Co, Cobalt (ppm)	11.5	1.8	9.7	13.4	11.1	11.9
Cr, Chromium (ppm)	599	40	575	623	584	614
Cr ₂ O ₃ , Chromium(III) oxide (ppm)	875	59	840	911	854	897
Cs, Cesium (ppm)	0.79	0.059	0.75	0.82	0.75	0.82
Cu, Copper (ppm)	60	20	41	80	IND	IND
Dy, Dysprosium (ppm)	34.8	1.44	34.0	35.6	33.7	36.0
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	40.0	1.66	39.1	40.9	38.6	41.3
Er, Erbium (ppm)	8.80	0.296	8.64	8.96	8.47	9.13
Er ₂ O ₃ , Erbium(III) oxide (ppm)	10.1	0.34	9.9	10.3	9.7	10.4
Eu, Europium (ppm)	46.7	2.19	45.4	48.0	45.1	48.4
Eu ₂ O ₃ , Europium(III) oxide (ppm)	54	2.5	53	56	52	56
Fe, Iron (wt.%)	31.77	1.301	30.82	32.73	31.24	32.31
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	45.43	1.860	44.06	46.79	44.66	46.19
Ga, Gallium (ppm)	50	6	43	57	48	52
Gd, Gadolinium (ppm)	100	8	95	105	98	103
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	116	9	110	121	112	119
Hf, Hafnium (ppm)	14.1	0.61	13.7	14.5	13.4	14.7
HfO ₂ , Hafnium dioxide (ppm)	16.6	0.72	16.2	17.1	15.9	17.4
Ho, Holmium (ppm)	4.56	0.306	4.38	4.74	4.36	4.76
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	5.22	0.350	5.02	5.43	4.99	5.45
In, Indium (ppm)	0.60	0.08	0.52	0.68	IND	IND
K, Potassium (wt.%)	0.237	0.033	0.213	0.261	0.224	0.249
K ₂ O, Potassium oxide (wt.%)	0.285	0.040	0.256	0.314	0.270	0.300

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)						
La, Lanthanum (ppm)	2690	158	2601	2779	2635	2745
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	3155	185	3051	3259	3090	3220
Li, Lithium (ppm)	12.0	1.5	10.3	13.7	IND	IND
Lu, Lutetium (ppm)	0.52	0.044	0.49	0.54	0.48	0.55
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.59	0.050	0.56	0.62	0.54	0.63
Mg, Magnesium (wt.%)	1.05	0.051	1.01	1.08	1.02	1.07
MgO, Magnesium oxide (wt.%)	1.74	0.085	1.68	1.79	1.70	1.78
Mn, Manganese (ppm)	653	49	616	689	633	673
MnO, Manganese oxide (ppm)	843	63	795	890	817	869
Mo, Molybdenum (ppm)	45.7	4.8	41.1	50.3	43.4	48.0
Nb, Niobium (ppm)	1296	98	1231	1361	1260	1333
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	1854	140	1762	1947	1803	1906
Nd, Neodymium (ppm)	1629	54	1597	1660	1584	1674
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	1900	63	1863	1937	1848	1952
Ni, Nickel (ppm)	64	5.5	61	66	53	74
NiO, Nickel oxide (ppm)	81	7.0	78	85	68	94
P, Phosphorus (wt.%)	0.359	0.019	0.344	0.373	0.344	0.374
P ₂ O ₅ , Phosphorus(V) oxide (wt.%)	0.822	0.044	0.789	0.855	0.787	0.857
Pb, Lead (ppm)	106	6	100	113	99	113
PbO, Lead oxide (ppm)	114	7	108	121	107	122
Pr, Praseodymium (ppm)	489	21	477	500	473	504
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	590	25	577	604	572	609
Rb, Rubidium (ppm)	13.5	0.54	13.2	13.8	13.0	14.0
S, Sulphur (ppm)	518	82	433	602	IND	IND
Sb, Antimony (ppm)	2.76	0.220	2.53	2.99	2.53	2.99
Si, Silicon (wt.%)	14.71	0.360	14.40	15.02	14.48	14.94
SiO ₂ , Silicon dioxide (wt.%)	31.46	0.770	30.80	32.13	30.97	31.96
Sm, Samarium (ppm)	220	7	216	223	213	226
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	255	9	251	259	247	263
Sn, Tin (ppm)	25.6	3.3	23.4	27.9	23.7	27.5
SnO ₂ , Tin dioxide (ppm)	32.5	4.2	29.7	35.4	30.1	34.9
Sr, Strontium (ppm)	579	15	570	588	567	591
SrO, Strontium oxide (ppm)	685	18	674	695	671	699
Ta, Tantalum (ppm)	25.1	1.04	24.6	25.7	24.0	26.2
Ta ₂ O ₅ , Tantalum(V) oxide (ppm)	30.7	1.27	30.0	31.4	29.4	32.0
Tb, Terbium (ppm)	9.08	0.305	8.92	9.23	8.76	9.39
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	10.7	0.36	10.5	10.9	10.3	11.0
Th, Thorium (ppm)	210	7	206	214	206	214
ThO ₂ , Thorium dioxide (ppm)	239	8	234	244	234	243
Ti, Titanium (wt.%)	1.84	0.055	1.80	1.88	1.81	1.87
TiO ₂ , Titanium dioxide (wt.%)	3.07	0.092	3.01	3.13	3.01	3.13
Tm, Thulium (ppm)	0.89	0.085	0.84	0.94	0.84	0.94

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)						
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.02	0.098	0.96	1.07	0.96	1.08
U, Uranium (ppm)	4.79	0.195	4.69	4.89	4.64	4.94
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	5.65	0.230	5.53	5.77	5.47	5.83
V, Vanadium (ppm)	385	16	376	393	375	394
V ₂ O ₅ , Vanadium(V) oxide (ppm)	687	29	671	702	670	704
W, Tungsten (ppm)	3.88	0.67	3.35	4.40	IND	IND
WO ₃ , Tungsten trioxide (ppm)	4.89	0.84	4.23	5.55	IND	IND
Y, Yttrium (ppm)	91	4.4	88	94	88	94
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	116	6	112	119	112	119
Yb, Ytterbium (ppm)	4.39	0.248	4.27	4.52	4.14	4.65
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	5.00	0.282	4.86	5.15	4.72	5.29
Zn, Zinc (ppm)	162	23	131	193	IND	IND
ZnO, Zinc oxide (ppm)	201	29	163	240	IND	IND
Zr, Zirconium (ppm)	603	27	585	621	582	624
ZrO ₂ , Zirconium dioxide (ppm)	815	36	790	839	786	843
4-Acid Digestion						
Ag, Silver (ppm)	< 2	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	5.96	0.180	5.86	6.06	5.86	6.06
As, Arsenic (ppm)	33.6	1.09	33.0	34.1	32.3	34.8
Ba, Barium (ppm)	957	44.1	931	983	936	978
Be, Beryllium (ppm)	2.09	0.24	1.96	2.22	1.98	2.20
Bi, Bismuth (ppm)	2.43	0.158	2.33	2.52	2.33	2.53
Ca, Calcium (wt.%)	1.21	0.090	1.16	1.27	1.20	1.23
Cd, Cadmium (ppm)	< 0.1	IND	IND	IND	IND	IND
Ce, Cerium (ppm)	3508	112.0	3420	3597	3411	3606
Co, Cobalt (ppm)	12.1	0.44	11.8	12.4	11.8	12.5
Cr, Chromium (ppm)	524	73	473	575	507	540
Cs, Cesium (ppm)	0.77	0.067	0.73	0.81	0.73	0.80
Cu, Copper (ppm)	56	2.3	55	57	54	57
Dy, Dysprosium (ppm)	33.5	0.86	33.1	33.9	32.7	34.4
Er, Erbium (ppm)	7.46	0.253	7.31	7.61	7.24	7.68
Eu, Europium (ppm)	47.6	3.26	45.7	49.6	46.6	48.7
Fe, Iron (wt.%)	31.52	0.959	30.95	32.09	31.08	31.96
Ga, Gallium (ppm)	45.5	8.0	37.7	53.3	44.1	46.9
Gd, Gadolinium (ppm)	98	3.5	95	100	95	100
Hf, Hafnium (ppm)	7.39	0.84	6.78	8.00	6.96	7.82
Ho, Holmium (ppm)	4.06	0.115	3.99	4.12	3.95	4.16
In, Indium (ppm)	0.57	0.027	0.55	0.58	0.54	0.60
K, Potassium (wt.%)	0.233	0.006	0.231	0.235	0.223	0.243
La, Lanthanum (ppm)	2476	228.0	2338	2615	2422	2531
Li, Lithium (ppm)	12.0	1.4	11.3	12.8	11.6	12.4
Lu, Lutetium (ppm)	0.39	0.07	0.35	0.43	0.36	0.41

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						
Mg, Magnesium (wt.%)	0.999	0.108	0.935	1.063	0.980	1.018
Mn, Manganese (ppm)	547	80	496	599	533	561
Mo, Molybdenum (ppm)	48.2	2.54	46.6	49.9	47.2	49.2
Na, Sodium (wt.%)	0.178	0.013	0.170	0.186	IND	IND
Nd, Neodymium (ppm)	1623	79.5	1579	1668	1566	1681
Ni, Nickel (ppm)	71	3.1	69	73	69	74
P, Phosphorus (wt.%)	0.338	0.022	0.324	0.352	0.333	0.343
Pb, Lead (ppm)	104	5.5	100	108	101	106
Pr, Praseodymium (ppm)	482	20.1	471	493	470	495
Rb, Rubidium (ppm)	13.3	0.99	12.8	13.8	13.0	13.6
Re, Rhenium (ppm)	< 0.01	IND	IND	IND	IND	IND
S, Sulphur (ppm)	475	49	446	504	IND	IND
Sb, Antimony (ppm)	2.42	0.174	2.30	2.53	2.30	2.54
Sc, Scandium (ppm)	38.8	2.50	37.3	40.4	37.7	40.0
Sm, Samarium (ppm)	208	7.4	204	212	203	214
Sn, Tin (ppm)	21.6	2.08	20.2	23.1	20.7	22.5
Sr, Strontium (ppm)	564	35.2	543	586	556	573
Ta, Tantalum (ppm)	20.9	1.96	19.2	22.6	19.7	22.2
Tb, Terbium (ppm)	9.09	0.503	8.76	9.43	8.87	9.32
Te, Tellurium (ppm)	0.34	0.06	0.30	0.38	0.29	0.40
Th, Thorium (ppm)	207	9.6	201	212	200	213
Ti, Titanium (wt.%)	1.06	0.12	0.96	1.15	1.01	1.10
Tl, Thallium (ppm)	0.10	0.008	0.10	0.11	IND	IND
Tm, Thulium (ppm)	0.70	0.046	0.67	0.73	0.68	0.72
U, Uranium (ppm)	4.40	0.161	4.31	4.49	4.26	4.54
V, Vanadium (ppm)	352	28.8	333	371	345	359
W, Tungsten (ppm)	2.50	0.36	2.25	2.74	2.35	2.65
Y, Yttrium (ppm)	83	3.7	81	85	81	86
Yb, Ytterbium (ppm)	3.06	0.149	2.98	3.15	2.96	3.17
Zn, Zinc (ppm)	139	13.3	131	148	136	142
Zr, Zirconium (ppm)	270	18.0	257	283	257	283

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 461.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al ₂ O ₃	wt.%	11.49	Lu ₂ O ₃	ppm	< 20	Ta ₂ O ₅	ppm	< 100
BaO	ppm	1117	MgO	wt.%	1.76	Tb ₄ O ₇	ppm	< 20
CaO	wt.%	1.82	MnO	ppm	904	TiO ₂	wt.%	3.08
Cr ₂ O ₃	ppm	628	Na ₂ O	wt.%	0.250	Tm ₂ O ₃	ppm	< 10
Dy ₂ O ₃	ppm	46.7	Nb ₂ O ₅	ppm	1863	U ₃ O ₈	ppm	< 100
Er ₂ O ₃	ppm	20.0	P ₂ O ₅	wt.%	0.850	V ₂ O ₅	ppm	728
Eu ₂ O ₃	ppm	< 100	SiO ₂	wt.%	31.65	WO ₃	ppm	< 100

Table 2 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF continued								
Gd ₂ O ₃	ppm	100	Sm ₂ O ₃	ppm	220	Y ₂ O ₃	ppm	146
HfO ₂	ppm	< 100	SnO ₂	ppm	< 100	Yb ₂ O ₃	ppm	10.0
Ho ₂ O ₃	ppm	30.0	SO ₃	wt.%	0.117	ZrO ₂	ppm	800
K ₂ O	wt.%	0.284	SrO	ppm	500			
Thermogravimetry								
H ₂ O-	wt.%	0.697						
Borate / Peroxide Fusion ICP								
Ag	ppm	15.2	Ge	ppm	2.50	Se	ppm	< 20
As	ppm	79	Na	wt.%	0.166	Te	ppm	< 1
B	ppm	151	Re	ppm	< 0.1	Tl	ppm	< 0.5
Cd	ppm	< 1	Sc	ppm	44.0			
4-Acid Digestion								
Ge	ppm	3.60	Nb	ppm	1153	Se	ppm	6.48

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 461 is an ore grade, rare earth element (TREO = 1.06%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 461 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% H₂O-).

OREAS 461 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 115 certified values (and 47 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 461 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 115 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 461. 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 115 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 47 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 461 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 461.

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	4376	55	4266	4486	4211	4541	1.26%	2.51%	3.77%	4157	4595
Fe ₂ O ₃ , wt.%	46.09	0.773	44.54	47.64	43.77	48.41	1.68%	3.35%	5.03%	43.79	48.39
La ₂ O ₃ , ppm	3191	64	3062	3319	2998	3383	2.01%	4.02%	6.03%	3031	3350
Nd ₂ O ₃ , ppm	1928	133	1663	2194	1530	2326	6.88%	13.77%	20.65%	1832	2025
Pr ₆ O ₁₁ , ppm	585	103	378	791	275	895	17.68%	35.36%	53.04%	555	614
ThO ₂ , ppm	265	52	161	369	109	420	19.60%	39.20%	58.80%	252	278
Thermogravimetry											
LOI, wt.%	0.996	0.175	0.646	1.346	0.471	1.521	17.56%	35.13%	52.69%	0.947	1.046
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)											
Al, wt.%	5.99	0.208	5.57	6.40	5.36	6.61	3.47%	6.94%	10.41%	5.69	6.29
Al ₂ O ₃ , wt.%	11.31	0.393	10.53	12.10	10.13	12.49	3.47%	6.94%	10.41%	10.74	11.88
Ba, ppm	929	55	818	1039	763	1094	5.94%	11.89%	17.83%	882	975
BaO, ppm	1037	62	914	1160	852	1222	5.94%	11.89%	17.83%	985	1089
Be, ppm	2.30	0.38	1.55	3.05	1.17	3.43	16.33%	32.67%	49.00%	2.19	2.42
Bi, ppm	2.49	0.39	1.71	3.26	1.33	3.65	15.56%	31.12%	46.68%	2.36	2.61
Ca, wt.%	1.26	0.057	1.14	1.37	1.09	1.43	4.55%	9.11%	13.66%	1.20	1.32
CaO, wt.%	1.76	0.080	1.60	1.92	1.52	2.00	4.55%	9.11%	13.66%	1.67	1.85

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
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
Ce, ppm	3510	132	3246	3774	3114	3906	3.76%	7.52%	11.28%	3334	3685
CeO ₂ , ppm	4311	162	3987	4636	3825	4798	3.76%	7.52%	11.28%	4096	4527
Co, ppm	11.5	1.8	8.0	15.1	6.2	16.8	15.39%	30.77%	46.16%	10.9	12.1
Cr, ppm	599	40	518	680	478	720	6.76%	13.51%	20.27%	569	629
Cr ₂ O ₃ , ppm	875	59	757	994	698	1053	6.76%	13.51%	20.27%	832	919
Cs, ppm	0.79	0.059	0.67	0.90	0.61	0.96	7.51%	15.01%	22.52%	0.75	0.83
Cu, ppm	60	20	21	100	1	119	32.85%	65.71%	98.56%	57	63
Dy, ppm	34.8	1.44	31.9	37.7	30.5	39.2	4.15%	8.29%	12.44%	33.1	36.6
Dy ₂ O ₃ , ppm	40.0	1.66	36.7	43.3	35.0	44.9	4.15%	8.29%	12.44%	38.0	42.0
Er, ppm	8.80	0.296	8.21	9.39	7.91	9.69	3.36%	6.73%	10.09%	8.36	9.24
Er ₂ O ₃ , ppm	10.1	0.34	9.4	10.7	9.1	11.1	3.36%	6.73%	10.09%	9.6	10.6
Eu, ppm	46.7	2.19	42.3	51.1	40.1	53.3	4.69%	9.37%	14.06%	44.4	49.1
Eu ₂ O ₃ , ppm	54	2.5	49	59	46	62	4.69%	9.37%	14.06%	51	57
Fe, wt.%	31.77	1.301	29.17	34.38	27.87	35.68	4.10%	8.19%	12.29%	30.18	33.36
Fe ₂ O ₃ , wt.%	45.43	1.860	41.71	49.15	39.85	51.01	4.10%	8.19%	12.29%	43.16	47.70
Ga, ppm	50	6	38	62	33	68	11.65%	23.29%	34.94%	48	53
Gd, ppm	100	8	84	117	76	125	8.04%	16.07%	24.11%	95	105
Gd ₂ O ₃ , ppm	116	9	97	134	88	144	8.04%	16.07%	24.11%	110	121
Hf, ppm	14.1	0.61	12.9	15.3	12.3	15.9	4.31%	8.61%	12.92%	13.4	14.8
HfO ₂ , ppm	16.6	0.72	15.2	18.1	14.5	18.8	4.31%	8.61%	12.92%	15.8	17.5
Ho, ppm	4.56	0.306	3.95	5.17	3.64	5.48	6.71%	13.42%	20.13%	4.33	4.79
Ho ₂ O ₃ , ppm	5.22	0.350	4.52	5.92	4.17	6.27	6.71%	13.42%	20.13%	4.96	5.48
In, ppm	0.60	0.08	0.45	0.75	0.37	0.83	12.64%	25.27%	37.91%	0.57	0.63
K, wt.%	0.237	0.033	0.170	0.303	0.137	0.336	14.01%	28.02%	42.02%	0.225	0.248
K ₂ O, wt.%	0.285	0.040	0.205	0.365	0.165	0.405	14.01%	28.02%	42.02%	0.271	0.299
La, ppm	2690	158	2375	3005	2218	3163	5.86%	11.71%	17.57%	2556	2825
La ₂ O ₃ , ppm	3155	185	2785	3524	2601	3709	5.86%	11.71%	17.57%	2997	3313
Li, ppm	12.0	1.5	8.9	15.1	7.4	16.6	12.77%	25.53%	38.30%	11.4	12.6
Lu, ppm	0.52	0.044	0.43	0.60	0.38	0.65	8.49%	16.99%	25.48%	0.49	0.54
Lu ₂ O ₃ , ppm	0.59	0.050	0.49	0.69	0.44	0.74	8.49%	16.99%	25.48%	0.56	0.62
Mg, wt.%	1.05	0.051	0.94	1.15	0.89	1.20	4.91%	9.82%	14.73%	0.99	1.10
MgO, wt.%	1.74	0.085	1.57	1.91	1.48	1.99	4.91%	9.82%	14.73%	1.65	1.82

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
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
Mn, ppm	653	49	555	750	506	799	7.49%	14.97%	22.46%	620	685
MnO, ppm	843	63	717	969	653	1032	7.49%	14.97%	22.46%	801	885
Mo, ppm	45.7	4.8	36.0	55.3	31.2	60.2	10.58%	21.16%	31.74%	43.4	48.0
Nb, ppm	1296	98	1101	1492	1003	1589	7.53%	15.06%	22.60%	1232	1361
Nb ₂ O ₅ , ppm	1854	140	1575	2134	1435	2274	7.53%	15.06%	22.60%	1762	1947
Nd, ppm	1629	54	1520	1737	1466	1792	3.33%	6.67%	10.00%	1547	1710
Nd ₂ O ₃ , ppm	1900	63	1773	2026	1710	2090	3.33%	6.67%	10.00%	1805	1995
Ni, ppm	64	5.5	53	75	47	80	8.59%	17.18%	25.76%	61	67
NiO, ppm	81	7.0	67	95	60	102	8.59%	17.18%	25.76%	77	85
P, wt.%	0.359	0.019	0.321	0.397	0.302	0.416	5.31%	10.62%	15.93%	0.341	0.377
P ₂ O ₅ , wt.%	0.822	0.044	0.735	0.909	0.691	0.953	5.31%	10.62%	15.93%	0.781	0.863
Pb, ppm	106	6	94	119	87	125	5.94%	11.87%	17.81%	101	112
PbO, ppm	114	7	101	128	94	135	5.94%	11.87%	17.81%	109	120
Pr, ppm	489	21	447	530	426	551	4.28%	8.56%	12.84%	464	513
Pr ₆ O ₁₁ , ppm	590	25	540	641	514	666	4.28%	8.56%	12.84%	561	620
Rb, ppm	13.5	0.54	12.4	14.6	11.9	15.1	4.01%	8.03%	12.04%	12.8	14.2
S, ppm	518	82	353	682	271	764	15.87%	31.75%	47.62%	492	544
Sb, ppm	2.76	0.220	2.32	3.20	2.10	3.42	7.96%	15.92%	23.89%	2.62	2.90
Si, wt.%	14.71	0.360	13.99	15.43	13.63	15.79	2.45%	4.89%	7.34%	13.97	15.44
SiO ₂ , wt.%	31.46	0.770	29.92	33.00	29.15	33.77	2.45%	4.89%	7.34%	29.89	33.04
Sm, ppm	220	7	205	234	198	242	3.35%	6.70%	10.05%	209	231
Sm ₂ O ₃ , ppm	255	9	238	272	229	280	3.35%	6.70%	10.05%	242	267
Sn, ppm	25.6	3.3	18.9	32.3	15.6	35.7	13.06%	26.11%	39.17%	24.3	26.9
SnO ₂ , ppm	32.5	4.2	24.0	41.0	19.8	45.3	13.06%	26.11%	39.17%	30.9	34.2
Sr, ppm	579	15	548	610	533	625	2.65%	5.29%	7.94%	550	608
SrO, ppm	685	18	648	721	630	739	2.65%	5.29%	7.94%	650	719
Ta, ppm	25.1	1.04	23.1	27.2	22.0	28.3	4.14%	8.28%	12.42%	23.9	26.4
Ta ₂ O ₅ , ppm	30.7	1.27	28.1	33.2	26.9	34.5	4.14%	8.28%	12.42%	29.2	32.2
Tb, ppm	9.08	0.305	8.46	9.69	8.16	9.99	3.36%	6.73%	10.09%	8.62	9.53
Tb ₄ O ₇ , ppm	10.7	0.36	10.0	11.4	9.6	11.8	3.36%	6.73%	10.09%	10.1	11.2
Th, ppm	210	7	196	223	189	230	3.25%	6.49%	9.74%	199	220
ThO ₂ , ppm	239	8	223	254	216	262	3.25%	6.49%	9.74%	227	251

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
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)											
Ti, wt.%	1.84	0.055	1.73	1.95	1.67	2.01	2.99%	5.99%	8.98%	1.75	1.93
TiO ₂ , wt.%	3.07	0.092	2.89	3.25	2.79	3.34	2.99%	5.99%	8.98%	2.92	3.22
Tm, ppm	0.89	0.085	0.72	1.06	0.63	1.15	9.61%	19.21%	28.82%	0.84	0.93
Tm ₂ O ₃ , ppm	1.02	0.098	0.82	1.21	0.72	1.31	9.61%	19.21%	28.82%	0.97	1.07
U, ppm	4.79	0.195	4.40	5.18	4.20	5.37	4.07%	8.14%	12.22%	4.55	5.03
U ₃ O ₈ , ppm	5.65	0.230	5.19	6.11	4.96	6.34	4.07%	8.14%	12.22%	5.36	5.93
V, ppm	385	16	352	418	335	434	4.28%	8.57%	12.85%	365	404
V ₂ O ₅ , ppm	687	29	628	745	598	775	4.28%	8.57%	12.85%	652	721
W, ppm	3.88	0.67	2.54	5.21	1.87	5.88	17.24%	34.48%	51.71%	3.68	4.07
WO ₃ , ppm	4.89	0.84	3.20	6.58	2.36	7.42	17.24%	34.48%	51.71%	4.65	5.13
Y, ppm	91	4.4	82	100	78	104	4.79%	9.58%	14.37%	86	96
Y ₂ O ₃ , ppm	116	6	104	127	99	132	4.79%	9.58%	14.37%	110	121
Yb, ppm	4.39	0.248	3.90	4.89	3.65	5.14	5.64%	11.28%	16.91%	4.17	4.61
Yb ₂ O ₃ , ppm	5.00	0.282	4.44	5.57	4.16	5.85	5.64%	11.28%	16.91%	4.75	5.25
Zn, ppm	162	23	115	208	92	232	14.44%	28.88%	43.32%	154	170
ZnO, ppm	201	29	143	259	114	289	14.44%	28.88%	43.32%	191	211
Zr, ppm	603	27	549	657	522	684	4.46%	8.92%	13.38%	573	633
ZrO ₂ , ppm	815	36	742	887	706	924	4.46%	8.92%	13.38%	774	855
4-Acid Digestion											
Ag, ppm	< 2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	5.96	0.180	5.60	6.32	5.42	6.50	3.02%	6.05%	9.07%	5.66	6.26
As, ppm	33.6	1.09	31.4	35.7	30.3	36.8	3.23%	6.47%	9.70%	31.9	35.2
Ba, ppm	957	44	869	1045	825	1089	4.60%	9.21%	13.81%	909	1005
Be, ppm	2.09	0.24	1.60	2.58	1.36	2.82	11.65%	23.31%	34.96%	1.99	2.20
Bi, ppm	2.43	0.158	2.11	2.74	1.95	2.90	6.52%	13.04%	19.56%	2.31	2.55
Ca, wt.%	1.21	0.090	1.03	1.40	0.94	1.49	7.44%	14.87%	22.31%	1.15	1.28
Cd, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ce, ppm	3508	112	3285	3732	3173	3844	3.19%	6.38%	9.57%	3333	3684
Co, ppm	12.1	0.44	11.2	13.0	10.8	13.5	3.66%	7.33%	10.99%	11.5	12.7
Cr, ppm	524	73	377	670	304	743	13.96%	27.92%	41.88%	497	550
Cs, ppm	0.77	0.067	0.63	0.90	0.57	0.97	8.71%	17.42%	26.14%	0.73	0.81
Cu, ppm	56	2.3	51	60	49	63	4.10%	8.20%	12.30%	53	59

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											
Dy, ppm	33.5	0.86	31.8	35.2	30.9	36.1	2.57%	5.14%	7.70%	31.8	35.2
Er, ppm	7.46	0.253	6.95	7.96	6.70	8.22	3.39%	6.77%	10.16%	7.09	7.83
Eu, ppm	47.6	3.26	41.1	54.2	37.9	57.4	6.85%	13.69%	20.54%	45.3	50.0
Fe, wt.%	31.52	0.959	29.60	33.44	28.64	34.40	3.04%	6.09%	9.13%	29.94	33.09
Ga, ppm	45.5	8.0	29.5	61.6	21.4	69.6	17.63%	35.26%	52.90%	43.2	47.8
Gd, ppm	98	3.5	91	105	87	108	3.57%	7.14%	10.71%	93	102
Hf, ppm	7.39	0.84	5.71	9.07	4.87	9.91	11.36%	22.71%	34.07%	7.02	7.76
Ho, ppm	4.06	0.115	3.83	4.29	3.71	4.40	2.83%	5.66%	8.48%	3.85	4.26
In, ppm	0.57	0.027	0.51	0.62	0.49	0.65	4.76%	9.53%	14.29%	0.54	0.60
K, wt.%	0.233	0.006	0.221	0.245	0.215	0.251	2.56%	5.13%	7.69%	0.221	0.245
La, ppm	2476	228	2021	2932	1793	3160	9.20%	18.41%	27.61%	2353	2600
Li, ppm	12.0	1.4	9.3	14.8	7.9	16.2	11.45%	22.90%	34.35%	11.4	12.6
Lu, ppm	0.39	0.07	0.25	0.52	0.19	0.58	17.01%	34.03%	51.04%	0.37	0.41
Mg, wt.%	0.999	0.108	0.783	1.215	0.675	1.323	10.81%	21.62%	32.43%	0.949	1.049
Mn, ppm	547	80	388	707	308	787	14.58%	29.16%	43.73%	520	575
Mo, ppm	48.2	2.54	43.2	53.3	40.6	55.9	5.26%	10.51%	15.77%	45.8	50.7
Na, wt.%	0.178	0.013	0.152	0.204	0.139	0.217	7.30%	14.60%	21.91%	0.169	0.187
Nd, ppm	1623	80	1464	1783	1385	1862	4.90%	9.80%	14.69%	1542	1705
Ni, ppm	71	3.1	65	77	62	81	4.42%	8.84%	13.26%	68	75
P, wt.%	0.338	0.022	0.294	0.381	0.273	0.403	6.42%	12.84%	19.26%	0.321	0.355
Pb, ppm	104	5	93	115	87	120	5.28%	10.55%	15.83%	99	109
Pr, ppm	482	20	442	523	422	543	4.17%	8.34%	12.51%	458	506
Rb, ppm	13.3	0.99	11.3	15.3	10.3	16.3	7.45%	14.90%	22.35%	12.6	14.0
Re, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, ppm	475	49	378	573	330	621	10.23%	20.46%	30.68%	452	499
Sb, ppm	2.42	0.174	2.07	2.76	1.90	2.94	7.18%	14.36%	21.54%	2.30	2.54
Sc, ppm	38.8	2.50	33.8	43.9	31.3	46.4	6.45%	12.90%	19.34%	36.9	40.8
Sm, ppm	208	7	193	223	186	230	3.56%	7.13%	10.69%	198	219
Sn, ppm	21.6	2.08	17.5	25.8	15.4	27.9	9.63%	19.25%	28.88%	20.6	22.7
Sr, ppm	564	35	494	634	459	670	6.23%	12.47%	18.70%	536	592
Ta, ppm	20.9	1.96	17.0	24.9	15.1	26.8	9.36%	18.73%	28.09%	19.9	22.0
Tb, ppm	9.09	0.503	8.09	10.10	7.59	10.60	5.53%	11.06%	16.59%	8.64	9.55

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											
Te, ppm	0.34	0.06	0.23	0.45	0.17	0.51	16.29%	32.57%	48.86%	0.32	0.36
Th, ppm	207	10	187	226	178	235	4.65%	9.30%	13.95%	196	217
Ti, wt. %	1.06	0.12	0.82	1.29	0.71	1.41	11.08%	22.16%	33.24%	1.00	1.11
Tl, ppm	0.10	0.008	0.09	0.12	0.08	0.13	8.15%	16.30%	24.44%	0.10	0.11
Tm, ppm	0.70	0.046	0.61	0.79	0.56	0.84	6.59%	13.19%	19.78%	0.67	0.74
U, ppm	4.40	0.161	4.08	4.72	3.92	4.88	3.66%	7.32%	10.98%	4.18	4.62
V, ppm	352	29	295	410	266	438	8.17%	16.34%	24.51%	334	370
W, ppm	2.50	0.36	1.78	3.22	1.41	3.58	14.45%	28.91%	43.36%	2.37	2.62
Y, ppm	83	3.7	76	91	72	94	4.51%	9.02%	13.53%	79	87
Yb, ppm	3.06	0.149	2.76	3.36	2.62	3.51	4.87%	9.75%	14.62%	2.91	3.22
Zn, ppm	139	13	113	166	99	179	9.54%	19.07%	28.61%	132	146
Zr, ppm	270	18	234	306	216	324	6.68%	13.36%	20.04%	256	283

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 3090 and 3220 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 461 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 115 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 461 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 461 has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd	Tel: +613-9729 0333
37A Hosie Street	Fax: +613-9729 8338
Bayswater North VIC 3153	Web: www.ore.com.au
AUSTRALIA	Email: info@ore.com.au

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

INTENDED USE

OREAS 461 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 461 has been prepared from a blend of barren weathered siltstone and low grade/waste REE bearing ore (TREO = 1.06%). 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 461 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.



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.

CERTIFYING OFFICER



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

REFERENCES

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