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#### CERTIFICATE OF ANALYSIS FOR

# GOLD-SILVER ORE CERTIFIED REFERENCE MATERIAL OREAS 60d

Constituent	Certified	190	95% Confic	lence Limits	95% Tolerance Limits						
Constituent	Value	130	Low	High	Low	High					
Fire Assay											
Au, Gold (ppm)	2.47	0.079	2.44	2.50	2.44*	2.51*					
Aqua Regia Digestion (sample weights 10-50g)											
Au, Gold (ppm)	2.43	0.110	2.38	2.48	2.40 <sup>†</sup>	2.47 <sup>†</sup>					
4-Acid Digestion											
Ag, Silver (ppm)	4.57	0.178	4.52	4.63	4.44	4.70					
S, Sulphur (wt.%)	0.573	0.023	0.563	0.582	0.561	0.584					
Aqua Regia Digestion											
Ag, Silver (ppm)	4.45	0.237	4.33	4.56	4.31	4.59					

#### Summary Statistics for Key Analytes.

\*Gold Tolerance Limits for typical 30g fire assay charge weight determined from 20 x 1g INAA results and the Sampling Constant (Ingamells & Switzer, 1973); <sup>†</sup>Gold Tolerance Limits for typical 25g aqua regia sample weight determined as above; Note: intervals may appear asymmetric due to rounding; Full certified elements list available in Table 1 below.



Constituent	Certified	150	95% Confid	ence Limits	95% Tolerance Limits		
Constituent	Value	150	Low	High	Low	High	
Fire Assay							
Au, Gold (ppm)	2.47	0.079	2.44	2.50	2.44*	2.51*	
Aqua Regia Digestion (sar	nple weights	; 10-50g)					
Au, Gold (ppm)	2.43	0.110	2.38	2.48	2.40 <sup>†</sup>	2.47 <sup>†</sup>	
4-Acid Digestion							
Ag, Silver (ppm)	4.57	0.178	4.52	4.63	4.44	4.70	
Al, Aluminium (wt.%)	3.56	0.176	3.49	3.64	3.49	3.63	
As, Arsenic (ppm)	15.7	1.39	15.1	16.3	14.9	16.5	
Ba, Barium (ppm)	215	8	212	218	210	220	
Be, Beryllium (ppm)	0.72	0.070	0.69	0.75	0.67	0.76	
Bi, Bismuth (ppm)	0.31	0.022	0.30	0.32	0.29	0.33	
Ca, Calcium (wt.%)	10.19	0.396	10.03	10.35	9.97	10.41	
Cd, Cadmium (ppm)	0.15	0.03	0.14	0.16	IND	IND	
Ce, Cerium (ppm)	18.6	1.67	17.8	19.4	18.0	19.1	
Co, Cobalt (ppm)	7.59	0.689	7.23	7.94	7.32	7.85	
Cr, Chromium (ppm)	13.6	2.7	12.5	14.8	12.6	14.7	
Cs, Cesium (ppm)	4.34	0.274	4.22	4.47	4.21	4.48	
Cu, Copper (ppm)	73	4.4	72	75	71	75	
Dy, Dysprosium (ppm)	1.37	0.060	1.32	1.41	1.32	1.42	
Er, Erbium (ppm)	0.81	0.050	0.76	0.85	0.75	0.86	
Eu, Europium (ppm)	0.50	0.06	0.46	0.55	0.47	0.54	
Fe, Iron (wt.%)	1.96	0.099	1.92	2.00	1.92	2.00	
Ga, Gallium (ppm)	8.15	0.562	7.84	8.46	7.86	8.43	
Gd, Gadolinium (ppm)	1.62	0.103	1.55	1.68	1.55	1.69	
Hf, Hafnium (ppm)	1.30	0.128	1.24	1.36	1.22	1.39	
Ho, Holmium (ppm)	0.28	0.022	0.26	0.30	0.27	0.30	
In, Indium (ppm)	0.023	0.003	0.021	0.024	0.020	0.025	
K, Potassium (wt.%)	1.51	0.068	1.49	1.54	1.48	1.55	
La, Lanthanum (ppm)	9.09	0.321	8.95	9.23	8.82	9.36	
Li, Lithium (ppm)	40.6	3.27	39.1	42.2	39.5	41.8	
Lu, Lutetium (ppm)	0.12	0.010	0.11	0.13	IND	IND	
Mg, Magnesium (wt.%)	0.590	0.033	0.576	0.604	0.578	0.602	
Mn, Manganese (wt.%)	0.059	0.002	0.058	0.060	0.057	0.060	
Mo, Molybdenum (ppm)	2.83	0.29	2.72	2.94	2.69	2.97	
Na, Sodium (wt.%)	0.565	0.039	0.548	0.582	0.553	0.577	
Nb, Niobium (ppm)	1.78	0.173	1.70	1.87	1.68	1.88	
Nd, Neodymium (ppm)	8.90	0.529	8.53	9.28	8.63	9.18	
Ni, Nickel (ppm)	9.56	0.945	9.17	9.95	9.05	10.07	
P, Phosphorus (wt.%)	0.042	0.002	0.041	0.043	0.041	0.043	
Pb, Lead (ppm)	10.6	1.1	10.1	11.0	9.9	11.2	
Pr, Praseodymium (ppm)	2.20	0.095	2.14	2.27	2.12	2.28	
Rb, Rubidium (ppm)	61	3.3	60	63	59	63	

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 60d.

\*Gold Tolerance Limits for typical 30g fire assay charge weight determined from 20 x 1g INAA results and the Sampling Constant (Ingamells & Switzer, 1973); <sup>†</sup>Gold Tolerance Limits for typical 25g aqua regia sample weight determined as above; Note: intervals may appear asymmetric due to rounding.



Table 1 continued.											
Constituent	Certified	400	95% Confid	ence Limits	95% Tolerance Limits						
	Value	150	Low	High	Low	High					
4-Acid Digestion											
S, Sulphur (wt.%)	0.573	0.023	0.563	0.582	0.561	0.584					
Sb, Antimony (ppm)	2.14	0.202	2.04	2.23	2.01	2.26					
Sc, Scandium (ppm)	6.85	0.72	6.55	7.14	6.66	7.03					
Sm, Samarium (ppm)	1.79	0.101	1.72	1.87	1.71	1.87					
Sn, Tin (ppm)	0.70	0.12	0.62	0.77	0.66	0.73					
Sr, Strontium (ppm)	244	12	239	249	239	249					
Ta, Tantalum (ppm)	0.13	0.02	0.11	0.14	IND	IND					
Tb, Terbium (ppm)	0.24	0.023	0.23	0.26	0.23	0.26					
Te, Tellurium (ppm)	2.15	0.23	2.04	2.27	2.01	2.30					
Th, Thorium (ppm)	2.15	0.198	2.06	2.24	2.04	2.26					
Ti, Titanium (wt.%)	0.171	0.006	0.169	0.174	0.166	0.176					
TI, Thallium (ppm)	0.64	0.034	0.62	0.66	0.61	0.68					
Tm, Thulium (ppm)	0.11	0.01	0.10	0.12	IND	IND					
U, Uranium (ppm)	0.65	0.052	0.63	0.67	0.60	0.69					
V, Vanadium (ppm)	62	2.2	61	63	61	64					
W, Tungsten (ppm)	2.28	0.220	2.17	2.39	2.06	2.49					
Y, Yttrium (ppm)	7.25	0.531	7.02	7.48	7.01	7.48					
Yb, Ytterbium (ppm)	0.75	0.047	0.72	0.77	0.71	0.78					
Zn, Zinc (ppm)	36.9	1.99	36.1	37.7	35.7	38.0					
Zr, Zirconium (ppm)	49.6	3.92	47.9	51.2	48.1	51.0					
Aqua Regia Digestion											
Ag, Silver (ppm)	4.45	0.237	4.33	4.56	4.31	4.59					
Al, Aluminium (wt.%)	1.00	0.083	0.96	1.04	0.97	1.03					
As, Arsenic (ppm)	15.3	0.89	14.9	15.7	14.9	15.7					
Ba, Barium (ppm)	36.4	2.09	35.3	37.5	35.5	37.2					
Be, Beryllium (ppm)	0.50	0.041	0.49	0.51	0.46	0.54					
Bi, Bismuth (ppm)	0.30	0.016	0.29	0.31	0.28	0.31					
Ca, Calcium (wt.%)	9.78	0.633	9.48	10.07	9.58	9.98					
Cd, Cadmium (ppm)	0.14	0.01	0.13	0.15	0.12	0.15					
Ce, Cerium (ppm)	15.8	0.71	15.4	16.2	15.4	16.2					
Co, Cobalt (ppm)	7.44	0.376	7.26	7.62	7.27	7.60					
Cr, Chromium (ppm)	13.3	1.9	12.4	14.1	12.9	13.6					
Cs, Cesium (ppm)	2.32	0.36	2.11	2.52	2.24	2.39					
Cu, Copper (ppm)	72	2.8	70	73	70	73					
Dy, Dysprosium (ppm)	0.97	0.050	0.93	1.02	0.93	1.02					
Er, Erbium (ppm)	0.51	0.032	0.47	0.55	0.48	0.55					
Fe, Iron (wt.%)	1.83	0.087	1.79	1.86	1.79	1.86					
Ga, Gallium (ppm)	3.96	0.396	3.75	4.17	3.80	4.12					
Hf, Hafnium (ppm)	0.22	0.04	0.20	0.25	0.21	0.24					
Hg, Mercury (ppm)	0.100	0.006	0.098	0.102	IND	IND					
Ho, Holmium (ppm)	0.19	0.008	0.19	0.20	0.19	0.20					



Constituent	Certified	1SD	95% Confid	ence Limits	95% Tolera	ance Limits						
	Value		Low	High	Low	High						
Aqua Regia Digestion con	tinued		T			1						
In, Indium (ppm)	0.018	0.003	0.017	0.020	IND	IND						
K, Potassium (wt.%)	0.199	0.022	0.189	0.209	0.194	0.205						
La, Lanthanum (ppm)	7.90	0.436	7.69	8.11	7.71	8.09						
Li, Lithium (ppm)	9.88	0.392	9.66	10.10	9.56	10.20						
Lu, Lutetium (ppm)	0.061	0.005	0.057	0.064	IND	IND						
Mg, Magnesium (wt.%)	0.516	0.018	0.508	0.524	0.505	0.527						
Mn, Manganese (wt.%)	0.057	0.003	0.056	0.058	0.056	0.058						
Mo, Molybdenum (ppm)	2.63	0.167	2.55	2.72	2.55	2.72						
Na, Sodium (wt.%)	0.061	0.008	0.056	0.065	0.058	0.063						
Nd, Neodymium (ppm)	7.64	0.264	7.40	7.89	7.49	7.80						
Ni, Nickel (ppm)	8.64	0.669	8.28	8.99	8.43	8.84						
P, Phosphorus (wt.%)	0.039	0.002	0.038	0.040	0.038	0.040						
Pb, Lead (ppm)	8.67	0.799	8.23	9.11	8.32	9.02						
Pr, Praseodymium (ppm)	1.91	0.078	1.84	1.98	1.85	1.97						
Rb, Rubidium (ppm)	9.91	1.40	9.12	10.70	9.59	10.23						
S, Sulphur (wt.%)	0.563	0.016	0.555	0.571	0.551	0.575						
Sb, Antimony (ppm)	0.43	0.04	0.40	0.45	0.38	0.47						
Sc, Scandium (ppm)	4.30	0.180	4.20	4.40	4.17	4.43						
Sm, Samarium (ppm)	1.51	0.067	1.46	1.57	1.45	1.58						
Sn, Tin (ppm)	0.39	0.032	0.37	0.40	0.36	0.41						
Sr, Strontium (ppm)	137	13	131	143	134	141						
Tb, Terbium (ppm)	0.19	0.005	0.18	0.19	0.17	0.20						
Te, Tellurium (ppm)	2.02	0.169	1.93	2.10	1.94	2.09						
Th, Thorium (ppm)	1.20	0.13	1.14	1.27	1.14	1.27						
Ti, Titanium (wt.%)	0.032	0.004	0.030	0.034	0.031	0.033						
TI, Thallium (ppm)	0.17	0.02	0.16	0.18	0.16	0.18						
U, Uranium (ppm)	0.29	0.014	0.29	0.30	0.28	0.31						
V, Vanadium (ppm)	37.8	2.88	36.4	39.2	37.2	38.5						
Y, Yttrium (ppm)	5.28	0.287	5.15	5.42	5.17	5.40						
Yb, Ytterbium (ppm)	0.42	0.030	0.41	0.43	0.39	0.45						
Zn, Zinc (ppm)	32.9	2.14	31.9	33.9	31.6	34.2						
Zr, Zirconium (ppm)	7.40	1.19	6.76	8.04	7.17	7.63						

Table 1 continued

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 60d was prepared from coarse reject splits of gold-silver ore samples blended with barren andesite. The gold-silver ore was sourced from the Cracow mine located 500km northwest of Brisbane in Queensland, Australia. The barren andesite was sourced from the Carboniferous Blair Duguid Hypersthene Andesite intrusive, located 70kms northwest of Newcastle, Australia. Cracow is a low sulphidation epithermal deposit hosted by meta-andesitic volcanics. High grade gold mineralisation occurs within fissure quartz veins and is associated with zones of silicification, present as quartz lode breccia and as quartz vein breccia. OREAS 60d is one of a suite of three CRMs ranging in gold content from 2.47 to 9.71ppm.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 60d was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of the barren andesite to >98% minus 75 microns;
- Crushing and multi stage milling of the ore materials to 100% passing 35 microns;
- Blending in appropriate proportions to achieve the desired grades;
- Packaging in 60g units sealed in laminated foil pouches and 500g units in plastic jars.

## ANALYTICAL PROGRAM

Twenty-seven commercial analytical laboratories participated in the program to certify the 110 elements reported in Table 1. The following methods were employed:

- Au by fire assay with AAS (23 labs) or ICP-OES (3 laboratories) finish;
- Instrumental neutron activation analysis for Au on 20 x 1g subsamples to confirm homogeneity (1 laboratory);
- Gold by aqua regia digestion on 10-50g sample weights with ICP-MS (11 laboratories), AAS (9 laboratories) or ICP-OES (1 laboratory) finish;
- 4-acid (HF-HNO<sub>3</sub>-HCIO<sub>4</sub>-HCI) digestion for full ICP-OES and ICP-MS elemental suites (up to 24 laboratories depending on the element);
- Aqua regia digestion for full ICP-OES and ICP-MS elemental suites (up to 23 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.



Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value			
Pb Fire As	ssay	I				I		I			
Pd	ppm	< 0.003	Pt	ppm	< 0.003						
4-Acid Dig	gestion										
Ge	ppm	0.10	Pt	ppm	< 0.01	Se	ppm	0.68			
Hg	ppm	0.19	Re	ppm	0.002	Та	ppm	0.13			
Ir	ppm	< 0.01	Rh	ppm	< 0.02						
Pd	ppm	< 0.01	Ru	ppm	< 0.02						
Aqua Reg	ia Diges	tion						1			
В	ppm	9.83	Pd	ppm	< 0.01	Та	ppm	< 0.01			
Eu	ppm	0.40	Pt	ppm	< 0.005	Tm	ppm	0.068			
Ge	ppm	< 0.05	Re	ppm	0.001	W	ppm	0.36			
Hf	ppm	0.22	Ru	ppm	< 0.005						
Nb	ppm	< 0.05	Se	ppm	0.38						
Borate Fusion XRF											
Al <sub>2</sub> O <sub>3</sub>	wt.%	6.77	K <sub>2</sub> O	wt.%	1.81	$P_2O_5$	wt.%	0.103			
BaO	ppm	200	MgO	wt.%	1.03	S	wt.%	0.538			
CaO	wt.%	14.55	MnO	wt.%	0.080	SiO <sub>2</sub>	wt.%	58.10			
Fe <sub>2</sub> O <sub>3</sub>	wt.%	2.81	Na <sub>2</sub> O	wt.%	0.765	TiO <sub>2</sub>	wt.%	0.295			
Thermogr	avimetry	<b>y</b>				L		1			
H <sub>2</sub> O-	wt.%	1.06	LOI <sup>1000</sup>	wt.%	12.71						
Laser Abl	ation ICI	P-MS									
Ag	ppm	5.00	Hf	ppm	1.41	Sm	ppm	1.84			
As	ppm	15.9	Но	ppm	0.30	Sn	ppm	0.60			
Ba	ppm	210	In	ppm	< 0.05	Sr	ppm	245			
Be	ppm	0.80	La	ppm	9.86	Та	ppm	0.13			
Bi	ppm	0.37	Lu	ppm	0.12	Tb	ppm	0.24			
Cd	ppm	0.30	Mn	wt.%	0.060	Те	ppm	2.30			
Ce	ppm	18.4	Мо	ppm	2.70	Th	ppm	2.44			
Co	ppm	8.05	Nb	ppm	1.83	Ti	wt.%	0.181			
Cr	ppm	15.0	Nd	ppm	9.15	TI	ppm	0.80			
Cs	ppm	4.39	Ni	ppm	10.0	Tm	ppm	0.13			
Cu	ppm	74	Pb	ppm	11.0	U	ppm	0.74			
Dy	ppm	1.41	Pr	ppm	2.29	V	ppm	65			
Er	ppm	0.85	Rb	ppm	59	W	ppm	2.50			
Eu	ppm	0.48	Re	ppm	< 0.01	Y	ppm	7.97			
Ga	ppm	7.95	Sb	ppm	2.10	Yb	ppm	0.83			
Gd	ppm	1.57	Sc	ppm	7.75	Zn	ppm	45.0			
Ge	ppm	0.68	Se	ppm	< 5	Zr	ppm	52			
Infrared C	ombusti	ion	·			·					
С	wt.%	3.11	S	wt.%	0.496						

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.



For the round robin program samples were taken at 20 predetermined sampling intervals during packaging and are considered representative of the entire batch of OREAS 60d. Six 120g samples were submitted to each laboratory for analysis. Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 92 indicative values including the 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**' on page 12). 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 60d DataPack.xlsx**).

Replicate	Au
No	ppm
1	2.56
2	2.50
3	2.43
4	2.56
5	2.57
6	2.51
7	2.64
8	2.55
9	2.54
10	2.51
11	2.49
12	2.57
13	2.52
14	2.48
15	2.65
16	2.67
17	2.47
18	2.58
19	2.56
20	2.55
Mean	2.55
Median	2.55
Std Dev.	0.061
Rel.Std.Dev.	2.39%
PDM <sup>3</sup>	2.94%

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

The 1RSD of 2.39% (or 0.445% at a 30g charge weight via the Sampling Constant (Ingamells & Switzer, 1973)) confirms the high level of gold homogeneity in OREAS 60d.



## STATISTICAL ANALYSIS

**Certified Values, Standard Deviations, Confidence and Tolerance Limits** have been determined for each analytical method following removal of individual and laboratory outliers (Table 1). Certified Values are the mean of means after outlier filtering. The 95% Confidence Limit is a measure of the reliability of the certified value, i.e. the narrower the Confidence Interval the greater the certainty in the Certified Value. *Confidence and Tolerance 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'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.

**Performance Gates** (Table 4) are 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.

	Certified		Absolute	Standard	Deviations	6	Relative	Standard D	eviations	5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Fire Assay											
Au, ppm	2.47	0.079	2.31	2.63	2.23	2.71	3.21%	6.42%	9.63%	2.35	2.60
Aqua Regia D	Digestion (sa	mple wei	ghts 10-5	0g)							
Au, ppm	2.43	0.110	2.21	2.65	2.10	2.76	4.52%	9.05%	13.57%	2.31	2.56
4-Acid Digest	tion										
Ag, ppm	4.57	0.178	4.22	4.93	4.04	5.11	3.89%	7.78%	11.67%	4.34	4.80
Al, wt.%	3.56	0.176	3.21	3.92	3.03	4.09	4.95%	9.89%	14.84%	3.38	3.74
As, ppm	15.7	1.39	12.9	18.5	11.5	19.9	8.87%	17.75%	26.62%	14.9	16.5
Ba, ppm	215	8	198	232	190	240	3.88%	7.76%	11.65%	204	226
Be, ppm	0.72	0.070	0.58	0.85	0.51	0.92	9.76%	19.53%	29.29%	0.68	0.75
Bi, ppm	0.31	0.022	0.26	0.35	0.24	0.37	7.12%	14.23%	21.35%	0.29	0.32
Ca, wt.%	10.19	0.396	9.40	10.98	9.00	11.38	3.88%	7.77%	11.65%	9.68	10.70
Cd, ppm	0.15	0.03	0.09	0.20	0.07	0.23	17.91%	35.81%	53.72%	0.14	0.15
Ce, ppm	18.6	1.67	15.3	21.9	13.6	23.6	8.97%	17.93%	26.90%	17.7	19.5
Co, ppm	7.59	0.689	6.21	8.96	5.52	9.65	9.08%	18.15%	27.23%	7.21	7.96
Cr, ppm	13.6	2.7	8.1	19.1	5.4	21.9	20.15%	40.30%	60.45%	12.9	14.3
Cs, ppm	4.34	0.274	3.80	4.89	3.52	5.16	6.30%	12.59%	18.89%	4.13	4.56
Cu, ppm	73	4.4	65	82	60	87	6.02%	12.04%	18.07%	70	77
Dy, ppm	1.37	0.060	1.25	1.49	1.19	1.55	4.35%	8.70%	13.05%	1.30	1.44
Er, ppm	0.81	0.050	0.71	0.91	0.65	0.96	6.24%	12.49%	18.73%	0.77	0.85
Eu, ppm	0.50	0.06	0.38	0.63	0.32	0.69	12.47%	24.94%	37.42%	0.48	0.53
Fe, wt.%	1.96	0.099	1.76	2.16	1.66	2.26	5.06%	10.11%	15.17%	1.86	2.06
Ga, ppm	8.15	0.562	7.02	9.27	6.46	9.83	6.90%	13.79%	20.69%	7.74	8.55
Gd, ppm	1.62	0.103	1.41	1.82	1.31	1.93	6.35%	12.71%	19.06%	1.54	1.70
Hf, ppm	1.30	0.128	1.05	1.56	0.92	1.68	9.80%	19.61%	29.41%	1.24	1.37
Ho, ppm	0.28	0.022	0.24	0.33	0.21	0.35	7.93%	15.86%	23.79%	0.27	0.29
In, ppm	0.023	0.003	0.016	0.029	0.013	0.032	14.41%	28.81%	43.22%	0.021	0.024
K, wt.%	1.51	0.068	1.38	1.65	1.31	1.72	4.50%	9.01%	13.51%	1.44	1.59
La, ppm	9.09	0.321	8.45	9.73	8.13	10.05	3.53%	7.07%	10.60%	8.64	9.54
Li, ppm	40.6	3.27	34.1	47.2	30.8	50.5	8.05%	16.11%	24.16%	38.6	42.7
Lu, ppm	0.12	0.010	0.10	0.14	0.09	0.15	8.20%	16.40%	24.61%	0.11	0.13
Mg, wt.%	0.590	0.033	0.525	0.655	0.492	0.687	5.52%	11.04%	16.57%	0.560	0.619
Mn, wt.%	0.059	0.002	0.054	0.063	0.052	0.065	3.76%	7.53%	11.29%	0.056	0.062
Mo, ppm	2.83	0.29	2.25	3.41	1.97	3.69	10.18%	20.37%	30.55%	2.69	2.97

Table 4. Performance Gates for OREAS 60d.



	Certified	Absolute Standard Deviations					Relative Standard Deviations			5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digest	ion continue	ed									
Na, wt.%	0.565	0.039	0.487	0.642	0.448	0.681	6.87%	13.74%	20.61%	0.537	0.593
Nb, ppm	1.78	0.173	1.44	2.13	1.26	2.30	9.70%	19.40%	29.09%	1.69	1.87
Nd, ppm	8.90	0.529	7.85	9.96	7.32	10.49	5.94%	11.89%	17.83%	8.46	9.35
Ni, ppm	9.56	0.945	7.67	11.45	6.72	12.39	9.89%	19.77%	29.66%	9.08	10.04
P, wt.%	0.042	0.002	0.038	0.047	0.036	0.049	5.21%	10.42%	15.63%	0.040	0.044
Pb, ppm	10.6	1.1	8.3	12.9	7.2	14.0	10.77%	21.54%	32.31%	10.0	11.1
Pr, ppm	2.20	0.095	2.01	2.39	1.92	2.49	4.30%	8.61%	12.91%	2.09	2.31
Rb, ppm	61	3.3	55	68	51	71	5.45%	10.90%	16.36%	58	64
S, wt.%	0.573	0.023	0.527	0.618	0.504	0.641	4.00%	8.01%	12.01%	0.544	0.601
Sb, ppm	2.14	0.202	1.73	2.54	1.53	2.74	9.46%	18.93%	28.39%	2.03	2.24
Sc, ppm	6.85	0.72	5.41	8.29	4.69	9.01	10.52%	21.04%	31.56%	6.51	7.19
Sm, ppm	1.79	0.101	1.59	1.99	1.49	2.10	5.65%	11.31%	16.96%	1.70	1.88
Sn, ppm	0.70	0.12	0.45	0.95	0.32	1.07	17.84%	35.69%	53.53%	0.66	0.73
Sr, ppm	244	12	221	267	209	279	4.75%	9.50%	14.26%	232	256
Ta, ppm	0.13	0.02	0.08	0.17	0.06	0.19	18.24%	36.49%	54.73%	0.12	0.13
Tb, ppm	0.24	0.023	0.20	0.29	0.18	0.31	9.42%	18.83%	28.25%	0.23	0.26
Te, ppm	2.15	0.23	1.69	2.62	1.45	2.85	10.82%	21.65%	32.47%	2.05	2.26
Th, ppm	2.15	0.198	1.75	2.54	1.55	2.74	9.23%	18.45%	27.68%	2.04	2.25
Ti, wt.%	0.171	0.006	0.160	0.183	0.154	0.189	3.44%	6.88%	10.32%	0.163	0.180
TI, ppm	0.64	0.034	0.57	0.71	0.54	0.75	5.34%	10.68%	16.03%	0.61	0.68
Tm, ppm	0.11	0.01	0.08	0.14	0.07	0.15	13.27%	26.55%	39.82%	0.10	0.11
U, ppm	0.65	0.052	0.54	0.75	0.49	0.80	8.03%	16.06%	24.09%	0.61	0.68
V, ppm	62	2.2	58	67	56	69	3.49%	6.99%	10.48%	59	65
W, ppm	2.28	0.220	1.84	2.72	1.62	2.94	9.64%	19.28%	28.91%	2.16	2.39
Y, ppm	7.25	0.531	6.18	8.31	5.65	8.84	7.32%	14.65%	21.97%	6.88	7.61
Yb, ppm	0.75	0.047	0.65	0.84	0.61	0.89	6.27%	12.54%	18.81%	0.71	0.78
Zn, ppm	36.9	1.99	32.9	40.9	30.9	42.9	5.40%	10.80%	16.21%	35.0	38.7
Zr, ppm	49.6	3.92	41.7	57.4	37.8	61.3	7.92%	15.83%	23.75%	47.1	52.0
Aqua Regia D	igestion										
Ag, ppm	4.45	0.237	3.97	4.92	3.74	5.16	5.32%	10.65%	15.97%	4.23	4.67
Al, wt.%	1.00	0.083	0.83	1.17	0.75	1.25	8.31%	16.61%	24.92%	0.95	1.05
As, ppm	15.3	0.89	13.5	17.1	12.6	17.9	5.79%	11.59%	17.38%	14.5	16.1
Ba, ppm	36.4	2.09	32.2	40.6	30.1	42.6	5.75%	11.50%	17.25%	34.6	38.2
Be, ppm	0.50	0.041	0.42	0.58	0.38	0.62	8.17%	16.34%	24.51%	0.47	0.52
Bi, ppm	0.30	0.016	0.27	0.33	0.25	0.35	5.45%	10.91%	16.36%	0.28	0.31
Ca, wt.%	9.78	0.633	8.51	11.04	7.88	11.68	6.48%	12.95%	19.43%	9.29	10.27
Cd, ppm	0.14	0.01	0.11	0.17	0.09	0.18	10.55%	21.11%	31.66%	0.13	0.14
Ce, ppm	15.8	0.71	14.4	17.2	13.7	17.9	4.49%	8.98%	13.47%	15.0	16.6

#### Table 4 continued.



Constituent	Certified		Absolute	Standard	Deviations	6	Relative Standard Deviations			5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Aqua Regia D	igestion co	ntinued									
Co, ppm	7.44	0.376	6.69	8.19	6.31	8.57	5.06%	10.11%	15.17%	7.07	7.81
Cr, ppm	13.3	1.9	9.4	17.1	7.5	19.0	14.52%	29.05%	43.57%	12.6	13.9
Cs, ppm	2.32	0.36	1.59	3.04	1.22	3.41	15.70%	31.40%	47.10%	2.20	2.43
Cu, ppm	72	2.8	66	77	63	80	3.97%	7.94%	11.92%	68	75
Dy, ppm	0.97	0.050	0.87	1.07	0.82	1.12	5.10%	10.19%	15.29%	0.92	1.02
Er, ppm	0.51	0.032	0.45	0.58	0.42	0.61	6.29%	12.57%	18.86%	0.49	0.54
Fe, wt.%	1.83	0.087	1.65	2.00	1.57	2.09	4.75%	9.50%	14.26%	1.73	1.92
Ga, ppm	3.96	0.396	3.17	4.75	2.77	5.15	9.99%	19.99%	29.98%	3.76	4.16
Hf, ppm	0.22	0.04	0.15	0.30	0.12	0.33	15.77%	31.55%	47.32%	0.21	0.24
Hg, ppm	0.100	0.006	0.088	0.112	0.082	0.118	5.90%	11.79%	17.69%	0.095	0.105
Ho, ppm	0.19	0.008	0.18	0.21	0.17	0.22	3.90%	7.79%	11.69%	0.18	0.20
In, ppm	0.018	0.003	0.013	0.024	0.010	0.026	14.98%	29.96%	44.95%	0.017	0.019
K, wt.%	0.199	0.022	0.155	0.243	0.133	0.265	11.03%	22.06%	33.09%	0.189	0.209
La, ppm	7.90	0.436	7.02	8.77	6.59	9.21	5.53%	11.05%	16.58%	7.50	8.29
Li, ppm	9.88	0.392	9.10	10.66	8.70	11.05	3.96%	7.93%	11.89%	9.39	10.37
Lu, ppm	0.061	0.005	0.051	0.070	0.047	0.074	7.61%	15.22%	22.83%	0.058	0.064
Mg, wt.%	0.516	0.018	0.480	0.551	0.463	0.569	3.43%	6.87%	10.30%	0.490	0.542
Mn, wt.%	0.057	0.003	0.052	0.063	0.049	0.065	4.81%	9.62%	14.43%	0.054	0.060
Mo, ppm	2.63	0.167	2.30	2.97	2.13	3.13	6.35%	12.70%	19.05%	2.50	2.76
Na, wt.%	0.061	0.008	0.044	0.077	0.036	0.085	13.28%	26.55%	39.83%	0.058	0.064
Nd, ppm	7.64	0.264	7.12	8.17	6.85	8.44	3.45%	6.91%	10.36%	7.26	8.03
Ni, ppm	8.64	0.669	7.30	9.98	6.63	10.64	7.75%	15.50%	23.25%	8.20	9.07
P, wt.%	0.039	0.002	0.035	0.043	0.033	0.045	4.88%	9.76%	14.64%	0.037	0.041
Pb, ppm	8.67	0.799	7.07	10.26	6.27	11.06	9.22%	18.44%	27.65%	8.23	9.10
Pr, ppm	1.91	0.078	1.75	2.07	1.68	2.14	4.08%	8.16%	12.24%	1.82	2.01
Rb, ppm	9.91	1.40	7.10	12.72	5.70	14.12	14.17%	28.34%	42.51%	9.41	10.40
S, wt.%	0.563	0.016	0.530	0.596	0.514	0.612	2.92%	5.84%	8.77%	0.535	0.591
Sb, ppm	0.43	0.04	0.34	0.52	0.29	0.56	10.54%	21.07%	31.61%	0.41	0.45
Sc, ppm	4.30	0.180	3.94	4.66	3.76	4.84	4.19%	8.38%	12.56%	4.08	4.51
Sm, ppm	1.51	0.067	1.38	1.65	1.31	1.71	4.45%	8.91%	13.36%	1.44	1.59
Sn, ppm	0.39	0.032	0.32	0.45	0.29	0.48	8.28%	16.56%	24.83%	0.37	0.40
Sr, ppm	137	13	112	163	100	175	9.17%	18.35%	27.52%	130	144
Tb, ppm	0.19	0.005	0.18	0.20	0.17	0.20	2.67%	5.33%	8.00%	0.18	0.20
Te, ppm	2.02	0.169	1.68	2.36	1.51	2.52	8.40%	16.81%	25.21%	1.92	2.12
Th, ppm	1.20	0.13	0.94	1.47	0.80	1.60	11.07%	22.15%	33.22%	1.14	1.26
Ti, wt.%	0.032	0.004	0.023	0.041	0.018	0.045	14.10%	28.20%	42.30%	0.030	0.033
TI, ppm	0.17	0.02	0.14	0.21	0.12	0.22	10.17%	20.34%	30.50%	0.16	0.18
U, ppm	0.29	0.014	0.26	0.32	0.25	0.34	4.84%	9.68%	14.51%	0.28	0.31

#### Table 4 continued.



Constituent	Certified Value		Absolute	Standard	Deviations	6	Relative Standard Deviations			5% window		
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High	
Aqua Regia Digestion continued												
V, ppm	37.8	2.88	32.1	43.6	29.2	46.5	7.62%	15.23%	22.85%	35.9	39.7	
W, ppm	0.36	0.08	0.20	0.52	0.12	0.61	22.28%	44.57%	66.85%	0.34	0.38	
Y, ppm	5.28	0.287	4.71	5.86	4.42	6.14	5.43%	10.86%	16.29%	5.02	5.55	
Yb, ppm	0.42	0.030	0.36	0.48	0.33	0.51	7.08%	14.16%	21.25%	0.40	0.44	
Zn, ppm	32.9	2.14	28.6	37.2	26.5	39.3	6.50%	13.01%	19.51%	31.2	34.5	
Zr, ppm	7.40	1.19	5.01	9.79	3.82	10.98	16.13%	32.26%	48.39%	7.03	7.77	

Table 4 continued.

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 silver 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 4.44 and 4.70ppm. 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 0.445% calculated for a 30g fire assay or aqua regia sample (2.39% at 1g weights) confirms the high level of gold homogeneity in OREAS 60d. The homogeneity is of a level such that **sampling error is negligible** for a conventional fire assay or aqua regia determination.

Please note that these INAA derived 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 60d 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 60d. The test was performed using the following parameters:

- Gold fire assay 156 samples (26 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion 126 samples (21 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis, H<sub>0</sub>: Between-unit variance is no greater than within-unit variance (reject H<sub>0</sub> if *p*-value < 0.05);</li>



• Alternative Hypothesis, H<sub>1</sub>: 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 *p*-values. This process derived *p*-values of 0.67 for Au by fire assay and 0.78 for Au by aqua regia digestion. Both *p*-values are insignificant and the Null Hypothesis is retained. Additionally, only six of the other 108 certified values showed significant p-values (Ca, Li, Nd, Pr, Sc and Sn; all by aqua regia digestion) and these elements (other than Ca) are all present in low concentration levels. Tin in particular is close to its lower level of detection (LLD) where reading resolution errors can lead to 'false positives' ('statistically significant' *p*-values that are in fact meaningless). Usually data becomes more reliable and meaningful when the concentration levels are at least twenty times the LLD. The other cases are also interpreted as false positives most likely due to i) acute differences in reading resolution where one or more laboratories report to a reduced number of significant figures resulting in an amplified effect on variance or ii) random statistical probability given the 5% significance level. As there is no other supporting evidence to suspect greater betweenunit 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 60d 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 60d is fit-for-purpose as a certified reference material (see 'Intended Use' below).

# PARTICIPATING LABORATORIES

- 1. Actlabs, Ancaster, Ontario, Canada
- 2. Actlabs, Kamloops, BC, Canada
- 3. Alex Stewart International, Mendoza, Argentina
- 4. ALS, Brisbane, QLD, Australia
- 5. ALS, Loughrea, Galway, Ireland
- 6. ALS, Perth, WA, Australia
- 7. ALS, Townsville, QLD, Australia
- 8. ALS, Vancouver, BC, Canada
- 9. American Assay Laboratories, Sparks, Nevada, USA
- 10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 11. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 12. Bureau Veritas Geoanalytical, Perth, WA, Australia
- 13. Gekko Assay Labs, Ballarat, VIC, Australia



- 14. Inspectorate (BV), Lima, Peru
- 15. Intertek Genalysis, Perth, WA, Australia
- 16. Intertek Tarkwa, Tarkwa, Ghana
- 17. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 18. MinAnalytical Services, Perth, WA, Australia
- 19. North Austalian Laboratories, Pine Creek, NT, Australia
- 20. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 21. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 22. PT SGS Indo Assay Laboratories, Jakarta, Indonesia
- 23. SGS Australia Mineral Services, Perth, WA, Australia
- 24. SGS del Peru, Lima, Peru
- 25. SGS Mineral Services, Townsville, QLD, Australia
- 26. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 27. Zarazma Mineral Studies Company, Tehran, Iran

# PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 60d has been prepared and certified by:

ORE Research & Exploration Pty Ltd 37A Hosie Street Bayswater North VIC 3153 AUSTRALIA 

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 Web:
 www.ore.com.au

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 info@ore.com.au

It has been packaged in 60g units into laminated foil pouches and 500g units in plastic jars.

#### **INTENDED USE**

OREAS 60d 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 60d has been prepared from primary gold-silver ore samples from the Cracow mine blended with barren andesite. It is low in reactive sulphide (0.57% 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 THE CORRECT USE**

The certified values for OREAS 60d 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.

#### LEGAL NOTICE

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

#### QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2015 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.



# **CERTIFYING OFFICER**

14<sup>th</sup> February, 2018

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

#### REFERENCES

Ingamells, C. O. and Switzer, P. (1973), Talanta 20, 547-568.

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