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CERTIFICATE OF ANALYSIS FOR

Glacial Till

CERTIFIED REFERENCE MATERIAL

OREAS 46

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 46.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Pb Fire Assay						
Au, Gold (ppb)	1.61	1.00	1.15	2.06	IND	IND
Pd, Palladium (ppb)	< 1	IND	IND	IND	IND	IND
Pt, Platinum (ppb)	< 1	IND	IND	IND	IND	IND
Borate / Peroxide Fusion ICP						
Al_2O_3, Aluminium(III) oxide (wt.%)	12.05	0.288	11.91	12.19	11.90	12.20
Ba, Barium (ppm)	461	15	452	469	452	469
Be, Beryllium (ppm)	0.92	0.16	0.81	1.02	IND	IND
CaO, Calcium oxide (wt.%)	3.40	0.087	3.36	3.44	3.34	3.46
Ce, Cerium (ppm)	37.2	2.65	35.8	38.7	35.4	39.1
Co, Cobalt (ppm)	9.93	0.742	9.29	10.57	9.40	10.46
Cr_2O_3, Chromium(III) oxide (ppm)	97	7.2	92	101	IND	IND
Cs, Cesium (ppm)	0.62	0.039	0.59	0.65	0.57	0.67
Dy, Dysprosium (ppm)	2.02	0.074	1.99	2.04	1.92	2.12
Er, Erbium (ppm)	1.12	0.050	1.10	1.14	1.07	1.17
Eu, Europium (ppm)	0.88	0.036	0.87	0.90	0.83	0.94
Fe_2O_3, Iron(III) oxide (wt.%)	3.79	0.091	3.74	3.84	3.72	3.86
Ga, Gallium (ppm)	14.1	0.76	13.6	14.6	13.7	14.4
Gd, Gadolinium (ppm)	2.60	0.154	2.53	2.66	2.42	2.77
Hf, Hafnium (ppm)	4.11	0.240	4.00	4.22	3.75	4.47
Ho, Holmium (ppm)	0.40	0.023	0.39	0.41	0.37	0.43

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 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate / Peroxide Fusion ICP continued						
K_2O, Potassium oxide (wt.%)	1.42	0.045	1.40	1.44	1.39	1.45
La, Lanthanum (ppm)	19.7	1.39	18.9	20.5	18.8	20.6
Lu, Lutetium (ppm)	0.16	0.011	0.16	0.16	IND	IND
MgO, Magnesium oxide (wt.%)	1.61	0.045	1.59	1.63	1.58	1.64
MnO, Manganese oxide (wt.%)	0.064	0.004	0.062	0.065	IND	IND
Mo, Molybdenum (ppm)	< 2	IND	IND	IND	IND	IND
Na_2O, Sodium oxide (wt.%)	3.49	0.070	3.45	3.52	3.44	3.54
Nb, Niobium (ppm)	5.42	0.338	5.20	5.64	5.01	5.83
Nd, Neodymium (ppm)	18.7	0.77	18.2	19.1	17.9	19.4
Ni, Nickel (ppm)	30.7	3.9	25.5	36.0	28.2	33.3
P_2O_5, Phosphorus(V) oxide (wt.%)	0.122	0.015	0.117	0.127	IND	IND
Pr, Praseodymium (ppm)	4.81	0.219	4.70	4.92	4.64	4.99
Rb, Rubidium (ppm)	33.5	1.64	32.6	34.4	32.7	34.4
Sc, Scandium (ppm)	9.17	0.461	8.58	9.75	IND	IND
SiO_2, Silicon dioxide (wt.%)	72.73	1.120	72.30	73.16	71.79	73.66
Sm, Samarium (ppm)	3.33	0.188	3.24	3.41	3.14	3.52
Sr, Strontium (ppm)	404	13	397	411	392	416
Ta, Tantalum (ppm)	0.31	0.09	0.26	0.36	IND	IND
Tb, Terbium (ppm)	0.36	0.020	0.35	0.37	0.33	0.40
Th, Thorium (ppm)	3.23	0.248	3.11	3.34	3.06	3.39
TiO_2, Titanium dioxide (wt.%)	0.372	0.013	0.366	0.378	0.360	0.384
Tm, Thulium (ppm)	0.16	0.012	0.16	0.17	IND	IND
U, Uranium (ppm)	0.74	0.036	0.72	0.76	0.68	0.79
V, Vanadium (ppm)	60	3.2	58	62	56	64
W, Tungsten (ppm)	< 1	IND	IND	IND	IND	IND
Y, Yttrium (ppm)	11.4	0.68	11.0	11.8	10.9	11.9
Yb, Ytterbium (ppm)	1.07	0.054	1.04	1.09	0.97	1.16
Zr, Zirconium (ppm)	159	12	152	166	147	171
Thermogravimetry						
LOI^{1000}, Loss on ignition @1000°C (wt.%)	1.06	0.22	0.91	1.22	1.02	1.11
4-Acid Digestion						
Ag, Silver (ppm)	0.038	0.007	0.032	0.043	0.034	0.041
Al, Aluminium (wt.%)	6.26	0.160	6.19	6.32	6.15	6.37
As, Arsenic (ppm)	1.01	0.24	0.80	1.22	0.88	1.14
Ba, Barium (ppm)	473	12	467	478	463	482
Be, Beryllium (ppm)	0.91	0.077	0.88	0.95	0.82	1.00
Bi, Bismuth (ppm)	0.054	0.006	0.053	0.056	0.050	0.059
Ca, Calcium (wt.%)	2.40	0.042	2.39	2.42	2.35	2.45
Cd, Cadmium (ppm)	0.059	0.014	0.050	0.067	0.052	0.066
Ce, Cerium (ppm)	36.4	1.68	35.6	37.2	35.6	37.2

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 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
4-Acid Digestion continued						
Co, Cobalt (ppm)	9.83	0.397	9.64	10.02	9.54	10.13
Cr, Chromium (ppm)	45.7	8.5	41.5	49.9	43.6	47.8
Cs, Cesium (ppm)	0.64	0.035	0.62	0.66	0.60	0.68
Cu, Copper (ppm)	23.1	0.60	22.9	23.3	22.3	23.8
Dy, Dysprosium (ppm)	2.03	0.112	1.97	2.08	1.95	2.11
Er, Erbium (ppm)	1.13	0.071	1.09	1.18	1.09	1.17
Eu, Europium (ppm)	0.89	0.052	0.86	0.92	0.86	0.92
Fe, Iron (wt.%)	2.61	0.067	2.58	2.64	2.56	2.66
Ga, Gallium (ppm)	14.0	0.51	13.7	14.2	13.6	14.3
Gd, Gadolinium (ppm)	2.66	0.220	2.55	2.77	2.54	2.79
Hf, Hafnium (ppm)	1.82	0.154	1.76	1.89	1.72	1.92
Ho, Holmium (ppm)	0.39	0.021	0.38	0.40	0.38	0.41
In, Indium (ppm)	0.026	0.005	0.023	0.029	0.022	0.029
K, Potassium (wt.%)	1.19	0.030	1.17	1.20	1.16	1.21
La, Lanthanum (ppm)	18.9	1.13	18.4	19.4	18.4	19.4
Li, Lithium (ppm)	10.4	0.46	10.2	10.6	10.1	10.8
Lu, Lutetium (ppm)	0.15	0.011	0.14	0.15	0.14	0.15
Mg, Magnesium (wt.%)	0.943	0.026	0.931	0.955	0.925	0.961
Mn, Manganese (wt.%)	0.049	0.002	0.048	0.050	0.048	0.050
Mo, Molybdenum (ppm)	0.77	0.065	0.73	0.81	0.71	0.84
Na, Sodium (wt.%)	2.61	0.084	2.57	2.65	2.56	2.67
Nb, Niobium (ppm)	4.56	0.411	4.36	4.77	4.37	4.76
Nd, Neodymium (ppm)	18.5	0.56	18.3	18.8	18.0	19.0
Ni, Nickel (ppm)	26.8	1.04	26.3	27.3	25.7	27.9
P, Phosphorus (wt.%)	0.054	0.002	0.053	0.055	0.053	0.056
Pb, Lead (ppm)	7.02	0.191	6.97	7.06	6.64	7.39
Pr, Praseodymium (ppm)	4.84	0.222	4.74	4.95	4.66	5.02
Rb, Rubidium (ppm)	33.5	1.59	32.8	34.3	32.7	34.4
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND	IND
S, Sulphur (wt.%)	< 0.005	IND	IND	IND	IND	IND
Sb, Antimony (ppm)	0.10	0.02	0.10	0.11	IND	IND
Sc, Scandium (ppm)	8.75	0.358	8.55	8.95	8.50	9.00
Sm, Samarium (ppm)	3.32	0.180	3.23	3.41	3.20	3.44
Sn, Tin (ppm)	0.78	0.066	0.73	0.83	0.73	0.83
Sr, Strontium (ppm)	408	13	402	413	400	415
Ta, Tantalum (ppm)	0.27	0.04	0.24	0.29	0.25	0.29
Tb, Terbium (ppm)	0.36	0.025	0.35	0.37	0.35	0.37
Th, Thorium (ppm)	3.26	0.220	3.17	3.35	3.12	3.40
Ti, Titanium (wt.%)	0.207	0.010	0.203	0.212	0.201	0.214
Tl, Thallium (ppm)	0.21	0.016	0.20	0.22	0.20	0.22
Tm, Thulium (ppm)	0.15	0.011	0.15	0.16	0.14	0.16

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 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
4-Acid Digestion continued						
U, Uranium (ppm)	0.70	0.042	0.68	0.72	0.66	0.74
V, Vanadium (ppm)	57	2.1	56	58	55	58
W, Tungsten (ppm)	0.21	0.04	0.18	0.24	0.17	0.26
Y, Yttrium (ppm)	10.5	0.36	10.4	10.6	10.2	10.7
Yb, Ytterbium (ppm)	1.01	0.060	0.98	1.03	0.96	1.06
Zn, Zinc (ppm)	35.5	1.36	35.0	36.0	34.3	36.7
Zr, Zirconium (ppm)	61	4.7	59	63	58	64
Aqua Regia Digestion (sample weights 0.15-50g)						
Ag, Silver (ppm)	0.025	0.002	0.023	0.026	0.022	0.028
Al, Aluminium (wt.%)	0.748	0.067	0.712	0.784	0.730	0.765
As, Arsenic (ppm)	0.72	0.10	0.66	0.77	0.61	0.83
Au, Gold (ppb)	< 2	IND	IND	IND	IND	IND
Ba, Barium (ppm)	55	3.6	53	57	54	57
Be, Beryllium (ppm)	0.17	0.02	0.15	0.18	IND	IND
Bi, Bismuth (ppm)	0.028	0.006	0.022	0.033	0.025	0.030
Ca, Calcium (wt.%)	0.600	0.053	0.571	0.630	0.586	0.615
Cd, Cadmium (ppm)	0.036	0.007	0.032	0.040	0.031	0.041
Ce, Cerium (ppm)	27.3	2.17	26.2	28.4	26.5	28.1
Co, Cobalt (ppm)	5.83	0.582	5.50	6.16	5.64	6.01
Cr, Chromium (ppm)	25.0	2.18	23.9	26.1	24.0	26.0
Cs, Cesium (ppm)	0.36	0.022	0.35	0.37	0.34	0.38
Cu, Copper (ppm)	23.4	1.21	22.8	23.9	22.6	24.1
Fe, Iron (wt.%)	1.45	0.108	1.40	1.51	1.43	1.48
Ga, Gallium (ppm)	2.99	0.40	2.74	3.23	2.88	3.10
Gd, Gadolinium (ppm)	1.64	0.29	1.32	1.96	1.57	1.71
Ge, Germanium (ppm)	0.061	0.008	0.052	0.070	0.051	0.070
Ho, Holmium (ppm)	0.20	0.04	0.15	0.25	IND	IND
K, Potassium (wt.%)	0.109	0.010	0.103	0.114	0.107	0.111
La, Lanthanum (ppm)	15.5	1.16	14.8	16.1	14.9	16.0
Li, Lithium (ppm)	6.78	0.669	6.35	7.22	6.53	7.03
Lu, Lutetium (ppm)	0.067	0.012	0.054	0.080	IND	IND
Mg, Magnesium (wt.%)	0.459	0.047	0.434	0.484	0.445	0.474
Mn, Manganese (wt.%)	0.025	0.002	0.024	0.026	0.024	0.026
Mo, Molybdenum (ppm)	0.66	0.041	0.64	0.67	0.62	0.69
Na, Sodium (wt.%)	0.077	0.011	0.070	0.084	0.074	0.080
Nd, Neodymium (ppm)	13.2	1.6	11.3	15.0	12.8	13.5
Ni, Nickel (ppm)	16.3	1.11	15.6	17.0	15.8	16.8
P, Phosphorus (wt.%)	0.053	0.002	0.052	0.054	0.052	0.055
Pb, Lead (ppm)	2.02	0.144	1.94	2.10	1.91	2.13
Pd, Palladium (ppb)	< 1	IND	IND	IND	IND	IND

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 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Aqua Regia Digestion (sample weights 0.15-50g) continued						
Pr, Praseodymium (ppm)	3.75	0.129	3.60	3.90	3.65	3.86
Pt, Platinum (ppb)	< 2	IND	IND	IND	IND	IND
Rb, Rubidium (ppm)	6.11	0.337	5.93	6.30	5.94	6.29
Re, Rhenium (ppm)	< 0.001	IND	IND	IND	IND	IND
S, Sulphur (wt.%)	< 0.005	IND	IND	IND	IND	IND
Sb, Antimony (ppm)	0.068	0.009	0.064	0.072	0.063	0.074
Sc, Scandium (ppm)	2.78	0.49	2.51	3.05	2.68	2.88
Se, Selenium (ppm)	< 0.2	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	2.11	0.34	1.73	2.49	1.99	2.23
Sn, Tin (ppm)	0.40	0.06	0.35	0.45	0.38	0.42
Sr, Strontium (ppm)	26.9	4.1	24.6	29.1	26.1	27.7
Tb, Terbium (ppm)	0.20	0.03	0.17	0.23	0.19	0.21
Th, Thorium (ppm)	2.87	0.224	2.76	2.99	2.77	2.98
Ti, Titanium (wt.%)	0.065	0.013	0.057	0.074	0.063	0.068
Tl, Thallium (ppm)	0.061	0.007	0.058	0.065	0.058	0.065
U, Uranium (ppm)	0.43	0.05	0.40	0.46	0.41	0.46
V, Vanadium (ppm)	22.1	2.5	20.8	23.3	21.1	23.0
W, Tungsten (ppm)	0.073	0.013	0.062	0.083	0.066	0.079
Y, Yttrium (ppm)	5.06	0.62	4.66	5.46	4.90	5.22
Yb, Ytterbium (ppm)	0.48	0.10	0.40	0.55	0.46	0.50
Zn, Zinc (ppm)	20.7	2.03	19.5	21.9	19.8	21.5
Zr, Zirconium (ppm)	5.66	1.34	4.82	6.51	5.47	5.85

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.

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

Certified Reference Material (CRM) OREAS 46 is a Canadian glacial basal till sourced from outside of Chibougamau, Quebec by IOS Services Geoscientifique. The till composition reflects the geochemistry of the surrounding Archean greenstone belts and felsic intrusives. Cobbles were removed prior to processing and all material has undergone a sterilisation procedure upon receipt in Australia according to soil import regulations (ISO 11137).

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 46 was prepared in the following manner:

- Sieving to remove larger cobbles (> 50mm);
- Gamma irradiation treatment upon receipt in Australia according to soil import regulations (Class 4.2, 50kgy gamma irradiation according to ISO 11137);
- Drying to constant mass at 105°C;
- Milling to 98% minus 75 microns;
- Homogenisation;
- Packaging in 10g and 60g units in laminated foil pouches and 1kg units in plastic wide-mouth jars.

ANALYTICAL PROGRAM

Twenty commercial analytical laboratories participated in the program to certify the analytes reported in Table 1. The following methods were employed:

- Gold by 25-50g fire assay with ICP-OES (10 laboratories), ICP-MS (5 laboratories) and AAS (3 laboratories) finish;
- Gold by 15-50g aqua regia digestion with ICP-MS (15 laboratories), graphite furnace AAS (1 laboratory) and ICP-OES (1 laboratory) finish;
- Lithium borate fusion or sodium peroxide fusion for full ICP-OES and ICP-MS elemental suites (up to 15 laboratories depending on the element; Only two laboratories used sodium peroxide fusion);
- Thermogravimetry for LOI at 1000° C; (6 laboratories used a conventional muffle furnace and 5 laboratories used a thermogravimetric analyser).
- Four acid digestion for full ICP-OES and ICP-MS elemental suites (up to 20 laboratories depending on the element);
- Aqua regia digestion using 0.15 to 50g sample weights (see note below) for full ICP-OES and ICP-MS elemental suites (up to 18 laboratories depending on the element).

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. Aqua regia is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions which can include the ratio of nitric to hydrochloric acids, acid strength, temperatures, leach times and secondary digestions. Recoveries for sulphide-hosted base metal sulphides approach total values, however, other analytes, in particular the lithophile elements, show greater sensitivity to method parameters. This can result in lack of consensus in an inter-laboratory certification program for these elements. The approach applied here is to report certified values in those instances where reasonable agreement exists amongst a majority of participating laboratories. The results of specific laboratories may differ significantly from the certified values, but will, nonetheless, be valid and reproducible in the context of the specifics of the aqua regia method in use. Users of this reference material should, therefore, be mindful of this limitation when applying the certified values in a quality control program.

For the round robin program twenty 700g test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered

representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking two 120g scoop splits from each of three separate 700g test units. 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 159 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 below shows 30 indicative values. Table 3 provides performance gate intervals for the certified values based on their associated pooled standard deviations. Tabulated results of all elements together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM^3) are presented in the detailed certification data for this CRM (**OREAS 46 DataPack.xlsx**).

Table 2. Indicative Values for OREAS 46.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate / Peroxide Fusion ICP								
Bi	ppm	*< 0.5*	Li	ppm	*8.83*	Te	ppm	*1.50*
Cu	ppm	*17.6*	Pb	ppm	*10.0*	Tl	ppm	*< 0.5*
Ge	ppm	*1.00*	Sb	ppm	*< 0.5*	Zn	ppm	*36.2*
In	ppm	*< 0.5*	Sn	ppm	*1.01*			
4-Acid Digestion								
Ge	ppm	*0.11*	Se	ppm	*0.20*			
Hg	ppm	*0.032*	Te	ppm	*< 0.04*			
Aqua Regia Digestion (sample weights 0.15-50g)								
B	ppm	*< 10*	Hg	ppm	*0.007*	Ta	ppm	*< 0.005*
Dy	ppm	*1.15*	In	ppm	*0.009*	Te	ppm	*0.010*
Er	ppm	*0.55*	Nb	ppm	*0.42*	Tm	ppm	*0.083*
Eu	ppm	*0.48*	Ru	ppm	*< 0.005*			
Hf	ppm	*0.17*	Si	wt.%	*0.037*			
Infrared Combustion								
C	wt.%	*0.090*	S	wt.%	*< 0.02*			

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 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.

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. The Certified Values are the means of accepted laboratory means after outlier filtering.

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.*

Indicative (uncertified) values (Table 2) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where inter-laboratory consensus is poor.

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's take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The SD values thus include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. OREAS prepared 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 percent 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.

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 copper (Cu) by 4-acid digestion, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 22.3 and 23.8 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). *Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.*

The homogeneity of OREAS 46 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty four round robin laboratories received six samples per CRM and these samples were made up of paired samples from three different, non-adjacent sampling intervals. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between-units to that of the variance within-units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 46. The test was performed using the following parameters:

- Null Hypothesis, H_0: Between-unit variance is no greater than within-unit variance (reject H_0 if *p*-value < 0.05);
- Alternative Hypothesis, H_1: Between-unit variance is greater than within-unit variance.

P-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of *p*-values. This process derived no significant *p*-values across the entire 159 certified values except for three elements (Pt by fire assay, MnO by fusion with ICP and Pr by aqua regia digestion). These elements are all present in low concentration levels close to their respective lower levels of detection (LLD) where reading resolution errors can easily lead to ‘false negatives’ (*p*-values detected as ‘significant’ that are in fact immaterial). Usually data becomes more reliable and meaningful when the concentration levels are at least twenty times the LLD. Furthermore, limited data was available for Pt and Pr so the ANOVA values are based on data from 6 laboratories only.

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 46 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 46 is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

Table 3. Pooled-Lab Performance Gates for OREAS 46.

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
Pb Fire Assay											
Au, ppb	1.61	1.00	0.00	3.61	0.00	4.61	62.3%	125%	187%	1.53	1.69
Pd, ppb	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pt, ppb	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Borate / Peroxide Fusion ICP											
Al_2O_3, wt.%	12.05	0.288	11.47	12.62	11.19	12.91	2.39%	4.77%	7.16%	11.45	12.65
Ba, ppm	461	15	430	491	414	507	3.34%	6.67%	10.0%	437	484
Be, ppm	0.92	0.16	0.60	1.24	0.44	1.40	17.4%	34.9%	52.3%	0.87	0.96
CaO, wt.%	3.40	0.087	3.22	3.57	3.14	3.66	2.57%	5.15%	7.72%	3.23	3.57
Ce, ppm	37.2	2.65	32.0	42.5	29.3	45.2	7.11%	14.2%	21.3%	35.4	39.1
Co, ppm	9.93	0.742	8.44	11.41	7.70	12.16	7.48%	15.0%	22.4%	9.43	10.42
Cr_2O_3, ppm	97	7.2	83	111	75	119	7.43%	14.9%	22.3%	92	102
Cs, ppm	0.62	0.039	0.54	0.70	0.50	0.74	6.30%	12.6%	18.9%	0.59	0.65
Dy, ppm	2.02	0.074	1.87	2.17	1.79	2.24	3.69%	7.38%	11.1%	1.92	2.12
Er, ppm	1.12	0.050	1.02	1.22	0.97	1.27	4.46%	8.92%	13.4%	1.07	1.18
Eu, ppm	0.88	0.036	0.81	0.95	0.77	0.99	4.07%	8.14%	12.2%	0.84	0.93
Fe_2O_3, wt.%	3.79	0.091	3.61	3.97	3.51	4.06	2.41%	4.82%	7.23%	3.60	3.98
Ga, ppm	14.1	0.76	12.6	15.6	11.8	16.4	5.38%	10.8%	16.1%	13.4	14.8
Gd, ppm	2.60	0.154	2.29	2.90	2.13	3.06	5.95%	11.9%	17.8%	2.47	2.72
Hf, ppm	4.11	0.240	3.63	4.59	3.39	4.83	5.85%	11.7%	17.5%	3.90	4.32
Ho, ppm	0.40	0.023	0.36	0.45	0.33	0.47	5.76%	11.5%	17.3%	0.38	0.42
K_2O, wt.%	1.42	0.045	1.33	1.51	1.29	1.55	3.13%	6.27%	9.40%	1.35	1.49
La, ppm	19.7	1.39	16.9	22.5	15.5	23.9	7.07%	14.1%	21.2%	18.7	20.7
Lu, ppm	0.16	0.011	0.14	0.18	0.13	0.19	6.84%	13.7%	20.5%	0.15	0.17
MgO, wt.%	1.61	0.045	1.52	1.70	1.48	1.75	2.79%	5.58%	8.37%	1.53	1.69
MnO, wt.%	0.064	0.004	0.056	0.071	0.052	0.075	5.98%	12.0%	18.0%	0.060	0.067
Mo, ppm	< 2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Na_2O, wt.%	3.49	0.070	3.35	3.63	3.28	3.70	2.00%	4.00%	6.00%	3.31	3.66
Nb, ppm	5.42	0.338	4.74	6.09	4.41	6.43	6.23%	12.5%	18.7%	5.15	5.69
Nd, ppm	18.7	0.77	17.1	20.2	16.4	21.0	4.12%	8.23%	12.35%	17.7	19.6
Ni, ppm	30.7	3.9	22.8	38.6	18.9	42.6	12.8%	25.7%	38.5%	29.2	32.3
P_2O_5, wt.%	0.122	0.015	0.092	0.152	0.076	0.167	12.4%	24.8%	37.2%	0.116	0.128
Pr, ppm	4.81	0.219	4.38	5.25	4.16	5.47	4.54%	9.09%	13.6%	4.57	5.06
Rb, ppm	33.5	1.64	30.3	36.8	28.6	38.4	4.88%	9.75%	14.6%	31.9	35.2
Sc, ppm	9.17	0.461	8.24	10.09	7.78	10.55	5.03%	10.1%	15.1%	8.71	9.63
SiO_2, wt.%	72.73	1.120	70.49	74.97	69.37	76.09	1.54%	3.08%	4.62%	69.09	76.36
Sm, ppm	3.33	0.188	2.95	3.71	2.76	3.89	5.66%	11.3%	17.0%	3.16	3.50
Sr, ppm	404	13	378	430	365	443	3.23%	6.46%	9.69%	384	424
Ta, ppm	0.31	0.09	0.13	0.48	0.04	0.57	28.5%	57.1%	85.6%	0.29	0.32
Tb, ppm	0.36	0.020	0.32	0.40	0.30	0.42	5.54%	11.1%	16.6%	0.34	0.38
Th, ppm	3.23	0.248	2.73	3.72	2.48	3.97	7.70%	15.4%	23.1%	3.06	3.39
TiO_2, wt.%	0.372	0.013	0.346	0.397	0.334	0.410	3.42%	6.85%	10.3%	0.353	0.390
Tm, ppm	0.16	0.012	0.14	0.19	0.13	0.20	7.44%	14.9%	22.3%	0.16	0.17

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 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											
U, ppm	0.74	0.036	0.67	0.81	0.63	0.85	4.86%	9.73%	14.6%	0.70	0.77
V, ppm	60	3.2	54	66	50	69	5.27%	10.5%	15.8%	57	63
W, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Y, ppm	11.4	0.68	10.0	12.7	9.3	13.4	6.01%	12.0%	18.0%	10.8	11.9
Yb, ppm	1.07	0.054	0.96	1.17	0.90	1.23	5.11%	10.2%	15.3%	1.01	1.12
Zr, ppm	159	12	135	183	123	195	7.58%	15.2%	22.7%	151	167
Thermogravimetry											
LOI^{1000} wt.%	1.06	0.22	0.62	1.50	0.40	1.72	20.8%	41.6%	62.3%	1.01	1.12
4-Acid Digestion											
Ag, ppm	0.038	0.007	0.023	0.052	0.016	0.059	19.0%	38.0%	57.0%	0.036	0.040
Al, wt.%	6.26	0.160	5.94	6.58	5.78	6.74	2.55%	5.10%	7.65%	5.95	6.57
As, ppm	1.01	0.24	0.52	1.50	0.28	1.74	24.2%	48.4%	72.6%	0.96	1.06
Ba, ppm	473	12	449	496	438	508	2.47%	4.93%	7.40%	449	496
Be, ppm	0.91	0.077	0.76	1.07	0.68	1.15	8.45%	16.9%	25.3%	0.87	0.96
Bi, ppm	0.054	0.006	0.043	0.066	0.038	0.071	10.2%	20.5%	30.7%	0.052	0.057
Ca, wt.%	2.40	0.042	2.32	2.49	2.28	2.53	1.74%	3.47%	5.21%	2.28	2.52
Cd, ppm	0.059	0.014	0.031	0.086	0.017	0.100	23.6%	47.2%	70.8%	0.056	0.062
Ce, ppm	36.4	1.68	33.0	39.8	31.4	41.4	4.62%	9.24%	13.9%	34.6	38.2
Co, ppm	9.83	0.397	9.04	10.62	8.64	11.02	4.04%	8.07%	12.1%	9.34	10.32
Cr, ppm	45.7	8.5	28.7	62.7	20.2	71.2	18.6%	37.2%	55.9%	43.4	48.0
Cs, ppm	0.64	0.035	0.57	0.71	0.53	0.75	5.49%	11.0%	16.5%	0.61	0.67
Cu, ppm	23.1	0.60	21.9	24.3	21.3	24.9	2.61%	5.23%	7.84%	21.9	24.2
Dy, ppm	2.03	0.112	1.80	2.25	1.69	2.36	5.52%	11.0%	16.6%	1.93	2.13
Er, ppm	1.13	0.071	0.99	1.28	0.92	1.35	6.27%	12.5%	18.8%	1.08	1.19
Eu, ppm	0.89	0.052	0.79	1.00	0.74	1.05	5.82%	11.6%	17.4%	0.85	0.94
Fe, wt.%	2.61	0.067	2.48	2.75	2.41	2.81	2.56%	5.11%	7.67%	2.48	2.75
Ga, ppm	14.0	0.51	12.9	15.0	12.4	15.5	3.63%	7.27%	10.9%	13.3	14.7
Gd, ppm	2.66	0.220	2.22	3.10	2.00	3.32	8.26%	16.5%	24.8%	2.53	2.80
Hf, ppm	1.82	0.154	1.52	2.13	1.36	2.29	8.44%	16.9%	25.3%	1.73	1.91
Ho, ppm	0.39	0.021	0.35	0.44	0.33	0.46	5.27%	10.5%	15.8%	0.37	0.41
In, ppm	0.026	0.005	0.016	0.035	0.011	0.040	18.9%	37.7%	56.6%	0.024	0.027
K, wt.%	1.19	0.030	1.13	1.25	1.10	1.28	2.53%	5.05%	7.58%	1.13	1.24
La, ppm	18.9	1.13	16.7	21.2	15.5	22.3	5.95%	11.9%	17.9%	18.0	19.9
Li, ppm	10.4	0.46	9.5	11.3	9.0	11.8	4.44%	8.88%	13.3%	9.9	10.9
Lu, ppm	0.15	0.011	0.13	0.17	0.11	0.18	7.19%	14.4%	21.6%	0.14	0.15
Mg, wt.%	0.943	0.026	0.890	0.996	0.864	1.022	2.80%	5.59%	8.39%	0.896	0.990
Mn, wt.%	0.049	0.002	0.045	0.053	0.043	0.054	3.77%	7.55%	11.3%	0.046	0.051
Mo, ppm	0.77	0.065	0.64	0.90	0.58	0.97	8.35%	16.7%	25.1%	0.73	0.81
Na, wt.%	2.61	0.084	2.44	2.78	2.36	2.86	3.20%	6.40%	9.60%	2.48	2.74
Nb, ppm	4.56	0.411	3.74	5.39	3.33	5.80	9.01%	18.0%	27.0%	4.34	4.79
Nd, ppm	18.5	0.56	17.4	19.6	16.8	20.2	3.03%	6.05%	9.08%	17.6	19.4
Ni, ppm	26.8	1.04	24.7	28.9	23.7	29.9	3.87%	7.73%	11.6%	25.5	28.1

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 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											
P, wt.%	0.054	0.002	0.050	0.058	0.048	0.060	3.75%	7.49%	11.2%	0.052	0.057
Pb, ppm	7.02	0.191	6.63	7.40	6.44	7.59	2.73%	5.45%	8.18%	6.67	7.37
Pr, ppm	4.84	0.222	4.40	5.29	4.17	5.51	4.59%	9.18%	13.8%	4.60	5.08
Rb, ppm	33.5	1.59	30.3	36.7	28.8	38.3	4.75%	9.50%	14.3%	31.9	35.2
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sb, ppm	0.10	0.02	0.06	0.15	0.04	0.17	19.8%	39.6%	59.3%	0.10	0.11
Sc, ppm	8.75	0.358	8.04	9.47	7.68	9.82	4.09%	8.17%	12.3%	8.31	9.19
Sm, ppm	3.32	0.180	2.96	3.68	2.78	3.86	5.42%	10.8%	16.3%	3.16	3.49
Sn, ppm	0.78	0.066	0.65	0.91	0.58	0.98	8.47%	16.9%	25.4%	0.74	0.82
Sr, ppm	408	13	382	433	370	445	3.10%	6.20%	9.29%	387	428
Ta, ppm	0.27	0.04	0.18	0.35	0.14	0.40	15.9%	31.7%	47.6%	0.26	0.28
Tb, ppm	0.36	0.025	0.31	0.41	0.29	0.43	6.84%	13.7%	20.5%	0.34	0.38
Th, ppm	3.26	0.220	2.82	3.70	2.60	3.92	6.73%	13.5%	20.2%	3.10	3.42
Ti, wt.%	0.207	0.010	0.188	0.227	0.178	0.237	4.69%	9.39%	14.1%	0.197	0.218
Tl, ppm	0.21	0.016	0.17	0.24	0.16	0.26	7.89%	15.8%	23.7%	0.20	0.22
Tm, ppm	0.15	0.011	0.13	0.18	0.12	0.19	7.34%	14.7%	22.0%	0.15	0.16
U, ppm	0.70	0.042	0.61	0.78	0.57	0.83	6.07%	12.1%	18.2%	0.66	0.73
V, ppm	57	2.1	52	61	50	63	3.76%	7.52%	11.3%	54	60
W, ppm	0.21	0.04	0.13	0.30	0.08	0.34	20.55%	41.1%	61.6%	0.20	0.22
Y, ppm	10.5	0.36	9.8	11.2	9.4	11.6	3.40%	6.80%	10.2%	10.0	11.0
Yb, ppm	1.01	0.060	0.89	1.13	0.83	1.19	5.96%	11.9%	17.9%	0.96	1.06
Zn, ppm	35.5	1.36	32.8	38.2	31.4	39.6	3.82%	7.64%	11.5%	33.7	37.3
Zr, ppm	61	4.7	52	71	47	76	7.74%	15.5%	23.2%	58	64
Aqua Regia Digestion (sample weights 0.15-50g)											
Ag, ppm	0.025	0.002	0.020	0.029	0.018	0.031	8.37%	16.7%	25.1%	0.023	0.026
Al, wt.%	0.748	0.067	0.614	0.882	0.546	0.949	8.98%	18.0%	26.9%	0.710	0.785
As, ppm	0.72	0.10	0.51	0.92	0.41	1.03	14.5%	29.0%	43.5%	0.68	0.75
Au, ppb	1.18	0.51	0.16	2.20	0.00	2.71	43.3%	86.7%	130.0%	1.12	1.24
Ba, ppm	55	3.6	48	62	44	66	6.59%	13.2%	19.8%	52	58
Be, ppm	0.17	0.02	0.13	0.21	0.11	0.23	11.4%	22.7%	34.1%	0.16	0.18
Bi, ppm	0.028	0.006	0.016	0.039	0.010	0.045	21.2%	42.4%	63.6%	0.026	0.029
Ca, wt.%	0.600	0.053	0.495	0.706	0.442	0.759	8.80%	17.6%	26.4%	0.570	0.630
Cd, ppm	0.036	0.007	0.022	0.050	0.015	0.058	19.7%	39.5%	59.2%	0.034	0.038
Ce, ppm	27.3	2.17	23.0	31.7	20.8	33.8	7.96%	15.9%	23.9%	25.9	28.7
Co, ppm	5.83	0.582	4.66	6.99	4.08	7.58	9.99%	20.0%	30.0%	5.54	6.12
Cr, ppm	25.0	2.18	20.7	29.4	18.5	31.6	8.72%	17.4%	26.2%	23.8	26.3
Cs, ppm	0.36	0.022	0.32	0.40	0.29	0.42	6.04%	12.1%	18.1%	0.34	0.38
Cu, ppm	23.4	1.21	20.9	25.8	19.7	27.0	5.16%	10.3%	15.5%	22.2	24.5
Fe, wt.%	1.45	0.108	1.24	1.67	1.13	1.78	7.42%	14.8%	22.3%	1.38	1.53
Ga, ppm	2.99	0.40	2.20	3.78	1.80	4.17	13.2%	26.4%	39.7%	2.84	3.14
Gd, ppm	1.64	0.29	1.07	2.21	0.78	2.50	17.4%	34.8%	52.3%	1.56	1.72

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 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
Aqua Regia Digestion (sample weights 0.15-50g) continued											
Ge, ppm	0.061	0.008	0.044	0.078	0.035	0.086	13.9%	27.8%	41.8%	0.058	0.064
Ho, ppm	0.20	0.04	0.13	0.28	0.09	0.32	18.7%	37.4%	56.1%	0.19	0.21
K, wt.%	0.109	0.010	0.089	0.128	0.079	0.138	9.06%	18.1%	27.2%	0.103	0.114
La, ppm	15.5	1.16	13.1	17.8	12.0	19.0	7.53%	15.1%	22.6%	14.7	16.2
Li, ppm	6.78	0.669	5.44	8.12	4.78	8.79	9.86%	19.7%	29.6%	6.44	7.12
Lu, ppm	0.067	0.012	0.044	0.090	0.032	0.102	17.3%	34.6%	52.0%	0.064	0.070
Mg, wt.%	0.459	0.047	0.365	0.553	0.319	0.600	10.2%	20.4%	30.6%	0.436	0.482
Mn, wt.%	0.025	0.002	0.021	0.029	0.019	0.031	8.63%	17.3%	25.9%	0.024	0.026
Mo, ppm	0.66	0.041	0.57	0.74	0.53	0.78	6.29%	12.6%	18.9%	0.62	0.69
Na, wt.%	0.077	0.011	0.056	0.098	0.045	0.109	13.9%	27.7%	41.6%	0.073	0.081
Nd, ppm	13.2	1.6	9.9	16.4	8.3	18.0	12.2%	24.4%	36.7%	12.5	13.8
Ni, ppm	16.3	1.11	14.1	18.5	13.0	19.6	6.80%	13.6%	20.4%	15.5	17.1
P, wt.%	0.053	0.002	0.049	0.058	0.047	0.060	4.12%	8.25%	12.4%	0.051	0.056
Pb, ppm	2.02	0.144	1.73	2.31	1.59	2.45	7.15%	14.3%	21.5%	1.92	2.12
Pd, ppb	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pr, ppm	3.75	0.129	3.49	4.01	3.36	4.14	3.44%	6.88%	10.3%	3.56	3.94
Pt, ppb	< 2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Rb, ppm	6.11	0.337	5.44	6.79	5.10	7.12	5.51%	11.0%	16.5%	5.81	6.42
Re, ppm	< 0.001	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sb, ppm	0.068	0.009	0.051	0.085	0.043	0.094	12.5%	25.0%	37.5%	0.065	0.072
Sc, ppm	2.78	0.49	1.80	3.76	1.31	4.25	17.6%	35.3%	52.9%	2.64	2.92
Se, ppm	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	2.11	0.34	1.43	2.79	1.09	3.13	16.1%	32.1%	48.2%	2.01	2.22
Sn, ppm	0.40	0.06	0.27	0.52	0.21	0.59	15.8%	31.6%	47.3%	0.38	0.42
Sr, ppm	26.9	4.1	18.7	35.1	14.6	39.1	15.2%	30.4%	45.5%	25.5	28.2
Tb, ppm	0.20	0.03	0.14	0.27	0.11	0.30	15.8%	31.7%	47.5%	0.19	0.21
Th, ppm	2.87	0.224	2.42	3.32	2.20	3.54	7.81%	15.6%	23.4%	2.73	3.02
Ti, wt.%	0.065	0.013	0.040	0.091	0.027	0.104	19.6%	39.3%	58.9%	0.062	0.069
Tl, ppm	0.061	0.007	0.048	0.075	0.041	0.081	10.8%	21.7%	32.5%	0.058	0.065
U, ppm	0.43	0.05	0.34	0.53	0.29	0.58	11.1%	22.2%	33.2%	0.41	0.45
V, ppm	22.1	2.5	17.1	27.0	14.7	29.4	11.1%	22.3%	33.4%	21.0	23.2
W, ppm	0.073	0.013	0.046	0.100	0.032	0.113	18.5%	37.0%	55.4%	0.069	0.076
Y, ppm	5.06	0.62	3.82	6.30	3.20	6.91	12.2%	24.5%	36.7%	4.81	5.31
Yb, ppm	0.48	0.10	0.28	0.68	0.18	0.78	20.8%	41.7%	62.5%	0.45	0.50
Zn, ppm	20.7	2.03	16.6	24.7	14.6	26.8	9.84%	19.7%	29.5%	19.6	21.7
Zr, ppm	5.66	1.34	2.99	8.33	1.65	9.67	23.6%	47.2%	70.8%	5.38	5.95

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.

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Lima, Peru
4. ALS, Loughrea, Galway, Ireland
5. ALS, Perth, WA, Australia
6. ALS, Vancouver, BC, Canada
7. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
8. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
9. Bureau Veritas Geoanalytical, Perth, WA, Australia
10. Inspectorate (BV), Lima, Peru
11. Intertek Genalysis, Adelaide, SA, Australia
12. Intertek Genalysis, Perth, WA, Australia
13. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
14. Labtium Oy, Saarenkylä, Rovaniemi, Finland
15. Nagrom, Perth, WA, Australia
16. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
17. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
18. SGS Mineral Services, Townsville, QLD, Australia
19. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
20. Zarazma Mineral Studies Company, Tehran, Iran

PREPARER AND SUPPLIER

Certified reference material OREAS 46 is prepared, certified and supplied by:

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It is packaged in in 10g and 60g units in laminated foil pouches and 1kg units in plastic wide-mouth jars.

METROLOGICAL 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 values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases,* ***only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results*** *(see ISO Guide 35:2006, Clause 10)."*

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their field samples.

INTENDED USE

OREAS 46 is intended to cover all activities needed to produce a measurement result. This includes extraction, possible separation steps and the actual measurement process (the signal producing step). OREAS 46 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 46 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 46 has been prepared from pure glacial till and contains negligible reactive sulphide (S = <0.005 wt.%). In its unopened state and under normal conditions of storage it 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 for OREAS 46 refer to the concentration levels in its packaged state. There is no need for drying prior to weighing and analysis.

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 9001:2015 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
0	5th March, 2018	First publication (ICP-OES and ICP-MS finishes separated for Fusion ICP, 4-Acid Digestion and Aqua Regia Digestion method groups).
1	2nd August, 2018	Combined ICP-OES and ICP-MS finishes (Fusion ICP, 4-Acid Digestion and Aqua Regia Digestion method groups).

CERTIFYING OFFICER

2nd August, 2018

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

REFERENCES

ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2015), Reference materials – Contents of certificates and labels.

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

ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.