

CERTIFICATE OF ANALYSIS FOR

CARBONATITE SUPERGENE REE-Nb ORE (TREO 5.10%)

CERTIFIED REFERENCE MATERIAL

OREAS 464

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 (wt.%)	1.88	0.065	1.84	1.92	1.83	1.92
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	204	9	199	210	198	210
Er ₂ O ₃ , Erbium(III) oxide (ppm)	43.6	1.49	42.8	44.5	42.0	45.2
Eu ₂ O ₃ , Europium(III) oxide (ppm)	375	10	371	379	364	386
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	779	35	758	801	762	797
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	24.4	1.54	23.5	25.3	23.7	25.1
La ₂ O ₃ , Lanthanum(III) oxide (wt.%)	1.37	0.037	1.36	1.39	1.34	1.41
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	1.92	0.112	1.87	1.97	1.79	2.05
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2723	115	2637	2809	2612	2833
Nd ₂ O ₃ , Neodymium(III) oxide (wt.%)	1.16	0.037	1.14	1.18	1.13	1.18
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	3138	128	3063	3213	3079	3197
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	1738	44	1719	1756	1677	1798
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	63	3.2	61	65	61	65
ThO ₂ , Thorium dioxide (ppm)	600	27	585	615	584	616
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	4.06	0.199	3.95	4.18	3.93	4.20
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	20.7	0.49	20.5	21.0	20.0	21.4
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	570	27	553	586	550	589
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	17.9	1.13	17.4	18.5	17.2	18.7
ZrO ₂ , Zirconium dioxide (ppm)	624	23	605	642	586	661

Note: intervals may appear asymmetric due to rounding.



Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 464.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO ₂ , Cerium(IV) oxide (wt.%)	1.89	0.027	1.87	1.92	1.87	1.92
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	54.38	1.031	52.99	55.77	53.94	54.82
La ₂ O ₃ , Lanthanum(III) oxide (wt.%)	1.41	0.020	1.39	1.42	1.38	1.43
Nd ₂ O ₃ , Neodymium(III) oxide (wt.%)	1.12	0.036	1.08	1.16	1.10	1.15
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	3070	99	2987	3152	2907	3232
Thermogravimetry						
LOI, Loss On Ignition @1000°C (wt.%)	0.697	0.071	0.608	0.786	0.652	0.742
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	4.63	0.150	4.52	4.74	4.53	4.73
Al ₂ O ₃ , Aluminium(III) oxide (wt.%)	8.75	0.284	8.54	8.96	8.57	8.93
Ba, Barium (ppm)	1657	69	1614	1699	1613	1701
BaO, Barium oxide (ppm)	1850	77	1802	1897	1801	1899
Be, Beryllium (ppm)	15.6	1.6	14.1	17.1	IND	IND
Bi, Bismuth (ppm)	3.80	0.43	3.34	4.25	IND	IND
Ca, Calcium (wt.%)	0.896	0.033	0.875	0.917	0.875	0.917
CaO, Calcium oxide (wt.%)	1.25	0.046	1.22	1.28	1.22	1.28
Ce, Cerium (wt.%)	1.53	0.053	1.50	1.56	1.49	1.57
CeO ₂ , Cerium(IV) oxide (wt.%)	1.88	0.065	1.84	1.92	1.83	1.92
Co, Cobalt (ppm)	18.0	3.4	14.8	21.3	15.6	20.5
Cr, Chromium (ppm)	395	22	381	409	381	409
Cr ₂ O ₃ , Chromium(III) oxide (ppm)	577	32	557	597	557	598
Cs, Cesium (ppm)	0.27	0.04	0.24	0.30	0.21	0.34
Dy, Dysprosium (ppm)	178	8	173	183	173	183
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	204	9	199	210	198	210
Er, Erbium (ppm)	38.2	1.30	37.4	38.9	36.8	39.6
Er ₂ O ₃ , Erbium(III) oxide (ppm)	43.6	1.49	42.8	44.5	42.0	45.2
Eu, Europium (ppm)	324	8	320	328	314	333
Eu ₂ O ₃ , Europium(III) oxide (ppm)	375	10	371	379	364	386
Fe, Iron (wt.%)	37.24	1.007	36.47	38.00	36.38	38.10
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	53.24	1.440	52.15	54.33	52.01	54.47
Ga, Gallium (ppm)	106	21	80	132	101	111
Gd, Gadolinium (ppm)	676	30	657	695	661	691
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	779	35	758	801	762	797
Hf, Hafnium (ppm)	12.5	0.77	12.1	13.0	11.8	13.2
HfO ₂ , Hafnium dioxide (ppm)	14.8	0.91	14.2	15.3	14.0	15.5
Ho, Holmium (ppm)	21.3	1.34	20.5	22.1	20.7	21.9
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	24.4	1.54	23.5	25.3	23.7	25.1
In, Indium (ppm)	2.14	0.163	2.00	2.28	IND	IND
K, Potassium (wt.%)	0.086	0.016	0.069	0.103	IND	IND
K ₂ O, Potassium oxide (wt.%)	0.104	0.019	0.083	0.125	IND	IND
La, Lanthanum (wt.%)	1.17	0.032	1.16	1.18	1.14	1.20
La ₂ O ₃ , Lanthanum(III) oxide (wt.%)	1.37	0.037	1.36	1.39	1.34	1.41

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
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)						
Lu, Lutetium (ppm)	1.69	0.098	1.64	1.73	1.57	1.80
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	1.92	0.112	1.87	1.97	1.79	2.05
Mg, Magnesium (wt.%)	0.846	0.038	0.818	0.873	0.833	0.858
MgO, Magnesium oxide (wt.%)	1.40	0.063	1.36	1.45	1.38	1.42
Mn, Manganese (wt.%)	0.282	0.019	0.267	0.298	0.272	0.292
MnO, Manganese oxide (wt.%)	0.365	0.025	0.345	0.384	0.352	0.378
Mo, Molybdenum (ppm)	77	4.3	73	81	74	81
Nb, Niobium (ppm)	1903	80	1843	1964	1826	1981
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2723	115	2637	2809	2612	2833
Nd, Neodymium (wt.%)	0.994	0.032	0.978	1.010	0.972	1.016
Nd ₂ O ₃ , Neodymium(III) oxide (wt.%)	1.16	0.037	1.14	1.18	1.13	1.18
Ni, Nickel (ppm)	82	16	67	97	67	97
NiO, Nickel oxide (ppm)	104	20	86	123	85	124
P, Phosphorus (wt.%)	1.41	0.045	1.37	1.44	1.37	1.44
P ₂ O ₅ , Phosphorus(V) oxide (wt.%)	3.23	0.103	3.15	3.31	3.15	3.30
Pb, Lead (ppm)	174	12	160	188	167	180
PbO, Lead oxide (ppm)	187	13	172	202	179	194
Pr, Praseodymium (ppm)	2597	106	2535	2659	2548	2646
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	3138	128	3063	3213	3079	3197
Rb, Rubidium (ppm)	4.39	0.46	4.17	4.60	4.09	4.68
S, Sulphur (ppm)	1028	91	940	1117	IND	IND
Sb, Antimony (ppm)	1.80	0.25	1.49	2.11	IND	IND
Si, Silicon (wt.%)	9.52	0.294	9.21	9.83	9.33	9.71
SiO ₂ , Silicon dioxide (wt.%)	20.36	0.629	19.69	21.03	19.95	20.78
Sm, Samarium (ppm)	1498	38	1482	1515	1446	1550
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	1738	44	1719	1756	1677	1798
Sn, Tin (ppm)	45.9	2.68	44.3	47.4	43.9	47.8
SnO ₂ , Tin dioxide (ppm)	58	3.4	56	60	56	61
Sr, Strontium (ppm)	2060	69	2019	2102	2016	2105
SrO, Strontium oxide (ppm)	2437	82	2387	2486	2384	2489
Ta, Tantalum (ppm)	24.3	3.5	22.0	26.6	21.8	26.8
Ta ₂ O ₅ , Tantalum(V) oxide (ppm)	29.7	4.2	26.9	32.5	26.6	32.8
Tb, Terbium (ppm)	54	2.7	52	55	52	55
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	63	3.2	61	65	61	65
Th, Thorium (ppm)	527	24	514	541	513	542
ThO ₂ , Thorium dioxide (ppm)	600	27	585	615	584	616
Ti, Titanium (wt.%)	1.95	0.059	1.91	2.00	1.91	2.00
TiO ₂ , Titanium dioxide (wt.%)	3.26	0.099	3.18	3.33	3.19	3.33
Tm, Thulium (ppm)	3.56	0.175	3.46	3.66	3.44	3.67
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	4.06	0.199	3.95	4.18	3.93	4.20
U, Uranium (ppm)	17.6	0.42	17.4	17.8	17.0	18.1
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	20.7	0.49	20.5	21.0	20.0	21.4

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, Vanadium (ppm)	231	19	220	242	222	240
V ₂ O ₅ , Vanadium(V) oxide (ppm)	412	34	393	432	396	429
W, Tungsten (ppm)	< 6	IND	IND	IND	IND	IND
WO ₃ , Tungsten trioxide (ppm)	< 7	IND	IND	IND	IND	IND
Y, Yttrium (ppm)	449	21	435	462	433	464
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	570	27	553	586	550	589
Yb, Ytterbium (ppm)	15.7	0.99	15.3	16.2	15.1	16.4
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	17.9	1.13	17.4	18.5	17.2	18.7
Zn, Zinc (ppm)	1232	96	1106	1358	1186	1277
ZnO, Zinc oxide (ppm)	1533	119	1376	1690	1476	1590
Zr, Zirconium (ppm)	462	17	448	475	434	490
ZrO ₂ , Zirconium dioxide (ppm)	624	23	605	642	586	661
4-Acid Digestion						
Ag, Silver (ppm)	4.4	0.36	4.2	4.7	4.1	4.8
Al, Aluminium (wt.%)	4.47	0.096	4.42	4.52	4.38	4.56
As, Arsenic (ppm)	32.9	4.7	29.0	36.8	31.0	34.9
Ba, Barium (ppm)	1648	71.6	1609	1687	1617	1679
Be, Beryllium (ppm)	15.2	1.19	14.4	16.0	14.8	15.7
Bi, Bismuth (ppm)	4.05	0.236	3.90	4.21	3.94	4.17
Ca, Calcium (wt.%)	0.851	0.059	0.811	0.891	0.835	0.867
Cd, Cadmium (ppm)	0.52	0.09	0.46	0.58	0.47	0.56
Ce, Cerium (wt.%)	1.55	0.048	1.50	1.60	1.50	1.59
Co, Cobalt (ppm)	17.0	0.93	16.6	17.4	16.4	17.6
Cr, Chromium (ppm)	335	37	303	367	319	351
Cs, Cesium (ppm)	0.29	0.018	0.28	0.30	0.27	0.31
Cu, Copper (ppm)	92	2.2	90	93	89	94
Dy, Dysprosium (ppm)	182	6.9	177	186	179	185
Er, Erbium (ppm)	36.9	1.55	35.9	38.0	36.1	37.8
Eu, Europium (ppm)	334	18.9	322	347	329	340
Fe, Iron (wt.%)	35.55	1.569	34.62	36.48	35.01	36.09
Gd, Gadolinium (ppm)	670	26.4	651	689	658	682
Hf, Hafnium (ppm)	7.79	1.32	6.74	8.85	7.35	8.23
Ho, Holmium (ppm)	21.3	0.93	20.7	21.9	20.8	21.8
In, Indium (ppm)	2.11	0.118	2.04	2.19	2.06	2.16
K, Potassium (wt.%)	0.081	0.014	0.072	0.090	IND	IND
La, Lanthanum (wt.%)	1.06	0.21	0.86	1.27	1.03	1.10
Li, Lithium (ppm)	8.26	1.07	7.58	8.93	7.91	8.60
Lu, Lutetium (ppm)	1.57	0.30	1.38	1.77	1.51	1.63
Mg, Magnesium (wt.%)	0.825	0.045	0.795	0.855	0.809	0.841
Mn, Manganese (wt.%)	0.270	0.016	0.258	0.282	0.263	0.277
Mo, Molybdenum (ppm)	77	4.4	74	80	75	79
Na, Sodium (wt.%)	0.149	0.011	0.142	0.155	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 (wt.%)	0.959	0.026	0.942	0.976	0.941	0.977
Ni, Nickel (ppm)	89	6.5	84	93	86	91
P, Phosphorus (wt.%)	1.36	0.067	1.31	1.41	1.32	1.40
Pb, Lead (ppm)	205	32	184	226	199	210
Pr, Praseodymium (ppm)	2601	86.8	2516	2685	2524	2678
Rb, Rubidium (ppm)	4.22	0.393	3.97	4.47	4.02	4.42
Re, Rhenium (ppm)	< 0.03	IND	IND	IND	IND	IND
S, Sulphur (ppm)	983	78.8	934	1033	IND	IND
Sb, Antimony (ppm)	1.87	0.111	1.80	1.94	1.75	1.99
Sc, Scandium (ppm)	141	5.8	137	145	138	145
Se, Selenium (ppm)	< 50	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	1485	49.4	1438	1532	1437	1533
Sn, Tin (ppm)	35.7	3.21	33.5	37.8	34.3	37.1
Sr, Strontium (ppm)	2043	144.3	1944	2142	2010	2075
Ta, Tantalum (ppm)	20.9	2.7	18.6	23.2	19.8	22.0
Tb, Terbium (ppm)	55	2.5	53	57	53	56
Te, Tellurium (ppm)	0.36	0.07	0.30	0.42	0.33	0.40
Th, Thorium (ppm)	506	60	459	554	496	517
Ti, Titanium (wt.%)	< 1	IND	IND	IND	IND	IND
Tl, Thallium (ppm)	0.15	0.013	0.14	0.15	IND	IND
Tm, Thulium (ppm)	3.30	0.321	3.08	3.51	3.19	3.40
U, Uranium (ppm)	17.6	0.61	17.2	18.0	17.2	18.1
V, Vanadium (ppm)	207	26	188	226	203	212
W, Tungsten (ppm)	2.37	0.40	2.10	2.64	2.24	2.51
Y, Yttrium (ppm)	449	33.3	425	473	443	455
Yb, Ytterbium (ppm)	14.3	0.94	13.6	15.0	13.8	14.8
Zn, Zinc (ppm)	1128	56.8	1090	1166	1110	1146
Zr, Zirconium (ppm)	210	40	179	240	200	219

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 464.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al ₂ O ₃	wt.%	8.87	Lu ₂ O ₃	ppm	< 20	SrO	ppm	2367
BaO	ppm	2025	MgO	wt.%	1.42	Ta ₂ O ₅	ppm	< 100
CaO	wt.%	1.28	MnO	wt.%	0.395	Tb ₄ O ₇	ppm	58
Cr ₂ O ₃	ppm	476	Na ₂ O	wt.%	0.235	TiO ₂	wt.%	3.29
Dy ₂ O ₃	ppm	198	Nb ₂ O ₅	ppm	2773	Tm ₂ O ₃	ppm	< 10
Er ₂ O ₃	ppm	40.0	NiO	ppm	200	U ₃ O ₈	ppm	< 100
Eu ₂ O ₃	ppm	333	P ₂ O ₅	wt.%	3.25	V ₂ O ₅	ppm	460
Gd ₂ O ₃	ppm	804	PbO	ppm	300	WO ₃	ppm	< 100
HfO ₂	ppm	< 100	SiO ₂	wt.%	20.01	Yb ₂ O ₃	ppm	20.0
Ho ₂ O ₃	ppm	20.0	SnO ₂	ppm	< 100	ZnO	ppm	1567
K ₂ O	wt.%	0.103	SO ₃	wt.%	0.248	ZrO ₂	ppm	617

Table 2 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Thermogravimetry								
H ₂ O-	wt.%	0.503						
Peroxide Fusion ICP								
Ag	ppm	10.4	Ge	ppm	43.3	Se	ppm	40.0
As	ppm	252	Li	ppm	8.13	Te	ppm	< 1
B	ppm	108	Na	wt.%	0.135	Tl	ppm	< 0.5
Cd	ppm	< 1	Re	ppm	< 0.1			
Cu	ppm	104	Sc	ppm	152			
4-Acid Digestion								
Ga	ppm	97	Ge	ppm	18.4	Nb	ppm	1598

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 464 is a high grade ore, rare earth element (TREO = 5.10%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 464 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 464 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 115 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%: 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%).

COMMINUTION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 464 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 464. 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 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 (PDM³) are presented in the detailed certification data for this CRM (**OREAS 464 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 464.

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 , wt.%	1.89	0.027	1.84	1.95	1.81	1.98	1.44%	2.87%	4.31%	1.80	1.99
Fe ₂ O ₃ , wt.%	54.38	1.031	52.32	56.44	51.29	57.47	1.90%	3.79%	5.69%	51.66	57.10
La ₂ O ₃ , wt.%	1.41	0.020	1.37	1.45	1.35	1.47	1.40%	2.81%	4.21%	1.34	1.48
Nd ₂ O ₃ , wt.%	1.12	0.036	1.05	1.20	1.02	1.23	3.21%	6.43%	9.64%	1.07	1.18
Pr ₆ O ₁₁ , ppm	3070	99	2873	3267	2774	3365	3.21%	6.42%	9.63%	2916	3223
Sm ₂ O ₃ , ppm	1635	55	1524	1745	1469	1800	3.38%	6.76%	10.14%	1553	1716
ThO ₂ , ppm	576	84	407	745	322	829	14.67%	29.34%	44.01%	547	605
Y ₂ O ₃ , ppm	562	69	424	699	355	768	12.24%	24.49%	36.73%	534	590
Thermogravimetry											
LOI, wt.%	0.697	0.071	0.555	0.839	0.484	0.910	10.20%	20.40%	30.61%	0.662	0.732
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)											
Al, wt.%	4.63	0.150	4.33	4.93	4.18	5.08	3.25%	6.49%	9.74%	4.40	4.86
Al ₂ O ₃ , wt.%	8.75	0.284	8.18	9.32	7.90	9.60	3.25%	6.49%	9.74%	8.31	9.19
Ba, ppm	1657	69	1518	1795	1449	1865	4.18%	8.36%	12.55%	1574	1740
BaO, ppm	1850	77	1695	2004	1618	2082	4.18%	8.36%	12.55%	1757	1942
Be, ppm	15.6	1.6	12.5	18.8	10.9	20.4	10.09%	20.17%	30.26%	14.8	16.4
Bi, ppm	3.80	0.43	2.94	4.65	2.52	5.08	11.23%	22.46%	33.69%	3.61	3.99
Ca, wt.%	0.896	0.033	0.830	0.962	0.796	0.996	3.70%	7.41%	11.11%	0.851	0.941
CaO, wt.%	1.25	0.046	1.16	1.35	1.11	1.39	3.70%	7.41%	11.11%	1.19	1.32

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)											
Ce, wt.%	1.53	0.053	1.42	1.63	1.37	1.69	3.46%	6.91%	10.37%	1.45	1.60
CeO ₂ , wt.%	1.88	0.065	1.75	2.01	1.68	2.07	3.46%	6.91%	10.37%	1.78	1.97
Co, ppm	18.0	3.4	11.2	24.8	7.9	28.2	18.80%	37.59%	56.39%	17.1	18.9
Cr, ppm	395	22	351	439	329	461	5.58%	11.16%	16.75%	375	415
Cr ₂ O ₃ , ppm	577	32	513	642	481	674	5.58%	11.16%	16.75%	549	606
Cs, ppm	0.27	0.04	0.18	0.36	0.14	0.40	16.06%	32.11%	48.17%	0.26	0.29
Dy, ppm	178	8	162	194	154	202	4.51%	9.03%	13.54%	169	187
Dy ₂ O ₃ , ppm	204	9	186	223	177	232	4.51%	9.03%	13.54%	194	215
Er, ppm	38.2	1.30	35.5	40.8	34.2	42.1	3.42%	6.83%	10.25%	36.2	40.1
Er ₂ O ₃ , ppm	43.6	1.49	40.6	46.6	39.2	48.1	3.42%	6.83%	10.25%	41.4	45.8
Eu, ppm	324	8	307	341	299	349	2.58%	5.16%	7.74%	308	340
Eu ₂ O ₃ , ppm	375	10	356	394	346	404	2.58%	5.16%	7.74%	356	394
Fe, wt.%	37.24	1.007	35.22	39.25	34.22	40.26	2.70%	5.41%	8.11%	35.38	39.10
Fe ₂ O ₃ , wt.%	53.24	1.440	50.36	56.12	48.92	57.56	2.70%	5.41%	8.11%	50.58	55.90
Ga, ppm	106	21	63	148	42	169	19.99%	39.99%	59.98%	101	111
Gd, ppm	676	30	616	736	585	767	4.47%	8.94%	13.40%	642	710
Gd ₂ O ₃ , ppm	779	35	710	849	675	884	4.47%	8.94%	13.40%	740	818
Hf, ppm	12.5	0.77	11.0	14.1	10.2	14.8	6.16%	12.31%	18.47%	11.9	13.1
HfO ₂ , ppm	14.8	0.91	12.9	16.6	12.0	17.5	6.16%	12.31%	18.47%	14.0	15.5
Ho, ppm	21.3	1.34	18.6	24.0	17.3	25.3	6.31%	12.63%	18.94%	20.2	22.4
Ho ₂ O ₃ , ppm	24.4	1.54	21.3	27.5	19.8	29.0	6.31%	12.63%	18.94%	23.2	25.6
In, ppm	2.14	0.163	1.81	2.47	1.65	2.63	7.63%	15.26%	22.89%	2.03	2.25
K, wt.%	0.086	0.016	0.055	0.118	0.039	0.134	18.35%	36.69%	55.04%	0.082	0.091
K ₂ O, wt.%	0.104	0.019	0.066	0.142	0.047	0.161	18.35%	36.69%	55.04%	0.099	0.109
La, wt.%	1.17	0.032	1.11	1.23	1.07	1.27	2.73%	5.46%	8.19%	1.11	1.23
La ₂ O ₃ , wt.%	1.37	0.037	1.30	1.45	1.26	1.49	2.73%	5.46%	8.19%	1.30	1.44
Lu, ppm	1.69	0.098	1.49	1.88	1.39	1.98	5.84%	11.68%	17.52%	1.60	1.77
Lu ₂ O ₃ , ppm	1.92	0.112	1.69	2.14	1.58	2.25	5.84%	11.68%	17.52%	1.82	2.01
Mg, wt.%	0.846	0.038	0.770	0.921	0.732	0.959	4.48%	8.97%	13.45%	0.803	0.888
MgO, wt.%	1.40	0.063	1.28	1.53	1.21	1.59	4.48%	8.97%	13.45%	1.33	1.47
Mn, wt.%	0.282	0.019	0.244	0.321	0.225	0.340	6.80%	13.61%	20.41%	0.268	0.297
MnO, wt.%	0.365	0.025	0.315	0.414	0.290	0.439	6.80%	13.61%	20.41%	0.346	0.383

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	77	4.3	69	86	64	90	5.54%	11.08%	16.61%	73	81
Nb, ppm	1903	80	1743	2064	1663	2144	4.22%	8.43%	12.65%	1808	1999
Nb ₂ O ₅ , ppm	2723	115	2493	2952	2378	3067	4.22%	8.43%	12.65%	2587	2859
Nd, wt.%	0.994	0.032	0.931	1.057	0.899	1.089	3.18%	6.36%	9.55%	0.944	1.044
Nd ₂ O ₃ , wt.%	1.16	0.037	1.09	1.23	1.05	1.27	3.18%	6.36%	9.55%	1.10	1.22
Ni, ppm	82	16	51	113	35	129	19.20%	38.40%	57.59%	78	86
NiO, ppm	104	20	64	144	44	164	19.20%	38.40%	57.59%	99	110
P, wt.%	1.41	0.045	1.32	1.50	1.27	1.54	3.21%	6.41%	9.62%	1.34	1.48
P ₂ O ₅ , wt.%	3.23	0.103	3.02	3.43	2.92	3.54	3.21%	6.41%	9.62%	3.07	3.39
Pb, ppm	174	12	149	198	137	211	7.11%	14.22%	21.34%	165	182
PbO, ppm	187	13	160	214	147	227	7.11%	14.22%	21.34%	178	196
Pr, ppm	2597	106	2385	2809	2279	2915	4.08%	8.16%	12.25%	2467	2727
Pr ₆ O ₁₁ , ppm	3138	128	2882	3394	2754	3522	4.08%	8.16%	12.25%	2981	3295
Rb, ppm	4.39	0.46	3.47	5.31	3.01	5.77	10.49%	20.98%	31.48%	4.17	4.61
S, ppm	1028	91	847	1210	756	1300	8.82%	17.64%	26.46%	977	1080
Sb, ppm	1.80	0.25	1.31	2.29	1.06	2.54	13.65%	27.30%	40.95%	1.71	1.89
Si, wt.%	9.52	0.294	8.93	10.11	8.64	10.40	3.09%	6.17%	9.26%	9.04	10.00
SiO ₂ , wt.%	20.36	0.629	19.11	21.62	18.48	22.25	3.09%	6.17%	9.26%	19.35	21.38
Sm, ppm	1498	38	1423	1574	1385	1611	2.52%	5.03%	7.55%	1423	1573
Sm ₂ O ₃ , ppm	1738	44	1650	1825	1606	1869	2.52%	5.03%	7.55%	1651	1824
Sn, ppm	45.9	2.68	40.5	51.2	37.8	53.9	5.84%	11.67%	17.51%	43.6	48.2
SnO ₂ , ppm	58	3.4	51	65	48	68	5.84%	11.67%	17.51%	55	61
Sr, ppm	2060	69	1922	2199	1853	2268	3.36%	6.71%	10.07%	1957	2163
SrO, ppm	2437	82	2273	2600	2191	2682	3.36%	6.71%	10.07%	2315	2558
Ta, ppm	24.3	3.5	17.4	31.3	13.9	34.7	14.26%	28.52%	42.77%	23.1	25.5
Ta ₂ O ₅ , ppm	29.7	4.2	21.2	38.2	17.0	42.4	14.26%	28.52%	42.77%	28.2	31.2
Tb, ppm	54	2.7	48	59	46	62	5.02%	10.03%	15.05%	51	56
Tb ₄ O ₇ , ppm	63	3.2	57	70	54	73	5.02%	10.03%	15.05%	60	66
Th, ppm	527	24	480	575	457	598	4.47%	8.94%	13.40%	501	554
ThO ₂ , ppm	600	27	547	654	520	681	4.47%	8.94%	13.40%	570	630
Ti, wt.%	1.95	0.059	1.84	2.07	1.78	2.13	3.04%	6.08%	9.12%	1.86	2.05
TiO ₂ , wt.%	3.26	0.099	3.06	3.46	2.96	3.56	3.04%	6.08%	9.12%	3.10	3.42

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, ppm	3.56	0.175	3.21	3.91	3.03	4.08	4.91%	9.82%	14.72%	3.38	3.73
Tm ₂ O ₃ , ppm	4.06	0.199	3.66	4.46	3.46	4.66	4.91%	9.82%	14.72%	3.86	4.27
U, ppm	17.6	0.42	16.7	18.4	16.3	18.8	2.38%	4.75%	7.13%	16.7	18.4
U ₃ O ₈ , ppm	20.7	0.49	19.7	21.7	19.2	22.2	2.38%	4.75%	7.13%	19.7	21.8
V, ppm	231	19	193	269	174	288	8.25%	16.50%	24.75%	219	243
V ₂ O ₅ , ppm	412	34	344	480	310	514	8.25%	16.50%	24.75%	392	433
W, ppm	< 6	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
WO ₃ , ppm	< 7	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Y, ppm	449	21	406	491	385	512	4.74%	9.49%	14.23%	426	471
Y ₂ O ₃ , ppm	570	27	516	624	489	651	4.74%	9.49%	14.23%	541	598
Yb, ppm	15.7	0.99	13.7	17.7	12.8	18.7	6.31%	12.63%	18.94%	14.9	16.5
Yb ₂ O ₃ , ppm	17.9	1.13	15.7	20.2	14.5	21.3	6.31%	12.63%	18.94%	17.0	18.8
Zn, ppm	1232	96	1041	1423	945	1518	7.76%	15.52%	23.28%	1170	1293
ZnO, ppm	1533	119	1295	1771	1176	1890	7.76%	15.52%	23.28%	1456	1610
Zr, ppm	462	17	427	496	410	514	3.76%	7.53%	11.29%	439	485
ZrO ₂ , ppm	624	23	577	671	553	694	3.76%	7.53%	11.29%	593	655
4-Acid Digestion											
Ag, ppm	4.43	0.362	3.71	5.15	3.35	5.52	8.17%	16.34%	24.51%	4.21	4.65
Al, wt. %	4.47	0.096	4.28	4.66	4.18	4.76	2.15%	4.31%	6.46%	4.25	4.69
As, ppm	32.9	4.7	23.5	42.4	18.8	47.1	14.32%	28.63%	42.95%	31.3	34.6
Ba, ppm	1648	72	1505	1791	1433	1863	4.34%	8.69%	13.03%	1566	1731
Be, ppm	15.2	1.19	12.9	17.6	11.7	18.8	7.83%	15.66%	23.49%	14.5	16.0
Bi, ppm	4.05	0.236	3.58	4.53	3.35	4.76	5.82%	11.65%	17.47%	3.85	4.26
Ca, wt. %	0.851	0.059	0.734	0.968	0.675	1.027	6.89%	13.78%	20.67%	0.808	0.893
Cd, ppm	0.52	0.09	0.34	0.69	0.26	0.78	16.88%	33.77%	50.65%	0.49	0.54
Ce, wt. %	1.55	0.048	1.45	1.65	1.40	1.69	3.13%	6.25%	9.38%	1.47	1.63
Co, ppm	17.0	0.93	15.1	18.9	14.2	19.8	5.49%	10.97%	16.46%	16.1	17.8
Cr, ppm	335	37	260	409	223	446	11.12%	22.23%	33.35%	318	351
Cs, ppm	0.29	0.018	0.25	0.32	0.23	0.34	6.21%	12.42%	18.63%	0.27	0.30
Cu, ppm	92	2.2	87	96	85	98	2.39%	4.78%	7.16%	87	96
Dy, ppm	182	7	168	196	161	202	3.80%	7.61%	11.41%	173	191
Er, ppm	36.9	1.55	33.8	40.0	32.3	41.6	4.21%	8.41%	12.62%	35.1	38.8

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											
Eu, ppm	334	19	297	372	278	391	5.66%	11.33%	16.99%	318	351
Fe, wt.%	35.55	1.569	32.41	38.69	30.84	40.26	4.41%	8.83%	13.24%	33.77	37.33
Gd, ppm	670	26	617	723	591	749	3.93%	7.87%	11.80%	637	704
Hf, ppm	7.79	1.32	5.15	10.44	3.82	11.76	16.98%	33.96%	50.94%	7.40	8.18
Ho, ppm	21.3	0.93	19.5	23.2	18.5	24.1	4.37%	8.74%	13.11%	20.2	22.4
In, ppm	2.11	0.118	1.88	2.35	1.76	2.47	5.60%	11.19%	16.79%	2.01	2.22
K, wt.%	0.081	0.014	0.053	0.109	0.040	0.123	17.10%	34.21%	51.31%	0.077	0.085
La, wt.%	1.06	0.21	0.64	1.49	0.43	1.70	20.01%	40.02%	60.03%	1.01	1.12
Li, ppm	8.26	1.07	6.12	10.39	5.06	11.46	12.92%	25.84%	38.76%	7.84	8.67
Lu, ppm	1.57	0.30	0.97	2.18	0.67	2.48	19.19%	38.38%	57.58%	1.49	1.65
Mg, wt.%	0.825	0.045	0.736	0.914	0.691	0.959	5.40%	10.80%	16.21%	0.784	0.866
Mn, wt.%	0.270	0.016	0.237	0.303	0.221	0.319	6.04%	12.09%	18.13%	0.256	0.283
Mo, ppm	77	4.4	68	86	64	90	5.70%	11.40%	17.10%	73	81
Na, wt.%	0.149	0.011	0.126	0.171	0.114	0.183	7.66%	15.31%	22.97%	0.141	0.156
Nd, wt.%	0.959	0.026	0.908	1.010	0.882	1.036	2.67%	5.34%	8.01%	0.911	1.007
Ni, ppm	89	6.5	75	102	69	108	7.39%	14.79%	22.18%	84	93
P, wt.%	1.36	0.067	1.23	1.49	1.16	1.56	4.90%	9.81%	14.71%	1.29	1.43
Pb, ppm	205	32	141	268	110	300	15.49%	30.98%	46.47%	195	215
Pr, ppm	2601	87	2427	2774	2340	2861	3.34%	6.68%	10.02%	2471	2731
Rb, ppm	4.22	0.393	3.44	5.01	3.04	5.40	9.30%	18.60%	27.90%	4.01	4.43
Re, ppm	< 0.03	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, ppm	983	79	826	1141	747	1220	8.02%	16.03%	24.05%	934	1033
Sb, ppm	1.87	0.111	1.65	2.09	1.53	2.20	5.95%	11.91%	17.86%	1.77	1.96
Sc, ppm	141	6	130	153	124	159	4.11%	8.22%	12.32%	134	148
Se, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	1485	49	1386	1584	1337	1633	3.33%	6.65%	9.98%	1411	1559
Sn, ppm	35.7	3.21	29.3	42.1	26.1	45.3	8.98%	17.97%	26.95%	33.9	37.5
Sr, ppm	2043	144	1754	2331	1610	2476	7.06%	14.12%	21.19%	1941	2145
Ta, ppm	20.9	2.7	15.5	26.3	12.8	29.0	12.89%	25.77%	38.66%	19.9	22.0
Tb, ppm	55	2.5	50	60	47	63	4.63%	9.26%	13.89%	52	58
Te, ppm	0.36	0.07	0.23	0.50	0.16	0.57	18.73%	37.45%	56.18%	0.34	0.38
Th, ppm	506	60	387	625	328	685	11.75%	23.51%	35.26%	481	532

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.%	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tl, ppm	0.15	0.013	0.12	0.17	0.11	0.18	8.82%	17.63%	26.45%	0.14	0.15
Tm, ppm	3.30	0.321	2.65	3.94	2.33	4.26	9.75%	19.49%	29.24%	3.13	3.46
U, ppm	17.6	0.61	16.4	18.9	15.8	19.5	3.48%	6.97%	10.45%	16.8	18.5
V, ppm	207	26	155	259	129	286	12.64%	25.29%	37.93%	197	217
W, ppm	2.37	0.40	1.57	3.18	1.17	3.58	16.90%	33.79%	50.69%	2.26	2.49
Y, ppm	449	33	382	515	349	549	7.41%	14.83%	22.24%	426	471
Yb, ppm	14.3	0.94	12.4	16.2	11.5	17.1	6.57%	13.13%	19.70%	13.6	15.0
Zn, ppm	1128	57	1015	1242	958	1298	5.03%	10.06%	15.10%	1072	1184
Zr, ppm	210	40	130	290	90	329	19.03%	38.05%	57.08%	199	220

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 La₂O₃ by fusion ICP, where 99% of the time (1- α =0.99) at least 95% of subsamples ($p=0.95$) will have concentrations lying between 1.34 and 1.41 wt.%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

The homogeneity of OREAS 464 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 464 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 464 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.

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

INTENDED USE

OREAS 464 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 464 has been prepared from ore grade/waste REE bearing ore (TREO = 5.10%). 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 464 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.

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.



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.

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., Mieзитis, Y. Ore Geology Reviews 62 (2014) 72-128 - Geological setting and resources of the major rare-earth-element deposits in Australia.