

CERTIFICATE OF ANALYSIS FOR

CARBONATITE SUPERGENE REE-Nb ORE (TREO 2.08%)

CERTIFIED REFERENCE MATERIAL

OREAS 463

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.%)	0.810	0.019	0.801	0.818	0.790	0.829
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	81	3.8	79	83	78	84
Er ₂ O ₃ , Erbium(III) oxide (ppm)	18.3	1.22	17.6	19.0	17.7	18.9
Eu ₂ O ₃ , Europium(III) oxide (ppm)	133	5	130	136	129	137
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	278	15	269	287	272	284
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	9.98	0.650	9.61	10.35	9.69	10.28
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	5824	163	5744	5904	5690	5957
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.90	0.046	0.88	0.92	0.83	0.97
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2139	105	2066	2213	2069	2209
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	4295	215	4173	4418	4217	4373
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	1212	52	1184	1241	1199	1225
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	624	13	619	628	613	634
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	23.9	1.12	23.3	24.5	23.2	24.5
ThO ₂ , Thorium dioxide (ppm)	332	13	325	340	325	340
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.80	0.115	1.74	1.85	1.71	1.88
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	9.26	0.346	9.08	9.44	9.02	9.49
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	229	10	223	235	222	236
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	8.00	0.471	7.76	8.24	7.63	8.37
ZrO ₂ , Zirconium dioxide (ppm)	778	33	756	801	746	811

SI unit equivalents: ppm (parts per million) ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 1000 ppb (parts per billion).

Note: intervals may appear asymmetric due to rounding.

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 463.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
CeO ₂ , Cerium(IV) oxide (wt.%)	0.815	0.016	0.803	0.828	0.795	0.835
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	49.48	1.113	47.98	50.98	49.22	49.74
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	5917	95.9	5835	5998	5772	6061
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	4330	229.7	4114	4546	4200	4460
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	1201	80.1	1113	1289	IND	IND
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	597	107	488	705	IND	IND
Thermogravimetry						
LOI, Loss On Ignition @1000°C (wt.%)	0.781	0.075	0.681	0.881	0.735	0.828
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)						
Al, Aluminium (wt.%)	5.63	0.210	5.48	5.78	5.53	5.73
Al ₂ O ₃ , Aluminium(III) oxide (wt.%)	10.64	0.396	10.35	10.93	10.46	10.82
Ba, Barium (ppm)	1106	70	1069	1144	1078	1135
BaO, Barium oxide (ppm)	1235	78	1193	1277	1203	1267
Be, Beryllium (ppm)	5.62	0.420	5.50	5.75	IND	IND
Bi, Bismuth (ppm)	2.75	0.38	2.39	3.12	IND	IND
Ca, Calcium (wt.%)	1.22	0.058	1.19	1.26	1.19	1.25
CaO, Calcium oxide (wt.%)	1.71	0.081	1.66	1.76	1.67	1.75
Ce, Cerium (wt.%)	0.659	0.015	0.652	0.666	0.643	0.675
CeO ₂ , Cerium(IV) oxide (wt.%)	0.810	0.019	0.801	0.818	0.790	0.829
Co, Cobalt (ppm)	15.2	2.6	12.6	17.7	13.9	16.4
Cr, Chromium (ppm)	574	32	552	595	555	592
Cr ₂ O ₃ , Chromium(III) oxide (ppm)	838	47	807	870	811	866
Cs, Cesium (ppm)	0.42	0.05	0.39	0.46	0.39	0.46
Cu, Copper (ppm)	74	16	67	82	IND	IND
Dy, Dysprosium (ppm)	70	3.3	69	72	68	73
Dy ₂ O ₃ , Dysprosium(III) oxide (ppm)	81	3.8	79	83	78	84
Er, Erbium (ppm)	16.0	1.07	15.4	16.6	15.4	16.5
Er ₂ O ₃ , Erbium(III) oxide (ppm)	18.3	1.22	17.6	19.0	17.7	18.9
Eu, Europium (ppm)	115	4	113	118	112	118
Eu ₂ O ₃ , Europium(III) oxide (ppm)	133	5	130	136	129	137
Fe, Iron (wt.%)	34.47	1.340	33.46	35.49	33.55	35.39
Fe ₂ O ₃ , Iron(III) oxide (wt.%)	49.29	1.915	47.84	50.73	47.97	50.60
Gd, Gadolinium (ppm)	241	13	233	249	236	246
Gd ₂ O ₃ , Gadolinium(III) oxide (ppm)	278	15	269	287	272	284
Hf, Hafnium (ppm)	13.8	0.45	13.6	14.0	13.3	14.2
HfO ₂ , Hafnium dioxide (ppm)	16.3	0.54	16.0	16.5	15.7	16.8
Ho, Holmium (ppm)	8.71	0.567	8.39	9.04	8.46	8.97
Ho ₂ O ₃ , Holmium(III) oxide (ppm)	9.98	0.650	9.61	10.35	9.69	10.28
In, Indium (ppm)	1.01	0.072	1.00	1.02	IND	IND
K, Potassium (wt.%)	0.119	0.020	0.102	0.136	IND	IND

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

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)						
K ₂ O, Potassium oxide (wt.%)	0.143	0.025	0.123	0.164	IND	IND
La, Lanthanum (ppm)	4966	139	4897	5034	4852	5080
La ₂ O ₃ , Lanthanum(III) oxide (ppm)	5824	163	5744	5904	5690	5957
Li, Lithium (ppm)	10.4	1.9	8.4	12.5	IND	IND
Lu, Lutetium (ppm)	0.79	0.040	0.78	0.81	0.73	0.86
Lu ₂ O ₃ , Lutetium(III) oxide (ppm)	0.90	0.046	0.88	0.92	0.83	0.97
Mg, Magnesium (wt.%)	1.02	0.054	0.99	1.06	1.01	1.04
MgO, Magnesium oxide (wt.%)	1.70	0.090	1.63	1.76	1.67	1.72
Mn, Manganese (wt.%)	0.121	0.009	0.114	0.127	0.117	0.125
MnO, Manganese oxide (wt.%)	0.156	0.012	0.147	0.165	0.151	0.161
Mo, Molybdenum (ppm)	56	2.4	54	58	54	59
Nb, Niobium (ppm)	1495	73	1444	1547	1447	1544
Nb ₂ O ₅ , Niobium(V) oxide (ppm)	2139	105	2066	2213	2069	2209
Nd, Neodymium (ppm)	3682	185	3577	3787	3616	3749
Nd ₂ O ₃ , Neodymium(III) oxide (ppm)	4295	215	4173	4418	4217	4373
Ni, Nickel (ppm)	71	11	60	81	59	82
NiO, Nickel oxide (ppm)	90	14	77	103	75	105
P, Phosphorus (wt.%)	0.629	0.024	0.609	0.648	0.608	0.649
P ₂ O ₅ , Phosphorus(V) oxide (wt.%)	1.44	0.054	1.40	1.49	1.39	1.49
Pb, Lead (ppm)	122	6	116	129	115	129
PbO, Lead oxide (ppm)	132	7	125	138	124	139
Pr, Praseodymium (ppm)	1004	43	980	1027	993	1014
Pr ₆ O ₁₁ , Praseodymium(III,IV) oxide (ppm)	1212	52	1184	1241	1199	1225
Rb, Rubidium (ppm)	6.08	0.336	5.92	6.24	5.75	6.41
S, Sulphur (ppm)	671	104	588	754	IND	IND
Sb, Antimony (ppm)	2.31	0.40	2.03	2.59	2.00	2.61
Si, Silicon (wt.%)	12.85	0.392	12.54	13.17	12.52	13.19
SiO ₂ , Silicon dioxide (wt.%)	27.50	0.839	26.82	28.17	26.78	28.21
Sm, Samarium (ppm)	538	11	534	542	529	547
Sm ₂ O ₃ , Samarium(III) oxide (ppm)	624	13	619	628	613	634
Sn, Tin (ppm)	31.4	3.09	29.4	33.4	29.4	33.5
SnO ₂ , Tin dioxide (ppm)	39.9	3.93	37.3	42.4	37.3	42.5
Sr, Strontium (ppm)	961	26	947	975	938	984
SrO, Strontium oxide (ppm)	1136	30	1120	1153	1109	1164
Ta, Tantalum (ppm)	25.2	1.28	24.7	25.8	24.1	26.4
Ta ₂ O ₅ , Tantalum(V) oxide (ppm)	30.8	1.56	30.1	31.5	29.4	32.3
Tb, Terbium (ppm)	20.3	0.95	19.8	20.8	19.7	20.8
Tb ₄ O ₇ , Terbium(III,IV) oxide (ppm)	23.9	1.12	23.3	24.5	23.2	24.5
Th, Thorium (ppm)	292	11	285	299	286	298
ThO ₂ , Thorium dioxide (ppm)	332	13	325	340	325	340
Ti, Titanium (wt.%)	1.92	0.068	1.87	1.97	1.88	1.96

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

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)						
TiO ₂ , Titanium dioxide (wt.%)	3.21	0.114	3.12	3.29	3.14	3.28
Tm, Thulium (ppm)	1.57	0.101	1.52	1.62	1.50	1.65
Tm ₂ O ₃ , Thulium(III) oxide (ppm)	1.80	0.115	1.74	1.85	1.71	1.88
U, Uranium (ppm)	7.85	0.294	7.70	8.01	7.65	8.05
U ₃ O ₈ , Uranium(V,VI) oxide (ppm)	9.26	0.346	9.08	9.44	9.02	9.49
V, Vanadium (ppm)	360	21	349	372	349	372
V ₂ O ₅ , Vanadium(V) oxide (ppm)	643	37	622	664	622	664
W, Tungsten (ppm)	3.74	0.70	3.20	4.28	IND	IND
WO ₃ , Tungsten trioxide (ppm)	4.71	0.88	4.03	5.40	IND	IND
Y, Yttrium (ppm)	180	8	175	185	174	186
Y ₂ O ₃ , Yttrium(III) oxide (ppm)	229	10	223	235	222	236
Yb, Ytterbium (ppm)	7.03	0.414	6.81	7.24	6.70	7.35
Yb ₂ O ₃ , Ytterbium(III) oxide (ppm)	8.00	0.471	7.76	8.24	7.63	8.37
Zn, Zinc (ppm)	422	54	364	481	397	448
ZnO, Zinc oxide (ppm)	526	67	453	599	494	558
Zr, Zirconium (ppm)	576	24	560	593	552	600
ZrO ₂ , Zirconium dioxide (ppm)	778	33	756	801	746	811
4-Acid Digestion						
Ag, Silver (ppm)	< 3	IND	IND	IND	IND	IND
Al, Aluminium (wt.%)	5.47	0.121	5.41	5.53	5.38	5.56
As, Arsenic (ppm)	31.3	1.75	30.0	32.5	29.4	33.1
Ba, Barium (ppm)	1135	46.0	1110	1160	1106	1164
Be, Beryllium (ppm)	5.32	0.56	4.99	5.65	5.10	5.54
Bi, Bismuth (ppm)	2.93	0.180	2.81	3.04	2.80	3.06
Ca, Calcium (wt.%)	1.19	0.072	1.14	1.23	1.17	1.21
Ce, Cerium (wt.%)	0.654	0.022	0.632	0.676	0.643	0.665
Co, Cobalt (ppm)	13.5	0.61	13.2	13.9	13.1	14.0
Cr, Chromium (ppm)	460	86	401	520	446	474
Cs, Cesium (ppm)	0.42	0.06	0.39	0.46	0.40	0.44
Cu, Copper (ppm)	65	3.2	64	67	63	68
Dy, Dysprosium (ppm)	71	2.4	69	72	69	73
Er, Erbium (ppm)	14.8	0.56	14.5	15.2	14.3	15.4
Eu, Europium (ppm)	121	7.2	116	126	118	123
Fe, Iron (wt.%)	33.33	1.079	32.68	33.99	32.87	33.79
Gd, Gadolinium (ppm)	244	11.3	237	252	239	250
Hf, Hafnium (ppm)	7.40	0.88	6.71	8.09	7.02	7.77
Ho, Holmium (ppm)	8.37	0.469	8.08	8.67	8.13	8.62
In, Indium (ppm)	0.95	0.061	0.92	0.99	0.91	1.00
K, Potassium (wt.%)	0.109	0.009	0.104	0.115	IND	IND
La, Lanthanum (ppm)	4773	277.0	4548	4997	4652	4893
Li, Lithium (ppm)	10.5	1.2	9.8	11.2	10.1	10.9

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

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						
Lu, Lutetium (ppm)	0.67	0.10	0.61	0.73	0.63	0.71
Mg, Magnesium (wt.%)	1.01	0.069	0.96	1.06	0.99	1.03
Mn, Manganese (wt.%)	0.112	0.008	0.106	0.117	0.109	0.114
Mo, Molybdenum (ppm)	57	3.6	55	60	56	59
Na, Sodium (wt.%)	0.173	0.011	0.166	0.179	IND	IND
Nd, Neodymium (ppm)	3611	108.5	3542	3680	3545	3678
Ni, Nickel (ppm)	76	4.4	74	79	74	79
P, Phosphorus (wt.%)	0.590	0.054	0.553	0.627	0.580	0.601
Pb, Lead (ppm)	130	5.5	126	134	128	132
Pr, Praseodymium (ppm)	986	35.9	963	1008	962	1010
Rb, Rubidium (ppm)	6.11	0.580	5.78	6.44	5.92	6.30
Re, Rhenium (ppm)	< 0.02	IND	IND	IND	IND	IND
S, Sulphur (ppm)	624	46.2	600	649	IND	IND
Sb, Antimony (ppm)	2.14	0.22	2.00	2.29	2.05	2.24
Sc, Scandium (ppm)	66	3.4	64	68	65	67
Se, Selenium (ppm)	< 20	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	531	21.0	518	543	521	540
Sn, Tin (ppm)	25.6	2.47	23.9	27.3	24.8	26.4
Sr, Strontium (ppm)	934	66.9	891	977	920	948
Ta, Tantalum (ppm)	21.7	2.3	19.7	23.6	21.0	22.3
Tb, Terbium (ppm)	20.6	0.87	20.1	21.1	19.9	21.3
Te, Tellurium (ppm)	0.35	0.06	0.31	0.40	0.32	0.39
Th, Thorium (ppm)	291	10.2	285	297	284	298
Ti, Titanium (wt.%)	0.876	0.163	0.762	0.989	0.844	0.908
Tl, Thallium (ppm)	0.094	0.011	0.091	0.097	IND	IND
Tm, Thulium (ppm)	1.33	0.117	1.26	1.41	1.28	1.38
U, Uranium (ppm)	7.55	0.368	7.32	7.77	7.35	7.75
V, Vanadium (ppm)	327	25.7	309	345	318	337
W, Tungsten (ppm)	2.45	0.38	2.19	2.70	2.30	2.59
Y, Yttrium (ppm)	176	10.0	169	183	172	181
Yb, Ytterbium (ppm)	5.86	0.346	5.65	6.07	5.63	6.09
Zn, Zinc (ppm)	391	29.6	371	411	385	398
Zr, Zirconium (ppm)	256	25.4	238	273	244	268

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 463.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al ₂ O ₃	wt. %	10.73	Lu ₂ O ₃	ppm	< 20	Tb ₄ O ₇	ppm	35.0
BaO	ppm	1333	MgO	wt. %	1.71	TiO ₂	wt. %	3.19
CaO	wt. %	1.75	MnO	wt. %	0.169	Tm ₂ O ₃	ppm	< 10
Cr ₂ O ₃	ppm	573	Na ₂ O	wt. %	0.260	U ₃ O ₈	ppm	< 100
Dy ₂ O ₃	ppm	72	Nb ₂ O ₅	ppm	2135	V ₂ O ₅	ppm	683
Er ₂ O ₃	ppm	20.0	P ₂ O ₅	wt. %	1.45	WO ₃	ppm	< 100
Eu ₂ O ₃	ppm	94	SiO ₂	wt. %	27.23	Y ₂ O ₃	ppm	212
Gd ₂ O ₃	ppm	283	SnO ₂	ppm	< 100	Yb ₂ O ₃	ppm	10.0
HfO ₂	ppm	< 100	SO ₃	wt. %	0.156	ZrO ₂	ppm	733
Ho ₂ O ₃	ppm	10.0	SrO	ppm	950			
K ₂ O	wt. %	0.142	Ta ₂ O ₅	ppm	< 100			
Thermogravimetry								
H ₂ O-	wt. %	0.640						
Borate / Peroxide Fusion ICP								
Ag	ppm	9.67	Ga [†]	ppm	63	Sc	ppm	92
As	ppm	179	Ge	ppm	9.08	Se	ppm	16.7
B	ppm	147	Na	wt. %	0.162	Te	ppm	< 1
Cd	ppm	< 1	Re	ppm	< 0.1	Tl	ppm	< 0.5
4-Acid Digestion								
Cd	ppm	0.17	Ge	ppm	8.27			
Ga	ppm	60	Nb	ppm	1296			

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt. % $\equiv$ 1000 ppb (parts per billion).

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

[†]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 463 is an ore grade, rare earth element (TREO = 2.08%) matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. The materials constituting OREAS 463 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 463 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%).

COMMINUTION AND HOMOGENISATION PROCEDURES

The source materials (waste, low and medium REE ores) constituting OREAS 463 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 463. 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 463 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 463.

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.%	0.815	0.016	0.783	0.848	0.767	0.864	1.98%	3.97%	5.95%	0.775	0.856
Fe ₂ O ₃ , wt.%	49.48	1.113	47.25	51.71	46.14	52.82	2.25%	4.50%	6.75%	47.01	51.95
La ₂ O ₃ , ppm	5917	96	5725	6108	5629	6204	1.62%	3.24%	4.86%	5621	6212
Nd ₂ O ₃ , ppm	4330	230	3871	4789	3641	5019	5.30%	10.61%	15.91%	4114	4547
Pr ₆ O ₁₁ , ppm	1201	80	1041	1361	961	1441	6.67%	13.34%	20.01%	1141	1261
Sm ₂ O ₃ , ppm	597	107	383	810	276	917	17.89%	35.79%	53.68%	567	626
Thermogravimetry											
LOI, wt.%	0.781	0.075	0.631	0.931	0.556	1.006	9.60%	19.21%	28.81%	0.742	0.820
Borate / Peroxide Fusion ICP (majors and REE's shown in both oxide and elemental format)											
Al, wt.%	5.63	0.210	5.21	6.05	5.00	6.26	3.73%	7.45%	11.18%	5.35	5.91

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

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)											
Al ₂ O ₃ , wt. %	10.64	0.396	9.85	11.43	9.45	11.83	3.73%	7.45%	11.18%	10.11	11.17
Ba, ppm	1106	70	967	1246	897	1316	6.31%	12.62%	18.93%	1051	1162
BaO, ppm	1235	78	1079	1391	1001	1469	6.31%	12.62%	18.93%	1173	1297
Be, ppm	5.62	0.420	4.78	6.46	4.36	6.88	7.48%	14.95%	22.43%	5.34	5.90
Bi, ppm	2.75	0.38	2.00	3.51	1.62	3.89	13.72%	27.44%	41.16%	2.62	2.89
Ca, wt. %	1.22	0.058	1.11	1.34	1.05	1.39	4.72%	9.43%	14.15%	1.16	1.28
CaO, wt. %	1.71	0.081	1.55	1.87	1.47	1.95	4.72%	9.43%	14.15%	1.62	1.79
Ce, wt. %	0.659	0.015	0.628	0.690	0.613	0.705	2.34%	4.69%	7.03%	0.626	0.692
CeO ₂ , wt. %	0.810	0.019	0.772	0.848	0.753	0.867	2.34%	4.69%	7.03%	0.769	0.850
Co, ppm	15.2	2.6	9.9	20.4	7.3	23.0	17.28%	34.56%	51.85%	14.4	15.9
Cr, ppm	574	32	509	638	476	671	5.65%	11.30%	16.94%	545	602
Cr ₂ O ₃ , ppm	838	47	744	933	696	980	5.65%	11.30%	16.94%	796	880
Cs, ppm	0.42	0.05	0.32	0.53	0.26	0.58	12.59%	25.19%	37.78%	0.40	0.44
Cu, ppm	74	16	42	107	26	123	21.72%	43.45%	65.17%	71	78
Dy, ppm	70	3.3	64	77	61	80	4.71%	9.42%	14.12%	67	74
Dy ₂ O ₃ , ppm	81	3.8	73	88	69	92	4.71%	9.42%	14.12%	77	85
Er, ppm	16.0	1.07	13.9	18.1	12.8	19.2	6.68%	13.36%	20.03%	15.2	16.8
Er ₂ O ₃ , ppm	18.3	1.22	15.8	20.7	14.6	21.9	6.68%	13.36%	20.03%	17.4	19.2
Eu, ppm	115	4	106	124	102	128	3.79%	7.57%	11.36%	109	121
Eu ₂ O ₃ , ppm	133	5	123	143	118	148	3.79%	7.57%	11.36%	127	140
Fe, wt. %	34.47	1.340	31.79	37.15	30.45	38.49	3.89%	7.77%	11.66%	32.75	36.20
Fe ₂ O ₃ , wt. %	49.29	1.915	45.45	53.12	43.54	55.03	3.89%	7.77%	11.66%	46.82	51.75
Gd, ppm	241	13	215	267	202	281	5.46%	10.93%	16.39%	229	253
Gd ₂ O ₃ , ppm	278	15	247	308	232	323	5.46%	10.93%	16.39%	264	292
Hf, ppm	13.8	0.45	12.9	14.7	12.4	15.1	3.29%	6.59%	9.88%	13.1	14.5
HfO ₂ , ppm	16.3	0.54	15.2	17.3	14.7	17.9	3.29%	6.59%	9.88%	15.4	17.1
Ho, ppm	8.71	0.567	7.58	9.85	7.01	10.42	6.51%	13.02%	19.54%	8.28	9.15
Ho ₂ O ₃ , ppm	9.98	0.650	8.68	11.28	8.03	11.93	6.51%	13.02%	19.54%	9.48	10.48
In, ppm	1.01	0.072	0.87	1.15	0.79	1.23	7.13%	14.27%	21.40%	0.96	1.06
K, wt. %	0.119	0.020	0.078	0.160	0.058	0.180	17.13%	34.26%	51.39%	0.113	0.125
K ₂ O, wt. %	0.143	0.025	0.094	0.192	0.070	0.217	17.13%	34.26%	51.39%	0.136	0.150
La, ppm	4966	139	4687	5244	4548	5384	2.81%	5.61%	8.42%	4717	5214
La ₂ O ₃ , ppm	5824	163	5497	6151	5334	6314	2.81%	5.61%	8.42%	5533	6115
Li, ppm	10.4	1.9	6.6	14.3	4.6	16.2	18.49%	36.98%	55.47%	9.9	10.9
Lu, ppm	0.79	0.040	0.71	0.87	0.67	0.91	5.09%	10.18%	15.27%	0.75	0.83
Lu ₂ O ₃ , ppm	0.90	0.046	0.81	0.99	0.76	1.04	5.09%	10.18%	15.27%	0.86	0.95
Mg, wt. %	1.02	0.054	0.91	1.13	0.86	1.19	5.31%	10.61%	15.92%	0.97	1.07
MgO, wt. %	1.70	0.090	1.52	1.88	1.43	1.97	5.31%	10.61%	15.92%	1.61	1.78
Mn, wt. %	0.121	0.009	0.102	0.140	0.093	0.149	7.75%	15.50%	23.24%	0.115	0.127
MnO, wt. %	0.156	0.012	0.132	0.180	0.120	0.192	7.75%	15.50%	23.24%	0.148	0.164
Mo, ppm	56	2.4	51	61	49	63	4.25%	8.49%	12.74%	53	59
Nb, ppm	1495	73	1349	1641	1276	1715	4.89%	9.77%	14.66%	1421	1570
Nb2O5, ppm	2139	105	1930	2348	1826	2453	4.89%	9.77%	14.66%	2032	2246
Nd, ppm	3682	185	3313	4051	3129	4236	5.01%	10.02%	15.03%	3498	3866
Nd2O3, ppm	4295	215	3865	4726	3649	4941	5.01%	10.02%	15.03%	4080	4510

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt. % $\equiv$ 1000 ppb (parts per billion).

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)											
Ni, ppm	71	11	48	93	37	104	15.94%	31.88%	47.82%	67	74
NiO, ppm	90	14	61	119	47	133	15.94%	31.88%	47.82%	85	94
P, wt. %	0.629	0.024	0.581	0.676	0.558	0.700	3.77%	7.54%	11.32%	0.597	0.660
P2O5, wt. %	1.44	0.054	1.33	1.55	1.28	1.60	3.77%	7.54%	11.32%	1.37	1.51
Pb, ppm	122	6	110	135	104	141	5.07%	10.14%	15.22%	116	128
PbO, ppm	132	7	118	145	112	152	5.07%	10.14%	15.22%	125	138
Pr, ppm	1004	43	918	1090	875	1133	4.28%	8.57%	12.85%	953	1054
Pr6O11, ppm	1212	52	1109	1316	1057	1368	4.28%	8.57%	12.85%	1152	1273
Rb, ppm	6.08	0.336	5.41	6.75	5.07	7.09	5.53%	11.06%	16.59%	5.78	6.38
S, ppm	671	104	463	879	359	983	15.52%	31.05%	46.57%	637	705
Sb, ppm	2.31	0.40	1.51	3.10	1.12	3.50	17.20%	34.40%	51.60%	2.19	2.42
Si, wt. %	12.85	0.392	12.07	13.64	11.68	14.03	3.05%	6.10%	9.16%	12.21	13.50
SiO2, wt. %	27.50	0.839	25.82	29.17	24.98	30.01	3.05%	6.10%	9.16%	26.12	28.87
Sm, ppm	538	11	516	559	505	570	2.01%	4.02%	6.03%	511	565
Sm2O3, ppm	624	13	598	649	586	661	2.01%	4.02%	6.03%	592	655
Sn, ppm	31.4	3.09	25.2	37.6	22.1	40.7	9.85%	19.69%	29.54%	29.8	33.0
SnO2, ppm	39.9	3.93	32.0	47.7	28.1	51.7	9.85%	19.69%	29.54%	37.9	41.9
Sr, ppm	961	26	910	1012	884	1038	2.67%	5.34%	8.02%	913	1009
SrO, ppm	1136	30	1076	1197	1045	1227	2.67%	5.34%	8.02%	1080	1193
Ta, ppm	25.2	1.28	22.7	27.8	21.4	29.1	5.06%	10.12%	15.17%	24.0	26.5
Ta2O5, ppm	30.8	1.56	27.7	33.9	26.1	35.5	5.06%	10.12%	15.17%	29.3	32.4
Tb, ppm	20.3	0.95	18.4	22.2	17.4	23.2	4.70%	9.40%	14.10%	19.3	21.3
Tb4O7, ppm	23.9	1.12	21.6	26.1	20.5	27.2	4.70%	9.40%	14.10%	22.7	25.1
Th, ppm	292	11	270	314	259	325	3.79%	7.57%	11.36%	278	307
ThO2, ppm	332	13	307	358	295	370	3.79%	7.57%	11.36%	316	349
Ti, wt. %	1.92	0.068	1.79	2.06	1.72	2.13	3.54%	7.08%	10.63%	1.83	2.02
TiO2, wt. %	3.21	0.114	2.98	3.43	2.87	3.55	3.54%	7.08%	10.63%	3.05	3.37
Tm, ppm	1.57	0.101	1.37	1.77	1.27	1.88	6.41%	12.82%	19.23%	1.49	1.65
Tm2O3,	1.80	0.115	1.57	2.03	1.45	2.14	6.41%	12.82%	19.23%	1.71	1.89
U, ppm	7.85	0.294	7.27	8.44	6.97	8.73	3.74%	7.48%	11.21%	7.46	8.24
U3O8, ppm	9.26	0.346	8.57	9.95	8.22	10.30	3.74%	7.48%	11.21%	8.80	9.72
V, ppm	360	21	319	402	298	423	5.78%	11.56%	17.34%	342	378
V2O5, ppm	643	37	569	718	532	755	5.78%	11.56%	17.34%	611	675
W, ppm	3.74	0.70	2.34	5.14	1.64	5.84	18.74%	37.48%	56.22%	3.55	3.93
WO3, ppm	4.71	0.88	2.95	6.48	2.06	7.36	18.74%	37.48%	56.22%	4.48	4.95
Y, ppm	180	8	164	196	156	204	4.48%	8.97%	13.45%	171	189
Y2O3, ppm	229	10	208	249	198	260	4.48%	8.97%	13.45%	217	240
Yb, ppm	7.03	0.414	6.20	7.85	5.78	8.27	5.89%	11.78%	17.67%	6.67	7.38
Yb2O3, ppm	8.00	0.471	7.06	8.94	6.59	9.41	5.89%	11.78%	17.67%	7.60	8.40
Zn, ppm	422	54	315	530	262	583	12.67%	25.34%	38.01%	401	444
ZnO, ppm	526	67	393	659	326	726	12.67%	25.34%	38.01%	500	552
Zr, ppm	576	24	528	625	504	649	4.19%	8.39%	12.58%	547	605
ZrO2, ppm	778	33	713	844	680	876	4.19%	8.39%	12.58%	739	817
4-Acid Digestion											
Ag, ppm	< 3	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt. % $\equiv$ 1000 ppb (parts per billion).

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											
Al, wt. %	5.47	0.121	5.23	5.71	5.11	5.83	2.21%	4.42%	6.62%	5.20	5.74
As, ppm	31.3	1.75	27.8	34.8	26.0	36.5	5.60%	11.19%	16.79%	29.7	32.8
Ba, ppm	1135	46	1043	1227	997	1273	4.05%	8.10%	12.15%	1078	1192
Be, ppm	5.32	0.56	4.20	6.44	3.64	7.00	10.52%	21.04%	31.56%	5.05	5.59
Bi, ppm	2.93	0.180	2.57	3.29	2.39	3.47	6.15%	12.30%	18.45%	2.78	3.07
Ca, wt. %	1.19	0.072	1.04	1.33	0.97	1.40	6.06%	12.13%	18.19%	1.13	1.25
Ce, wt. %	0.654	0.022	0.610	0.698	0.588	0.721	3.39%	6.78%	10.17%	0.621	0.687
Co, ppm	13.5	0.61	12.3	14.8	11.7	15.4	4.50%	8.99%	13.49%	12.9	14.2
Cr, ppm	460	86	288	632	202	718	18.67%	37.33%	56.00%	437	483
Cs, ppm	0.42	0.06	0.31	0.53	0.26	0.59	13.15%	26.30%	39.45%	0.40	0.44
Cu, ppm	65	3.2	59	72	56	75	4.88%	9.76%	14.64%	62	69
Dy, ppm	71	2.4	66	76	63	78	3.43%	6.85%	10.28%	67	74
Er, ppm	14.8	0.56	13.7	15.9	13.1	16.5	3.80%	7.60%	11.41%	14.1	15.6
Eu, ppm	121	7	107	135	99	143	5.97%	11.95%	17.92%	115	127
Fe, wt. %	33.33	1.079	31.18	35.49	30.10	36.57	3.24%	6.47%	9.71%	31.67	35.00
Gd, ppm	244	11	222	267	210	279	4.64%	9.28%	13.92%	232	257
Hf, ppm	7.40	0.88	5.64	9.16	4.76	10.04	11.89%	23.77%	35.66%	7.03	7.77
Ho, ppm	8.37	0.469	7.44	9.31	6.97	9.78	5.60%	11.19%	16.79%	7.96	8.79
In, ppm	0.95	0.061	0.83	1.08	0.77	1.14	6.40%	12.80%	19.20%	0.91	1.00
K, wt. %	0.109	0.009	0.091	0.128	0.081	0.138	8.54%	17.08%	25.62%	0.104	0.115
La, ppm	4773	277	4219	5327	3942	5604	5.80%	11.61%	17.41%	4534	5011
Li, ppm	10.5	1.2	8.0	13.0	6.8	14.2	11.68%	23.37%	35.05%	10.0	11.0
Lu, ppm	0.67	0.10	0.48	0.86	0.39	0.96	14.11%	28.22%	42.33%	0.64	0.71
Mg, wt. %	1.01	0.069	0.87	1.15	0.80	1.22	6.85%	13.70%	20.54%	0.96	1.06
Mn, wt. %	0.112	0.008	0.097	0.127	0.089	0.134	6.73%	13.46%	20.19%	0.106	0.117
Mo, ppm	57	3.6	50	64	46	68	6.27%	12.54%	18.81%	54	60
Na, wt. %	0.173	0.011	0.150	0.195	0.139	0.207	6.49%	12.99%	19.48%	0.164	0.182
Nd, ppm	3611	108	3394	3828	3286	3937	3.00%	6.01%	9.01%	3431	3792
Ni, ppm	76	4.4	68	85	63	90	5.72%	11.43%	17.15%	73	80
P, wt. %	0.590	0.054	0.482	0.699	0.428	0.753	9.17%	18.33%	27.50%	0.561	0.620
Pb, ppm	130	5	119	141	114	147	4.22%	8.44%	12.67%	124	137
Pr, ppm	986	36	914	1058	878	1093	3.64%	7.29%	10.93%	936	1035
Rb, ppm	6.11	0.580	4.95	7.27	4.37	7.85	9.49%	18.98%	28.47%	5.80	6.42
Re, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, ppm	624	46	532	717	486	763	7.40%	14.79%	22.19%	593	656
Sb, ppm	2.14	0.22	1.70	2.59	1.48	2.81	10.38%	20.76%	31.14%	2.04	2.25
Sc, ppm	66	3.4	59	73	56	76	5.16%	10.33%	15.49%	63	69
Se, ppm	< 20	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	531	21	489	573	468	594	3.95%	7.90%	11.85%	504	557
Sn, ppm	25.6	2.47	20.7	30.5	18.2	33.0	9.64%	19.28%	28.92%	24.3	26.9
Sr, ppm	934	67	800	1068	733	1135	7.16%	14.32%	21.48%	887	981
Ta, ppm	21.7	2.3	17.0	26.3	14.6	28.7	10.80%	21.59%	32.39%	20.6	22.7
Tb, ppm	20.6	0.87	18.9	22.3	18.0	23.2	4.22%	8.43%	12.65%	19.6	21.6
Te, ppm	0.35	0.06	0.23	0.48	0.17	0.54	17.45%	34.90%	52.35%	0.33	0.37
Th, ppm	291	10	271	312	261	322	3.51%	7.01%	10.52%	277	306

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt. % $\equiv$ 1000 ppb (parts per billion).

Note: intervals may appear asymmetric due to rounding.

Table 3 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion continued											
Ti, wt. %	0.876	0.163	0.549	1.202	0.386	1.365	18.64%	37.28%	55.91%	0.832	0.919
Tl, ppm	0.094	0.011	0.071	0.117	0.060	0.128	12.08%	24.17%	36.25%	0.089	0.099
Tm, ppm	1.33	0.117	1.10	1.57	0.98	1.68	8.82%	17.65%	26.47%	1.26	1.40
U, ppm	7.55	0.368	6.81	8.28	6.44	8.65	4.88%	9.76%	14.64%	7.17	7.93
V, ppm	327	26	276	379	250	404	7.85%	15.70%	23.55%	311	344
W, ppm	2.45	0.38	1.68	3.21	1.30	3.59	15.65%	31.29%	46.94%	2.32	2.57
Y, ppm	176	10	156	196	146	206	5.69%	11.37%	17.06%	167	185
Yb, ppm	5.86	0.346	5.17	6.55	4.82	6.90	5.90%	11.80%	17.70%	5.57	6.15
Zn, ppm	391	30	332	450	303	480	7.56%	15.11%	22.67%	372	411
Zr, ppm	256	25	205	307	179	332	9.94%	19.88%	29.83%	243	268

SI unit equivalents: ppm (parts per million) $\equiv$ mg/kg $\equiv$ μ g/g $\equiv$ 0.0001 wt.% $\equiv$ 1000 ppb (parts per billion).

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 ($p=0.95$) will have concentrations lying between 5690 and 5957 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 463 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 463 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 463 has been prepared, certified and is supplied by:

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

INTENDED USE

OREAS 463 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 463 has been prepared from ore grade/waste REE bearing ore (TREO = 2.08%). 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 463 has been packaged in single-use, 10g units in laminated foil pouches and 1kg units in plastic jars. In its unopened state and under normal conditions of storage the CRM has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

INSTRUCTIONS FOR CORRECT USE

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

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

TRACEABILITY

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

HANDLING INSTRUCTIONS

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

LEGAL NOTICE

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

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 th September, 2025	Ga reclassified from Certified Value to Indicative Value.
0	28 th April, 2015	First publication.

CERTIFYING OFFICER

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

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.

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