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OREAS
Certified Reference Materials

CERTIFICATE OF ANALYSIS FOR

GOLD OXIDE ORE
CERTIFIED REFERENCE MATERIAL
OREAS 250

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 250.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fire Assay						
Au, Gold (ppm)	0.309	0.013	0.304	0.313	*0.299	*0.319
Aqua Regia Digestion						
Ag, Silver (ppm)	0.258	0.036	0.242	0.275	0.231	0.286
Al, Aluminium (wt.%)	1.34	0.16	1.26	1.41	1.30	1.37
As, Arsenic (ppm)	11.8	1.15	11.4	12.2	11.3	12.3
Au, Gold (ppm)	0.303	0.013	0.298	0.307	*0.293	*0.312
B, Boron (ppm)	< 10	IND	IND	IND	IND	IND
Ba, Barium (ppm)	69	2.7	68	71	67	72
Be, Beryllium (ppm)	0.60	0.057	0.57	0.63	0.58	0.62
Bi, Bismuth (ppm)	0.081	0.011	0.074	0.088	IND	IND
Ca, Calcium (wt.%)	0.884	0.048	0.860	0.909	0.866	0.902
Cd, Cadmium (ppm)	0.12	0.01	0.11	0.12	IND	IND
Ce, Cerium (ppm)	39.7	2.74	37.9	41.4	38.7	40.6
Co, Cobalt (ppm)	33.0	0.91	32.6	33.4	32.0	33.9
Cr, Chromium (ppm)	49.5	2.88	48.1	51.0	48.1	51.0
Cs, Cesium (ppm)	0.85	0.14	0.76	0.94	0.82	0.87
Cu, Copper (ppm)	44.7	1.27	44.2	45.3	43.6	45.9
Dy, Dysprosium (ppm)	2.90	0.146	2.72	3.08	2.73	3.08
Er, Erbium (ppm)	1.33	0.082	1.23	1.43	1.29	1.38
Eu, Europium (ppm)	0.75	0.10	0.64	0.86	0.71	0.79
Fe, Iron (wt.%)	5.08	0.311	4.94	5.23	5.00	5.17
Ga, Gallium (ppm)	4.18	0.56	3.83	4.52	3.99	4.37

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Aqua Regia Digestion continued						
Gd, Gadolinium (ppm)	3.63	0.41	3.17	4.09	3.50	3.75
Hf, Hafnium (ppm)	0.87	0.09	0.78	0.96	0.79	0.95
Ho, Holmium (ppm)	0.49	0.06	0.43	0.55	0.47	0.52
In, Indium (ppm)	< 0.04	IND	IND	IND	IND	IND
K, Potassium (wt.%)	0.111	0.013	0.105	0.118	0.107	0.116
La, Lanthanum (ppm)	17.5	2.1	16.4	18.6	17.2	17.9
Li, Lithium (ppm)	7.23	1.41	6.41	8.06	6.90	7.57
Lu, Lutetium (ppm)	0.14	0.013	0.13	0.15	IND	IND
Mg, Magnesium (wt.%)	1.73	0.060	1.70	1.75	1.70	1.75
Mn, Manganese (wt.%)	0.058	0.003	0.056	0.059	0.057	0.059
Mo, Molybdenum (ppm)	0.94	0.072	0.91	0.97	0.89	0.99
Na, Sodium (wt.%)	0.186	0.015	0.179	0.194	0.180	0.193
Nb, Niobium (ppm)	< 1	IND	IND	IND	IND	IND
Nd, Neodymium (ppm)	17.5	1.33	16.0	18.9	16.9	18.0
Ni, Nickel (ppm)	119	6.3	116	122	117	121
P, Phosphorus (wt.%)	0.099	0.004	0.097	0.101	0.097	0.101
Pb, Lead (ppm)	8.06	1.09	7.65	8.48	7.76	8.37
Pr, Praseodymium (ppm)	4.39	0.61	3.70	5.07	4.22	4.56
Rb, Rubidium (ppm)	11.2	1.8	10.0	12.4	10.8	11.6
S, Sulphur (wt.%)	0.012	0.002	0.010	0.013	0.011	0.012
Sb, Antimony (ppm)	0.44	0.08	0.38	0.50	0.42	0.46
Sc, Scandium (ppm)	5.08	0.54	4.82	5.35	4.94	5.23
Se, Selenium (ppm)	< 1	IND	IND	IND	IND	IND
Sm, Samarium (ppm)	3.86	0.368	3.45	4.27	3.63	4.09
Sn, Tin (ppm)	0.71	0.11	0.64	0.78	0.65	0.77
Sr, Strontium (ppm)	66	9	61	70	64	67
Ta, Tantalum (ppm)	< 0.05	IND	IND	IND	IND	IND
Tb, Terbium (ppm)	0.52	0.07	0.47	0.57	0.50	0.54
Te, Tellurium (ppm)	< 0.05	IND	IND	IND	IND	IND
Th, Thorium (ppm)	3.06	0.232	2.91	3.21	2.98	3.13
Ti, Titanium (wt.%)	0.171	0.026	0.157	0.185	0.165	0.177
Tl, Thallium (ppm)	0.068	0.010	0.061	0.075	IND	IND
Tm, Thulium (ppm)	0.17	0.02	0.14	0.19	IND	IND
U, Uranium (ppm)	0.59	0.046	0.56	0.61	0.56	0.62
V, Vanadium (ppm)	41.4	1.91	40.4	42.4	40.3	42.6
Y, Yttrium (ppm)	12.3	1.02	11.8	12.9	12.1	12.6
Yb, Ytterbium (ppm)	1.00	0.061	0.96	1.05	0.96	1.04
Zn, Zinc (ppm)	82	4.3	80	84	79	84
Zr, Zirconium (ppm)	< 50	IND	IND	IND	IND	IND

Note: intervals may appear asymmetric due to rounding; *determined from RSD of gold INAA data for 30g analytical subsample weight.

Table 2. Indicative Values for OREAS 250.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Pd	ppb	*< 5*	Pt	ppb	*3*			
Borate Fusion XRF								
Al_2O_3	wt.%	*14.40*	Fe_2O_3	wt.%	*11.41*	Pb	ppm	*20.0*
As	ppm	*20.0*	K_2O	wt.%	*1.03*	SiO_2	wt.%	*53.85*
Ba	ppm	*325*	MgO	wt.%	*5.27*	Sn	ppm	*< 10*
CaO	wt.%	*6.81*	MnO	wt.%	*0.140*	SO_3	wt.%	*0.042*
Co	ppm	*55*	Na_2O	wt.%	*2.51*	TiO_2	wt.%	*1.70*
Cr	ppm	*230*	Ni	ppm	*170*	U	ppm	*25.0*
Cu	ppm	*75*	P_2O_5	wt.%	*0.282*	Zn	ppm	*135*
Thermogravimetry								
LOI^{1000}	wt.%	*2.48*						
Laser Ablation ICP-MS								
Ag	ppm	*0.350*	Ho	ppm	*0.87*	Sn	ppm	*2.60*
As	ppm	*15.3*	In	ppm	*< 0.05*	Sr	ppm	*361*
Ba	ppm	*311*	La	ppm	*23.3*	Ta	ppm	*1.42*
Be	ppm	*1.80*	Lu	ppm	*0.28*	Tb	ppm	*0.80*
Bi	ppm	*0.10*	Mn	wt.%	*0.102*	Te	ppm	*< 0.2*
Cd	ppm	*0.18*	Mo	ppm	*1.90*	Th	ppm	*5.09*
Ce	ppm	*48.6*	Nb	ppm	*20.7*	Ti	wt.%	*1.02*
Co	ppm	*45.3*	Nd	ppm	*22.8*	Tl	ppm	*< 0.2*
Cr	ppm	*216*	Ni	ppm	*136*	Tm	ppm	*0.32*
Cs	ppm	*1.80*	Pb	ppm	*10.0*	U	ppm	*1.24*
Cu	ppm	*59*	Pr	ppm	*6.03*	V	ppm	*153*
Dy	ppm	*4.96*	Rb	ppm	*37.2*	W	ppm	*1.58*
Er	ppm	*2.59*	Re	ppm	*< 0.01*	Y	ppm	*23.3*
Eu	ppm	*1.96*	Sb	ppm	*0.85*	Yb	ppm	*1.94*
Ga	ppm	*19.3*	Sc	ppm	*20.1*	Zn	ppm	*113*
Gd	ppm	*5.27*	Se	ppm	*3.75*	Zr	ppm	*160*
Hf	ppm	*4.53*	Sm	ppm	*5.76*			
Aqua Regia Digestion								
Ge	ppm	*0.14*	Pd	ppb	*< 10*	Re	ppm	*< 0.01*
Hg	ppm	*< 0.01*	Pt	ppb	*< 5*	W	ppm	*0.12*

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.

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 250 was prepared from a blend of gold-bearing Wilber Lode oxide ore from the Andy Well Gold Project and barren basaltic saprolite and siltstone sourced from quarries north of Melbourne, Australia. The Wilber Lode is a shear- hosted, narrow vein, quartz lode-style gold deposit situated within the Meekatharra-Wydgee greenstone belt in the Archaean Yilgarn Craton of Western Australia. The common primary mineral assemblage as stated by Mason and Harris (2011, 2012, cited in Hingston et al, 2014) is quartz, calcite, chlorite, fuchsite, pyrite, galena, sphalerite, chalcopyrite and gold. The host rock consists of a complex sequence of weathered Archaean meta-basalt and meta-porphyritic rocks derived from a primary mineralogy of albite, actinolite, chlorite, sericite, biotite, calcite, zoisite, muscovite, quartz and titanate. The Andy Well deposit is located approximately 45km north of Meekatharra in the Murchison region of Western Australia. OREAS 250 is one of a suite of three oxide gold CRMs ranging in gold content from 0.31 to 0.67ppm.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 250 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and milling of the barren material to 99% minus 75 microns;
- crushing and milling of the ore material to 100% minus 30 microns;
- blending in appropriate proportions to achieve the desired grade;
- packaging in 60g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

Twenty four commercial analytical laboratories participated in the program to certify the 60 elements reported in Table 1. The following methods were employed:

- Gold via 25-40g fire assay with AAS (11 labs) or ICP-OES (11 labs) finish;
- Instrumental neutron activation analysis for Au on 1g subsamples to confirm homogeneity (1 laboratory);
- Gold via 15-40g aqua regia digestion with ICP-MS (16 labs) or AAS (4 labs) finish;
- Aqua regia digestion (see note below) for full elemental suite ICP-OES and ICP-MS (up to 22 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 1kg lot samples were taken at predetermined intervals during the bagging stage, immediately following final blending and are considered representative of the entire batch. The six samples received by each laboratory were obtained by taking two 110g scoop splits from each of three separate 1kg lots. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance. Table 1 presents the 60 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 80 indicative values for major and trace element composition.

Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty ~1 gram sample portions (see Table 3) and by a nested ANOVA program for both fire assay and aqua regia digestion (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[3]) are presented in the detailed certification data for this CRM (**OREAS 250 DataPack.xlsx**).

STATISTICAL ANALYSIS

Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

Certified Values are the means of accepted laboratory means after outlier filtering. The NAA 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 250.

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 provided for the major and trace elements determined by borate fusion XRF (Al_2O_3 to Zn) and laser ablation with ICP-MS (Ag to Zr) and are the means of duplicate assays from Bureau Veritas, Perth. Additional indicative values by other analytical methods 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 values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

The SD for each analyte’s certified value is calculated from the same filtered data set used to determine the certified value, i.e. after removal of any individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. **The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.**

In the application of SD’s in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD’s thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD’s the ‘pooled’ SD’s provided in this report include inter-lab bias. This ‘one size fits all’ approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

Table 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.

Table 3. Neutron Activation Analysis of Au (ppm) on 20 x 1g subsamples.

Replicate No	Au ppm
1	0.347
2	0.324
3	0.321
4	0.290
5	0.328
6	0.320
7	0.290
8	0.336
9	0.315
10	0.317
11	0.314
12	0.293
13	0.322
14	0.326
15	0.286
16	0.342
17	0.320
18	0.335
19	0.343
20	0.316
Mean	0.319
Median	0.321
Std Dev.	0.018
Rel.Std.Dev.	5.61%
PDM[3]	3.42%

Table 4. Performance Gates for OREAS 250.

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
Fire Assay											
Au, ppm	0.309	0.013	0.283	0.335	0.270	0.348	4.21%	8.42%	12.62%	0.293	0.324
Aqua Regia Digestion											
Ag, ppm	0.258	0.036	0.186	0.330	0.151	0.366	13.88%	27.76%	41.65%	0.245	0.271
Al, wt.%	1.34	0.16	1.02	1.65	0.86	1.81	11.77%	23.54%	35.30%	1.27	1.40
As, ppm	11.8	1.15	9.5	14.1	8.3	15.3	9.77%	19.54%	29.31%	11.2	12.4
Au, ppm	0.303	0.013	0.276	0.329	0.263	0.342	4.39%	8.78%	13.17%	0.287	0.318
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	69	2.7	64	75	61	77	3.83%	7.65%	11.48%	66	73
Be, ppm	0.60	0.057	0.49	0.72	0.43	0.77	9.45%	18.90%	28.35%	0.57	0.63
Bi, ppm	0.081	0.011	0.059	0.103	0.049	0.113	13.34%	26.67%	40.01%	0.077	0.085
Ca, wt.%	0.884	0.048	0.788	0.981	0.739	1.029	5.47%	10.93%	16.40%	0.840	0.928

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
Aqua Regia Digestion continued											
Cd, ppm	0.12	0.01	0.09	0.15	0.07	0.16	12.50%	25.00%	37.51%	0.11	0.12
Ce, ppm	39.7	2.74	34.2	45.1	31.4	47.9	6.91%	13.82%	20.73%	37.7	41.6
Co, ppm	33.0	0.91	31.2	34.8	30.3	35.7	2.76%	5.52%	8.28%	31.3	34.6
Cr, ppm	49.5	2.88	43.8	55.3	40.9	58.2	5.82%	11.64%	17.46%	47.1	52.0
Cs, ppm	0.85	0.14	0.58	1.12	0.44	1.25	15.96%	31.92%	47.88%	0.81	0.89
Cu, ppm	44.7	1.27	42.2	47.3	40.9	48.6	2.84%	5.67%	8.51%	42.5	47.0
Dy, ppm	2.90	0.146	2.61	3.19	2.46	3.34	5.02%	10.04%	15.06%	2.76	3.05
Er, ppm	1.33	0.082	1.17	1.50	1.09	1.58	6.16%	12.33%	18.49%	1.27	1.40
Eu, ppm	0.75	0.10	0.56	0.94	0.46	1.04	12.82%	25.63%	38.45%	0.71	0.79
Fe, wt.%	5.08	0.311	4.46	5.71	4.15	6.02	6.11%	12.22%	18.33%	4.83	5.34
Ga, ppm	4.18	0.56	3.05	5.30	2.49	5.87	13.48%	26.96%	40.44%	3.97	4.39
Gd, ppm	3.63	0.41	2.80	4.46	2.39	4.87	11.40%	22.81%	34.21%	3.45	3.81
Hf, ppm	0.87	0.09	0.68	1.06	0.58	1.15	10.90%	21.79%	32.69%	0.82	0.91
Ho, ppm	0.49	0.06	0.38	0.61	0.32	0.67	11.84%	23.69%	35.53%	0.47	0.52
In, ppm	< 0.04	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	0.111	0.013	0.086	0.137	0.073	0.150	11.51%	23.03%	34.54%	0.106	0.117
La, ppm	17.5	2.1	13.3	21.7	11.2	23.8	11.92%	23.85%	35.77%	16.6	18.4
Li, ppm	7.23	1.41	4.42	10.05	3.02	11.45	19.43%	38.86%	58.29%	6.87	7.60
Lu, ppm	0.14	0.013	0.11	0.16	0.10	0.18	9.37%	18.75%	28.12%	0.13	0.15
Mg, wt.%	1.73	0.060	1.61	1.85	1.55	1.91	3.48%	6.96%	10.44%	1.64	1.81
Mn, wt.%	0.058	0.003	0.051	0.064	0.048	0.067	5.43%	10.85%	16.28%	0.055	0.061
Mo, ppm	0.94	0.072	0.80	1.08	0.73	1.15	7.61%	15.22%	22.84%	0.89	0.99
Na, wt.%	0.186	0.015	0.157	0.216	0.142	0.231	7.92%	15.85%	23.77%	0.177	0.196
Nb, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Nd, ppm	17.5	1.33	14.8	20.1	13.5	21.5	7.62%	15.23%	22.85%	16.6	18.3
Ni, ppm	119	6	106	132	100	138	5.33%	10.65%	15.98%	113	125
P, wt.%	0.099	0.004	0.091	0.107	0.087	0.110	3.89%	7.79%	11.68%	0.094	0.104
Pb, ppm	8.06	1.09	5.89	10.24	4.80	11.33	13.50%	27.01%	40.51%	7.66	8.47
Pr, ppm	4.39	0.61	3.16	5.61	2.55	6.22	13.96%	27.91%	41.87%	4.17	4.61
Rb, ppm	11.2	1.8	7.7	14.7	5.9	16.5	15.75%	31.50%	47.25%	10.6	11.7
S, wt.%	0.012	0.002	0.008	0.015	0.006	0.017	15.01%	30.03%	45.04%	0.011	0.012
Sb, ppm	0.44	0.08	0.27	0.61	0.19	0.70	19.30%	38.59%	57.89%	0.42	0.46
Sc, ppm	5.08	0.54	4.01	6.16	3.47	6.70	10.60%	21.20%	31.81%	4.83	5.34
Se, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

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
Aqua Regia Digestion continued											
Sm, ppm	3.86	0.368	3.12	4.59	2.76	4.96	9.53%	19.06%	28.59%	3.67	4.05
Sn, ppm	0.71	0.11	0.50	0.92	0.39	1.03	14.94%	29.87%	44.81%	0.68	0.75
Sr, ppm	66	9	48	83	39	92	13.26%	26.52%	39.78%	62	69
Ta, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.52	0.07	0.39	0.66	0.32	0.72	12.89%	25.78%	38.67%	0.50	0.55
Te, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	3.06	0.232	2.59	3.52	2.36	3.75	7.58%	15.17%	22.75%	2.90	3.21
Ti, wt.%	0.171	0.026	0.118	0.223	0.092	0.250	15.40%	30.79%	46.19%	0.162	0.179
Tl, ppm	0.068	0.010	0.048	0.088	0.038	0.098	14.59%	29.19%	43.78%	0.065	0.072
Tm, ppm	0.17	0.02	0.12	0.21	0.10	0.23	12.88%	25.75%	38.63%	0.16	0.17
U, ppm	0.59	0.046	0.50	0.68	0.45	0.72	7.82%	15.63%	23.45%	0.56	0.62
V, ppm	41.4	1.91	37.6	45.3	35.7	47.2	4.62%	9.24%	13.86%	39.4	43.5
Y, ppm	12.3	1.02	10.3	14.4	9.3	15.4	8.30%	16.61%	24.91%	11.7	13.0
Yb, ppm	1.00	0.061	0.88	1.12	0.82	1.18	6.07%	12.13%	18.20%	0.95	1.05
Zn, ppm	82	4.3	73	90	69	95	5.24%	10.48%	15.72%	78	86
Zr, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

Note: intervals may appear asymmetric due to rounding

Tolerance Limits (ISO Guide 3207) were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for copper by aqua regia digestion, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 43.6 and 45.9ppm. 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).

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 1 gram was employed and the 1RSD of 1.03% calculated for a 30g fire assay or aqua regia sample (5.61% at 1g weight) confirms the high level of gold homogeneity in OREAS 250. The homogeneity is of a level such that ***sampling error is minor*** for a conventional fire assay or aqua regia determination.

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 gold homogeneity of OREAS 250 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 250. The test was performed using the following parameters:

- Gold fire assay – 132 samples (22 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion – 120 samples (20 laboratories each providing analyses on 3 pairs of samples);
- 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 dataset was filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the *p*-value. This process derived *p*-values of 0.67 for Au by fire assay and 0.96 for Au by aqua regia digestion. Both p-values are insignificant and the Null Hypothesis is retained. Additionally, none of the other certified values showed significant *p*-values.

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 250 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 250 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Johannesburg, South Africa
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. Bureau Veritas Kalassay, Perth, WA, Australia
11. Intertek Genalysis, Adelaide, SA, Australia
12. Intertek Genalysis, Perth, WA, Australia
13. Intertek Minerals (IMI), Jakarta, Indonesia
14. Intertek Testing Services, Cupang, Muntinlupa, Philippines
15. Intertek Testing Services, Shunyi, Beijing, China
16. Intertek Testing Services, Townsville, QLD, Australia
17. Nagrom, Perth, WA, Australia
18. Newmont Metallurgical Services, Engelwood, Colorado, USA
19. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
20. SGS Canada Inc., Vancouver, BC, Canada
21. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
22. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
23. TSL Laboratories Inc., Saskatoon, Saskatchewan, Canada

PREPARER AND SUPPLIER

Certified reference material OREAS 250 is prepared, certified and supplied by:

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It is available in unit sizes of 60g (single-use laminated foil pouches) and 1kg (plastic jars).

INTENDED USE

OREAS 250 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 250 has been prepared from a blend of gold-bearing Wilber Lode oxide ore from the Andy Well Gold Project and barren saprolite and siltstone sourced from quarries north of Melbourne, Australia. It is low in reactive sulphide (0.012% S) and in its unopened state and under normal conditions of storage 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 250 refer to the concentration level in its packaged state. It should not be dried 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.

TRACEABILITY

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

LEGAL NOTICE

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

QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2008 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.

CERTIFYING OFFICER

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

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