

**CERTIFICATE OF ANALYSIS FOR**

**CARBONATITE SUPERGENE REE-Nb ORE (TREO 9.88%)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 465**

**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 <sub>2</sub> , Cerium(IV) oxide (wt.%)	4.86	0.166	4.75	4.96	4.76	4.95
Dy <sub>2</sub> O <sub>3</sub> , Dysprosium(III) oxide (ppm)	249	14	240	257	241	256
Er <sub>2</sub> O <sub>3</sub> , Erbium(III) oxide (ppm)	58	3.6	56	60	56	60
Eu <sub>2</sub> O <sub>3</sub> , Europium(III) oxide (ppm)	331	13	324	338	321	341
Gd <sub>2</sub> O <sub>3</sub> , Gadolinium(III) oxide (ppm)	674	35	654	693	656	691
Ho <sub>2</sub> O <sub>3</sub> , Holmium(III) oxide (ppm)	31.7	2.44	30.4	33.1	30.6	32.8
La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)	2.83	0.097	2.78	2.88	2.77	2.89
Lu <sub>2</sub> O <sub>3</sub> , Lutetium(III) oxide (ppm)	2.39	0.138	2.34	2.45	2.27	2.52
Nb <sub>2</sub> O <sub>5</sub> , Niobium(V) oxide (ppm)	6695	431	6383	7007	6341	7049
Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)	1.37	0.059	1.34	1.41	1.34	1.40
Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm)	4557	198	4443	4672	4435	4679
Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)	1578	42	1560	1597	1527	1630
Tb <sub>4</sub> O <sub>7</sub> , Terbium(III,IV) oxide (ppm)	67	3.7	65	69	64	69
ThO <sub>2</sub> , Thorium dioxide (ppm)	985	49	956	1013	961	1008
Tm <sub>2</sub> O <sub>3</sub> , Thulium(III) oxide (ppm)	5.16	0.299	5.02	5.31	4.90	5.43
U <sub>3</sub> O <sub>8</sub> , Uranium(V,VI) oxide (ppm)	16.0	0.47	15.8	16.3	15.4	16.7
Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)	665	42	640	691	639	691
Yb <sub>2</sub> O <sub>3</sub> , Ytterbium(III) oxide (ppm)	21.6	0.83	21.2	22.0	20.4	22.8
ZrO <sub>2</sub> , Zirconium dioxide (ppm)	2539	274	2280	2798	2382	2696

Note: intervals may appear asymmetric due to rounding.

**Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 465.**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO <sub>2</sub> , Cerium(IV) oxide (wt.%)	4.88	0.045	4.84	4.92	4.84	4.91
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	49.96	1.309	48.22	51.70	49.62	50.29
La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)	2.84	0.033	2.81	2.87	2.82	2.86
Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)	1.38	0.102	1.28	1.48	1.35	1.40
Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm)	4534	143.9	4362	4705	4353	4714
Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)	1640	135.7	1500	1780	IND	IND
ThO <sub>2</sub> , Thorium dioxide (ppm)	901	128	757	1045	IND	IND
Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)	637	45.0	614	661	IND	IND
Thermogravimetry						
LOI, Loss On Ignition @1000°C (wt.%)	0.824	0.133	0.697	0.950	0.774	0.874
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	6.60	0.261	6.39	6.80	6.47	6.72
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	12.47	0.494	12.08	12.85	12.23	12.70
Ba, Barium (ppm)	4397	464	4127	4667	4278	4516
BaO, Barium oxide (ppm)	4909	518	4608	5211	4776	5042
Be, Beryllium (ppm)	13.2	0.89	12.5	14.0	IND	IND
Bi, Bismuth (ppm)	16.9	1.04	16.0	17.9	16.1	17.7
Ca, Calcium (wt.%)	0.900	0.060	0.861	0.938	0.871	0.929
CaO, Calcium oxide (wt.%)	1.26	0.084	1.21	1.31	1.22	1.30
Ce, Cerium (wt.%)	3.95	0.135	3.87	4.03	3.88	4.03
CeO <sub>2</sub> , Cerium(IV) oxide (wt.%)	4.86	0.166	4.75	4.96	4.76	4.95
Cr, Chromium (ppm)	544	42	516	572	530	558
Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)	795	61	755	836	775	815
Cs, Cesium (ppm)	< 0.1	IND	IND	IND	IND	IND
Dy, Dysprosium (ppm)	217	13	209	224	210	223
Dy <sub>2</sub> O <sub>3</sub> , Dysprosium(III) oxide (ppm)	249	14	240	257	241	256
Er, Erbium (ppm)	50	3.1	49	52	49	52
Er <sub>2</sub> O <sub>3</sub> , Erbium(III) oxide (ppm)	58	3.6	56	60	56	60
Eu, Europium (ppm)	286	11	280	292	277	294
Eu <sub>2</sub> O <sub>3</sub> , Europium(III) oxide (ppm)	331	13	324	338	321	341
Fe, Iron (wt.%)	34.71	0.634	34.16	35.26	33.82	35.60
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	49.63	0.906	48.84	50.41	48.36	50.90
Gd, Gadolinium (ppm)	584	31	568	601	569	600
Gd <sub>2</sub> O <sub>3</sub> , Gadolinium(III) oxide (ppm)	674	35	654	693	656	691
Hf, Hafnium (ppm)	41.4	7.2	35.8	47.0	38.5	44.3
HfO <sub>2</sub> , Hafnium dioxide (ppm)	48.8	8.5	42.3	55.4	45.4	52.2
Ho, Holmium (ppm)	27.7	2.13	26.5	28.9	26.7	28.7
Ho <sub>2</sub> O <sub>3</sub> , Holmium(III) oxide (ppm)	31.7	2.44	30.4	33.1	30.6	32.8
In, Indium (ppm)	3.47	0.227	3.32	3.62	2.94	4.00
La, Lanthanum (wt.%)	2.41	0.082	2.37	2.46	2.36	2.47
La <sub>2</sub> O <sub>3</sub> , Lanthanum(III) oxide (wt.%)	2.83	0.097	2.78	2.88	2.77	2.89
Lu, Lutetium (ppm)	2.10	0.122	2.06	2.15	1.99	2.21

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 <sub>2</sub> O <sub>3</sub> , Lutetium(III) oxide (ppm)	2.39	0.138	2.34	2.45	2.27	2.52
Mg, Magnesium (wt.%)	0.392	0.021	0.377	0.406	0.380	0.404
MgO, Magnesium oxide (wt.%)	0.650	0.034	0.625	0.674	0.629	0.670
Mn, Manganese (wt.%)	0.263	0.023	0.247	0.279	0.254	0.271
MnO, Manganese oxide (wt.%)	0.339	0.030	0.318	0.360	0.328	0.350
Mo, Molybdenum (ppm)	114	6	109	119	108	120
Nb, Niobium (ppm)	4680	301	4462	4898	4433	4928
Nb <sub>2</sub> O <sub>5</sub> , Niobium(V) oxide (ppm)	6695	431	6383	7007	6341	7049
Nd, Neodymium (wt.%)	1.18	0.050	1.15	1.20	1.15	1.20
Nd <sub>2</sub> O <sub>3</sub> , Neodymium(III) oxide (wt.%)	1.37	0.059	1.34	1.41	1.34	1.40
P, Phosphorus (wt.%)	3.81	0.122	3.70	3.92	3.69	3.94
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	8.74	0.280	8.49	8.98	8.45	9.02
Pb, Lead (ppm)	506	19	496	516	480	532
PbO, Lead oxide (ppm)	545	20	534	556	517	573
Pr, Praseodymium (ppm)	3772	164	3677	3867	3671	3873
Pr <sub>6</sub> O <sub>11</sub> , Praseodymium(III,IV) oxide (ppm)	4557	198	4443	4672	4435	4679
S, Sulphur (ppm)	1941	132	1787	2095	IND	IND
Si, Silicon (wt.%)	1.53	0.043	1.48	1.58	1.49	1.58
SiO <sub>2</sub> , Silicon dioxide (wt.%)	3.28	0.093	3.18	3.39	3.19	3.37
Sm, Samarium (ppm)	1361	36	1345	1377	1317	1405
Sm <sub>2</sub> O <sub>3</sub> , Samarium(III) oxide (ppm)	1578	42	1560	1597	1527	1630
Sn, Tin (ppm)	136	20	124	148	129	143
SnO <sub>2</sub> , Tin dioxide (ppm)	172	26	157	188	164	181
Sr, Strontium (ppm)	5204	182	5085	5322	5102	5305
SrO, Strontium oxide (ppm)	6154	215	6014	6294	6034	6274
Ta, Tantalum (ppm)	79	5.0	75	82	74	83
Ta <sub>2</sub> O <sub>5</sub> , Tantalum(V) oxide (ppm)	96	6.2	91	101	91	102
Tb, Terbium (ppm)	57	3.1	55	58	54	59
Tb <sub>4</sub> O <sub>7</sub> , Terbium(III,IV) oxide (ppm)	67	3.7	65	69	64	69
Th, Thorium (ppm)	866	43	840	891	845	886
ThO <sub>2</sub> , Thorium dioxide (ppm)	985	49	956	1013	961	1008
Ti, Titanium (wt.%)	6.30	0.184	6.16	6.44	6.14	6.46
TiO <sub>2</sub> , Titanium dioxide (wt.%)	10.51	0.307	10.28	10.74	10.25	10.78
Tm, Thulium (ppm)	4.52	0.262	4.40	4.65	4.29	4.75
Tm <sub>2</sub> O <sub>3</sub> , Thulium(III) oxide (ppm)	5.16	0.299	5.02	5.31	4.90	5.43
U, Uranium (ppm)	13.6	0.40	13.4	13.8	13.0	14.2
U <sub>3</sub> O <sub>8</sub> , Uranium(V,VI) oxide (ppm)	16.0	0.47	15.8	16.3	15.4	16.7
V, Vanadium (ppm)	534	36	512	557	518	551
V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)	953	64	913	994	924	983
W, Tungsten (ppm)	7.52	1.24	6.51	8.53	IND	IND
WO <sub>3</sub> , Tungsten trioxide (ppm)	9.48	1.56	8.20	10.76	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
Borate / Peroxide Fusion ICP continued (majors and REE's shown in both oxide and elemental format)						
Y, Yttrium (ppm)	524	33	504	544	503	544
Y <sub>2</sub> O <sub>3</sub> , Yttrium(III) oxide (ppm)	665	42	640	691	639	691
Yb, Ytterbium (ppm)	19.0	0.72	18.6	19.4	17.9	20.1
Yb <sub>2</sub> O <sub>3</sub> , Ytterbium(III) oxide (ppm)	21.6	0.83	21.2	22.0	20.4	22.8
Zr, Zirconium (ppm)	1880	203	1688	2071	1764	1996
ZrO <sub>2</sub> , Zirconium dioxide (ppm)	2539	274	2280	2798	2382	2696
4-Acid Digestion						
Ag, Silver (ppm)	5.5	0.35	5.3	5.7	5.1	5.9
Al, Aluminium (wt.%)	6.21	0.399	6.00	6.42	6.02	6.40
Ba, Barium (ppm)	4359	158.0	4270	4448	4291	4427
Be, Beryllium (ppm)	11.6	0.76	11.2	12.1	11.2	12.1
Bi, Bismuth (ppm)	17.3	1.03	16.7	17.9	16.7	17.8
Ca, Calcium (wt.%)	0.872	0.062	0.830	0.914	0.850	0.894
Cd, Cadmium (ppm)	1.20	0.14	1.12	1.28	1.08	1.32
Ce, Cerium (wt.%)	3.91	0.149	3.76	4.05	3.81	4.00
Co, Cobalt (ppm)	18.7	1.26	18.1	19.3	18.1	19.3
Cs, Cesium (ppm)	< 0.1	IND	IND	IND	IND	IND
Cu, Copper (ppm)	128	5.3	125	130	123	132
Dy, Dysprosium (ppm)	215	9.3	210	220	210	221
Er, Erbium (ppm)	47.3	2.51	45.8	48.7	45.5	49.0
Eu, Europium (ppm)	282	11.8	275	290	275	289
Fe, Iron (wt.%)	29.55	2.025	28.38	30.73	28.92	30.19
Ga, Gallium (ppm)	188	31	157	220	175	201
Gd, Gadolinium (ppm)	581	35.8	556	606	570	592
Hf, Hafnium (ppm)	14.4	1.9	13.7	15.1	13.0	15.8
Ho, Holmium (ppm)	26.8	0.94	26.3	27.2	25.9	27.7
In, Indium (ppm)	3.18	0.208	3.06	3.31	3.05	3.32
La, Lanthanum (wt.%)	2.27	0.093	2.15	2.39	2.22	2.33
Li, Lithium (ppm)	3.04	0.33	2.80	3.28	2.86	3.22
Lu, Lutetium (ppm)	1.72	0.28	1.54	1.90	1.62	1.82
Mg, Magnesium (wt.%)	0.374	0.026	0.356	0.391	0.362	0.386
Mn, Manganese (wt.%)	0.198	0.022	0.181	0.215	0.191	0.204
Mo, Molybdenum (ppm)	98	10	92	105	95	101
Na, Sodium (wt.%)	< 0.2	IND	IND	IND	IND	IND
Nd, Neodymium (wt.%)	1.10	0.043	1.05	1.14	1.04	1.15
Ni, Nickel (ppm)	106	14	95	116	95	116
P, Phosphorus (wt.%)	3.15	0.38	2.83	3.47	3.06	3.24
Pb, Lead (ppm)	573	16.1	562	583	561	584
Pr, Praseodymium (ppm)	3670	192.8	3488	3852	3561	3778
Rb, Rubidium (ppm)	0.43	0.05	0.40	0.45	0.35	0.50
Re, Rhenium (ppm)	< 0.05	IND	IND	IND	IND	IND
Sc, Scandium (ppm)	149	9.1	143	154	143	154

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						
Sm, Samarium (ppm)	1307	80.8	1230	1383	1241	1373
Sr, Strontium (wt.%)	0.505	0.026	0.487	0.523	0.490	0.520
Tb, Terbium (ppm)	57	3.3	55	59	55	59
Te, Tellurium (ppm)	< 1	IND	IND	IND	IND	IND
Th, Thorium (ppm)	805	150	693	916	773	837
Tl, Thallium (ppm)	0.087	0.009	0.082	0.093	IND	IND
Tm, Thulium (ppm)	3.82	0.274	3.65	4.00	3.70	3.95
U, Uranium (ppm)	12.6	0.68	12.2	13.0	12.1	13.1
V, Vanadium (ppm)	427	69	380	473	410	443
Y, Yttrium (ppm)	478	32.4	455	500	464	492
Yb, Ytterbium (ppm)	14.9	0.95	14.3	15.5	14.3	15.5
Zn, Zinc (ppm)	921	133	841	1001	894	949

Note: intervals may appear asymmetric due to rounding.

**Table 2. Indicative Values for OREAS 465.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al <sub>2</sub> O <sub>3</sub>	wt. %	12.62	Lu <sub>2</sub> O <sub>3</sub>	ppm	< 20	SrO	wt. %	0.603
BaO	ppm	5342	MgO	wt. %	0.664	Ta <sub>2</sub> O <sub>5</sub>	ppm	< 100
CaO	wt. %	1.31	MnO	wt. %	0.369	Tb <sub>4</sub> O <sub>7</sub>	ppm	67
Cr <sub>2</sub> O <sub>3</sub>	ppm	735	Na <sub>2</sub> O	wt. %	0.172	TiO <sub>2</sub>	wt. %	10.59
Dy <sub>2</sub> O <sub>3</sub>	ppm	329	Nb <sub>2</sub> O <sub>5</sub>	ppm	6651	Tm <sub>2</sub> O <sub>3</sub>	ppm	< 10
Er <sub>2</sub> O <sub>3</sub>	ppm	62	NiO	ppm	200	U <sub>3</sub> O <sub>8</sub>	ppm	< 100
Eu <sub>2</sub> O <sub>3</sub>	ppm	350	P <sub>2</sub> O <sub>5</sub>	wt. %	8.77	V <sub>2</sub> O <sub>5</sub>	ppm	1088
Gd <sub>2</sub> O <sub>3</sub>	ppm	870	PbO	ppm	650	WO <sub>3</sub>	ppm	< 100
HfO <sub>2</sub>	ppm	< 100	SiO <sub>2</sub>	wt. %	3.20	Yb <sub>2</sub> O <sub>3</sub>	ppm	80
Ho <sub>2</sub> O <sub>3</sub>	ppm	30.0	SnO <sub>2</sub>	ppm	217	ZnO	ppm	1500
K <sub>2</sub> O	wt. %	0.015	SO <sub>3</sub>	wt. %	0.485	ZrO <sub>2</sub>	ppm	2617
Thermogravimetry								
H <sub>2</sub> O-	wt. %	0.312						
Borate / Peroxide Fusion ICP								
Ag	ppm	6.50	Ge	ppm	60	Sb	ppm	0.98
As	ppm	420	K <sub>2</sub> O	wt. %	0.119	Sc	ppm	171
B	ppm	23.3	Li	ppm	3.33	Se	ppm	33.6
Cd	ppm	1.04	Na	wt. %	0.078	Te	ppm	< 1
Co	ppm	23.0	NiO	ppm	128	Tl	ppm	< 0.5
Cu	ppm	128	Rb	ppm	0.98	ZnO	ppm	1366
Ga <sup>†</sup>	ppm	237	Re	ppm	< 0.1			
4-Acid Digestion								
As	ppm	31.7	S	ppm	1103	Ti	wt. %	1.30
Cr	ppm	357	Sb	ppm	0.57	W	ppm	2.40
Ge	ppm	23.6	Se	ppm	35.4	Zr	ppm	367
K	wt. %	0.023	Sn	ppm	48.8			
Nb	ppm	399	Ta	ppm	50			

<sup>†</sup>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 465 is a high grade ore, rare earth element (TREO = 9.88%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 465 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% H<sub>2</sub>O-).

OREAS 465 is one of six MMCRMs ranging 0.53 - 9.88% TREO and contains 99 certified values (and 66 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<sub>2</sub> (46.7%), La<sub>2</sub>O<sub>3</sub> (25.5%), Nd<sub>2</sub>O<sub>3</sub> (18.5%), Pr<sub>6</sub>O<sub>11</sub> (5.32%), Sm<sub>2</sub>O<sub>3</sub> (2.27%) and Eu<sub>2</sub>O<sub>3</sub> (0.443%), together with minor components of HREE: Dy<sub>2</sub>O<sub>3</sub> (0.124%) and Tb<sub>4</sub>O<sub>7</sub> (0.068%).

## COMMINUTION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 465 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 99 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<sub>3</sub>-HClO<sub>4</sub>-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 465. 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 99 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 66 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<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 465 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 465.**

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 <sub>2</sub> , wt. %	4.88	0.045	4.79	4.97	4.74	5.01	0.93%	1.85%	2.78%	4.63	5.12
Fe <sub>2</sub> O <sub>3</sub> , wt. %	49.96	1.309	47.34	52.58	46.03	53.89	2.62%	5.24%	7.86%	47.46	52.46
La <sub>2</sub> O <sub>3</sub> , wt. %	2.84	0.033	2.77	2.90	2.74	2.94	1.16%	2.33%	3.49%	2.70	2.98
Nd <sub>2</sub> O <sub>3</sub> , wt. %	1.38	0.102	1.17	1.58	1.07	1.68	7.43%	14.86%	22.29%	1.31	1.45
Pr <sub>6</sub> O <sub>11</sub> , ppm	4534	144	4246	4821	4102	4965	3.17%	6.35%	9.52%	4307	4760
Sm <sub>2</sub> O <sub>3</sub> , ppm	1640	136	1368	1911	1233	2047	8.27%	16.55%	24.82%	1558	1722
ThO <sub>2</sub> , ppm	901	128	645	1158	517	1286	14.23%	28.46%	42.68%	856	946
Y <sub>2</sub> O <sub>3</sub> , ppm	637	45	547	728	502	773	7.06%	14.13%	21.19%	606	669
Thermogravimetry											
LOI, wt. %	0.824	0.133	0.558	1.089	0.425	1.222	16.12%	32.23%	48.35%	0.783	0.865
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)											
Al, wt. %	6.60	0.261	6.08	7.12	5.81	7.38	3.96%	7.92%	11.88%	6.27	6.93
Al <sub>2</sub> O <sub>3</sub> , wt. %	12.47	0.494	11.48	13.45	10.99	13.95	3.96%	7.92%	11.88%	11.84	13.09
Ba, ppm	4397	464	3469	5325	3005	5789	10.56%	21.11%	31.67%	4177	4617
BaO, ppm	4909	518	3873	5946	3355	6464	10.56%	21.11%	31.67%	4664	5155
Be, ppm	13.2	0.89	11.5	15.0	10.6	15.9	6.74%	13.49%	20.23%	12.6	13.9
Bi, ppm	16.9	1.04	14.8	19.0	13.8	20.0	6.16%	12.31%	18.47%	16.1	17.8
Ca, wt. %	0.900	0.060	0.780	1.020	0.720	1.080	6.67%	13.33%	20.00%	0.855	0.945
CaO, wt. %	1.26	0.084	1.09	1.43	1.01	1.51	6.67%	13.33%	20.00%	1.20	1.32
Ce, wt. %	3.95	0.135	3.68	4.22	3.55	4.36	3.42%	6.83%	10.25%	3.75	4.15
CeO <sub>2</sub> , wt. %	4.86	0.166	4.52	5.19	4.36	5.35	3.42%	6.83%	10.25%	4.61	5.10
Cr, ppm	544	42	460	628	418	670	7.70%	15.40%	23.10%	517	571
Cr <sub>2</sub> O <sub>3</sub> , ppm	795	61	673	918	612	979	7.70%	15.40%	23.10%	756	835
Cs, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Dy, ppm	217	13	192	242	179	254	5.80%	11.61%	17.41%	206	228
Dy <sub>2</sub> O <sub>3</sub> , ppm	249	14	220	278	205	292	5.80%	11.61%	17.41%	236	261
Er, ppm	50	3.1	44	57	41	60	6.22%	12.44%	18.66%	48	53
Er <sub>2</sub> O <sub>3</sub> , ppm	58	3.6	51	65	47	68	6.22%	12.44%	18.66%	55	61
Eu, ppm	286	11	263	309	251	320	4.02%	8.03%	12.05%	271	300

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)											
Eu <sub>2</sub> O <sub>3</sub> , ppm	331	13	304	357	291	371	4.02%	8.03%	12.05%	314	347
Fe, wt. %	34.71	0.634	33.45	35.98	32.81	36.61	1.83%	3.65%	5.48%	32.98	36.45
Fe <sub>2</sub> O <sub>3</sub> , wt. %	49.63	0.906	47.82	51.44	46.91	52.35	1.83%	3.65%	5.48%	47.15	52.11
Gd, ppm	584	31	523	646	492	676	5.25%	10.50%	15.76%	555	614
Gd <sub>2</sub> O <sub>3</sub> , ppm	674	35	603	744	567	780	5.25%	10.50%	15.76%	640	707
Hf, ppm	41.4	7.2	27.0	55.8	19.8	63.0	17.41%	34.82%	52.22%	39.3	43.5
HfO <sub>2</sub> , ppm	48.8	8.5	31.8	65.8	23.3	74.3	17.41%	34.82%	52.22%	46.4	51.3
Ho, ppm	27.7	2.13	23.4	32.0	21.3	34.1	7.70%	15.39%	23.09%	26.3	29.1
Ho <sub>2</sub> O <sub>3</sub> , ppm	31.7	2.44	26.8	36.6	24.4	39.1	7.70%	15.39%	23.09%	30.1	33.3
In, ppm	3.47	0.227	3.02	3.92	2.79	4.15	6.53%	13.07%	19.60%	3.30	3.64
La, wt. %	2.41	0.082	2.25	2.58	2.17	2.66	3.41%	6.83%	10.24%	2.29	2.53
La <sub>2</sub> O <sub>3</sub> , wt. %	2.83	0.097	2.64	3.02	2.54	3.12	3.41%	6.83%	10.24%	2.69	2.97
Lu, ppm	2.10	0.122	1.86	2.35	1.74	2.47	5.78%	11.56%	17.34%	2.00	2.21
Lu <sub>2</sub> O <sub>3</sub> , ppm	2.39	0.138	2.12	2.67	1.98	2.81	5.78%	11.56%	17.34%	2.27	2.51
Mg, wt. %	0.392	0.021	0.351	0.433	0.330	0.453	5.23%	10.47%	15.70%	0.372	0.411
MgO, wt. %	0.650	0.034	0.582	0.718	0.548	0.752	5.23%	10.47%	15.70%	0.617	0.682
Mn, wt. %	0.263	0.023	0.217	0.308	0.194	0.331	8.72%	17.43%	26.15%	0.249	0.276
MnO, wt. %	0.339	0.030	0.280	0.398	0.250	0.428	8.72%	17.43%	26.15%	0.322	0.356
Mo, ppm	114	6	103	125	97	131	4.97%	9.95%	14.92%	108	120
Nb, ppm	4680	301	4078	5283	3776	5584	6.44%	12.87%	19.31%	4446	4914
Nb <sub>2</sub> O <sub>5</sub> , ppm	6695	431	5833	7557	5402	7988	6.44%	12.87%	19.31%	6360	7030
Nd, wt. %	1.18	0.050	1.08	1.28	1.03	1.33	4.29%	8.57%	12.86%	1.12	1.24
Nd <sub>2</sub> O <sub>3</sub> , wt. %	1.37	0.059	1.25	1.49	1.20	1.55	4.29%	8.57%	12.86%	1.30	1.44
P, wt. %	3.81	0.122	3.57	4.06	3.45	4.18	3.21%	6.42%	9.63%	3.62	4.00
P <sub>2</sub> O <sub>5</sub> , wt. %	8.74	0.280	8.18	9.30	7.89	9.58	3.21%	6.42%	9.63%	8.30	9.17
Pb, ppm	506	19	469	543	450	562	3.68%	7.36%	11.04%	481	531
PbO, ppm	545	20	505	585	485	605	3.68%	7.36%	11.04%	518	572
Pr, ppm	3772	164	3444	4100	3280	4264	4.35%	8.70%	13.05%	3583	3960
Pr <sub>6</sub> O <sub>11</sub> , ppm	4557	198	4161	4953	3963	5152	4.35%	8.70%	13.05%	4329	4785
S, ppm	1941	132	1676	2206	1544	2338	6.82%	13.65%	20.47%	1844	2038
Si, wt. %	1.53	0.043	1.45	1.62	1.40	1.66	2.83%	5.65%	8.48%	1.46	1.61

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)											
SiO <sub>2</sub> , wt. %	3.28	0.093	3.10	3.47	3.00	3.56	2.83%	5.65%	8.48%	3.12	3.45
Sm, ppm	1361	36	1289	1433	1252	1470	2.66%	5.32%	7.97%	1293	1429
Sm <sub>2</sub> O <sub>3</sub> , ppm	1578	42	1494	1662	1452	1704	2.66%	5.32%	7.97%	1499	1657
Sn, ppm	136	20	95	176	75	196	14.89%	29.78%	44.68%	129	143
SnO <sub>2</sub> , ppm	172	26	121	224	95	249	14.89%	29.78%	44.68%	164	181
Sr, ppm	5204	182	4840	5568	4658	5750	3.50%	7.00%	10.50%	4944	5464
SrO, ppm	6154	215	5723	6585	5508	6800	3.50%	7.00%	10.50%	5846	6462
Ta, ppm	79	5.0	69	89	64	94	6.41%	12.82%	19.24%	75	83
Ta <sub>2</sub> O <sub>5</sub> , ppm	96	6.2	84	108	78	115	6.41%	12.82%	19.24%	91	101
Tb, ppm	57	3.1	50	63	47	66	5.52%	11.03%	16.55%	54	59
Tb <sub>4</sub> O <sub>7</sub> , ppm	67	3.7	59	74	56	78	5.52%	11.03%	16.55%	63	70
Th, ppm	866	43	780	951	737	994	4.95%	9.89%	14.84%	822	909
ThO <sub>2</sub> , ppm	985	49	887	1082	839	1131	4.95%	9.89%	14.84%	936	1034
Ti, wt. %	6.30	0.184	5.93	6.67	5.75	6.85	2.92%	5.83%	8.75%	5.99	6.62
TiO <sub>2</sub> , wt. %	10.51	0.307	9.90	11.13	9.59	11.43	2.92%	5.83%	8.75%	9.99	11.04
Tm, ppm	4.52	0.262	4.00	5.05	3.74	5.31	5.80%	11.59%	17.39%	4.30	4.75
Tm <sub>2</sub> O <sub>3</sub> , ppm	5.16	0.299	4.57	5.76	4.27	6.06	5.80%	11.59%	17.39%	4.91	5.42
U, ppm	13.6	0.40	12.8	14.4	12.4	14.8	2.95%	5.89%	8.84%	12.9	14.3
U <sub>3</sub> O <sub>8</sub> , ppm	16.0	0.47	15.1	17.0	14.6	17.5	2.95%	5.89%	8.84%	15.2	16.8
V, ppm	534	36	463	605	427	641	6.66%	13.32%	19.99%	507	561
V <sub>2</sub> O <sub>5</sub> , ppm	953	64	826	1081	763	1144	6.66%	13.32%	19.99%	906	1001
W, ppm	7.52	1.24	5.04	10.00	3.81	11.23	16.47%	32.93%	49.40%	7.14	7.90
WO <sub>3</sub> , ppm	9.48	1.56	6.36	12.61	4.80	14.17	16.47%	32.93%	49.40%	9.01	9.96
Y, ppm	524	33	457	590	424	623	6.34%	12.67%	19.01%	498	550
Y <sub>2</sub> O <sub>3</sub> , ppm	665	42	581	749	539	792	6.34%	12.67%	19.01%	632	698
Yb, ppm	19.0	0.72	17.5	20.4	16.8	21.2	3.82%	7.63%	11.45%	18.0	19.9
Yb <sub>2</sub> O <sub>3</sub> , ppm	21.6	0.83	20.0	23.3	19.1	24.1	3.82%	7.63%	11.45%	20.5	22.7
Zr, ppm	1880	203	1473	2286	1270	2489	10.81%	21.62%	32.42%	1786	1974
ZrO <sub>2</sub> , ppm	2539	274	1990	3088	1716	3362	10.81%	21.62%	32.42%	2412	2666
4-Acid Digestion											
Ag, ppm	5.48	0.353	4.78	6.19	4.42	6.54	6.44%	12.88%	19.32%	5.21	5.76
Al, wt. %	6.21	0.399	5.41	7.01	5.01	7.41	6.43%	12.86%	19.30%	5.90	6.52

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											
Ba, ppm	4359	158	4043	4675	3885	4833	3.62%	7.25%	10.87%	4141	4577
Be, ppm	11.6	0.76	10.1	13.1	9.3	13.9	6.52%	13.04%	19.56%	11.0	12.2
Bi, ppm	17.3	1.03	15.2	19.3	14.2	20.4	5.96%	11.92%	17.89%	16.4	18.1
Ca, wt. %	0.872	0.062	0.748	0.995	0.687	1.057	7.08%	14.15%	21.23%	0.828	0.915
Cd, ppm	1.20	0.14	0.92	1.49	0.78	1.63	11.83%	23.66%	35.49%	1.14	1.26
Ce, wt. %	3.91	0.149	3.61	4.20	3.46	4.35	3.81%	7.62%	11.43%	3.71	4.10
Co, ppm	18.7	1.26	16.2	21.2	14.9	22.5	6.74%	13.49%	20.23%	17.8	19.6
Cs, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, ppm	128	5	117	138	112	144	4.17%	8.34%	12.51%	121	134
Dy, ppm	215	9	197	234	187	243	4.30%	8.61%	12.91%	204	226
Er, ppm	47.3	2.51	42.2	52.3	39.7	54.8	5.31%	10.61%	15.92%	44.9	49.6
Eu, ppm	282	12	259	306	247	318	4.18%	8.37%	12.55%	268	296
Fe, wt. %	29.55	2.025	25.50	33.60	23.48	35.63	6.85%	13.70%	20.55%	28.08	31.03
Ga, ppm	188	31	127	250	96	281	16.34%	32.69%	49.03%	179	198
Gd, ppm	581	36	509	653	474	689	6.16%	12.33%	18.49%	552	610
Hf, ppm	14.4	1.9	10.5	18.3	8.6	20.2	13.42%	26.84%	40.26%	13.7	15.1
Ho, ppm	26.8	0.94	24.9	28.6	24.0	29.6	3.49%	6.99%	10.48%	25.4	28.1
In, ppm	3.18	0.208	2.77	3.60	2.56	3.81	6.54%	13.08%	19.62%	3.02	3.34
La, wt. %	2.27	0.093	2.09	2.46	2.00	2.55	4.09%	8.18%	12.27%	2.16	2.39
Li, ppm	3.04	0.33	2.38	3.71	2.05	4.04	10.91%	21.83%	32.74%	2.89	3.19
Lu, ppm	1.72	0.28	1.16	2.28	0.88	2.57	16.37%	32.74%	49.11%	1.63	1.81
Mg, wt. %	0.374	0.026	0.322	0.426	0.296	0.452	6.98%	13.96%	20.95%	0.355	0.393
Mn, wt. %	0.198	0.022	0.153	0.243	0.130	0.265	11.36%	22.71%	34.07%	0.188	0.208
Mo, ppm	98	10	78	118	68	128	10.35%	20.71%	31.06%	93	103
Na, wt. %	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Nd, wt. %	1.10	0.043	1.01	1.18	0.97	1.22	3.93%	7.86%	11.78%	1.04	1.15
Ni, ppm	106	14	79	133	65	146	12.80%	25.61%	38.41%	101	111
P, wt. %	3.15	0.38	2.40	3.90	2.02	4.28	11.92%	23.84%	35.75%	2.99	3.31
Pb, ppm	573	16	540	605	524	621	2.81%	5.61%	8.42%	544	601
Pr, ppm	3670	193	3284	4055	3092	4248	5.25%	10.50%	15.76%	3486	3853
Rb, ppm	0.43	0.05	0.32	0.53	0.26	0.59	12.75%	25.50%	38.25%	0.40	0.45
Re, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

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											
Sc, ppm	149	9	130	167	121	176	6.11%	12.23%	18.34%	141	156
Sm, ppm	1307	81	1145	1468	1064	1549	6.18%	12.37%	18.55%	1241	1372
Sr, wt. %	0.505	0.026	0.453	0.558	0.427	0.584	5.19%	10.39%	15.58%	0.480	0.531
Tb, ppm	57	3.3	50	64	47	67	5.83%	11.66%	17.50%	54	60
Te, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	805	150	505	1104	356	1254	18.60%	37.20%	55.79%	764	845
Tl, ppm	0.087	0.009	0.069	0.105	0.061	0.114	10.22%	20.44%	30.66%	0.083	0.092
Tm, ppm	3.82	0.274	3.28	4.37	3.00	4.65	7.17%	14.34%	21.51%	3.63	4.01
U, ppm	12.6	0.68	11.2	14.0	10.6	14.6	5.38%	10.77%	16.15%	12.0	13.2
V, ppm	427	69	289	565	220	634	16.17%	32.34%	48.51%	405	448
Y, ppm	478	32	413	543	381	575	6.77%	13.54%	20.32%	454	502
Yb, ppm	14.9	0.95	13.0	16.8	12.0	17.8	6.39%	12.78%	19.17%	14.2	15.6
Zn, ppm	921	133	655	1187	522	1320	14.44%	28.87%	43.31%	875	967

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  $\text{La}_2\text{O}_3$  by fusion ICP, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 2.77 and 2.89 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 465 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 99 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 465 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 465 has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd  
37A Hosie Street  
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AUSTRALIA

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

## **INTENDED USE**

OREAS 465 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 465 has been prepared from ore grade/waste REE bearing ore (TREO = 9.88%). 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<sub>2</sub>O-).

OREAS 465 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	24 <sup>th</sup> September, 2025	Ga reclassified from Certified Value to Indicative Value.
0	28 <sup>th</sup> April, 2015	First publication.

## 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



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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.

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