

**CERTIFICATE OF ANALYSIS FOR**

**Lateritic Soil Lithochem**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 45h**

**Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 45h.**

Constituent	Certified Value	SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Pb Fire Assay</b>						
Au, Gold (ppb)	41.1	2.3	40.3	41.9	40.1*	42.1*
Pd, Palladium (ppb)	128	7	125	130	125	131
Pt, Platinum (ppb)	87.5	4.6	85.6	89.3	85.0	89.9
<b>Aqua Regia Digestion (sample weights 10-50g)</b>						
Au, Gold (ppb)	39.4	2.4	38.4	40.3	38.3*	40.4*
<b>Borate Fusion XRF</b>						
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	15.88	0.170	15.81	15.96	15.78	15.99
BaO, Barium oxide (ppm)	417	74	334	499	382	451
CaO, Calcium oxide (wt.%)	0.197	0.007	0.194	0.200	0.193	0.201
Co, Cobalt (ppm)	90	8.9	79	101	IND	IND
Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)	1019	45	1000	1038	991	1046
Cu, Copper (ppm)	777	25	751	803	743	810
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	29.31	0.426	29.12	29.50	29.13	29.49
K <sub>2</sub> O, Potassium oxide (wt.%)	0.257	0.005	0.255	0.259	0.251	0.262
MgO, Magnesium oxide (wt.%)	0.435	0.011	0.430	0.439	0.424	0.446
MnO, Manganese oxide (wt.%)	0.051	0.002	0.050	0.051	0.049	0.052
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.127	0.012	0.122	0.133	0.116	0.139

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 and 25g aqua regia sample weight are 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>Borate Fusion XRF continued</b>						
Ni, Nickel (ppm)	455	13	445	465	436	473
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.055	0.005	0.052	0.059	0.054	0.057
S, Sulphur (wt.%)	0.036	0.002	0.034	0.038	0.035	0.037
SiO <sub>2</sub> , Silicon dioxide (wt.%)	41.45	0.212	41.35	41.55	41.28	41.62
Sr, Strontium (ppm)	33.9	6.7	31.0	36.9	IND	IND
TiO <sub>2</sub> , Titanium dioxide (wt.%)	1.61	0.014	1.60	1.61	1.59	1.62
V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)	466	42	423	508	445	486
Zr, Zirconium (ppm)	199	18	182	217	191	208
<b>Thermogravimetry</b>						
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	10.17	0.363	10.00	10.35	10.11	10.23
<b>Borate / Peroxide Fusion ICP</b>						
Al, Aluminium (wt.%)	8.04	0.184	7.95	8.13	7.93	8.16
Ba, Barium (ppm)	330	15	323	337	319	340
Bi, Bismuth (ppm)	0.18	0.04	0.15	0.21	IND	IND
Ca, Calcium (wt.%)	0.143	0.016	0.132	0.153	0.137	0.148
Ce, Cerium (ppm)	24.3	0.70	23.9	24.8	23.5	25.2
Co, Cobalt (ppm)	90	7.1	85	94	87	93
Cr, Chromium (ppm)	677	40	652	701	655	698
Cs, Cesium (ppm)	2.34	0.132	2.27	2.41	2.21	2.47
Cu, Copper (ppm)	760	20	749	770	739	781
Dy, Dysprosium (ppm)	2.63	0.255	2.41	2.85	2.45	2.81
Er, Erbium (ppm)	1.62	0.19	1.48	1.76	1.52	1.72
Eu, Europium (ppm)	0.65	0.055	0.62	0.69	0.61	0.70
Fe, Iron (wt.%)	20.14	0.467	19.92	20.36	19.87	20.41
Ga, Gallium (ppm)	20.6	1.94	19.3	21.9	19.8	21.5
Gd, Gadolinium (ppm)	2.41	0.204	2.24	2.58	2.22	2.60
Hf, Hafnium (ppm)	4.95	0.90	4.08	5.82	4.61	5.28
Ho, Holmium (ppm)	0.56	0.036	0.53	0.60	0.53	0.60
K, Potassium (wt.%)	0.214	0.018	0.204	0.224	0.206	0.222
La, Lanthanum (ppm)	12.6	1.14	12.1	13.1	11.9	13.2
Li, Lithium (ppm)	13.5	1.08	12.9	14.2	IND	IND
Lu, Lutetium (ppm)	0.24	0.05	0.19	0.28	0.21	0.26
Mg, Magnesium (wt.%)	0.255	0.006	0.252	0.258	0.248	0.262
Mn, Manganese (wt.%)	0.040	0.002	0.039	0.041	0.039	0.041
Na, Sodium (wt.%)	0.095	0.012	0.083	0.107	IND	IND
Nb, Niobium (ppm)	15.6	2.6	14.1	17.1	14.9	16.3
Nd, Neodymium (ppm)	11.3	0.93	10.4	12.2	10.9	11.7
Ni, Nickel (ppm)	439	26	424	455	421	457
P, Phosphorus (wt.%)	0.023	0.004	0.021	0.026	0.020	0.027
Pr, Praseodymium (ppm)	2.93	0.130	2.81	3.05	2.80	3.06

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>Borate / Peroxide Fusion ICP continued</b>						
Rb, Rubidium (ppm)	22.7	1.48	21.8	23.6	22.0	23.4
Sb, Antimony (ppm)	0.57	0.10	0.50	0.65	IND	IND
Sc, Scandium (ppm)	53	6	47	58	51	55
Si, Silicon (wt.%)	19.00	0.492	18.76	19.25	18.77	19.24
Sm, Samarium (ppm)	2.50	0.26	2.28	2.73	2.38	2.62
Sr, Strontium (ppm)	29.9	4.3	26.5	33.2	27.5	32.2
Ta, Tantalum (ppm)	0.99	0.099	0.93	1.06	0.89	1.10
Tb, Terbium (ppm)	0.40	0.019	0.39	0.41	0.36	0.44
Th, Thorium (ppm)	7.40	0.565	7.07	7.74	7.15	7.66
Ti, Titanium (wt.%)	0.933	0.027	0.921	0.946	0.919	0.947
Tm, Thulium (ppm)	0.26	0.03	0.23	0.29	0.24	0.28
U, Uranium (ppm)	1.82	0.151	1.73	1.91	1.75	1.89
V, Vanadium (ppm)	275	10	271	278	268	281
Y, Yttrium (ppm)	12.4	1.3	11.7	13.2	12.0	12.9
Yb, Ytterbium (ppm)	1.66	0.20	1.51	1.81	1.55	1.77
Zn, Zinc (ppm)	36.7	6.9	31.1	42.3	30.4	43.0
Zr, Zirconium (ppm)	200	7	193	206	191	208
<b>4-Acid Digestion</b>						
Ag, Silver (ppm)	0.147	0.028	0.133	0.161	0.130	0.165
Al, Aluminium (wt.%)	7.99	0.261	7.90	8.08	7.78	8.21
As, Arsenic (ppm)	16.9	1.25	16.6	17.3	16.2	17.6
Ba, Barium (ppm)	332	13	328	337	325	340
Be, Beryllium (ppm)	1.09	0.15	1.03	1.15	1.01	1.17
Bi, Bismuth (ppm)	0.17	0.015	0.17	0.18	0.16	0.18
Ca, Calcium (wt.%)	0.135	0.010	0.132	0.139	0.130	0.140
Ce, Cerium (ppm)	23.6	1.63	23.0	24.3	22.6	24.6
Co, Cobalt (ppm)	88	3.8	86	89	86	89
Cr, Chromium (ppm)	602	50	584	620	583	620
Cs, Cesium (ppm)	2.29	0.124	2.24	2.34	2.19	2.39
Cu, Copper (ppm)	767	18	761	772	753	781
Dy, Dysprosium (ppm)	2.42	0.187	2.29	2.55	2.32	2.53
Er, Erbium (ppm)	1.44	0.102	1.39	1.49	1.37	1.51
Eu, Europium (ppm)	0.65	0.08	0.60	0.70	0.61	0.69
Fe, Iron (wt.%)	19.52	0.658	19.25	19.80	19.16	19.89
Ga, Gallium (ppm)	21.3	1.57	20.7	22.0	20.7	21.9
Gd, Gadolinium (ppm)	2.34	0.143	2.24	2.44	2.23	2.46
Hf, Hafnium (ppm)	3.60	0.284	3.49	3.72	3.48	3.73
Ho, Holmium (ppm)	0.48	0.031	0.46	0.51	0.46	0.51
In, Indium (ppm)	0.10	0.009	0.10	0.10	0.09	0.11
K, Potassium (wt.%)	0.205	0.006	0.203	0.207	0.199	0.211
La, Lanthanum (ppm)	12.4	0.89	12.1	12.8	12.0	12.9

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>						
Li, Lithium (ppm)	13.1	0.95	12.7	13.4	12.4	13.7
Lu, Lutetium (ppm)	0.21	0.019	0.20	0.22	0.20	0.22
Mg, Magnesium (wt.%)	0.238	0.012	0.234	0.243	0.231	0.245
Mn, Manganese (wt.%)	0.038	0.001	0.038	0.039	0.037	0.039
Mo, Molybdenum (ppm)	1.55	0.23	1.46	1.63	1.46	1.63
Na, Sodium (wt.%)	0.090	0.005	0.087	0.093	0.088	0.092
Nb, Niobium (ppm)	14.8	0.86	14.5	15.1	14.2	15.3
Nd, Neodymium (ppm)	11.2	1.06	10.5	11.9	10.7	11.8
Ni, Nickel (ppm)	423	20	416	430	414	432
P, Phosphorus (wt.%)	0.023	0.002	0.023	0.024	0.022	0.025
Pb, Lead (ppm)	11.9	0.84	11.6	12.2	11.6	12.3
Pr, Praseodymium (ppm)	2.91	0.148	2.80	3.01	2.75	3.06
Rb, Rubidium (ppm)	22.5	1.13	22.1	23.0	21.7	23.3
S, Sulphur (wt.%)	0.035	0.004	0.034	0.037	0.034	0.036
Sb, Antimony (ppm)	0.63	0.07	0.60	0.66	0.59	0.67
Sc, Scandium (ppm)	57	3.6	55	58	55	59
Se, Selenium (ppm)	2.02	0.39	1.86	2.18	1.90	2.14
Sm, Samarium (ppm)	2.50	0.155	2.40	2.61	2.36	2.64
Sn, Tin (ppm)	1.93	0.160	1.86	1.99	1.82	2.04
Sr, Strontium (ppm)	27.1	1.48	26.6	27.6	26.0	28.2
Ta, Tantalum (ppm)	1.08	0.12	1.03	1.13	1.03	1.13
Tb, Terbium (ppm)	0.39	0.034	0.37	0.40	0.36	0.41
Th, Thorium (ppm)	7.26	0.78	6.96	7.55	6.96	7.56
Ti, Titanium (wt.%)	0.878	0.027	0.867	0.888	0.861	0.894
Tl, Thallium (ppm)	0.15	0.013	0.14	0.15	0.14	0.16
Tm, Thulium (ppm)	0.23	0.02	0.21	0.24	0.21	0.24
U, Uranium (ppm)	1.68	0.144	1.62	1.73	1.63	1.73
V, Vanadium (ppm)	263	9	260	266	257	269
W, Tungsten (ppm)	0.99	0.088	0.96	1.03	0.94	1.04
Y, Yttrium (ppm)	10.4	0.71	10.1	10.6	9.9	10.8
Yb, Ytterbium (ppm)	1.44	0.116	1.39	1.49	1.39	1.49
Zn, Zinc (ppm)	39.7	1.81	39.0	40.4	38.4	40.9
Zr, Zirconium (ppm)	131	6	129	134	127	135
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>						
Ag, Silver (ppm)	0.092	0.011	0.085	0.098	0.085	0.098
Al, Aluminium (wt.%)	3.89	0.55	3.65	4.13	3.77	4.01
As, Arsenic (ppm)	8.36	1.16	7.80	8.91	7.92	8.79
Ba, Barium (ppm)	271	13	265	276	264	277
Be, Beryllium (ppm)	0.94	0.091	0.90	0.97	0.89	0.99
Bi, Bismuth (ppm)	0.14	0.010	0.13	0.14	0.13	0.14
Ca, Calcium (wt.%)	0.106	0.009	0.103	0.110	0.104	0.108

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>Aqua Regia Digestion (sample weights 0.15-50g) continued</b>						
Ce, Cerium (ppm)	18.4	1.63	17.8	19.1	18.0	18.9
Co, Cobalt (ppm)	79	6.2	77	82	78	81
Cr, Chromium (ppm)	508	36	494	522	498	518
Cs, Cesium (ppm)	1.22	0.15	1.14	1.29	1.17	1.27
Cu, Copper (ppm)	717	24	708	727	706	728
Dy, Dysprosium (ppm)	1.83	0.109	1.75	1.90	1.76	1.89
Er, Erbium (ppm)	0.99	0.066	0.94	1.04	0.96	1.02
Eu, Europium (ppm)	0.54	0.054	0.50	0.58	0.52	0.57
Fe, Iron (wt.%)	18.18	1.019	17.75	18.61	17.85	18.51
Ga, Gallium (ppm)	16.1	1.37	15.5	16.6	15.6	16.5
Gd, Gadolinium (ppm)	1.86	0.167	1.74	1.97	1.79	1.92
Ge, Germanium (ppm)	0.15	0.03	0.12	0.18	0.14	0.17
Hf, Hafnium (ppm)	0.90	0.16	0.83	0.97	0.86	0.94
Hg, Mercury (ppm)	0.024	0.003	0.022	0.025	0.021	0.026
Ho, Holmium (ppm)	0.35	0.025	0.33	0.37	0.34	0.36
In, Indium (ppm)	0.086	0.006	0.084	0.088	0.079	0.093
K, Potassium (wt.%)	0.084	0.009	0.081	0.088	0.082	0.087
La, Lanthanum (ppm)	8.91	0.581	8.68	9.15	8.67	9.15
Lu, Lutetium (ppm)	0.13	0.008	0.13	0.14	0.12	0.15
Mg, Magnesium (wt.%)	0.165	0.016	0.159	0.171	0.161	0.169
Mn, Manganese (wt.%)	0.026	0.002	0.025	0.027	0.025	0.026
Mo, Molybdenum (ppm)	0.92	0.13	0.87	0.97	0.87	0.97
Na, Sodium (wt.%)	0.036	0.005	0.034	0.039	0.035	0.038
Nd, Neodymium (ppm)	8.92	0.541	8.56	9.27	8.58	9.25
Ni, Nickel (ppm)	348	31	336	360	341	355
P, Phosphorus (wt.%)	0.018	0.001	0.018	0.019	0.018	0.019
Pb, Lead (ppm)	10.2	0.57	10.0	10.4	9.9	10.4
Pd, Palladium (ppb)	94.5	8.7	88.6	100.4	86.6	102.4
Pr, Praseodymium (ppm)	2.33	0.25	2.14	2.51	2.24	2.41
Pt, Platinum (ppb)	81.3	4.2	78.5	84.0	76.3	86.2
Rb, Rubidium (ppm)	11.5	1.8	10.7	12.4	11.1	11.9
S, Sulphur (wt.%)	0.032	0.003	0.030	0.033	0.031	0.032
Sc, Scandium (ppm)	50	3.3	49	52	49	52
Sm, Samarium (ppm)	1.95	0.174	1.82	2.08	1.88	2.03
Sn, Tin (ppm)	1.34	0.131	1.28	1.40	1.26	1.41
Sr, Strontium (ppm)	15.5	1.05	15.1	15.9	15.0	16.0
Tb, Terbium (ppm)	0.29	0.012	0.29	0.30	0.28	0.31
Th, Thorium (ppm)	5.52	0.542	5.30	5.75	5.37	5.67
Ti, Titanium (wt.%)	0.116	0.021	0.107	0.126	0.112	0.121
Tl, Thallium (ppm)	0.085	0.011	0.080	0.090	0.078	0.091
Tm, Thulium (ppm)	0.14	0.010	0.13	0.15	0.13	0.15

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>Aqua Regia Digestion (sample weights 0.15-50g) continued</b>						
U, Uranium (ppm)	0.92	0.060	0.90	0.95	0.90	0.95
V, Vanadium (ppm)	233	11	229	237	228	238
Y, Yttrium (ppm)	7.24	0.313	7.13	7.36	6.90	7.58
Yb, Ytterbium (ppm)	0.95	0.063	0.92	0.98	0.92	0.99
Zn, Zinc (ppm)	27.7	4.1	26.0	29.3	26.6	28.7
Zr, Zirconium (ppm)	28.1	4.1	26.4	29.8	26.8	29.4
<b>Infrared Combustion</b>						
C, Carbon (wt.%)	0.487	0.019	0.479	0.494	0.474	0.499

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

OREAS 45h was prepared from a blend of mineralised ferruginous soil, barren mature soil and minor additions of gold and nickel ores. The ferruginous soil was developed over a Ni-Cu-PGE mineralised contact between gabbro and pyroxenite in a layered mafic intrusive from the Southern Murchison region of Western Australia. It contains anomalous precious and base metal values (Au, PGE's, Cu and Ni). The barren soil was taken from a layer of mature soil developed in situ over early Tertiary tholeiitic basalt in outer eastern Melbourne, Victoria, Australia.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 45h was prepared in the following manner:

- Drying to constant mass at 105°C;
- Milling of barren material to >98% minus 75 microns;
- Milling of mineralised ferruginous soil and ore materials to 100% minus 35 microns;
- Preliminary homogenisation and check assaying of source materials;
- Pre-equilibration of material to typical laboratory atmosphere (~3.48% H<sub>2</sub>O: 20 degrees Celsius, 60% humidity);
- 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 500g units in plastic wide-mouth jars.

## ANALYTICAL PROGRAM

Thirty-three 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 and/or ICP-MS (23 laboratories) and AAS (6 laboratories) finish;
- Instrumental neutron activation analysis for Au on 20 x 85mg subsamples to confirm homogeneity (1 laboratory);
- Gold by 10-50g aqua regia digestion with ICP-OES and/or ICP-MS finish (15 laboratories), AAS finish (4 laboratories);
- Lithium borate fusion followed by X-ray fluorescence (up to 21 laboratories);
- Thermogravimetry: Moisture at 105°C (15 laboratories oven dried and 12 laboratories used a thermogravimetric analyser). LOI at 1000°C (19 laboratories used a thermogravimetric analyser and 6 laboratories used conventional muffle furnace);
- Lithium borate fusion (7 laboratories) or sodium peroxide fusion (11 laboratories) followed by full elemental suites ICP-OES and/or ICP-MS finish;
- Four acid digestion followed by full elemental suites ICP-OES and/or ICP-MS finish (up to 31 laboratories depending on the element);
- Aqua regia digestion using 0.15 to 50g sample weights followed by full elemental suites ICP-OES and/or ICP-MS finish (up to 31 laboratories depending on the element);
- Total C and S by infra-red combustion furnace (27 laboratories);
- Gold by 25g nickel sulphide (NiS) collection fire assay with ICP-OES and/or ICP-MS finish (1 laboratory).

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 1.5kg 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 1.5kg 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 181 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 below shows 61 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 45h DataPack-1.0.180813\_122016.xlsx**).

**Table 2. Indicative Values for OREAS 45h.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>NiS Fire Assay</b>								
Au	ppb	39.5	Pd	ppb	127	Rh	ppb	7.66
Ir	ppb	3.24	Pt	ppb	84.3	Ru	ppb	7.48
<b>Borate Fusion XRF</b>								
As	ppm	15.7	F	ppm	1318	Sn	ppm	< 10
Cl	ppm	212	Rb	ppm	27.4	Zn	ppm	40.1
<b>Thermogravimetry</b>								
H <sub>2</sub> O	wt.%	3.48						
<b>Borate / Peroxide Fusion ICP</b>								
Ag	ppm	< 1	In	ppm	< 0.2	Ru	ppb	< 30
As	ppm	16.1	Ir	ppb	< 15	S	wt.%	0.037
B	ppm	< 20	Mo	ppm	1.54	Se	ppm	< 20
Be	ppm	1.02	Pb	ppm	12.8	Sn	ppm	2.25
Cd	ppm	< 0.2	Pt	ppb	86.7	Te	ppm	< 0.5
Ge	ppm	2.40	Re	ppm	< 0.01	Tl	ppm	< 0.5
I	ppm	12.8	Rh	ppb	< 30	W	ppm	1.10
<b>4-Acid Digestion</b>								
B	ppm	8.49	I	ppm	13.8	Re	ppm	< 0.002
Cd	ppm	0.027	Ir	ppb	< 300	Rh	ppb	< 5
Ge	ppm	0.31	Pd	ppb	109	Ru	ppb	< 10
Hg	ppm	< 0.02	Pt	ppb	71.2	Te	ppm	0.10
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>								
B	ppm	< 10	Nb	ppm	0.20	Se	ppm	0.83
Cd	ppm	0.015	Re	ppm	< 0.001	Ta	ppm	< 0.05
I	ppm	14.8	Rh	ppb	< 10	Te	ppm	0.056
Ir	ppb	6.14	Ru	ppb	< 10	W	ppm	< 0.1
Li	ppm	6.37	Sb	ppm	0.30			
<b>Infrared Combustion</b>								
S	wt.%	0.030						

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 45h.

**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. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL)  $\pm$  10%.

*i.e. Certified Value  $\pm$  10%  $\pm$  2DL (adapted from Govett, 1983)*

**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 ( $\rho=0.95$ ) will have concentrations lying between 753 and 781 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 0.77% calculated for a 30g fire assay or aqua regia sample (14.38% at 85mg weights) confirms the high level of gold homogeneity in OREAS 45h. Given the low concentration level of gold (41.1ppb) and that those laboratories mostly reported to the nearest ppb, this level of homogeneity is more than sufficient for its intended purpose.

*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.*

Table 3 below shows the INAA data determined on 20 x 85mg subsamples of OREAS 45h. 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	42.3	45.1
2	66.1	44.9
3	41.8	44.8
4	42.0	44.8
5	37.2	44.9
6	45.3	45.0
7	41.3	45.1
8	42.7	45.0
9	47.8	45.5
10	52.3	44.9
11	52.6	45.5
12	39.5	44.7
13	42.3	45.0
14	38.5	45.0
15	45.3	45.4
16	44.6	45.3
17	43.9	45.1
18	43.0	46.2
19	51.1	45.0
20	42.6	45.0
Mean	45.1	45.1
Median	42.8	45.0
Std Dev.	6.5	0.35
<b>Rel.Std.Dev.</b>	<b>14.38%</b>	<b>0.77%</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 45h 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 45h. 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 181 certified values except for Bi by fusion with ICP and Co by aqua regia digestion. Bi is present in low concentration close to its respective lower level 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. For the case of Co by aqua regia digestion the ‘significant’ *p*-value is most likely due to random statistical probability given the high number of analytes considered for this ANOVA test. As there is no other supporting evidence to suspect greater between-unit variance compared with within-unit variance 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 45h 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 45h is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

**Table 4. Pooled-Lab Performance Gates for OREAS 45h.**

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	41.1	2.3	36.5	45.7	34.3	47.9	5.55%	11.10%	16.65%	39.0	43.2
Pd, ppb	128	7	115	141	108	147	5.13%	10.25%	15.38%	121	134
Pt, ppb	87.5	4.6	78.2	96.7	73.6	101.3	5.27%	10.54%	15.81%	83.1	91.8
<b>Aqua Regia Digestion (sample weights 10-50g)</b>											
Au, ppb	39.4	2.4	34.6	44.1	32.2	46.5	6.09%	12.17%	18.26%	37.4	41.3
<b>Borate Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	15.88	0.170	15.54	16.23	15.37	16.40	1.07%	2.14%	3.21%	15.09	16.68
BaO, ppm	417	74	269	564	196	637	17.67%	35.34%	53.01%	396	437
CaO, wt. %	0.197	0.007	0.183	0.212	0.175	0.219	3.66%	7.32%	10.98%	0.187	0.207
Co, ppm	90	8.9	72	108	63	117	9.88%	19.77%	29.65%	85	94
Cr <sub>2</sub> O <sub>3</sub> , ppm	1019	45	929	1108	884	1153	4.40%	8.80%	13.20%	968	1070
Cu, ppm	777	25	726	827	701	852	3.24%	6.49%	9.73%	738	816
Fe <sub>2</sub> O <sub>3</sub> , wt. %	29.31	0.426	28.46	30.16	28.03	30.59	1.45%	2.91%	4.36%	27.85	30.78
K <sub>2</sub> O, wt. %	0.257	0.005	0.246	0.268	0.240	0.273	2.13%	4.26%	6.39%	0.244	0.269
MgO, wt. %	0.435	0.011	0.413	0.456	0.403	0.467	2.44%	4.88%	7.32%	0.413	0.456
MnO, wt. %	0.051	0.002	0.047	0.054	0.045	0.056	3.80%	7.60%	11.40%	0.048	0.053
Na <sub>2</sub> O, wt. %	0.127	0.012	0.103	0.151	0.091	0.163	9.49%	18.97%	28.46%	0.121	0.134

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 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 Fusion XRF continued</b>											
Ni, ppm	455	13	428	481	415	494	2.88%	5.76%	8.65%	432	477
P <sub>2</sub> O <sub>5</sub> , wt. %	0.055	0.005	0.046	0.065	0.042	0.069	8.37%	16.75%	25.12%	0.053	0.058
S, wt. %	0.036	0.002	0.032	0.040	0.030	0.042	5.78%	11.56%	17.35%	0.034	0.038
SiO <sub>2</sub> , wt. %	41.45	0.212	41.03	41.87	40.81	42.09	0.51%	1.02%	1.54%	39.38	43.52
Sr, ppm	33.9	6.7	20.6	47.2	13.9	53.9	19.63%	39.26%	58.89%	32.2	35.6
TiO <sub>2</sub> , wt. %	1.61	0.014	1.58	1.63	1.56	1.65	0.86%	1.72%	2.58%	1.52	1.69
V <sub>2</sub> O <sub>5</sub> , ppm	466	42	382	550	340	592	9.01%	18.01%	27.02%	442	489
Zr, ppm	199	18	164	235	147	252	8.80%	17.60%	26.40%	190	209
<b>Thermogravimetry</b>											
LOI <sup>1000</sup> , wt. %	10.17	0.363	9.45	10.90	9.08	11.26	3.57%	7.14%	10.72%	9.67	10.68
<b>Borate / Peroxide Fusion ICP</b>											
Al, wt. %	8.04	0.184	7.67	8.41	7.49	8.59	2.29%	4.59%	6.88%	7.64	8.44
Ba, ppm	330	15	300	359	286	374	4.45%	8.91%	13.36%	313	346
Bi, ppm	0.18	0.04	0.09	0.27	0.05	0.31	24.24%	48.48%	72.73%	0.17	0.19
Ca, wt. %	0.143	0.016	0.111	0.174	0.095	0.190	11.20%	22.39%	33.59%	0.135	0.150
Ce, ppm	24.3	0.70	22.9	25.7	22.2	26.4	2.86%	5.73%	8.59%	23.1	25.6
Co, ppm	90	7.1	76	104	69	111	7.86%	15.72%	23.58%	85	94
Cr, ppm	677	40	596	757	556	797	5.93%	11.85%	17.78%	643	710
Cs, ppm	2.34	0.132	2.08	2.61	1.94	2.74	5.65%	11.31%	16.96%	2.22	2.46
Cu, ppm	760	20	719	800	699	820	2.65%	5.31%	7.96%	722	798
Dy, ppm	2.63	0.255	2.12	3.14	1.87	3.39	9.68%	19.36%	29.04%	2.50	2.76
Er, ppm	1.62	0.19	1.24	2.00	1.05	2.19	11.80%	23.61%	35.41%	1.54	1.70
Eu, ppm	0.65	0.055	0.55	0.76	0.49	0.82	8.32%	16.65%	24.97%	0.62	0.69
Fe, wt. %	20.14	0.467	19.21	21.07	18.74	21.54	2.32%	4.63%	6.95%	19.13	21.15
Ga, ppm	20.6	1.94	16.8	24.5	14.8	26.5	9.40%	18.81%	28.21%	19.6	21.7
Gd, ppm	2.41	0.204	2.00	2.82	1.80	3.02	8.47%	16.95%	25.42%	2.29	2.53
Hf, ppm	4.95	0.90	3.15	6.75	2.24	7.65	18.20%	36.40%	54.61%	4.70	5.19
Ho, ppm	0.56	0.036	0.49	0.64	0.45	0.67	6.46%	12.92%	19.38%	0.54	0.59
K, wt. %	0.214	0.018	0.179	0.249	0.161	0.267	8.20%	16.40%	24.59%	0.203	0.225
La, ppm	12.6	1.14	10.3	14.8	9.1	16.0	9.09%	18.18%	27.27%	11.9	13.2
Li, ppm	13.5	1.08	11.4	15.7	10.3	16.8	8.02%	16.03%	24.05%	12.9	14.2
Lu, ppm	0.24	0.05	0.14	0.33	0.10	0.37	19.71%	39.41%	59.12%	0.22	0.25
Mg, wt. %	0.255	0.006	0.243	0.267	0.237	0.273	2.31%	4.61%	6.92%	0.242	0.268
Mn, wt. %	0.040	0.002	0.036	0.044	0.034	0.046	5.03%	10.06%	15.09%	0.038	0.042
Na, wt. %	0.095	0.012	0.071	0.118	0.059	0.130	12.58%	25.16%	37.74%	0.090	0.099
Nb, ppm	15.6	2.6	10.3	20.8	7.7	23.5	16.85%	33.70%	50.54%	14.8	16.4
Nd, ppm	11.3	0.93	9.4	13.1	8.5	14.1	8.28%	16.57%	24.85%	10.7	11.8
Ni, ppm	439	26	388	491	362	517	5.87%	11.75%	17.62%	417	461
P, wt. %	0.023	0.004	0.015	0.032	0.010	0.037	18.68%	37.35%	56.03%	0.022	0.025
Pr, ppm	2.93	0.130	2.67	3.19	2.54	3.32	4.43%	8.86%	13.29%	2.78	3.08
Rb, ppm	22.7	1.48	19.7	25.7	18.3	27.2	6.53%	13.07%	19.60%	21.6	23.9
Sb, ppm	0.57	0.10	0.37	0.78	0.27	0.88	17.70%	35.41%	53.11%	0.54	0.60

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>											
Sc, ppm	53	6	41	64	36	70	10.84%	21.68%	32.52%	50	56
Si, wt. %	19.00	0.492	18.02	19.99	17.53	20.48	2.59%	5.17%	7.76%	18.05	19.95
Sm, ppm	2.50	0.26	1.98	3.02	1.72	3.28	10.37%	20.75%	31.12%	2.38	2.63
Sr, ppm	29.9	4.3	21.3	38.4	17.1	42.7	14.31%	28.62%	42.92%	28.4	31.4
Ta, ppm	0.99	0.099	0.80	1.19	0.70	1.29	9.97%	19.94%	29.91%	0.95	1.04
Tb, ppm	0.40	0.019	0.36	0.44	0.35	0.46	4.65%	9.29%	13.94%	0.38	0.42
Th, ppm	7.40	0.565	6.27	8.54	5.71	9.10	7.63%	15.27%	22.90%	7.03	7.78
Ti, wt. %	0.933	0.027	0.880	0.987	0.853	1.014	2.88%	5.76%	8.65%	0.887	0.980
Tm, ppm	0.26	0.03	0.19	0.33	0.16	0.36	12.90%	25.80%	38.70%	0.25	0.27
U, ppm	1.82	0.151	1.52	2.12	1.37	2.27	8.32%	16.65%	24.97%	1.73	1.91
V, ppm	275	10	254	295	244	305	3.71%	7.43%	11.14%	261	288
Y, ppm	12.4	1.3	9.9	15.0	8.6	16.2	10.16%	20.33%	30.49%	11.8	13.1
Yb, ppm	1.66	0.20	1.27	2.06	1.07	2.25	11.89%	23.77%	35.66%	1.58	1.75
Zn, ppm	36.7	6.9	22.9	50.4	16.0	57.3	18.75%	37.50%	56.26%	34.8	38.5
Zr, ppm	200	7	186	213	180	219	3.29%	6.59%	9.88%	190	209
<b>4-Acid Digestion</b>											
Ag, ppm	0.147	0.028	0.090	0.204	0.062	0.233	19.35%	38.70%	58.05%	0.140	0.155
Al, wt. %	7.99	0.261	7.47	8.52	7.21	8.78	3.27%	6.54%	9.81%	7.59	8.39
As, ppm	16.9	1.25	14.4	19.4	13.2	20.7	7.36%	14.72%	22.07%	16.1	17.8
Ba, ppm	332	13	306	359	293	372	3.96%	7.93%	11.89%	316	349
Be, ppm	1.09	0.15	0.78	1.40	0.63	1.55	14.05%	28.11%	42.16%	1.04	1.15
Bi, ppm	0.17	0.015	0.14	0.20	0.13	0.22	8.75%	17.51%	26.26%	0.16	0.18
Ca, wt. %	0.135	0.010	0.114	0.156	0.104	0.167	7.75%	15.50%	23.25%	0.128	0.142
Ce, ppm	23.6	1.63	20.4	26.9	18.7	28.5	6.90%	13.81%	20.71%	22.4	24.8
Co, ppm	88	3.8	80	95	76	99	4.33%	8.66%	12.99%	83	92
Cr, ppm	602	50	502	701	453	751	8.26%	16.52%	24.78%	572	632
Cs, ppm	2.29	0.124	2.04	2.54	1.92	2.66	5.41%	10.82%	16.23%	2.18	2.41
Cu, ppm	767	18	731	802	713	820	2.32%	4.64%	6.96%	728	805
Dy, ppm	2.42	0.187	2.05	2.80	1.86	2.98	7.70%	15.41%	23.11%	2.30	2.54
Er, ppm	1.44	0.102	1.24	1.64	1.13	1.74	7.07%	14.14%	21.21%	1.37	1.51
Eu, ppm	0.65	0.08	0.49	0.81	0.41	0.89	12.38%	24.75%	37.13%	0.62	0.68
Fe, wt. %	19.52	0.658	18.21	20.84	17.55	21.50	3.37%	6.74%	10.10%	18.55	20.50
Ga, ppm	21.3	1.57	18.2	24.4	16.6	26.0	7.35%	14.69%	22.04%	20.2	22.4
Gd, ppm	2.34	0.143	2.06	2.63	1.91	2.77	6.09%	12.18%	18.28%	2.22	2.46
Hf, ppm	3.60	0.284	3.04	4.17	2.75	4.46	7.88%	15.76%	23.64%	3.42	3.78
Ho, ppm	0.48	0.031	0.42	0.54	0.39	0.58	6.46%	12.92%	19.39%	0.46	0.51
In, ppm	0.10	0.009	0.08	0.12	0.07	0.13	8.87%	17.74%	26.60%	0.10	0.11
K, wt. %	0.205	0.006	0.193	0.217	0.187	0.223	2.91%	5.82%	8.72%	0.195	0.215
La, ppm	12.4	0.89	10.6	14.2	9.7	15.1	7.17%	14.35%	21.52%	11.8	13.0
Li, ppm	13.1	0.95	11.2	15.0	10.2	15.9	7.27%	14.53%	21.80%	12.4	13.7
Lu, ppm	0.21	0.019	0.17	0.25	0.16	0.27	8.90%	17.81%	26.71%	0.20	0.22
Mg, wt. %	0.238	0.012	0.214	0.262	0.202	0.274	5.05%	10.11%	15.16%	0.226	0.250

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>											
Mn, wt. %	0.038	0.001	0.035	0.041	0.034	0.043	3.88%	7.76%	11.64%	0.036	0.040
Mo, ppm	1.55	0.23	1.08	2.01	0.84	2.25	15.13%	30.27%	45.40%	1.47	1.62
Na, wt. %	0.090	0.005	0.080	0.101	0.075	0.106	5.77%	11.53%	17.30%	0.086	0.095
Nb, ppm	14.8	0.86	13.1	16.5	12.2	17.4	5.80%	11.61%	17.41%	14.0	15.5
Nd, ppm	11.2	1.06	9.1	13.4	8.0	14.4	9.45%	18.91%	28.36%	10.7	11.8
Ni, ppm	423	20	384	463	364	482	4.67%	9.35%	14.02%	402	444
P, wt. %	0.023	0.002	0.020	0.027	0.018	0.028	7.22%	14.44%	21.66%	0.022	0.025
Pb, ppm	11.9	0.84	10.3	13.6	9.4	14.5	7.03%	14.07%	21.10%	11.3	12.5
Pr, ppm	2.91	0.148	2.61	3.20	2.46	3.35	5.10%	10.21%	15.31%	2.76	3.05
Rb, ppm	22.5	1.13	20.3	24.8	19.1	25.9	5.02%	10.05%	15.07%	21.4	23.7
S, wt. %	0.035	0.004	0.027	0.044	0.022	0.048	12.23%	24.47%	36.70%	0.033	0.037
Sb, ppm	0.63	0.07	0.49	0.77	0.42	0.84	10.89%	21.77%	32.66%	0.60	0.66
Sc, ppm	57	3.6	49	64	46	68	6.39%	12.78%	19.17%	54	60
Se, ppm	2.02	0.39	1.24	2.79	0.86	3.18	19.21%	38.43%	57.64%	1.92	2.12
Sm, ppm	2.50	0.155	2.19	2.81	2.04	2.97	6.18%	12.35%	18.53%	2.38	2.63
Sn, ppm	1.93	0.160	1.61	2.25	1.45	2.41	8.31%	16.62%	24.94%	1.83	2.02
Sr, ppm	27.1	1.48	24.1	30.0	22.7	31.5	5.45%	10.90%	16.35%	25.7	28.4
Ta, ppm	1.08	0.12	0.83	1.32	0.70	1.45	11.53%	23.06%	34.59%	1.02	1.13
Tb, ppm	0.39	0.034	0.32	0.45	0.28	0.49	8.84%	17.68%	26.51%	0.37	0.40
Th, ppm	7.26	0.78	5.70	8.81	4.93	9.59	10.70%	21.41%	32.11%	6.89	7.62
Ti, wt. %	0.878	0.027	0.823	0.932	0.795	0.960	3.13%	6.25%	9.38%	0.834	0.921
Tl, ppm	0.15	0.013	0.12	0.17	0.11	0.19	8.47%	16.94%	25.41%	0.14	0.16
Tm, ppm	0.23	0.02	0.18	0.28	0.15	0.30	10.67%	21.35%	32.02%	0.22	0.24
U, ppm	1.68	0.144	1.39	1.97	1.25	2.11	8.56%	17.12%	25.68%	1.59	1.76
V, ppm	263	9	245	280	236	289	3.34%	6.68%	10.02%	250	276
W, ppm	0.99	0.088	0.82	1.17	0.73	1.26	8.89%	17.77%	26.66%	0.94	1.04
Y, ppm	10.4	0.71	8.9	11.8	8.2	12.5	6.82%	13.63%	20.45%	9.8	10.9
Yb, ppm	1.44	0.116	1.21	1.67	1.09	1.79	8.08%	16.16%	24.24%	1.37	1.51
Zn, ppm	39.7	1.81	36.1	43.3	34.2	45.1	4.57%	9.14%	13.71%	37.7	41.7
Zr, ppm	131	6	119	144	113	150	4.75%	9.49%	14.24%	125	138
<b>Aqua Regia Digestion (sample weights 0.15-50g)</b>											
Ag, ppm	0.092	0.011	0.069	0.114	0.058	0.126	12.33%	24.66%	36.99%	0.087	0.096
Al, wt. %	3.89	0.55	2.79	4.99	2.24	5.54	14.10%	28.20%	42.30%	3.70	4.08
As, ppm	8.36	1.16	6.04	10.67	4.89	11.82	13.84%	27.67%	41.51%	7.94	8.77
Ba, ppm	271	13	245	297	231	310	4.83%	9.66%	14.48%	257	284
Be, ppm	0.94	0.091	0.75	1.12	0.66	1.21	9.72%	19.45%	29.17%	0.89	0.98
Bi, ppm	0.14	0.010	0.12	0.16	0.11	0.17	7.52%	15.04%	22.56%	0.13	0.14
Ca, wt. %	0.106	0.009	0.089	0.124	0.080	0.133	8.37%	16.73%	25.10%	0.101	0.112
Ce, ppm	18.4	1.63	15.2	21.7	13.6	23.3	8.84%	17.68%	26.52%	17.5	19.4
Co, ppm	79	6.2	67	92	61	98	7.76%	15.53%	23.29%	75	83
Cr, ppm	508	36	436	580	400	616	7.08%	14.17%	21.25%	483	533
Cs, ppm	1.22	0.15	0.93	1.51	0.78	1.66	11.96%	23.91%	35.87%	1.16	1.28

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>											
Cu, ppm	717	24	668	766	644	790	3.39%	6.78%	10.17%	681	753
Dy, ppm	1.83	0.109	1.61	2.04	1.50	2.15	5.97%	11.93%	17.90%	1.74	1.92
Er, ppm	0.99	0.066	0.86	1.12	0.79	1.19	6.66%	13.32%	19.98%	0.94	1.04
Eu, ppm	0.54	0.054	0.44	0.65	0.38	0.70	9.88%	19.76%	29.63%	0.52	0.57
Fe, wt.%	18.18	1.019	16.14	20.22	15.12	21.24	5.60%	11.20%	16.81%	17.27	19.09
Ga, ppm	16.1	1.37	13.3	18.8	12.0	20.2	8.52%	17.05%	25.57%	15.3	16.9
Gd, ppm	1.86	0.167	1.52	2.19	1.36	2.36	9.00%	18.00%	27.00%	1.77	1.95
Ge, ppm	0.15	0.03	0.09	0.21	0.06	0.24	19.80%	39.59%	59.39%	0.14	0.16
Hf, ppm	0.90	0.16	0.59	1.21	0.43	1.37	17.41%	34.82%	52.23%	0.85	0.94
Hg, ppm	0.024	0.003	0.018	0.029	0.015	0.032	12.25%	24.51%	36.76%	0.022	0.025
Ho, ppm	0.35	0.025	0.30	0.40	0.28	0.42	7.04%	14.07%	21.11%	0.33	0.37
In, ppm	0.086	0.006	0.073	0.099	0.067	0.105	7.50%	14.99%	22.49%	0.082	0.090
K, wt.%	0.084	0.009	0.067	0.102	0.058	0.110	10.35%	20.70%	31.05%	0.080	0.088
La, ppm	8.91	0.581	7.75	10.07	7.17	10.65	6.52%	13.04%	19.56%	8.47	9.36
Lu, ppm	0.13	0.008	0.12	0.15	0.11	0.16	5.65%	11.30%	16.95%	0.13	0.14
Mg, wt.%	0.165	0.016	0.133	0.197	0.117	0.213	9.73%	19.46%	29.19%	0.157	0.173
Mn, wt.%	0.026	0.002	0.021	0.031	0.019	0.033	9.29%	18.59%	27.88%	0.025	0.027
Mo, ppm	0.92	0.13	0.67	1.17	0.55	1.30	13.57%	27.13%	40.70%	0.88	0.97
Na, wt.%	0.036	0.005	0.027	0.046	0.022	0.051	13.03%	26.05%	39.08%	0.035	0.038
Nd, ppm	8.92	0.541	7.83	10.00	7.29	10.54	6.07%	12.14%	18.21%	8.47	9.36
Ni, ppm	348	31	287	410	256	440	8.84%	17.68%	26.52%	331	366
P, wt.%	0.018	0.001	0.017	0.020	0.016	0.021	5.02%	10.03%	15.05%	0.018	0.019
Pb, ppm	10.2	0.57	9.0	11.3	8.4	11.9	5.61%	11.22%	16.84%	9.6	10.7
Pd, ppb	94.5	8.7	77.1	111.9	68.4	120.6	9.20%	18.40%	27.60%	89.8	99.2
Pr, ppm	2.33	0.25	1.83	2.82	1.59	3.06	10.57%	21.14%	31.71%	2.21	2.44
Pt, ppb	81.3	4.2	72.9	89.6	68.8	93.7	5.12%	10.24%	15.36%	77.2	85.3
Rb, ppm	11.5	1.8	7.9	15.2	6.0	17.1	15.98%	31.96%	47.94%	11.0	12.1
S, wt.%	0.032	0.003	0.026	0.037	0.023	0.040	9.01%	18.02%	27.03%	0.030	0.033
Sc, ppm	50	3.3	44	57	40	60	6.55%	13.10%	19.65%	48	53
Sm, ppm	1.95	0.174	1.60	2.30	1.43	2.47	8.90%	17.80%	26.70%	1.85	2.05
Sn, ppm	1.34	0.131	1.08	1.60	0.94	1.73	9.81%	19.63%	29.44%	1.27	1.40
Sr, ppm	15.5	1.05	13.4	17.6	12.3	18.7	6.80%	13.59%	20.39%	14.7	16.3
Tb, ppm	0.29	0.012	0.27	0.32	0.26	0.33	3.97%	7.95%	11.92%	0.28	0.31
Th, ppm	5.52	0.542	4.44	6.61	3.89	7.15	9.82%	19.64%	29.46%	5.24	5.80
Ti, wt.%	0.116	0.021	0.075	0.158	0.055	0.178	17.70%	35.41%	53.11%	0.111	0.122
Tl, ppm	0.085	0.011	0.064	0.106	0.053	0.117	12.58%	25.17%	37.75%	0.081	0.089
Tm, ppm	0.14	0.010	0.12	0.16	0.11	0.17	7.23%	14.46%	21.68%	0.13	0.15
U, ppm	0.92	0.060	0.80	1.04	0.74	1.10	6.50%	13.00%	19.49%	0.88	0.97
V, ppm	233	11	212	254	201	264	4.54%	9.07%	13.61%	221	244
Y, ppm	7.24	0.313	6.62	7.87	6.31	8.18	4.32%	8.63%	12.95%	6.88	7.61
Yb, ppm	0.95	0.063	0.83	1.08	0.76	1.14	6.59%	13.19%	19.78%	0.90	1.00
Zn, ppm	27.7	4.1	19.5	35.8	15.4	39.9	14.79%	29.58%	44.36%	26.3	29.0

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>											
Zr, ppm	28.1	4.1	19.9	36.2	15.9	40.3	14.51%	29.02%	43.53%	26.7	29.5
<b>Infrared Combustion</b>											
C, wt. %	0.487	0.019	0.448	0.525	0.428	0.545	3.99%	7.98%	11.98%	0.462	0.511

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. AGAT Laboratories, Mississauga, Ontario, Canada
2. Alex Stewart International, Mendoza, Argentina
3. ALS, Brisbane, QLD, Australia
4. ALS, Johannesburg, South Africa
5. ALS, Lima, Peru
6. ALS, Loughrea, Galway, Ireland
7. ALS, Perth, WA, Australia
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ANSTO, Lucas Heights, NSW, Australia
11. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
12. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. Inspectorate (BV), Lima, Peru
15. Intertek Genalysis, Perth, WA, Australia
16. Intertek Testing Services, Townsville, QLD, Australia
17. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
18. MinAnalytical Services, Perth, WA, Australia
19. Nagrom, Perth, WA, Australia
20. Ontario Geological Survey, Sudbury, Ontario, Canada
21. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
22. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
23. PT SGS Indo Assay Laboratories, Jakarta, Indonesia
24. SGS, Randfontein, Gauteng, South Africa
25. SGS Australia Mineral Services, Perth, WA, Australia
26. SGS Canada Inc., Vancouver, BC, Canada
27. SGS del Peru, Lima, Peru
28. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
29. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada

30. SGS Mineral Services, Townsville, QLD, Australia
31. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
32. UIS Analytical Services, Centurion , South Africa
33. Zarazma Mineral Studies Company, Tehran, Iran

## PREPARER AND SUPPLIER

Certified reference material OREAS 45h is prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd  
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Email: [info@ore.com.au](mailto:info@ore.com.au)

It is packaged in in 10g and 60g units in laminated foil pouches and 500g 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 45h 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 45h may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 45h 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 45h contains negligible reactive sulphide ( $S = 0.035 \text{ wt.}\%$ ) 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 will be notified if any changes are observed. OREAS 45h is moderately hygroscopic and has been pre-equilibrated to a normal laboratory atmosphere (20 degrees Celsius, 60% humidity). This pre-equilibration yielded a moisture level of ~3.48% and facilitates ease of use by reducing the potential change in moisture content upon exposure of the CRM to different laboratory atmospheres. If the CRM is exposed and left to equilibrate in extremely dry or humid laboratory atmospheres a significant change in hygroscopic moisture is likely. Care should be taken in these circumstances to limit exposure of the CRM prior to assay.

## **INSTRUCTIONS FOR CORRECT USE**

The certified values for OREAS 45h 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

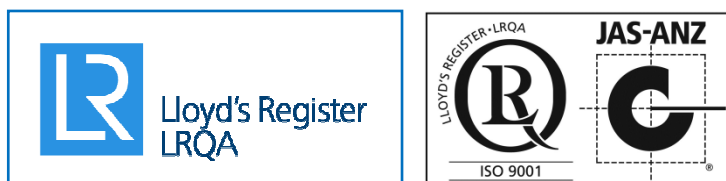
Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the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

## DOCUMENT HISTORY

Revision No	Date	Changes applied
1	4 <sup>th</sup> September, 2018	Corrected Table 1 method group title (for Borate Fusion XRF).
0	13 <sup>th</sup> August, 2018	First publication.

## 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.



## CERTIFYING OFFICER

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

3<sup>rd</sup> September, 2018

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

## REFERENCES

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