

**CERTIFICATE OF ANALYSIS FOR**

**Anomalous Glacial Till**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 47**

**Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 47.**

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Pb Fire Assay</b>						
Au, Gold (ppb)	44.3	2.5	43.3	45.3	43.7*	44.9*
Pd, Palladium (ppb)	44.2	2.6	41.3	47.2	42.3	46.2
Pt, Platinum (ppb)	29.2	2.1	27.1	31.2	27.6	30.7
<b>Borate / Peroxide Fusion ICP</b>						
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	11.96	0.322	11.79	12.12	11.74	12.17
Ba, Barium (ppm)	473	23	462	484	462	484
Be, Beryllium (ppm)	0.97	0.10	0.92	1.02	IND	IND
CaO, Calcium oxide (wt.%)	3.27	0.094	3.22	3.32	3.21	3.32
Ce, Cerium (ppm)	56	3.9	54	58	54	59
Co, Cobalt (ppm)	52	2.5	49	55	50	54
Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)	165	16	157	173	IND	IND
Cs, Cesium (ppm)	2.01	0.100	1.96	2.05	1.92	2.09
Dy, Dysprosium (ppm)	2.11	0.102	2.06	2.15	1.98	2.23
Er, Erbium (ppm)	1.16	0.047	1.14	1.17	1.09	1.23
Eu, Europium (ppm)	1.01	0.047	0.98	1.03	0.96	1.05
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	3.97	0.100	3.92	4.02	3.91	4.03
Ga, Gallium (ppm)	14.1	0.90	13.5	14.7	13.4	14.8
Gd, Gadolinium (ppm)	2.83	0.142	2.77	2.89	2.68	2.97
Hf, Hafnium (ppm)	4.10	0.319	3.92	4.29	3.80	4.41
Ho, Holmium (ppm)	0.42	0.021	0.40	0.43	0.40	0.44

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

\*Gold Tolerance Limits for typical 30g fire assay charge weight determined from 20 x 1g INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.



Project: COA-1251-OREAS47\_Rev1

2-August-2018

Table 1 continued.

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Borate / Peroxide Fusion ICP continued</b>						
K <sub>2</sub> O, Potassium oxide (wt.%)	1.42	0.044	1.40	1.44	1.39	1.45
La, Lanthanum (ppm)	30.9	1.98	29.8	32.0	30.2	31.7
Lu, Lutetium (ppm)	0.16	0.009	0.16	0.16	IND	IND
MgO, Magnesium oxide (wt.%)	1.67	0.047	1.65	1.70	1.65	1.70
MnO, Manganese oxide (wt.%)	0.064	0.003	0.062	0.065	IND	IND
Mo, Molybdenum (ppm)	12.7	1.18	12.3	13.2	IND	IND
Na <sub>2</sub> O, Sodium oxide (wt.%)	3.47	0.059	3.44	3.49	3.40	3.53
Nb, Niobium (ppm)	17.9	1.09	17.2	18.6	17.2	18.6
Nd, Neodymium (ppm)	24.0	1.12	23.4	24.6	23.1	25.0
Ni, Nickel (ppm)	91	6.3	86	97	85	98
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.129	0.012	0.124	0.133	IND	IND
Pr, Praseodymium (ppm)	6.58	0.233	6.46	6.71	6.37	6.80
Rb, Rubidium (ppm)	37.6	1.81	36.6	38.6	36.5	38.7
Sc, Scandium (ppm)	9.27	0.521	8.67	9.86	IND	IND
SiO <sub>2</sub> , Silicon dioxide (wt.%)	72.78	0.995	72.34	73.23	72.04	73.52
Sm, Samarium (ppm)	4.01	0.138	3.94	4.07	3.78	4.23
Sn, Tin (ppm)	6.14	0.580	5.90	6.38	IND	IND
Sr, Strontium (ppm)	402	12	395	408	392	411
Ta, Tantalum (ppm)	0.46	0.10	0.41	0.51	IND	IND
Tb, Terbium (ppm)	0.39	0.018	0.38	0.40	0.37	0.41
Th, Thorium (ppm)	3.84	0.205	3.75	3.93	3.63	4.06
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.379	0.012	0.373	0.385	0.367	0.392
Tm, Thulium (ppm)	0.17	0.012	0.16	0.17	IND	IND
U, Uranium (ppm)	0.79	0.038	0.76	0.81	0.74	0.83
V, Vanadium (ppm)	61	4.1	59	63	57	65
W, Tungsten (ppm)	< 1	IND	IND	IND	IND	IND
Y, Yttrium (ppm)	11.6	0.52	11.3	11.9	11.3	12.0
Yb, Ytterbium (ppm)	1.08	0.058	1.05	1.10	1.00	1.16
Zn, Zinc (ppm)	217	13	206	229	196	238
Zr, Zirconium (ppm)	161	13	154	169	152	170
<b>Thermogravimetry</b>						
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	1.02	0.22	0.88	1.15	0.95	1.08
<b>4-Acid Digestion</b>						
Ag, Silver (ppm)	0.130	0.019	0.116	0.144	0.118	0.143
Al, Aluminium (wt.%)	6.25	0.183	6.17	6.33	6.16	6.34
As, Arsenic (ppm)	9.57	0.435	9.40	9.73	9.14	9.99
Ba, Barium (ppm)	485	14	479	491	474	496
Be, Beryllium (ppm)	1.04	0.11	0.99	1.08	0.95	1.13
Bi, Bismuth (ppm)	0.17	0.014	0.17	0.18	0.17	0.18
Ca, Calcium (wt.%)	2.31	0.056	2.29	2.33	2.27	2.35

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ 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	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>4-Acid Digestion continued</b>						
Cd, Cadmium (ppm)	0.50	0.036	0.48	0.51	0.47	0.53
Ce, Cerium (ppm)	55	2.0	54	55	53	56
Co, Cobalt (ppm)	53	1.9	52	54	52	54
Cr, Chromium (ppm)	82	12	76	89	79	86
Cs, Cesium (ppm)	2.09	0.079	2.05	2.13	2.02	2.16
Cu, Copper (ppm)	159	4	158	160	155	162
Dy, Dysprosium (ppm)	2.12	0.118	2.07	2.17	2.03	2.21
Er, Erbium (ppm)	1.18	0.088	1.12	1.23	1.13	1.23
Eu, Europium (ppm)	1.03	0.080	0.98	1.08	0.99	1.08
Fe, Iron (wt.%)	2.78	0.067	2.75	2.80	2.72	2.83
Ga, Gallium (ppm)	14.1	0.65	13.7	14.4	13.7	14.5
Gd, Gadolinium (ppm)	3.01	0.249	2.88	3.14	2.89	3.13
Hf, Hafnium (ppm)	1.87	0.173	1.79	1.94	1.75	1.99
Ho, Holmium (ppm)	0.41	0.024	0.39	0.42	0.39	0.43
In, Indium (ppm)	0.055	0.006	0.052	0.057	0.050	0.060
K, Potassium (wt.%)	1.18	0.029	1.17	1.19	1.15	1.20
La, Lanthanum (ppm)	30.1	1.23	29.6	30.6	29.2	31.0
Li, Lithium (ppm)	42.5	2.38	41.3	43.7	41.1	44.0
Lu, Lutetium (ppm)	0.15	0.02	0.14	0.16	0.14	0.16
Mg, Magnesium (wt.%)	0.979	0.022	0.970	0.987	0.957	1.001
Mn, Manganese (wt.%)	0.051	0.002	0.050	0.052	0.050	0.052
Mo, Molybdenum (ppm)	12.9	0.44	12.7	13.1	12.4	13.4
Na, Sodium (wt.%)	2.61	0.057	2.59	2.63	2.56	2.66
Nb, Niobium (ppm)	17.0	0.84	16.5	17.5	16.5	17.5
Nd, Neodymium (ppm)	24.3	1.08	23.7	24.9	23.8	24.7
Ni, Nickel (ppm)	90	2.8	88	91	87	92
P, Phosphorus (wt.%)	0.056	0.002	0.056	0.057	0.055	0.058
Pb, Lead (ppm)	284	10	279	289	275	292
Pr, Praseodymium (ppm)	6.68	0.245	6.60	6.77	6.46	6.90
Rb, Rubidium (ppm)	37.9	2.14	36.9	38.9	37.0	38.7
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND	IND
S, Sulphur (wt.%)	0.044	0.005	0.041	0.046	0.042	0.045
Sb, Antimony (ppm)	0.32	0.03	0.30	0.34	0.30	0.35
Sc, Scandium (ppm)	9.11	0.320	8.92	9.31	8.75	9.47
Sm, Samarium (ppm)	4.03	0.281	3.90	4.15	3.81	4.25
Sn, Tin (ppm)	4.30	0.45	4.11	4.50	4.10	4.51
Sr, Strontium (ppm)	408	17	400	415	399	417
Ta, Tantalum (ppm)	0.42	0.08	0.38	0.47	0.40	0.45
Tb, Terbium (ppm)	0.38	0.018	0.37	0.39	0.37	0.39
Th, Thorium (ppm)	3.86	0.236	3.76	3.97	3.72	4.00
Ti, Titanium (wt.%)	0.213	0.008	0.208	0.217	0.206	0.219

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ 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	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>4-Acid Digestion continued</b>						
Tl, Thallium (ppm)	0.25	0.022	0.24	0.26	0.24	0.26
Tm, Thulium (ppm)	0.15	0.006	0.15	0.15	0.14	0.16
U, Uranium (ppm)	0.76	0.052	0.73	0.79	0.72	0.80
V, Vanadium (ppm)	58	2.1	57	59	57	60
W, Tungsten (ppm)	0.26	0.024	0.24	0.28	0.23	0.28
Y, Yttrium (ppm)	10.7	0.41	10.5	10.9	10.4	10.9
Yb, Ytterbium (ppm)	1.01	0.053	0.99	1.04	0.97	1.06
Zn, Zinc (ppm)	226	14	219	232	220	231
Zr, Zirconium (ppm)	63	7	60	66	61	65
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>						
Ag, Silver (ppm)	0.107	0.010	0.101	0.113	0.102	0.112
Al, Aluminium (wt.%)	0.810	0.067	0.775	0.845	0.788	0.831
As, Arsenic (ppm)	9.53	0.648	9.14	9.92	9.22	9.84
Au, Gold (ppb)	32.4	5.5	29.5	35.3	31.9 <sup>†</sup>	32.9 <sup>†</sup>
Ba, Barium (ppm)	62	2.6	60	63	60	64
Be, Beryllium (ppm)	0.19	0.02	0.17	0.21	0.17	0.20
Bi, Bismuth (ppm)	0.15	0.014	0.14	0.16	0.14	0.16
Ca, Calcium (wt.%)	0.547	0.056	0.516	0.579	0.532	0.563
Cd, Cadmium (ppm)	0.50	0.041	0.48	0.52	0.48	0.53
Ce, Cerium (ppm)	44.7	3.05	43.2	46.3	43.7	45.8
Co, Cobalt (ppm)	49.9	3.00	48.4	51.4	48.8	51.1
Cr, Chromium (ppm)	30.4	1.96	29.4	31.4	29.6	31.2
Cs, Cesium (ppm)	1.19	0.062	1.15	1.22	1.15	1.23
Cu, Copper (ppm)	160	6	157	163	157	163
Fe, Iron (wt.%)	1.65	0.125	1.58	1.71	1.61	1.69
Ga, Gallium (ppm)	3.28	0.41	3.04	3.53	3.15	3.41
Gd, Gadolinium (ppm)	1.92	0.33	1.54	2.29	1.85	1.99
Ge, Germanium (ppm)	0.075	0.011	0.063	0.086	0.067	0.082
Ho, Holmium (ppm)	0.21	0.04	0.16	0.27	0.20	0.23
In, Indium (ppm)	0.037	0.006	0.033	0.041	0.032	0.041
K, Potassium (wt.%)	0.116	0.010	0.110	0.121	0.113	0.118
La, Lanthanum (ppm)	25.2	1.16	24.5	25.8	24.4	25.9
Li, Lithium (ppm)	8.83	0.89	8.25	9.42	8.52	9.14
Lu, Lutetium (ppm)	0.070	0.011	0.058	0.081	IND	IND
Mg, Magnesium (wt.%)	0.484	0.046	0.459	0.508	0.472	0.496
Mn, Manganese (wt.%)	0.027	0.002	0.026	0.028	0.026	0.028
Mo, Molybdenum (ppm)	12.7	0.89	12.3	13.2	12.4	13.1
Na, Sodium (wt.%)	0.091	0.016	0.081	0.100	0.088	0.093
Nd, Neodymium (ppm)	17.8	2.0	15.5	20.0	17.2	18.3

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

<sup>†</sup>Gold Tolerance Limits for typical 25g aqua regia sample weight determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973);

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
<b>Aqua Regia Digestion (sample weights 0.15-50g) continued</b>						
Ni, Nickel (ppm)	80	4.1	78	82	78	81
P, Phosphorus (wt.%)	0.055	0.002	0.054	0.055	0.053	0.056
Pb, Lead (ppm)	284	15	276	291	277	290
Pd, Palladium (ppb)	43.1	4.3	40.2	46.0	38.9	47.3
Pr, Praseodymium (ppm)	5.30	0.261	4.97	5.63	5.09	5.51
Pt, Platinum (ppb)	25.7	1.9	24.3	27.1	23.3	28.2
Rb, Rubidium (ppm)	7.15	0.512	6.88	7.42	6.92	7.39
Re, Rhenium (ppm)	< 0.001	IND	IND	IND	IND	IND
S, Sulphur (wt.%)	0.046	0.004	0.044	0.048	0.045	0.047
Sb, Antimony (ppm)	0.20	0.03	0.18	0.22	0.18	0.22
Sc, Scandium (ppm)	3.17	0.54	2.88	3.47	3.05	3.30
Se, Selenium (ppm)	< 0.4	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	2.66	0.37	2.25	3.08	2.54	2.79
Sn, Tin (ppm)	2.54	0.34	2.35	2.74	2.43	2.66
Sr, Strontium (ppm)	31.4	4.1	29.2	33.7	30.5	32.4
Tb, Terbium (ppm)	0.23	0.04	0.20	0.26	0.21	0.25
Th, Thorium (ppm)	3.25	0.211	3.14	3.36	3.13	3.37
Ti, Titanium (wt.%)	0.070	0.014	0.062	0.079	0.068	0.073
Tl, Thallium (ppm)	0.083	0.009	0.077	0.088	0.075	0.090
U, Uranium (ppm)	0.47	0.05	0.43	0.50	0.44	0.49
V, Vanadium (ppm)	24.7	2.28	23.4	25.9	23.8	25.5
W, Tungsten (ppm)	0.11	0.02	0.10	0.13	0.11	0.12
Y, Yttrium (ppm)	5.75	0.84	5.26	6.23	5.55	5.95
Yb, Ytterbium (ppm)	0.50	0.09	0.44	0.57	0.48	0.53
Zn, Zinc (ppm)	213	10	208	218	208	217
Zr, Zirconium (ppm)	6.70	0.93	6.07	7.33	6.45	6.95

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  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 47 is an augmented 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 but with minor additions of various ores (PGE + REE + Li) and concentrates (base metals). 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 47 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, 50kg gamma irradiation according to ISO 11137);
- Drying of till material to constant mass at 105°C;
- Drying of base metal concentrates (Cu, Pb, Zn, Ni-Co and Mo) to constant mass at 85°C;
- Milling of till material to 98% minus 75 microns;
- Milling of ore and concentrate materials to 100% minus 35 microns;
- Preliminary homogenisation and check assaying of source materials;
- Final homogenisation by blending the source materials in specific ratios to achieve target grades;
- 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;
- Instrumental neutron activation analysis for Au on 20 x 85mg subsamples to confirm homogeneity (1 laboratory);
- Gold by 15-50g aqua regia digestion with ICP-MS (14 laboratories), graphite furnace AAS (1 laboratory), 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 162 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 below shows 28 indicative values. Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty ~85 milligram sample portions (see Table 3) and by a nested ANOVA program (see '**nested ANOVA**' section). Table 4 provides performance gate intervals for the certified values of each method group based on their pooled 1SD's. Tabulated results of all elements (including Au INAA analyses) together with uncorrected means, medians, standard deviations, relative standard deviations and percent 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 47 DataPack.xlsx**).

**Table 2. Indicative Values for OREAS 47.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Borate / Peroxide ICP</b>								
As	ppm	9.67	In	ppm	< 0.5	Te	ppm	2.25
Bi	ppm	< 0.5	Li	ppm	42.3	Tl	ppm	< 0.5
Cu	ppm	149	Pb	ppm	303			
Ge	ppm	1.06	Sb	ppm	< 0.5			
<b>4-Acid Digestion ICP</b>								
Ge	ppm	0.12	Se	ppm	0.27			
Hg	ppm	0.040	Te	ppm	< 0.05			
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>								
B	ppm	< 10	Hf	ppm	0.20	Si	wt.%	0.037
Dy	ppm	1.26	Hg	ppm	0.014	Ta	ppm	< 0.005
Er	ppm	0.58	Nb	ppm	0.92	Te	ppm	0.016
Eu	ppm	0.60	Ru	ppm	< 0.005	Tm	ppm	0.084
<b>Infrared Combustion</b>								
C	wt.%	0.070	S	wt.%	0.045			

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ 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.



## 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. The INAA data (see Table 3) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 47.

**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 4 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 ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 155 and 162 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.*

For gold the tolerance has been determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.42% calculated for a 30g fire assay or aqua regia sample (7.82% at 85mg weights) confirms the high level of gold homogeneity in OREAS 47.

*Please note that these RSD's and tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.*

The homogeneity is of a level such that **sampling error is essentially negligible** for a conventional fire assay or aqua regia determination. Using the INAA data, the sampling constant (defined as the minimum sample mass required to achieve a 1% RSD) is 5.3g. Table 3 below shows the INAA data determined on 20 x 85mg subsamples of OREAS 47. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30g fire assay determinations were undertaken without the normal measurement error associated with this methodology.

**Table 3. Neutron Activation Analysis of Au (in ppb) on 20 x 85mg subsamples showing the equivalent results scaled to a 30g sample mass typical of fire assay determination.**

Replicate No	Au 85mg actual	Au 30g equivalent*
1	47.67	44.64
2	42.90	44.38
3	42.62	44.37
4	47.15	44.61
5	40.74	44.27
6	41.02	44.28
7	50.83	44.80
8	44.44	44.46
9	49.52	44.73
10	38.57	44.15
11	43.72	44.42
12	42.98	44.39
13	42.35	44.35
14	45.72	44.53
15	45.01	44.49
16	48.90	44.70
17	41.43	44.30
18	39.38	44.19
19	46.46	44.57
20	47.89	44.65
Mean	44.46	44.46
Median	44.08	44.44
Std Dev.	3.48	0.19
<b>Rel.Std.Dev.</b>	<b>7.82%</b>	<b>0.42%</b>

\*Results calculated for a 30g equivalent sample mass using the formula:  $x^{30g Eq} = \frac{(x^{INAA}) - RSD@30g}{RSD@85mg} + \bar{X}$

where  $x^{30g Eq}$  = equivalent result calculated for a 30g sample mass

$(x^{INAA})$  = raw INAA result at 85mg

$\bar{X}$  = mean of 85mg INAA results

The homogeneity of OREAS 47 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 47. 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 162 certified values. The null hypothesis is retained.

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 47 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 47 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

**Table 4. Pooled-Lab Performance Gates for OREAS 47.**

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
<b>Pb Fire Assay</b>											
Au, ppb	44.3	2.5	39.2	49.4	36.7	51.9	5.75%	11.50%	17.25%	42.1	46.5
Pd, ppb	44.2	2.6	39.1	49.4	36.5	52.0	5.82%	11.63%	17.45%	42.0	46.5
Pt, ppb	29.2	2.1	25.0	33.3	23.0	35.3	7.08%	14.15%	21.23%	27.7	30.6
<b>Borate / Peroxide Fusion ICP</b>											
Al <sub>2</sub> O <sub>3</sub> , wt.%	11.96	0.322	11.31	12.60	10.99	12.92	2.69%	5.39%	8.08%	11.36	12.55
Ba, ppm	473	23	427	518	404	541	4.84%	9.68%	14.52%	449	496
Be, ppm	0.97	0.10	0.77	1.17	0.67	1.27	10.35%	20.69%	31.04%	0.92	1.02
CaO, wt.%	3.27	0.094	3.08	3.46	2.98	3.55	2.89%	5.77%	8.66%	3.10	3.43
Ce, ppm	56	3.9	48	64	44	68	6.97%	13.93%	20.90%	53	59
Co, ppm	52	2.5	47	57	45	60	4.85%	9.70%	14.55%	50	55
Cr <sub>2</sub> O <sub>3</sub> , ppm	165	16	133	197	117	214	9.75%	19.51%	29.26%	157	173
Cs, ppm	2.01	0.100	1.81	2.21	1.70	2.31	5.00%	10.00%	15.00%	1.91	2.11
Dy, ppm	2.11	0.102	1.90	2.31	1.80	2.41	4.82%	9.65%	14.47%	2.00	2.21
Er, ppm	1.16	0.047	1.06	1.25	1.02	1.30	4.04%	8.08%	12.11%	1.10	1.21
Eu, ppm	1.01	0.047	0.91	1.10	0.86	1.15	4.70%	9.40%	14.10%	0.96	1.06
Fe <sub>2</sub> O <sub>3</sub> , wt.%	3.97	0.100	3.77	4.17	3.67	4.27	2.51%	5.02%	7.52%	3.77	4.17
Ga, ppm	14.1	0.90	12.3	15.9	11.4	16.8	6.37%	12.73%	19.10%	13.4	14.8
Gd, ppm	2.83	0.142	2.54	3.11	2.40	3.25	5.02%	10.04%	15.06%	2.69	2.97
Hf, ppm	4.10	0.319	3.47	4.74	3.15	5.06	7.77%	15.53%	23.30%	3.90	4.31
Ho, ppm	0.42	0.021	0.37	0.46	0.35	0.48	5.10%	10.19%	15.29%	0.39	0.44
K <sub>2</sub> O, wt.%	1.42	0.044	1.33	1.51	1.29	1.55	3.08%	6.17%	9.25%	1.35	1.49
La, ppm	30.9	1.98	26.9	34.9	25.0	36.9	6.41%	12.82%	19.24%	29.4	32.5
Lu, ppm	0.16	0.009	0.14	0.18	0.13	0.19	5.81%	11.61%	17.42%	0.15	0.17
MgO, wt.%	1.67	0.047	1.58	1.77	1.53	1.81	2.79%	5.58%	8.38%	1.59	1.76
MnO, wt.%	0.064	0.003	0.057	0.070	0.054	0.073	5.01%	10.01%	15.02%	0.060	0.067
Mo, ppm	12.7	1.18	10.4	15.1	9.2	16.3	9.30%	18.60%	27.90%	12.1	13.4
Na <sub>2</sub> O, wt.%	3.47	0.059	3.35	3.59	3.29	3.65	1.71%	3.42%	5.13%	3.29	3.64
Nb, ppm	17.9	1.09	15.7	20.1	14.7	21.2	6.06%	12.12%	18.18%	17.0	18.8
Nd, ppm	24.0	1.12	21.8	26.3	20.6	27.4	4.68%	9.35%	14.03%	22.8	25.2
Ni, ppm	91	6.3	79	104	72	110	6.92%	13.85%	20.77%	87	96

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

Note: intervals may appear asymmetric due to rounding.

Table 4 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
<b>Borate / Peroxide Fusion ICP continued</b>											
P <sub>2</sub> O <sub>5</sub> , wt. %	0.129	0.012	0.105	0.152	0.094	0.163	9.04%	18.09%	27.13%	0.122	0.135
Pr, ppm	6.58	0.233	6.12	7.05	5.89	7.28	3.53%	7.07%	10.60%	6.26	6.91
Rb, ppm	37.6	1.81	34.0	41.2	32.2	43.0	4.82%	9.64%	14.45%	35.7	39.5
Sc, ppm	9.27	0.521	8.23	10.31	7.70	10.83	5.62%	11.24%	16.86%	8.80	9.73
SiO <sub>2</sub> , wt. %	72.78	0.995	70.79	74.77	69.80	75.77	1.37%	2.73%	4.10%	69.14	76.42
Sm, ppm	4.01	0.138	3.73	4.28	3.59	4.42	3.43%	6.87%	10.30%	3.81	4.21
Sn, ppm	6.14	0.580	4.98	7.30	4.40	7.88	9.45%	18.91%	28.36%	5.83	6.45
Sr, ppm	402	12	377	426	365	438	3.05%	6.09%	9.14%	381	422
Ta, ppm	0.46	0.10	0.26	0.65	0.17	0.74	21.00%	41.99%	62.99%	0.43	0.48
Tb, ppm	0.39	0.018	0.35	0.42	0.33	0.44	4.71%	9.43%	14.14%	0.37	0.41
Th, ppm	3.84	0.205	3.43	4.25	3.23	4.46	5.34%	10.68%	16.02%	3.65	4.04
TiO <sub>2</sub> , wt. %	0.379	0.012	0.355	0.404	0.342	0.416	3.26%	6.52%	9.77%	0.360	0.398
Tm, ppm	0.17	0.012	0.14	0.19	0.13	0.20	6.94%	13.88%	20.81%	0.16	0.17
U, ppm	0.79	0.038	0.71	0.86	0.67	0.90	4.80%	9.60%	14.40%	0.75	0.82
V, ppm	61	4.1	53	70	49	74	6.71%	13.41%	20.12%	58	64
W, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Y, ppm	11.6	0.52	10.6	12.6	10.1	13.2	4.44%	8.88%	13.32%	11.0	12.2
Yb, ppm	1.08	0.058	0.96	1.19	0.90	1.25	5.37%	10.75%	16.12%	1.02	1.13
Zn, ppm	217	13	191	243	178	256	5.99%	11.97%	17.96%	206	228
Zr, ppm	161	13	136	186	123	199	7.79%	15.58%	23.36%	153	169
<b>Thermogravimetry</b>											
LOI <sup>1000</sup> , wt. %	1.02	0.22	0.58	1.46	0.36	1.68	21.66%	43.32%	64.97%	0.97	1.07
<b>4-Acid Digestion</b>											
Ag, ppm	0.130	0.019	0.091	0.169	0.072	0.188	14.83%	29.67%	44.50%	0.124	0.137
Al, wt. %	6.25	0.183	5.88	6.62	5.70	6.80	2.93%	5.87%	8.80%	5.94	6.56
As, ppm	9.57	0.435	8.70	10.44	8.26	10.87	4.55%	9.09%	13.64%	9.09	10.04
Ba, ppm	485	14	457	513	444	527	2.87%	5.73%	8.60%	461	510
Be, ppm	1.04	0.11	0.81	1.26	0.70	1.38	10.91%	21.81%	32.72%	0.98	1.09
Bi, ppm	0.17	0.014	0.15	0.20	0.13	0.22	8.29%	16.58%	24.87%	0.17	0.18
Ca, wt. %	2.31	0.056	2.20	2.42	2.14	2.48	2.44%	4.87%	7.31%	2.20	2.43
Cd, ppm	0.50	0.036	0.43	0.57	0.39	0.61	7.30%	14.60%	21.91%	0.47	0.52
Ce, ppm	55	2.0	50	59	48	61	3.74%	7.48%	11.21%	52	57
Co, ppm	53	1.9	49	57	47	58	3.53%	7.06%	10.60%	50	56
Cr, ppm	82	12	59	106	47	118	14.31%	28.61%	42.92%	78	86
Cs, ppm	2.09	0.079	1.93	2.25	1.85	2.33	3.78%	7.57%	11.35%	1.99	2.19
Cu, ppm	159	4	152	166	148	170	2.24%	4.48%	6.71%	151	167
Dy, ppm	2.12	0.118	1.88	2.35	1.77	2.47	5.56%	11.11%	16.67%	2.01	2.22
Er, ppm	1.18	0.088	1.00	1.35	0.91	1.44	7.44%	14.89%	22.33%	1.12	1.24
Eu, ppm	1.03	0.080	0.87	1.19	0.79	1.27	7.77%	15.55%	23.32%	0.98	1.08
Fe, wt. %	2.78	0.067	2.64	2.91	2.57	2.98	2.43%	4.85%	7.28%	2.64	2.91
Ga, ppm	14.1	0.65	12.8	15.4	12.1	16.0	4.59%	9.19%	13.78%	13.4	14.8

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

Note: intervals may appear asymmetric due to rounding.

**Table 4 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
<b>4-Acid Digestion continued</b>											
Gd, ppm	3.01	0.249	2.51	3.51	2.26	3.76	8.29%	16.58%	24.87%	2.86	3.16
Hf, ppm	1.87	0.173	1.52	2.21	1.35	2.38	9.25%	18.50%	27.76%	1.77	1.96
Ho, ppm	0.41	0.024	0.36	0.46	0.34	0.48	5.85%	11.70%	17.55%	0.39	0.43
In, ppm	0.055	0.006	0.043	0.066	0.038	0.072	10.44%	20.88%	31.32%	0.052	0.057
K, wt. %	1.18	0.029	1.12	1.24	1.09	1.27	2.46%	4.93%	7.39%	1.12	1.24
La, ppm	30.1	1.23	27.6	32.6	26.4	33.8	4.08%	8.16%	12.25%	28.6	31.6
Li, ppm	42.5	2.38	37.8	47.3	35.4	49.7	5.60%	11.20%	16.80%	40.4	44.6
Lu, ppm	0.15	0.02	0.12	0.18	0.10	0.20	11.01%	22.02%	33.03%	0.14	0.16
Mg, wt. %	0.979	0.022	0.934	1.023	0.912	1.046	2.28%	4.55%	6.83%	0.930	1.028
Mn, wt. %	0.051	0.002	0.048	0.054	0.046	0.056	3.24%	6.48%	9.71%	0.048	0.053
Mo, ppm	12.9	0.44	12.0	13.8	11.6	14.2	3.38%	6.76%	10.14%	12.2	13.5
Na, wt. %	2.61	0.057	2.50	2.72	2.44	2.78	2.18%	4.35%	6.53%	2.48	2.74
Nb, ppm	17.0	0.84	15.3	18.7	14.5	19.5	4.95%	9.90%	14.86%	16.1	17.8
Nd, ppm	24.3	1.08	22.1	26.5	21.0	27.5	4.46%	8.92%	13.39%	23.1	25.5
Ni, ppm	90	2.8	84	95	81	98	3.18%	6.36%	9.53%	85	94
P, wt. %	0.056	0.002	0.053	0.060	0.051	0.061	2.94%	5.89%	8.83%	0.054	0.059
Pb, ppm	284	10	263	304	253	315	3.61%	7.23%	10.84%	270	298
Pr, ppm	6.68	0.245	6.19	7.17	5.95	7.42	3.67%	7.34%	11.01%	6.35	7.02
Rb, ppm	37.9	2.14	33.6	42.2	31.5	44.3	5.65%	11.29%	16.94%	36.0	39.8
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	0.044	0.005	0.033	0.054	0.028	0.059	11.81%	23.62%	35.43%	0.041	0.046
Sb, ppm	0.32	0.03	0.26	0.39	0.22	0.42	10.07%	20.14%	30.21%	0.31	0.34
Sc, ppm	9.11	0.320	8.47	9.75	8.15	10.07	3.52%	7.03%	10.55%	8.66	9.57
Sm, ppm	4.03	0.281	3.46	4.59	3.18	4.87	6.98%	13.96%	20.94%	3.83	4.23
Sn, ppm	4.30	0.45	3.40	5.20	2.96	5.65	10.44%	20.87%	31.31%	4.09	4.52
Sr, ppm	408	17	374	442	357	458	4.14%	8.28%	12.42%	387	428
Ta, ppm	0.42	0.08	0.27	0.58	0.19	0.65	18.33%	36.66%	54.99%	0.40	0.44
Tb, ppm	0.38	0.018	0.35	0.42	0.33	0.44	4.72%	9.44%	14.16%	0.36	0.40
Th, ppm	3.86	0.236	3.39	4.33	3.15	4.57	6.11%	12.22%	18.34%	3.67	4.05
Ti, wt. %	0.213	0.008	0.196	0.229	0.188	0.238	3.89%	7.78%	11.68%	0.202	0.223
Tl, ppm	0.25	0.022	0.21	0.30	0.19	0.32	8.77%	17.54%	26.31%	0.24	0.26
Tm, ppm	0.15	0.006	0.14	0.16	0.13	0.17	4.19%	8.38%	12.58%	0.14	0.16
U, ppm	0.76	0.052	0.66	0.87	0.60	0.92	6.89%	13.78%	20.68%	0.72	0.80
V, ppm	58	2.1	54	62	52	65	3.55%	7.11%	10.66%	55	61
W, ppm	0.26	0.024	0.21	0.30	0.18	0.33	9.24%	18.48%	27.72%	0.24	0.27
Y, ppm	10.7	0.41	9.8	11.5	9.4	11.9	3.88%	7.77%	11.65%	10.1	11.2
Yb, ppm	1.01	0.053	0.91	1.12	0.85	1.17	5.25%	10.49%	15.74%	0.96	1.06
Zn, ppm	226	14	198	254	184	268	6.20%	12.41%	18.61%	215	237
Zr, ppm	63	7	50	76	43	83	10.56%	21.11%	31.67%	60	66
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>											
Ag, ppm	0.107	0.010	0.087	0.127	0.077	0.137	9.43%	18.86%	28.29%	0.102	0.112

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

Note: intervals may appear asymmetric due to rounding.

Table 4 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
<b>Aqua Regia Digestion (sample weights 0.15-50g) continued</b>											
Al, wt.%	0.810	0.067	0.676	0.943	0.610	1.010	8.24%	16.47%	24.71%	0.769	0.850
As, ppm	9.53	0.648	8.24	10.83	7.59	11.47	6.80%	13.59%	20.39%	9.05	10.01
Au, ppb	32.4	5.5	21.4	43.4	15.8	49.0	17.03%	34.07%	51.10%	30.8	34.0
Ba, ppm	62	2.6	56	67	54	70	4.28%	8.56%	12.83%	59	65
Be, ppm	0.19	0.02	0.14	0.24	0.11	0.26	13.26%	26.53%	39.79%	0.18	0.20
Bi, ppm	0.15	0.014	0.12	0.18	0.11	0.19	9.30%	18.60%	27.90%	0.14	0.16
Ca, wt.%	0.547	0.056	0.435	0.660	0.379	0.716	10.27%	20.55%	30.82%	0.520	0.575
Cd, ppm	0.50	0.041	0.42	0.59	0.38	0.63	8.24%	16.47%	24.71%	0.48	0.53
Ce, ppm	44.7	3.05	38.6	50.8	35.6	53.9	6.83%	13.65%	20.48%	42.5	47.0
Co, ppm	49.9	3.00	43.9	55.9	40.9	58.9	6.01%	12.02%	18.03%	47.4	52.4
Cr, ppm	30.4	1.96	26.5	34.3	24.5	36.3	6.43%	12.87%	19.30%	28.9	31.9
Cs, ppm	1.19	0.062	1.06	1.31	1.00	1.37	5.21%	10.42%	15.63%	1.13	1.25
Cu, ppm	160	6	149	171	143	177	3.48%	6.96%	10.44%	152	168
Fe, wt.%	1.65	0.125	1.40	1.90	1.27	2.02	7.59%	15.18%	22.77%	1.57	1.73
Ga, ppm	3.28	0.41	2.45	4.11	2.04	4.53	12.65%	25.29%	37.94%	3.12	3.45
Gd, ppm	1.92	0.33	1.25	2.58	0.91	2.92	17.44%	34.89%	52.33%	1.82	2.01
Ge, ppm	0.075	0.011	0.053	0.096	0.043	0.106	14.19%	28.39%	42.58%	0.071	0.078
Ho, ppm	0.21	0.04	0.13	0.30	0.09	0.34	19.82%	39.64%	59.47%	0.20	0.22
In, ppm	0.037	0.006	0.025	0.048	0.019	0.054	15.79%	31.57%	47.36%	0.035	0.039
K, wt.%	0.116	0.010	0.095	0.136	0.085	0.146	8.76%	17.52%	26.28%	0.110	0.121
La, ppm	25.2	1.16	22.8	27.5	21.7	28.6	4.62%	9.23%	13.85%	23.9	26.4
Li, ppm	8.83	0.89	7.06	10.61	6.17	11.49	10.04%	20.08%	30.12%	8.39	9.27
Lu, ppm	0.070	0.011	0.048	0.091	0.037	0.102	15.72%	31.44%	47.15%	0.066	0.073
Mg, wt.%	0.484	0.046	0.393	0.575	0.347	0.620	9.41%	18.83%	28.24%	0.460	0.508
Mn, wt.%	0.027	0.002	0.023	0.031	0.020	0.034	8.10%	16.20%	24.30%	0.026	0.028
Mo, ppm	12.7	0.89	11.0	14.5	10.1	15.4	6.99%	13.98%	20.97%	12.1	13.4
Na, wt.%	0.091	0.016	0.059	0.122	0.044	0.138	17.32%	34.63%	51.95%	0.086	0.095
Nd, ppm	17.8	2.0	13.7	21.8	11.6	23.9	11.47%	22.94%	34.41%	16.9	18.6
Ni, ppm	80	4.1	71	88	67	92	5.16%	10.32%	15.49%	76	84
P, wt.%	0.055	0.002	0.051	0.058	0.049	0.060	3.34%	6.68%	10.01%	0.052	0.057
Pb, ppm	284	15	253	314	238	329	5.31%	10.63%	15.94%	269	298
Pd, ppb	43.1	4.3	34.6	51.7	30.3	55.9	9.91%	19.83%	29.74%	41.0	45.3
Pr, ppm	5.30	0.261	4.78	5.82	4.52	6.09	4.93%	9.86%	14.80%	5.04	5.57
Pt, ppb	25.7	1.9	22.0	29.5	20.1	31.3	7.29%	14.58%	21.86%	24.4	27.0
Rb, ppm	7.15	0.512	6.13	8.18	5.62	8.69	7.16%	14.31%	21.47%	6.80	7.51
Re, ppm	< 0.001	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.046	0.004	0.038	0.053	0.034	0.057	8.51%	17.02%	25.54%	0.043	0.048
Sb, ppm	0.20	0.03	0.14	0.27	0.10	0.30	16.23%	32.47%	48.70%	0.19	0.21
Sc, ppm	3.17	0.54	2.10	4.24	1.57	4.78	16.86%	33.71%	50.57%	3.02	3.33
Se, ppm	< 0.4	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sm, ppm	2.66	0.37	1.92	3.41	1.54	3.78	14.01%	28.02%	42.03%	2.53	2.80

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

Note: intervals may appear asymmetric due to rounding.



**Table 4 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
<b>Aqua Regia Digestion (sample weights 0.15-50g) continued</b>											
Sn, ppm	2.54	0.34	1.86	3.22	1.53	3.56	13.33%	26.66%	39.98%	2.42	2.67
Sr, ppm	31.4	4.1	23.2	39.7	19.1	43.8	13.09%	26.17%	39.26%	29.9	33.0
Tb, ppm	0.23	0.04	0.16	0.30	0.12	0.34	16.23%	32.45%	48.68%	0.22	0.24
Th, ppm	3.25	0.211	2.83	3.67	2.62	3.88	6.49%	12.98%	19.46%	3.09	3.41
Ti, wt. %	0.070	0.014	0.043	0.098	0.030	0.111	19.22%	38.44%	57.65%	0.067	0.074
Tl, ppm	0.083	0.009	0.064	0.101	0.055	0.110	11.23%	22.45%	33.68%	0.078	0.087
U, ppm	0.47	0.05	0.36	0.58	0.30	0.63	11.72%	23.44%	35.16%	0.44	0.49
V, ppm	24.7	2.28	20.1	29.2	17.8	31.5	9.23%	18.46%	27.69%	23.4	25.9
W, ppm	0.11	0.02	0.07	0.15	0.06	0.17	16.85%	33.71%	50.56%	0.11	0.12
Y, ppm	5.75	0.84	4.06	7.43	3.22	8.28	14.67%	29.34%	44.01%	5.46	6.03
Yb, ppm	0.50	0.09	0.33	0.68	0.24	0.76	17.41%	34.82%	52.22%	0.48	0.53
Zn, ppm	213	10	192	234	181	244	4.90%	9.80%	14.69%	202	223
Zr, ppm	6.70	0.93	4.85	8.55	3.92	9.48	13.81%	27.63%	41.44%	6.37	7.04

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt. %  $\equiv$  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 47 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 undertaken by ORE Pty Ltd) 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 47 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 47 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 47 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 47 has been prepared from glacial till augmented with minor additions of various ores (PGE + REE + Li) and concentrates (base metals). It contains negligible reactive sulphide (S = 0.04 wt.%) and 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 47 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	5 <sup>th</sup> March, 2018	First publication (ICP-OES and ICP-MS finishes separated for Fusion ICP, 4-Acid Digestion and Aqua Regia Digestion method groups).
1	2 <sup>nd</sup> August, 2018	Combined ICP-OES and ICP-MS finishes (Fusion ICP, 4-Acid Digestion and Aqua Regia Digestion method groups).

## CERTIFYING OFFICER

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

2<sup>nd</sup> August, 2018

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

## REFERENCES

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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.