



Certified Reference Materials

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**CERTIFICATE OF ANALYSIS FOR**

**HIGH SULPHIDATION EPITHERMAL Ag-Cu-Au ORE**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 601**

**Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 601.**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fire Assay</b>						
Au, Gold (ppm)	0.780	0.031	0.769	0.791	0.769*	0.791*
<b>4-Acid Digestion</b>						
Ag, Silver (ppm)	49.2	2.02	48.3	50.1	48.1	50.3
Al, Aluminium (wt.%)	6.30	0.284	6.18	6.42	6.15	6.45
As, Arsenic (ppm)	307	22.9	296	318	296	317
Be, Beryllium (ppm)	2.07	0.170	2.00	2.15	1.96	2.18
Bi, Bismuth (ppm)	20.9	1.79	20.2	21.6	20.0	21.8
Ca, Calcium (wt.%)	1.31	0.049	1.29	1.33	1.28	1.34
Cd, Cadmium (ppm)	7.86	0.528	7.62	8.10	7.54	8.18
Ce, Cerium (ppm)	63	2.8	61	65	61	65
Co, Cobalt (ppm)	5.14	0.64	4.87	5.41	4.86	5.41
Cr, Chromium (ppm)	42.0	7.7	39.1	44.9	38.0	46.0
Cs, Cesium (ppm)	6.72	0.384	6.46	6.98	6.52	6.92
Cu, Copper (wt.%)	0.101	0.004	0.099	0.102	0.098	0.103
Dy, Dysprosium (ppm)	2.69	0.096	2.58	2.79	2.59	2.79
Er, Erbium (ppm)	0.92	0.061	0.85	1.00	0.87	0.98
Eu, Europium (ppm)	1.23	0.21	0.95	1.50	1.17	1.29
Fe, Iron (wt.%)	2.48	0.159	2.41	2.55	2.42	2.54
Ga, Gallium (ppm)	20.4	0.60	20.1	20.6	19.7	21.1
Gd, Gadolinium (ppm)	4.50	0.48	3.98	5.02	4.39	4.61
Hf, Hafnium (ppm)	4.52	0.268	4.34	4.69	4.34	4.70
Ho, Holmium (ppm)	0.40	0.026	0.37	0.43	0.37	0.42

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

Please note: intervals may appear asymmetric due to rounding.



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Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>4-Acid Digestion continued</b>						
In, Indium (ppm)	1.73	0.150	1.63	1.84	1.67	1.80
K, Potassium (wt.%)	2.10	0.083	2.07	2.14	2.04	2.17
La, Lanthanum (ppm)	30.9	2.50	29.6	32.2	30.0	31.9
Li, Lithium (ppm)	20.5	1.34	19.7	21.3	19.7	21.3
Lu, Lutetium (ppm)	0.095	0.007	0.087	0.103	IND	IND
Mg, Magnesium (wt.%)	0.389	0.019	0.381	0.397	0.377	0.401
Mn, Manganese (wt.%)	0.048	0.002	0.047	0.049	0.047	0.050
Mo, Molybdenum (ppm)	3.87	0.56	3.65	4.09	3.51	4.22
Na, Sodium (wt.%)	1.45	0.069	1.42	1.49	1.42	1.49
Nb, Niobium (ppm)	12.6	0.96	12.0	13.1	12.0	13.1
Nd, Neodymium (ppm)	27.0	0.94	26.2	27.8	25.8	28.2
Ni, Nickel (ppm)	24.3	2.28	23.6	24.9	21.5	27.0
P, Phosphorus (wt.%)	0.047	0.003	0.046	0.048	0.045	0.049
Pb, Lead (ppm)	329	20.4	320	338	321	338
Pr, Praseodymium (ppm)	7.55	0.590	6.90	8.20	7.25	7.84
Rb, Rubidium (ppm)	97	3.6	95	99	94	100
S, Sulphur (wt.%)	1.07	0.040	1.05	1.09	1.03	1.10
Sb, Antimony (ppm)	30.4	2.57	29.3	31.4	28.8	32.0
Sc, Scandium (ppm)	4.85	0.374	4.67	5.02	4.61	5.08
Se, Selenium (ppm)	12.0	2.0	10.6	13.5	10.6	13.4
Sm, Samarium (ppm)	5.12	0.245	4.88	5.37	4.99	5.25
Sn, Tin (ppm)	4.16	0.297	3.93	4.40	3.97	4.36
Sr, Strontium (ppm)	230	10.9	225	235	224	236
Ta, Tantalum (ppm)	1.04	0.23	0.85	1.24	0.95	1.14
Tb, Terbium (ppm)	0.56	0.07	0.49	0.63	0.54	0.58
Te, Tellurium (ppm)	16.0	3.3	13.3	18.6	15.0	16.9
Th, Thorium (ppm)	11.7	0.75	11.1	12.2	11.2	12.2
Ti, Titanium (wt.%)	0.180	0.012	0.174	0.185	0.173	0.187
Tl, Thallium (ppm)	1.21	0.049	1.19	1.24	1.15	1.27
U, Uranium (ppm)	4.00	0.340	3.75	4.26	3.88	4.13
V, Vanadium (ppm)	25.6	1.33	25.0	26.1	24.4	26.7
W, Tungsten (ppm)	5.80	0.79	5.21	6.38	5.36	6.23
Y, Yttrium (ppm)	11.2	0.92	10.7	11.7	10.9	11.5
Yb, Ytterbium (ppm)	0.68	0.042	0.65	0.71	0.60	0.75
Zn, Zinc (ppm)	1330	64.2	1303	1358	1298	1362
Zr, Zirconium (ppm)	155	7.4	151	159	150	160
<b>Aqua Regia Digestion</b>						
Ag, Silver (ppm)	49.4	1.47	48.8	50.1	48.1	50.7
Al, Aluminium (wt.%)	0.826	0.085	0.783	0.869	0.799	0.853
As, Arsenic (ppm)	305	17.2	297	312	297	312
Au, Gold (ppm)	0.774	0.044	0.749	0.798	0.761 <sup>†</sup>	0.786 <sup>†</sup>

<sup>†</sup>Gold Tolerance Limits for typical 25g aqua regia sample weight determined as above.  
Please note: intervals may appear asymmetric due to rounding.

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Aqua Regia Digestion continued</b>						
B, Boron (ppm)	< 10	IND	IND	IND	IND	IND
Be, Beryllium (ppm)	0.62	0.059	0.58	0.65	0.57	0.67
Bi, Bismuth (ppm)	20.6	1.90	19.7	21.5	19.9	21.4
Ca, Calcium (wt.%)	1.07	0.064	1.04	1.10	1.04	1.09
Cd, Cadmium (ppm)	7.81	0.575	7.53	8.09	7.57	8.05
Ce, Cerium (ppm)	44.8	3.64	42.0	47.5	43.6	46.0
Co, Cobalt (ppm)	4.70	0.48	4.54	4.87	4.57	4.84
Cr, Chromium (ppm)	44.2	5.2	42.1	46.3	40.5	47.9
Cs, Cesium (ppm)	1.98	0.39	1.66	2.29	1.90	2.05
Cu, Copper (wt.%)	0.101	0.003	0.100	0.102	0.099	0.103
Dy, Dysprosium (ppm)	1.53	0.105	1.40	1.67	1.49	1.58
Er, Erbium (ppm)	0.47	0.038	0.42	0.52	0.44	0.50
Eu, Europium (ppm)	0.66	0.07	0.57	0.75	0.62	0.70
Fe, Iron (wt.%)	2.20	0.116	2.14	2.25	2.14	2.25
Ga, Gallium (ppm)	5.17	0.484	4.85	5.49	5.03	5.31
Gd, Gadolinium (ppm)	2.65	0.29	2.32	2.97	2.54	2.76
Hf, Hafnium (ppm)	< 1	IND	IND	IND	IND	IND
Hg, Mercury (ppm)	< 3	IND	IND	IND	IND	IND
Ho, Holmium (ppm)	0.21	0.021	0.19	0.24	0.20	0.23
In, Indium (ppm)	1.68	0.143	1.56	1.80	1.63	1.73
K, Potassium (wt.%)	0.251	0.018	0.242	0.261	0.241	0.262
La, Lanthanum (ppm)	21.2	1.55	20.4	21.9	20.4	21.9
Li, Lithium (ppm)	7.95	0.94	7.38	8.51	7.57	8.33
Lu, Lutetium (ppm)	0.040	0.006	0.035	0.046	IND	IND
Mg, Magnesium (wt.%)	0.195	0.021	0.185	0.205	0.187	0.203
Mn, Manganese (wt.%)	0.045	0.002	0.044	0.046	0.044	0.046
Mo, Molybdenum (ppm)	3.80	0.64	3.57	4.03	3.32	4.27
Na, Sodium (wt.%)	0.070	0.006	0.067	0.073	IND	IND
Nb, Niobium (ppm)	< 1	IND	IND	IND	IND	IND
Nd, Neodymium (ppm)	18.8	1.58	17.0	20.6	18.1	19.5
Ni, Nickel (ppm)	24.1	2.7	23.0	25.2	22.2	26.0
P, Phosphorus (wt.%)	0.036	0.002	0.035	0.037	0.034	0.038
Pb, Lead (ppm)	283	9.5	279	287	275	291
Pr, Praseodymium (ppm)	5.37	0.313	4.98	5.76	5.23	5.51
Rb, Rubidium (ppm)	16.0	2.8	13.8	18.3	15.4	16.7
S, Sulphur (wt.%)	1.04	0.045	1.02	1.06	1.01	1.07
Sb, Antimony (ppm)	21.1	4.2	19.0	23.1	19.9	22.2
Sc, Scandium (ppm)	1.83	0.33	1.70	1.95	1.73	1.92
Se, Selenium (ppm)	12.3	1.6	11.3	13.2	11.7	12.8
Sm, Samarium (ppm)	3.42	0.318	3.07	3.77	3.33	3.52
Sn, Tin (ppm)	2.61	0.118	2.49	2.72	2.50	2.71
Sr, Strontium (ppm)	36.2	4.2	34.1	38.2	35.1	37.2

Please note: intervals may appear asymmetric due to rounding.

Table 1 continued.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Aqua Regia Digestion continued</b>						
Tb, Terbium (ppm)	0.33	0.04	0.30	0.37	0.32	0.34
Te, Tellurium (ppm)	15.4	1.11	14.3	16.4	14.5	16.3
Th, Thorium (ppm)	6.70	0.522	6.29	7.10	6.49	6.91
Ti, Titanium (ppm)	103	14	94	113	IND	IND
Tl, Thallium (ppm)	0.74	0.08	0.68	0.80	0.70	0.78
U, Uranium (ppm)	1.94	0.191	1.79	2.08	1.84	2.03
V, Vanadium (ppm)	9.24	1.34	8.59	9.89	8.53	9.95
W, Tungsten (ppm)	1.06	0.17	0.88	1.24	0.99	1.14
Y, Yttrium (ppm)	5.87	0.549	5.51	6.22	5.67	6.07
Yb, Ytterbium (ppm)	0.30	0.020	0.29	0.31	0.28	0.32
Zn, Zinc (ppm)	1293	78.6	1259	1328	1270	1317
Zr, Zirconium (ppm)	26.7	5.2	23.4	29.9	25.6	27.7
<b>Infrared Combustion</b>						
S, Sulphur (wt.%)	1.10	0.030	1.09	1.12	1.08	1.13

Please note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 601.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Ag	ppm	46.9	Pd	ppb	< 5	Pt	ppb	< 5
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt.%	12.46	Fe <sub>2</sub> O <sub>3</sub>	wt.%	3.63	Pb	ppm	355
As	ppm	310	K <sub>2</sub> O	wt.%	2.60	SiO <sub>2</sub>	wt.%	71.46
Ba	ppm	4230	MgO	wt.%	0.690	Sn	ppm	20.0
CaO	wt.%	1.86	MnO	wt.%	0.070	SO <sub>3</sub>	wt.%	2.79
Co	ppm	20.0	Na <sub>2</sub> O	wt.%	2.04	TiO <sub>2</sub>	wt.%	0.308
Cr	ppm	50	Ni	ppm	20.0	U	ppm	12.5
Cu	ppm	1005	P <sub>2</sub> O <sub>5</sub>	wt.%	0.112	Zn	ppm	1285
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt.%	2.71						
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	45.3	Ho	ppm	0.44	Sn	ppm	5.30
As	ppm	300	In	ppm	1.68	Sr	ppm	236
Ba	ppm	4235	La	ppm	34.6	Ta	ppm	1.04
Be	ppm	2.20	Lu	ppm	0.095	Tb	ppm	0.52
Bi	ppm	21.3	Mn	wt.%	0.048	Te	ppm	14.5
Cd	ppm	6.85	Mo	ppm	3.20	Th	ppm	12.1
Ce	ppm	62	Nb	ppm	12.2	Ti	wt.%	0.180
Co	ppm	5.55	Nd	ppm	26.6	Tl	ppm	1.10
Cr	ppm	48.5	Ni	ppm	26.0	Tm	ppm	0.13
Cs	ppm	6.77	Pb	ppm	330	U	ppm	4.10
Cu	ppm	978	Pr	ppm	7.56	V	ppm	23.5
Dy	ppm	2.50	Rb	ppm	94	W	ppm	5.48
Er	ppm	1.06	Re	ppm	0.008	Y	ppm	12.4

Table 2 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Laser Ablation ICP-MS continued								
Eu	ppm	0.99	Sb	ppm	28.1	Yb	ppm	0.77
Ga	ppm	22.2	Sc	ppm	4.55	Zn	ppm	1230
Gd	ppm	3.90	Se	ppm	6.25	Zr	ppm	187
Hf	ppm	5.56	Sm	ppm	5.02			
4-Acid Digestion								
B	ppm	< 20	Ge	ppm	1.36	Re	ppb	3
Ba	ppm	4128	Hg	ppm	< 1	Tm	ppm	0.11
Aqua Regia Digestion								
Ba	ppm	2714	Pt	ppb	< 5	Ta	ppm	0.099
Ge	ppm	< 0.1	Re	ppb	< 1	Tm	ppm	0.049
Pd	ppb	13	Ru	ppb	< 2			
Infrared Combustion								
C	wt.%	0.291						

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

OREAS 601 was prepared from gold-silver-copper bearing ore from Evolution Mining's Mount Carlton Operation in Queensland, Australia and blended with argillic rhyodacite waste rock to achieve the desired grades. The mineralisation assemblage consists of pyrite, enargite/tennantite, tetrahedrite, digenite, covellite, sphalerite, galena, alunite, dickite, kaolinite and vuggy silica, hosted in advanced argillic altered rhyodacite containing sulphur-salts. OREAS 601 is one of a suite of six CRMs ranging in grades from 24ppm Ag, 0.2 ppm Au and 0.05% Cu to 980ppm Ag, 1.7ppm Au and 5.0% Cu.

## COMMUNITION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 601 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and milling of the barren material to 95% minus 75 microns;
- crushing and milling of the ore material to 100% minus 30 microns;
- blending in appropriate proportions to achieve the desired grades;
- packaging in 60g and 10g units sealed under nitrogen in laminated foil pouches and 1kg units in plastic jars.

## ANALYTICAL PROGRAM

Twenty eight commercial analytical laboratories participated in the program to certify the 116 elements reported in Table 1. The following methods were employed:

- Gold via 20-40g\* fire assay with AAS (20 labs), ICP-OES (4 labs) or gravimetric (3 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 (7 labs) or AAS (5 labs) finish;
- 4-Acid digestion for full elemental suite ICP-OES and ICP-MS (up to 21 laboratories depending on the element).
- Aqua regia digestion (see note below) for full elemental suite ICP-OES and ICP-MS (up to 22 laboratories depending on the element).
- Sulphur via Infrared Combustion Analysis (16 labs).

\*The certified values (and 95% Confidence Interval and SD) for Au are also applicable to 50g charge weights.

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 test units 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 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 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 90 indicative values for major and trace element composition. Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty ~1g sample portions (see Table 3 below) 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 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 601 DataPack.xlsx**).

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

Replicate No	NAA 1g
1	0.745
2	0.737
3	0.714
4	0.724
5	0.739
6	0.711
7	0.716
8	0.743
9	0.726
10	0.693
11	0.720
12	0.704
13	0.746
14	0.734
15	0.743
16	0.711
17	0.699
18	0.723
19	0.698
20	0.690
Mean	0.721
Median	0.722
Std Dev.	0.018
Rel.Std.Dev.	2.52%
PDM <sup>3</sup>	-7.59%

## 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 601.

**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 ( $\text{Al}_2\text{O}_3$  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 per cent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates



calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow.

**Table 4. Performance Gates for OREAS 601.**

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>Fire Assay</b>											
Au, ppm	0.780	0.031	0.717	0.843	0.686	0.874	4.03%	8.06%	12.08%	0.741	0.819
<b>4-Acid Digestion</b>											
Ag, ppm	49.2	2.02	45.1	53.2	43.1	55.2	4.11%	8.23%	12.34%	46.7	51.6
Al, wt. %	6.30	0.284	5.73	6.87	5.45	7.15	4.51%	9.02%	13.52%	5.98	6.61
As, ppm	307	23	261	353	238	376	7.47%	14.95%	22.42%	291	322
Be, ppm	2.07	0.170	1.73	2.41	1.56	2.58	8.21%	16.42%	24.63%	1.97	2.18
Bi, ppm	20.9	1.79	17.3	24.5	15.5	26.3	8.59%	17.17%	25.76%	19.9	21.9
Ca, wt. %	1.31	0.049	1.21	1.41	1.16	1.46	3.76%	7.52%	11.28%	1.25	1.38
Cd, ppm	7.86	0.528	6.80	8.91	6.27	9.44	6.72%	13.44%	20.16%	7.46	8.25
Ce, ppm	63	2.8	57	68	55	71	4.38%	8.77%	13.15%	60	66
Co, ppm	5.14	0.64	3.85	6.43	3.20	7.07	12.55%	25.10%	37.65%	4.88	5.39
Cr, ppm	42.0	7.7	26.6	57.4	18.9	65.2	18.35%	36.70%	55.05%	39.9	44.1
Cs, ppm	6.72	0.384	5.95	7.49	5.57	7.87	5.71%	11.43%	17.14%	6.38	7.05
Cu, wt. %	0.101	0.004	0.093	0.108	0.090	0.111	3.55%	7.10%	10.65%	0.096	0.106
Dy, ppm	2.69	0.096	2.50	2.88	2.40	2.98	3.57%	7.13%	10.70%	2.55	2.82
Er, ppm	0.92	0.061	0.80	1.05	0.74	1.11	6.62%	13.24%	19.85%	0.88	0.97
Eu, ppm	1.23	0.21	0.82	1.64	0.61	1.85	16.79%	33.59%	50.38%	1.17	1.29
Fe, wt. %	2.48	0.159	2.17	2.80	2.01	2.96	6.39%	12.78%	19.17%	2.36	2.61
Ga, ppm	20.4	0.60	19.2	21.6	18.6	22.2	2.95%	5.90%	8.85%	19.3	21.4
Gd, ppm	4.50	0.48	3.54	5.46	3.06	5.94	10.66%	21.32%	31.97%	4.28	4.73
Hf, ppm	4.52	0.268	3.98	5.05	3.71	5.32	5.93%	11.86%	17.79%	4.29	4.74
Ho, ppm	0.40	0.026	0.35	0.45	0.32	0.48	6.63%	13.25%	19.88%	0.38	0.42
In, ppm	1.73	0.150	1.43	2.03	1.28	2.18	8.66%	17.31%	25.97%	1.65	1.82
K, wt. %	2.10	0.083	1.94	2.27	1.85	2.35	3.95%	7.90%	11.85%	2.00	2.21
La, ppm	30.9	2.50	25.9	35.9	23.4	38.4	8.07%	16.14%	24.21%	29.4	32.5
Li, ppm	20.5	1.34	17.8	23.2	16.5	24.5	6.53%	13.05%	19.58%	19.5	21.5
Lu, ppm	0.095	0.007	0.081	0.109	0.074	0.116	7.47%	14.94%	22.40%	0.090	0.100
Mg, wt. %	0.389	0.019	0.351	0.427	0.332	0.446	4.91%	9.83%	14.74%	0.369	0.408
Mn, wt. %	0.048	0.002	0.044	0.052	0.042	0.054	4.18%	8.37%	12.55%	0.046	0.051

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>											
Mo, ppm	3.87	0.56	2.75	4.98	2.19	5.54	14.45%	28.90%	43.35%	3.67	4.06
Na, wt. %	1.45	0.069	1.32	1.59	1.25	1.66	4.71%	9.43%	14.14%	1.38	1.53
Nb, ppm	12.6	0.96	10.6	14.5	9.7	15.4	7.63%	15.26%	22.89%	11.9	13.2
Nd, ppm	27.0	0.94	25.1	28.9	24.2	29.8	3.50%	7.00%	10.49%	25.7	28.4
Ni, ppm	24.3	2.28	19.7	28.8	17.4	31.1	9.41%	18.82%	28.22%	23.0	25.5
P, wt. %	0.047	0.003	0.040	0.053	0.037	0.056	6.83%	13.65%	20.48%	0.044	0.049
Pb, ppm	329	20	288	370	268	390	6.19%	12.38%	18.57%	313	346
Pr, ppm	7.55	0.590	6.37	8.73	5.78	9.32	7.81%	15.62%	23.44%	7.17	7.93
Rb, ppm	97	3.6	90	105	86	108	3.74%	7.48%	11.22%	92	102
S, wt. %	1.07	0.040	0.99	1.15	0.95	1.19	3.78%	7.55%	11.33%	1.01	1.12
Sb, ppm	30.4	2.57	25.2	35.5	22.7	38.1	8.47%	16.94%	25.40%	28.8	31.9
Sc, ppm	4.85	0.374	4.10	5.59	3.72	5.97	7.72%	15.44%	23.16%	4.60	5.09
Se, ppm	12.0	2.0	8.1	16.0	6.1	18.0	16.44%	32.88%	49.32%	11.4	12.6
Sm, ppm	5.12	0.245	4.63	5.61	4.39	5.86	4.79%	9.58%	14.37%	4.87	5.38
Sn, ppm	4.16	0.297	3.57	4.76	3.27	5.05	7.13%	14.27%	21.40%	3.95	4.37
Sr, ppm	230	11	208	252	197	263	4.74%	9.49%	14.23%	219	242
Ta, ppm	1.04	0.23	0.58	1.51	0.35	1.74	22.29%	44.57%	66.86%	0.99	1.10
Tb, ppm	0.56	0.07	0.42	0.70	0.35	0.77	12.35%	24.71%	37.06%	0.53	0.59
Te, ppm	16.0	3.3	9.4	22.5	6.2	25.7	20.41%	40.82%	61.24%	15.2	16.8
Th, ppm	11.7	0.75	10.2	13.2	9.4	13.9	6.41%	12.82%	19.23%	11.1	12.2
Ti, wt. %	0.180	0.012	0.155	0.204	0.143	0.216	6.80%	13.61%	20.41%	0.171	0.189
Tl, ppm	1.21	0.049	1.11	1.31	1.06	1.36	4.07%	8.13%	12.20%	1.15	1.27
U, ppm	4.00	0.340	3.32	4.68	2.98	5.02	8.48%	16.96%	25.45%	3.80	4.20
V, ppm	25.6	1.33	22.9	28.2	21.6	29.5	5.20%	10.40%	15.60%	24.3	26.8
W, ppm	5.80	0.79	4.21	7.38	3.42	8.17	13.65%	27.30%	40.95%	5.51	6.09
Y, ppm	11.2	0.92	9.4	13.1	8.4	14.0	8.23%	16.46%	24.69%	10.7	11.8
Yb, ppm	0.68	0.042	0.59	0.76	0.55	0.80	6.16%	12.31%	18.47%	0.64	0.71
Zn, ppm	1330	64	1202	1459	1137	1523	4.83%	9.66%	14.49%	1264	1397
Zr, ppm	155	7	141	170	133	177	4.73%	9.47%	14.20%	148	163
<b>Aqua Regia Digestion</b>											
Ag, ppm	49.4	1.47	46.5	52.4	45.0	53.8	2.98%	5.97%	8.95%	46.9	51.9
Al, wt. %	0.826	0.085	0.655	0.996	0.570	1.082	10.33%	20.67%	31.00%	0.784	0.867

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 continued</b>											
As, ppm	305	17	270	339	253	356	5.66%	11.31%	16.97%	289	320
Au, ppm	0.774	0.044	0.687	0.861	0.643	0.904	5.63%	11.26%	16.89%	0.735	0.812
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.62	0.059	0.50	0.74	0.44	0.80	9.62%	19.24%	28.86%	0.59	0.65
Bi, ppm	20.6	1.90	16.8	24.4	14.9	26.3	9.23%	18.45%	27.68%	19.6	21.7
Ca, wt. %	1.07	0.064	0.94	1.20	0.87	1.26	6.03%	12.05%	18.08%	1.01	1.12
Cd, ppm	7.81	0.575	6.66	8.96	6.09	9.54	7.36%	14.72%	22.08%	7.42	8.20
Ce, ppm	44.8	3.64	37.5	52.1	33.9	55.7	8.13%	16.26%	24.39%	42.5	47.0
Co, ppm	4.70	0.48	3.75	5.66	3.27	6.14	10.19%	20.39%	30.58%	4.47	4.94
Cr, ppm	44.2	5.2	33.8	54.6	28.6	59.8	11.80%	23.59%	35.39%	42.0	46.4
Cs, ppm	1.98	0.39	1.19	2.76	0.80	3.15	19.80%	39.60%	59.40%	1.88	2.08
Cu, wt. %	0.101	0.003	0.094	0.108	0.091	0.112	3.47%	6.93%	10.40%	0.096	0.106
Dy, ppm	1.53	0.105	1.32	1.74	1.22	1.85	6.85%	13.70%	20.55%	1.46	1.61
Er, ppm	0.47	0.038	0.39	0.54	0.35	0.58	8.15%	16.31%	24.46%	0.44	0.49
Eu, ppm	0.66	0.07	0.52	0.80	0.45	0.87	10.51%	21.01%	31.52%	0.63	0.69
Fe, wt. %	2.20	0.116	1.96	2.43	1.85	2.54	5.29%	10.58%	15.87%	2.09	2.31
Ga, ppm	5.17	0.484	4.20	6.14	3.72	6.62	9.37%	18.74%	28.10%	4.91	5.43
Gd, ppm	2.65	0.29	2.06	3.23	1.77	3.52	11.03%	22.06%	33.09%	2.52	2.78
Hf, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Hg, ppm	< 3	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ho, ppm	0.21	0.021	0.17	0.25	0.15	0.28	9.71%	19.43%	29.14%	0.20	0.22
In, ppm	1.68	0.143	1.39	1.97	1.25	2.11	8.53%	17.05%	25.58%	1.60	1.76
K, wt. %	0.251	0.018	0.215	0.287	0.197	0.305	7.16%	14.33%	21.49%	0.239	0.264
La, ppm	21.2	1.55	18.1	24.3	16.5	25.8	7.34%	14.68%	22.03%	20.1	22.2
Li, ppm	7.95	0.94	6.06	9.84	5.11	10.78	11.89%	23.77%	35.66%	7.55	8.34
Lu, ppm	0.040	0.006	0.029	0.052	0.023	0.058	14.39%	28.78%	43.17%	0.038	0.042
Mg, wt. %	0.195	0.021	0.153	0.237	0.132	0.258	10.73%	21.47%	32.20%	0.185	0.205
Mn, wt. %	0.045	0.002	0.041	0.049	0.039	0.051	4.44%	8.87%	13.31%	0.043	0.048
Mo, ppm	3.80	0.64	2.53	5.07	1.89	5.70	16.73%	33.46%	50.19%	3.61	3.99
Na, wt. %	0.070	0.006	0.058	0.082	0.052	0.088	8.52%	17.04%	25.56%	0.067	0.074
Nb, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Nd, ppm	18.8	1.58	15.7	22.0	14.1	23.6	8.42%	16.84%	25.26%	17.9	19.8

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 continued</b>											
Ni, ppm	24.1	2.7	18.7	29.5	16.0	32.2	11.15%	22.29%	33.44%	22.9	25.3
P, wt. %	0.036	0.002	0.033	0.039	0.031	0.041	4.27%	8.54%	12.81%	0.034	0.038
Pb, ppm	283	9	264	302	254	311	3.36%	6.72%	10.07%	269	297
Pr, ppm	5.37	0.313	4.75	6.00	4.43	6.31	5.83%	11.66%	17.48%	5.10	5.64
Rb, ppm	16.0	2.8	10.3	21.7	7.5	24.5	17.70%	35.40%	53.11%	15.2	16.8
S, wt. %	1.04	0.045	0.95	1.13	0.91	1.17	4.29%	8.58%	12.88%	0.99	1.09
Sb, ppm	21.1	4.2	12.7	29.4	8.5	33.6	19.81%	39.61%	59.42%	20.0	22.1
Sc, ppm	1.83	0.33	1.16	2.49	0.83	2.82	18.14%	36.29%	54.43%	1.74	1.92
Se, ppm	12.3	1.6	9.0	15.5	7.4	17.1	13.14%	26.28%	39.41%	11.6	12.9
Sm, ppm	3.42	0.318	2.79	4.06	2.47	4.38	9.29%	18.59%	27.88%	3.25	3.59
Sn, ppm	2.61	0.118	2.37	2.84	2.25	2.96	4.52%	9.04%	13.56%	2.48	2.74
Sr, ppm	36.2	4.2	27.8	44.5	23.6	48.7	11.56%	23.12%	34.68%	34.3	38.0
Tb, ppm	0.33	0.04	0.25	0.41	0.21	0.45	12.16%	24.32%	36.48%	0.31	0.35
Te, ppm	15.4	1.11	13.1	17.6	12.0	18.7	7.25%	14.50%	21.75%	14.6	16.1
Th, ppm	6.70	0.522	5.65	7.74	5.13	8.26	7.79%	15.59%	23.38%	6.36	7.03
Ti, ppm	103	14	75	132	60	147	13.97%	27.95%	41.92%	98	109
Tl, ppm	0.74	0.08	0.58	0.90	0.50	0.98	10.89%	21.79%	32.68%	0.70	0.78
U, ppm	1.94	0.191	1.55	2.32	1.36	2.51	9.86%	19.72%	29.58%	1.84	2.03
V, ppm	9.24	1.34	6.57	11.91	5.23	13.25	14.45%	28.91%	43.36%	8.78	9.70
W, ppm	1.06	0.17	0.73	1.39	0.56	1.56	15.59%	31.17%	46.76%	1.01	1.11
Y, ppm	5.87	0.549	4.77	6.97	4.22	7.51	9.36%	18.72%	28.07%	5.57	6.16
Yb, ppm	0.30	0.020	0.26	0.34	0.24	0.36	6.58%	13.16%	19.74%	0.29	0.32
Zn, ppm	1293	79	1136	1451	1058	1529	6.08%	12.15%	18.23%	1229	1358
Zr, ppm	26.7	5.2	16.2	37.2	11.0	42.4	19.65%	39.29%	58.94%	25.3	28.0
<b>Infrared Combustion</b>											
S, wt. %	1.10	0.030	1.04	1.16	1.01	1.19	2.73%	5.46%	8.20%	1.05	1.16

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 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 0.098 and 0.103wt.%. 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 1g was employed and the 1RSD of 0.46% calculated at a 30g charge weight (2.52% at 1g weights) confirms the high level of gold homogeneity in OREAS 601. Au by fire assay is reported by 27 laboratories and the charge weights range from 20-40g. The most common charge weight used in this round robin was 30g (19 labs) and tolerance intervals have been calculated at this sample weight. For Au by aqua regia digestion, tolerance limits have been calculated at a 25g sample weight (mode from the 25-50g sample weights used at 13 laboratories).

The gold homogeneity of OREAS 601 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty-eight 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 601. The test was performed using the following parameters:

- Gold fire assay – 162 samples (27 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion – 78 samples (13 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 0.99 for Au by fire assay and 0.94 for Au by aqua regia digestion. Both  $p$ -values are insignificant and the Null Hypothesis is retained. Additionally, none of the other 114 certified values showed significant  $p$ -values except for Ti by aqua regia digestion which is present in trace levels close to the lower level of detection. Its failure can be rationalised as an artifact of reading resolution error of the analytical methods employed at the laboratories.

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 601 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 601 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PARTICIPATING LABORATORIES

1. Accurassay, Thunder Bay, Ontario, Canada
2. Acme (BV), Santiago, Chile
3. Actlabs, Ancaster, Ontario, Canada
4. AH Knight, Spartanburg, SC, USA
5. ALS, Johannesburg, South Africa
6. ALS, Lima, Peru
7. ALS, Reno, Nevada, USA
8. ALS, Townsville, QLD, Australia
9. ALS, Val-d'or, Quebec, Canada
10. ALS, Vancouver, BC, Canada
11. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
12. Bureau Veritas Geoanalytical, Kalgoorlie, WA, Australia
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. Bureau Veritas Kalassay, Kalgoorlie, WA, Australia
15. Inspectorate (BV), Lima, Peru
16. Inspectorate (BV), Sparks, Nevada, USA
17. Intertek Genalysis, Adelaide, SA, Australia
18. Intertek Genalysis, Perth, WA, Australia
19. Intertek Testing Services, Cupang, Muntinlupa, Philippines
20. Intertek Testing Services, Shunyi, Beijing, China
21. MINTEK Analytical Services, Randburg, South Africa
22. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
23. SGS de Mexico, Durango, Mexico
24. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
25. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
26. SGS South Africa Pty Ltd, Booyens, Gauteng, South Africa
27. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
28. SRL (Bureau Veritas), Perth, WA, Australia

## PREPARER AND SUPPLIER

Certified reference material OREAS 601 is prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd  
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It is available in unit sizes of 10 and 60g (single-use laminated foil pouches) and 1kg (plastic jars).

## INTENDED USE

OREAS 601 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 601 has been prepared from gold-silver-copper bearing ore from Evolution Mining's Mount Carlton Operation in Queensland, Australia and blended with argillic altered rhyodacite waste rock. It is low in reactive sulphide (1.10% S) however, as a precaution has been packaged under a nitrogen environment (single use laminated foil pouches only). In its unopened state and under normal conditions of storage the CRM has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

## INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 601 refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis. The certified values for gold by fire assay and aqua regia digestion are applicable to charge/sample weights ranging 20-50g.

## 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

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## 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

A handwritten signature in blue ink, appearing to read 'S. Hamlyn', is positioned above a horizontal line.

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

## REFERENCES

- Ingamells, C. O. and Switzer, P. (1973), Talanta 20, 547-568.
- ISO Guide 30 (1992), Terms and definitions used in connection with reference materials.
- ISO Guide 31 (2000), Reference materials – Contents of certificates and labels.
- ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.
- ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.